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INSTITUTIONS ADVANCED

ON APPEAL FROM THE SUPREME COURT OF CANADA

BETWEEN :

MINERALS SEPARATION NORTH AMERICAN CORPORATION,

Appellants

—AND—

NORANDA MINES, LIMITED,

Respondents.

RECORD OF PROCEEDINGS

VOLUME 4

PAGES 690—911

BIRD & BIRD,
5-11 Theobald's Road,
London, W.C.1,
Appellants' Solicitors.

LAWRENCE JONES & CO.,
Winchester House,
Old Broad Street,
London, E.C.2,
Respondents' Solicitors.

In The Supreme Court of Canada

ON APPEAL FROM THE EXCHEQUER COURT OF CANADA

BETWEEN :

NORANDA MINES, LIMITED,

Appellant;

— AND —

MINERALS SEPARATION NORTH AMERICAN
CORPORATION,

Respondent.

APPEAL CASE

— VOLUME 4 —

HOLDEN, MURDOCH, WALTON, FINLAY & ROBINSON,
Solicitors for Appellant.

SMART & BIGGAR,
Ottawa Agents for Solicitors for Appellant.

EWART, SCOTT, KELLEY & HOWARD,
Solicitors for Respondent.

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INSTITUTE OF ADVANCED
LEGAL STUDIES

INDEX

PART I. — PLEADINGS, ETC.

DOCUMENT	DATE	VOL.	PAGE
Statement of Case	June 24, 1947	I	1
Statement of Claim	Mar. 1, 1943	I	1
Particulars of Breaches	Mar. 1, 1943	I	3
Demand for Particulars	May 6, 1943	I	3
Answer to Demand for Particulars	May 21, 1943	I	5
Answer to Demand under Rule 22A	Nov. 10, 1943	I	5
Statement of Defence	June 19, 1943	I	6
Particulars of Objection	June 19, 1943	I	7
Schedule I to Particulars of Objection	June 19, 1943	I	10
Schedule II to Particulars of Objection	June 19, 1943	I	11A

PART II. — EVIDENCE

DOCUMENT	DATE	VOL.	PAGE
Opening of Plaintiff's Case by Mr. Gowling		I	12
Mr. Gowling reads from examination for discovery of Murdoch		I	31
FOR PLAINTIFF—			
ARTHUR HOWARD HIGGINS :			
Examination in Chief		I	36
Cross-Examination		I	79
Re-Examination		I	107
Re-Cross-Examination		I	118
Remarks of Counsel		I	122
FOR PLAINTIFF—			
ELTOFT WRAY WILKINSON :			
Direct Examination		I	125
Cross-Examination		I	148
Re-Examination		I	173
FOR PLAINTIFF—			
CORNELIUS HORACE KELLER :			
Direct Examination		I	176
Cross-Examination		I	219
Re-Examination		I	236
FOR PLAINTIFF—			
HENRY D. WILLIAMS :			
Direct Examination		II	241
Cross-Examination		II	255
Re-Examination		II	275
FOR PLAINTIFF—			
SETH GREGORY :			
Direct Examination		II	280
Cross-Examination		II	299
Extract from letter R. S. Smart to Ewart Scott & Kelley, Re: Commission Evidence	Mar. 11, 1944	II	303
Letter in reply from Ewart Scott & Kelley	Mar. 18, 1944	II	304
Opening of Defendant's Case by Mr. Biggar		II	308

EVIDENCE — (Continued)

DOCUMENT	DATE	VOL.	PAGE
FOR DEFENDANT—			
CLIFFORD B. PURVES :			
Examination in Chief		II	419
Cross-Examination		II	458
Re-Examination		III	485
Re-Cross-Examination		III	494
FOR DEFENDANT—			
ROBERT L. BENNETT :			
Examination in Chief		III	503
Cross-Examination		III	548
Re-Examination		III	573
Rebuttal			
FOR PLAINTIFF—			
A. H. HIGGINS — Recalled			
Examination in Chief		III	583
Cross-Examination		III	650
Remarks of Counsel and filing of additional exhibits		III	666

PART III — EXHIBITS

EXHIBIT No.	DESCRIPTION	DATE	APPEAL BOOK		EXHIBIT BOOK		FIRST REFERRED TO	
			VOL.	PAGE	VOL.	PAGE	VOL.	PAGE
P-50	Patent No. 247,576 in suit	Mar. 10, 1925	V	935			I	30
M-2	Statement of flotation agents used by Defendant	Mar. 13, 1944			I	26	I	32
M-3	Duplicate of M-4EE							
P-51	Bottle containing piece of ore						I	40
P-52	Bottle of fine ground ore						I	40
P-53	Bottle of ground ore with slimes washed out						I	40
P-54	Chart-Structural formulae of carbonic acid, its sulphur derivatives and some alkyl radicals				I	27	I	46
P-55	Chart-Calculation of quantities for forma- tion of xanthate				I	28	I	50
D-56	Results of tests conducted by Higgins. . . .		V	1043			I	85
D-57	Chart-sulphur derivatives of carbonic acid.				I	29-31	I	100
D-58	Chart-Relation of classes of compounds referred to in patent 247,576.				I	32	I	106
D-59	Results of flotation tests at Noranda.	Aug. 29, 1944	V	1035			I	107
D-60	Chart of Sulphur Derivatives of carbonic acid showing inclusion of compounds. . .				I	33	I	116
D-61	Agreement as to list of xanthates.	Nov. 16, 1944	V	1039			I	122
K-1	Drawing of MSNAC—Sub. A. machine. . .				I	12	I	138

EXHIBITS — (Continued)

EXHIBIT No.	DESCRIPTION	DATE	APPEAL BOOK		EXHIBIT BOOK		FIRST REFERRED TO	
			VOL.	PAGE	VOL.	PAGE	VOL.	PAGE
K-2	Report of Lewis & Keller	Mar. 28, 1923	IV	771	I	143
K-3	Report	May 3, 1923	IV	785	I	143
K-4	Report	May 11, 1923	IV	794	I	143
	Table 1		IV	798		
	Table 2		IV	799		
K-5	Telegram Nutter to N.Y. office	June 15, 1923	IV	805	I	143
K-6	Article in Mining Journal Press	Feb. 9, 1924	IV	856	I	145
K-7	Editorial in Mining Journal Press	Aug. 2, 1924	IV	858	I	145
K-8	Abstract from Mining Journal Press	Oct. 18, 1924	IV	859	I	145
K-9	Editorial from Mining Journal Press	Nov. 22, 1924	IV	860	I	145
K-10	Extract from Mining Journal Press	Nov. 1, 1924	IV	860	I	146
K-11	Article in Mining Journal Press	Dec. 20, 1924	IV	861	I	146
K-12	Keller Notebook	May 1919- June 1924					I	178
	Page 14					II	I	181
	" 16					II	I	182
	" 27					II	I	222
	" 29					II	I	222
	" 30					II	I	184
	" 31					II	I	184
	" 32					II	I	184
	" 33					II	I	184
	" 34					II	I	184
	" 36					II	I	184
	" 37					II	I	184
	" 38					II	I	184
	" 39					II	I	184
	" 40					II	I	190
	" 41					II	I	191
	" 42					II	I	191
	" 43					II	I	192
	" 44					II	I	192
	" 45					II	I	193
	" 46					II	I	193
	" 47					II	I	179
	" 48					II	I	198
	" 49					II	I	198
	" 50					II	I	198
	" 52					II	I	198
	" 53					II	I	198
	" 54					II	I	198
	" 55					II	I	198
	" 57					II	I	200
	" 58					II	I	199
	" 59					II	I	200
	" 60					II	I	200
	" 61					II	I	200
	" 63					II	I	200
	" 64					II	I	200
	" 65					II	I	200
	" 66					II	I	200
	" 67					II	I	
	" 68					II	I	
	" 69					II	I	240
	" 70					II	I	235
	" 71					II	I	
	" 76					II	I	232
	" 77					II	I	233
	" 78					II	I	233
	" 81					II	I	236
K-13	Report of Keller	Sept. 1922	IV	761	I	189
K-14	Lewis Notebook	July 1922			I	196

EXHIBITS — (Continued)

EXHIBIT No.	DESCRIPTION	DATE	APPEAL BOOK	EXHIBIT BOOK	FIRST REFERRED TO
K-14 (con'd)	Page 67			II	I 233
	" 68			II	I 199
	" 75			II	I 196
	" 76			II	I 196
	" 77			II	I 196
	" 86			II	I 196
	" 92			II	I 196
	" 116			II	I 199
	" 131			II	I 200
	" 132			II	I 225
" 136			II	I 200	
K-15	Lewis Notebook	Jan. 4, 1923-			
		May 10, 1923			I 196
K-16	Lewis Notebook	May 4, 1923-			
		Feb. 1, 1924			I 196
K-17	Keller report	May 7, 1923	IV 792		I 199
K-18	Keller report	Dec. 11, 1922	IV 765		I 200
K-19	Laboratory Record Book	Jan. 12, 1922-			
		Oct. 27, 1922			I 201
K-20	Laboratory Record Book	Oct. 20, 1922-		II	I 201
		Aug. 25, 1923			
K-21	3 sheets of tabulated tests	1922 — 23		I 14 I 15 I 16	I 202
K-22	Page 199 of Laboratory Record Book (Ex. K-20)	Feb. 3, 1923		I 17	I 206
K-23	Page 905 of Lewis Notebook (Ex. K-15)	Feb. 3, 1923		I 16A	I 208
K-24	Three assay certificates	Mar. 15, 1923	IV 769		I 209
K-25	Tabulation of tests with xanthate	Mar. 2, 1923-		I 18-22	I 210
		Aug. 23, 1923			
K-26	Lr. Nutter to N.Y. office	May 7, 1923	IV 791		I 211
K-27	Lr. Keller to Nutter	May 15, 1923	IV 800		I 211
K-28	Lr. Keller & Lewis to Nutter	May 16, 1923	IV 802		I 211
K-29	Lrs. Rosenstein to MSNAC, San Francisco	July 21, 1923	IV 821		I 213
K-30	Lr. Keller to Nutter	July 25, 1923	IV 824		I 213
K-31	Tel. Nutter to Lewis	Aug. 3, 1923	IV 832		I 214
K-32	Lr. Nutter to G. W. Electro Chemical Company	Aug. 9, 1923	IV 838		I 215
	Flow sheet for xanthate preparation		IV 839		
K-33	Lr. Nutter to Anaconda	Aug. 9, 1923	IV 840		I 215
K-34	Lr. Nutter to MSNAC — N.Y.	Aug. 9, 1923	IV 837		I 215
K-35	Lr. Keller to Nutter	July 23, 1923	IV 823		I 215
K-36	Lr. Keller to Nutter	July 27, 1923	IV 826		I 215
K-37	2 lrs. Keller to Nutter	Aug. 2, 1923	IV 829		I 216
K-38	3 lrs. Keller to Nutter	Aug. 2, 1923	IV 830		I 216
K-39	4 lrs. Keller to Nutter	Oct. 15, 1923	IV 847		I 216
		Oct. 17, 1923	IV 848		
		Oct. 29, 1923	IV 849		
		Oct. 29, 1923	IV 851		
K-40	Lr. Keller to Nutter	Nov. 26, 1923	IV 852		I 217
K-41	Report Keller & Lewis to Nutter	Feb. 7, 1924	IV 854		I 217
K-42	Tabulation re: benzyl xanthate	1924		I 23	I 217
K-43	U.S. Patent 2,044,851	June 23, 1936	V 970		I 218
K-44	U.S. Patent 1,728,764	Sept. 17, 1929	V 961		I 218
K-45	Report Keller to Nutter	Sept. 11, 1925	IV 865		I 218
K-46	Tabulation re mercaptans	1922 — 25		I 13	I 218
K-47	File wrapper and contents of Keller U.S. Application :				
	U.S. Patent 1,554,216	Sept. 22, 1925	IV 875		I 218
	Certificate of Commissioner		IV 875		
	Cover		IV 876		
	Petition		IV 877		
	Specification		IV 877		
	Oath	Oct. 15, 1923	IV 882		

EXHIBITS — (Continued)

EXHIBIT No.	DESCRIPTION	DATE	APPEAL BOOK		EXHIBIT BOOK		FIRST REFERRED TO	
			VOL.	PAGE	VOL.	PAGE	VOL.	PAGE
K-47 (con'd)	Official Action.....	Oct. 31, 1923	IV	883				
	Reply to above action.....		IV	885				
	Official action.....	Dec. 3, 1923	IV	886				
	Reply to above action.....		IV	887				
	Interference record 50,394.....	Aug. 4, 1925	IV	887				
	Action advising of interference.....		IV	888				
	Amendment.....		IV	890				
	Amended petition.....		IV	893				
	Supplemental oath of Lewis.....	Mar. 26, 1924	IV	894				
	Supplemental oath of Keller.....	Mar. 25, 1924	IV	895				
	Official action.....		IV	897				
	Substitute power of Attorney.....		IV	897				
	Amendment.....		IV	898				
	Notice of Allowance.....	Aug. 5, 1925	IV	899				
	Covering lr. for final fee.....	Aug. 19, 1925	IV	901				
	The patent as issued.....	Aug. 20, 1925	IV	902				
	Title report.....	June 29, 1927	IV	908				
Official letter advising of Martin inter- ference 55642.....		IV	909					
Interference record.....		IV	911					
K-A	Page 30 of Laboratory Record Book (Ex. K-20)	Nov. 17, 1922			I	16B	I	227
K-B	Page from Keller notebook.....	Jan. 1924			I	25	I	231
K-C	Page from Keller notebook.....	Jan. 1924			I	24	I	232
W-1	Agreement Martin — Minerals Separation Ltd.....		III	687			II	243
W-2	Agreement Martin—Min. Sep. American Syndicate (1913) Ltd.....		III	684			II	243
W-3	2 sheets Williams notes on Martin inter- view.....		III	683			II	244
W-4	Lr. Martin to Gregory.....	Mar. 19, 1915	IV	690			II	246
W-5	Martin's specifications.....	Mar. 19, 1915	IV	691			II	246
	Kotrix.....		IV	691				
	Stanol.....		IV	695				
	Minola.....		IV	699				
	Grabanol.....		IV	702				
	Pyrox.....		IV	704				
	Unnamed specification.....		IV	709				
W-6	Tel. Williams to Nutter.....	Nov. 13, 1929	V	981			II	247
W-7	Tel. Nutter to Williams.....	Nov. 14, 1929	V	981			II	248
W-8	Lr. Martin to Gregory.....	Feb. 23, 1917	IV	738			II	249
W-9	U.S. Patent 1,236,856 Martin.....	Aug. 14, 1917	IV	747			II	249
W-10	U.S. Patent 1,236,857 Martin.....	Aug. 14, 1917	IV	744			II	249
W-11	Lr. Williams to MSNAC.....	Mar. 21, 1917	IV	738			II	250
W-12	Photostat copy receipt of Martin for \$5000	Mar. 21, 1917	IV	738			II	250
W-13	List of Martin's patent applications.....		IV	743			II	250
W-14	Report of Keller & Lewis.....	Mar. 30, 1923	IV	782			II	251
W-15	Lr. Nutter to MSNAC (duplicate of K-2)	Mar. 28, 1923	IV	771			II	251
W-16	Lr. Williams & Pritchard to MSNAC.....	April 30, 1923	IV	789			II	251
W-17	Tel. Williams to Evans.....	Aug. 20, 1926	V	980			II	253
W-18	Lr. Williams to Counsel for Metals Recovery.....	Aug. 20, 1926	V	980			II	253
G-1	Lr. Martin to Gregory.....	Mar. 19, 1915	III	689			II	283
G-2	Lr. Gregory to Martin.....	May 6, 1915	IV	714			II	283
G-3	Martin's Bulletin No. 2.....		IV	725			II	288
G-4	Pages from "Recipe Book".....		IV	751			II	289
G-5	Lr. Gregory to Ballot.....	July 28, 1915	IV	720			II	289
G-6	Lr. Higgins to Gregory re Martin.....	July 30, 1915	IV	721			II	290
G-7	Lr. Gregory to Ballot.....	Aug. 3, 1915	IV	723			II	290
G-8	Lr. Gregory to Ballot.....	Aug. 26, 1915	IV	732			II	290
G-9	Report.....	Aug. 14, 1915	IV	724			II	291
G-10	Lr. Gregory to Ballot.....	Sept. 17, 1915	IV	733			II	291
G-11	Lr. Ballot to Gregory.....	Oct. 1, 1915	IV	736			II	291

EXHIBITS — (Continued)

EXHIBIT No.	DESCRIPTION	DATE	APPEAL BOOK		EXHIBIT BOOK		FIRST REFERRED TO	
			VOL.	PAGE	VOL.	PAGE	VOL.	PAGE
G-12	Lr. Gregory to Ballot	Oct. 15, 1915	IV	736	II	291
G-13	Lr. Gregory to Ballot	Oct. 22, 1915	IV	737	II	291
G-14	List of Plaintiff's licensees in Canada.....	V	982	II	295
G-15	Licence form of Plaintiff.....	V	1012	II	295
G-16	Excerpts from minutes of MSNAC direc- tors' meeting.....	1936 — 43	V	1009	II	296
G-17	Lr. Ballot to Gregory	July 6, 1915	IV	717	II	300
G-18	Lr. Ballot to Gregory	July 20, 1915	IV	718	II	300
G-19	Lr. Gregory to Ballot	July 21, 1915	IV	719	II	300
G-20	Lr. Ballot to Gregory	Aug. 10, 1915	IV	723	II	300
G-21	Lr. Ballot to Gregory	Aug. 31, 1915	IV	732	II	300
D-62	Memo Rosenstein to Nutter	May 10, 1923	IV	793	II	320, 365
D-63	Lr. Nutter to Plaintiff	May 25, 1923	IV	803	II	320
D-64	Lr. Nutter to Plaintiff	May 29, 1923	IV	804	II	320, 367
D-65	Lr. Nutter to Plaintiff	June 18, 1923	IV	806	II	320, 369
D-66	Report of tests at Anaconda	IV	808	II	320
D-67	Lr. Nutter to Plaintiff	June 20, 1923	IV	813	II	320
D-68	Lr. Plaintiff to Nutter	June 26, 1923	IV	816	II	320, 369
D-69	Lr. Morrow to Nutter	July 6, 1923	IV	818	II	320
D-70	Lr. Nutter to Morrow	July 19, 1923	IV	819	II	320
D-71	Lr. Nutter to Morrow	July 21, 1923	IV	819	II	320
D-72	Lr. Morrow to Nutter	July 26, 1923	IV	825	II	320
D-73	Lr. Nutter to Anaconda	Aug. 2, 1923	IV	827	II	320
D-74	Lr. Nutter to Gregory	Sept. 4, 1923	IV	842	II	320, 373
D-75	Tel. Nutter to G. W. El. Chem. Co.	Sept. 6, 1923	IV	845	II	320
D-76	Tel. Nutter to G. W. El. Chem. Co.	Sept. 6, 1923	IV	845	II	320
D-77	Lr. G. W. El. Chem. Co. to Plaintiff	Sept. 15, 1923	IV	846	II	320, 373
D-78	Tel. Nutter to Gregory	Sept. 27, 1923	IV	847	II	320
D-79	Lr. C. B. A. to Janney	Feb. 14, 1924	IV	857	II	320, 376
D-80	Lr. Lewis to Nutter	Aug. 4, 1923	IV	832	II	353
D-81	U.S. Patent 1,154,220 Lewis	Sept. 22, 1925	V	952	II	360
D-82	Can. Patent 247,791 Lewis	Mar. 7, 1925	V	943	II	360
D-83	File Wrapper Can. Patent 247,576 in suit ..	Mar. 10, 1925	V	912	II	407
	Certificate of Commissioner	V	912		
	File Record	V	912		
	Petition	Dec. 5, 1924	V	912		
	Petition	Oct. 11, 1924	V	913		
	Power of Attorney to Ridout & Maybee ..	Dec. 5, 1924	V	914		
	Assoc. Power of Attorney to Ridout & Maybee	Oct. 21, 1924	V	914		
	Assoc. Power of Attorney to Caron & Caron	Oct. 23, 1924	V	915		
	Oath	Oct. 11, 1924	V	915		
	Specification	V	916		
	Covering lr. for application	Oct. 23, 1924	V	922		
	Oath of Albert Caron	Oct. 23, 1924	V	923		
	Receipt for filing fee	V	924		
	Covering lr. for new petition	Oct. 24, 1924	V	924		
	Petition	Oct. 23, 1924	V	925		
	Specification as filed	Oct. 23, 1924	V	926		
	Receipt for assignment fee	V	926		
	Official filing receipt	Oct. 28, 1924	V	927		
	Covering lr. to powers of attorney	Oct. 29, 1924	V	928		
	Acknowledgement of above	Nov. 14, 1924	V	929		
	Official objection to appointment of re- presentative	Nov. 25, 1924	V	929		
	Covering lr. for new petition and power of attorney	Dec. 13, 1924	V	930		
	Acknowledgement of above	Dec. 18, 1924	V	931		
	Notice of Allowance	Jan. 6, 1925	V	931		
	Covering lr. for final fee	Jan. 16, 1925	V	932		
	Receipt for final fee	V	933		
	File cover	934		

EXHIBITS — (Continued)

EXHIBIT No.	DESCRIPTION	DATE	APPEAL BOOK		EXHIBIT BOOK		FIRST REFERRED TO	
			VOL.	PAGE	VOL.	PAGE	VOL.	PAGE
D-84	U.S. Patent 835,120 Sulman	Nov. 6, 1906	III	673	II	419
D-85	U.S. Patent 962,678 Sulman	June 28, 1910	III	679	II	419
D-86	Chart of Organic radicals	I	34	II	420
D-87	Chart of Glycerol and Cellulose and their radicals	I	35	II	435
P-88	Copy of pages of Richter's Chemistry 1929	V	1019	II	459
D-89A	Assay certificate	Oct. 13, 1944	V	1037	III	506
D-89B	Assay certificate	Oct. 17, 1944	V	1038	III	506
D-59A	Result of flotation tests at Noranda	Oct. 29, 1944	V	1036	III	506
D-90	Laboratory notebook of R. L. Bennett	Aug.— Sept. 1944	III	507
D-91	Lr. Quigley to Nutter	April 28, 1921	IV	760	III	577
D-92	Tel. Plaintiff to Nutter	May 25, 1923	IV	803	III	577
D-93	Tel. Nutter to Morrow	July 5, 1923	IV	817	III	577
D-94	Tel. Lewis to Nutter	Aug. 6, 1923	IV	833	III	577
D-95	Tel. Nutter to Lewis	Aug. 6, 1923	IV	834	III	578
D-96	Lr. Anaconda to Nutter	Aug. 8, 1923	IV	836	III	578
D-97	Lr. Nutter to Plaintiff	Aug. 30, 1923	IV	841	III	578
D-98	Tel. G. W. El. Chem. Co. to Nutter	Sept. 5, 1923	IV	844	III	578
D-99	U.S. Patent 1,364,304 Perkins	Jan. 4, 1921	IV	754	III	579
P-100	Supplemental report of tests on Anaconda ore	Jun.-Jul. 1923	IV	814	III	581
P-101	Lr. Lewis to Nutter	Aug. 7, 1923	IV	835	III	582
P-102	Lr. Martin to Gregory	May 25, 1915	IV	716	III	636
P-103	Martin Bulletin No. 1	1915	III	640
P-104	Martin's specifications (Higgins' copy) (Duplicate of W-5 except for Higgins' notes)	Mar. 9, 1915	I	1-12	III	640
P-105	Martin's Bulletin No. 3	Sept. 20, 1915	IV	733	III	644
P-106	Martin's Bulletin No. 4	Sept. 20, 1915	IV	735	III	645
P-107	Lr. Higgins to Williams	Dec. 11, 1915	IV	737	III	646
P-108	Index to Martin Bulletins	1915 — 23	IV	739	III	648
D-109	Lr. Martin to Gregory	June 3, 1926	V	976	III	667
D-110	Lr. Plaintiff to Martin	June 3, 1926	V	977	III	667
D-111	Lr. Martin to Roberts	June 6, 1926	V	977	III	667
D-112	Lr. Plaintiff to Martin	June 7, 1926	V	978	III	667
D-113	Lr. Martin to Plaintiff	June 12, 1926	V	978	III	667
D-114	Lr. Plaintiff to Martin	June 14, 1926	V	979	III	667
D-115	Lr. Martin to Nutter	June 28, 1926	V	979	III	668
D-116	Lr. Nutter to Martin	July 6, 1926	V	980	III	668
P-117	Table of consumption of flotation reagents in 1923	April 1926	IV	875
M-4A	Lr. Dft. to Plf.	Dec. 8, 1925	V	983
M-4B	Lr. Plf. N.Y. to Plf. San Francisco	Dec. 11, 1925	V	983
M-4C	Lr. Dft. to Plf.	Dec. 28, 1925	V	984
M-4D	Lr. Plf. to Dft.	Mar. 9, 1926	V	985
M-4E	Lr. Plf. to Dft.	Aug. 2, 1930	V	986
M-4F	Lr. Plf. to Dft.	Aug. 12, 1930	V	987
M-4G	Lr. Plf. to Dft.	Nov. 1, 1931	V	988
M-4H	Lr. Dft. to Plf.	Nov. 10, 1932	V	990
M-4I	Lr. Plf. to Dft.	Nov. 16, 1932	V	992
M-4J	Lr. Plf. to Dft.	Nov. 17, 1932	V	993
M-4K	Lr. Plf. to Dft.	April 4, 1934	V	994
M-4L	Lr. Plf. to Dft.	April 24, 1934	V	994
M-4M	Lr. Dft. to Plf.	May 23, 1934	V	995
M-4N	Lr. Plf. to Dft.	May 25, 1934	V	995
M-4O	Lr. Plf. to Dft.	July 16, 1934	V	996
M-4P	Lr. Dft. to Plf.	July 18, 1934	V	996
M-4Q	Lr. Plf. to Dft.	July 20, 1934	V	997
M-4R	Lr. Dft. to Plf.	Nov. 2, 1934	V	997
M-4S	Lr. Plf. to Dft.	Nov. 14, 1934	V	998
M-4T	Lr. Dft. to Plf.	Nov. 21, 1934	V	999

EXHIBITS — (Continued)

EXHIBIT No.	DESCRIPTION	DATE	APPEAL BOOK		EXHIBIT BOOK		FIRST REFERRED TO	
			VOL.	PAGE	VOL.	PAGE	VOL.	PAGE
M-4U	Lr. Plf. to Dft.	Nov. 26, 1934	V	999				
M-4V	Lr. Dft. to Plf.	Jan. 15, 1935	V	1000				
M-4W	Lr. Dft. to Plf.	Feb. 12, 1935	V	1000				
M-4X	Lr. Plf. to Dft.	Feb. 14, 1935	V	1001				
M-4Y	Lr. Plf. to Dft.	Feb. 7, 1936	V	1001				
M-4Z	Lr. Dft. to Plf.	Feb. 12, 1936	V	1002				
M-4AA	Lr. Plf. to Dft.	Feb. 13, 1936	V	1003				
M-4BB	Lr. Dft. to Plf.	Feb. 17, 1936	V	1004				
M-4CC	Lr. Plf. to Dft.	Feb. 18, 1936	V	1004				
M-4DD	Lr. Plf. to Dft.	Mar. 31, 1936	V	1005				
M-4EE	Lr. Dft. to Plf. (Duplicate of Ex. M-3) ...	April 1, 1936	V	1005				
M-4FF	Lr. Plf. to Dft.	April 2, 1936	V	1006				
M-4GG	Lr. Plf. to Dft.	April 23, 1936	V	1006				
M-4HH	Lr. Dft. to Plf.	April 25, 1936	V	1007				
M-4II	Lr. Plf. to Dft.	April 27, 1936	V	1007				
M-4JJ	Lr. Plf. to Dft.	May 26, 1936	V	1008				
M-4KK	Lr. Dft. to Plf.	May 28, 1936	V	1008				

PART IV — JUDGMENTS, ETC.

DESCRIPTION	DATE	PAGE
Reasons for Judgment, Thorson, P.	May 28, 1947	1044
Formal Judgment of Exchequer Court.	May 28, 1947	1110
Consent as to contents of case.	Aug. 23, 1948	1111
Order of Supreme Court dispensing with printing of certain exhibits.	June 25, 1948	1113
Order of Supreme Court dispensing with printing of Exhibit P-104.	Aug. 23, 1948	1114
Solicitor's certificate.		1114
Registrar's certificate.		1115

DESCRIPTION	DATE	PAGE
IN THE SUPREME COURT OF CANADA		
Reasons for Judgment,		
Kerwin, J.	Dec. 5, 1949	1116
Kellock, J.	Dec. 5, 1949	1121
Rand and Locke, JJ.	Dec. 5, 1949	1131
Estey, J.	Dec. 5, 1949	1141
Formal Judgment of Supreme Court of Canada	Dec. 5, 1949	1149
IN THE PRIVY COUNCIL		
Order granting special leave to appeal	July 21, 1950	1150

Exhibit—W-4.

EXHIBIT—W 4

— C O P Y —

M. S. A. S. Ltd.
N.Y.

RBM/CF

March 19th, 1915.

Dr. S. Gregory, Managing Director,
Minerals Separation American Syndicate (1913), Ltd.,
61 Broadway, N.Y. City.

Dear Sir:—

10 I beg to hand you the following patent specifications covering the production and application of:

KOTRIX, an alkaline sulphohydrate solution. This solution has met with a great deal of success in connection with reconstructed sulphonated mineral or other oils in governing and maintaining the high and uniform extraction of copper, sulphides, oxides, hydrates, carbonates and silicates.

20 STANOL, an alkaline organic sulphide containing a great many complex organic compounds produced from organic sulphides and an alkali. This solution should be of vital importance to the flotation industry, especially so should your Company decide to manufacture it as a patent flotation agent.

GRABANOL, a hydrolized alkali colloidal compound, designed and prepared especially for the production of a spontaneous bubbling froth.

MINOLA, a sulphonated oil. Either mineral, pine organic or other oils, can by the process of affixing sulphur thereto be fashioned to materially effect the savings of valuable constituents of ores.

30 PYROX: The description of permissible reagents that have meritorious value in promoting flotation.

The five enclosed specifications for the application of patents do not fully cover or comprehend the scope of work that I have performed in designing means of promoting differential flotation. As soon as experimental facilities are provided, I will prepare and execute in form five other applications which I now have in mind. Several of the embryo patents resemble the ones now prepared and only differ as to interpretation. This will be in view of strengthening the ones now submitted.

Yours very truly,

40

(Sgd.) R. B. M.

Encl.

Research Chemical Engineer.

Exhibit—W-5.

EXHIBIT—W 5

PROPOSED NAMES FOR FLOTATION COMPOUNDS.

1. KOTRIX.
Alkali sulpho hydrate.
2. STANOL.
Organic Sulphide.
3. GRABANOL.
Hydrolized colloidal compound.
4. MINOLA.
Reconstructed petroleum oil.
- 10 5. ARANOL.
Reconstructed coal tar oil.
6. CINOL.
Reconstructed Pine Oil.
7. PYROX.
Organic and inorganic reagents that assist flotation.

MJB

March 9th, 1915.

KOTRIX

20 Rhetherford B. Martin of Salt Lake City, assigns shop rights or privilege of being employed to the Utah Copper Co. of Salt Lake City, Utah.

Method of treating copper sulphides, carbonates, hydrates, oxides, silicates and such other combination of minerals that accomplish copper ores previous to concentration by the flotation process.

To all whom it may concern:

30 Be it known that I, Rhetherford B. Martin, a citizen of the United States, residing in Salt Lake City, in the County of Salt Lake, and State of Utah, have invented certain new, useful and novel improvement in ore concentration, of which the following is a full, clear, concise and exact description.

My invention has relation to improvement in facilitating ore dressing, and it consists in the novel influence of certain chemical reagents added to aqueous ore pulp, previous to their submission to the flotation cell, of which the specific clauses are more fully set forth in the attached claims.

40 The invention relates to the novel use of an alkali-sulpho-hydrate, specially prepared and more preferably obtained from the intimate mixture of calcium oxide, flowers of sulphur, calcium carbonate and sodium hydrate with certain impurities consisting of aluminum oxide,

Exhibit—W-5.

magnesium oxide and carbonate silica dioxide and the natural occurring salts of iron.

The proportion of these salts are allotted to chemical laws which govern the union of making possible a calcium sulpho hydrate compound. The influence of the sodium hydrate, calcium carbonate and the associated impurities of aluminum oxide, magnesium oxide and carbonate silica dioxide and the oxides of iron shall be proportionately in regards to influences that it is desired to impart to the particular ore under treatment.

10 The main object of compounding these reagents, or selecting certain reagents that contain the impurities hereabove mentioned, is for the obvious reason of having present a compound that will go into solution in boiling water and besides embodying certain selective qualities, that will be requisite (in coating metallic sulphides, carbonates, hydrates, oxides, and silicates with a thin film of amorpheus sulphides), whereby the attachment of oil or other agents to the sulphides, carbonates, etc. are greatly accelerated in accomplishing the ultimate purpose of annexing air to the so affected sulphides.

20 A more preferable concrete example may be defined in the way of explanation by citing that when an intimate mixture of

calcium hydrate or oxide
Flowers or ground sulphur
Calcium carbonate
Sodium hydrate
containing their impurities

are subjected to the presence of water, either hot or cold, and afterwards brought to the boiling point and maintained at this temperature for a definite length of time, decomposition takes place and transmutation of the solid into the liquid state becomes established, resulting into a dark golden yellow alkaline sulpho hydrate solution with a specific gravity greater than water. The compounding system is generally characterized by several controlling features, which may be set forth as follows, namely: That the lime hydrate can be replaced by its equivalent, salt of sodium, likewise the calcium carbonate can be replaced with its equivalent, sodium salt, the sodium hydrate remaining the same or can be entirely dispensed, when the sodium salts are employed in larger quantities than is necessary to combine with the equivalent sulphur

30 to form sodium sulpho hydrate. The method of transmutating the requisite mix alkaline salts to sulphur for the immediate purpose of forming the alkaline sulpho hydrate solution, is most suitably carried out in the presence of eight gallons of water per pound of reagent employed; more or less water does not materially affect the solubility. While in practise it is found to be more expedient

40

Exhibit—W-5.

to limit the dilution to two ounces of solids per gallon of water, a stronger or weaker solution will not interfere with the qualities of this compound. The application of heat is necessary in order to promote the alkalies and sulphur to combine in forming an aqueous solution. Most preferable the heat to promote the reaction may be applied by conducting live steam at normal pressure into a vessel containing the reagents and water for a sufficient duration, or until the water has been brought to a boil, at this temperature the solution of the solids will be accomplished and the continuation of prolonged boiling will be unnecessary. In promoting the solubility of the reagents, several novel procedures may be instituted of applying direct heat to the metallic vessel containing the solution or the innovation of heating under pressure. After cotrix (calcium sulpho hydrate) has been prepared, it may be introduced into the flotation cell either hot or cold. The point of admission should be in advance of the mixing cells and the quantity admitted should be in proportion to other factors, of including the character of the ore and water as well as the nature of the reagent employed in order to combine air under the stress of agitation to the sulphides. The introduction of oil or other flotation agents should not be permitted to enter the charge until cotrix (calcium sulpho hydrate) has been thoroughly incorporated upon the surface of the valuable particles of the ore that it is desired to affect.

In the furtherance of influencing the various flatable constituents of ores to respond to flotation through the medium of employing cotrix (calcium sulpho hydrate) it is preferable to employ in connection with this reagent reconstructed oil (the reconstruction of oil is fully described under separate patent specification.)

30 What I claim is:

- 1st. The compounding of an alkaline sulpho hydrate solution.
- 2nd. More specifically prepared by boiling with a large quantity of water the theoretical equivalent quantities of calcium hydrate and sulphur containing lesser quantities of sodium hydrate in the presence of impurities that contaminate the commercial occurrence of lime and sulphur along with calcium carbonate or sodium carbonate as the case at issue may direct.
- 40 3rd. The manufacture of an alkaline sulpho hydrate solution by increasing or diminishing its component parts in view of instituting certain selective reaction that is requisite to govern the furtherance of treating the numerous occurring complex ores.
- 4th. The production of an alkaline sulpho hydrate solution, whose composition will lend special influence to the

Exhibit—W-5.

- decomposition of such a solution when brought in contact with atomized air.
- 5th. The production of an alkaline sulpho hydrate solution from calcium hydrate for the single purpose of producing a strong alkali sulpho hydrate which will take the place of the more drastic sodium salts in neutralizing acid ores.
- 10 6th. A method of preparing an aqueous sulphonated alkaline solution for the primary purpose of neutralizing acid ores, or otherwise coat such valuable constituents of these ores with a thin film of amorphous sulphides.
- 7th. A process of preparing an aqueous sulphonated alkali solution for the immediate object of influencing sulphides, hydrates, carbonates, oxides, and silicates to combine with flotation agents such as reconstructed oil or other natural occurring oils for the purpose of separating the valuable constituents from the less valuable portion by means of flotation.
- 20 8th. A process of preparing an aqueous alkali sulpho hydrate solution whose properties would be sufficiently flexible as to be commendable in the treatment of complex ores having present either an acid or basic reaction, containing the more complex constituents of carbonates, hydrates, oxides, silicates and sulphides.
- 30 9th. The process of commercially preparing an alkaline sulpho hydrate solution, whose virtue is invested in the special properties of coating metallic carbonates, hydrates, oxides, silicates and sulphides with an infinitesimal coating of amorphous sulphides whose astatic influence of combining with electro-static positive oil is greatly enhanced.
- 10th. The process of preparing an aqueous solution of calcium sulpho hydrate to facilitate and assist oil in the flotation of valuable constituents from colloidal slimes.
- 11th. The process of preparing an aqueous solution of an alkali sulpho hydrate, whereby the same may be used in connection with a sulphonated oil.
- 40 12th. The process of preparing an aqueous solution of an alkali sulpho hydrate for the purpose of dissipating colloidal matter of mineral slimes and at the same time affecting the valuable constituents of these slimes as above mentioned for the ultimate recovery through the means of flotation.

Exhibit—W-5.

STANOL

My invention consists of a process of producing and means for applying the same, in the elimination of certain valuable constituents from ores and other mineral compounds. During the ordinary flotation process by bringing an organic alkali sulphide salt in ultimate contact by the ordinary means or other specific methods while the ore is being transmitted through the numerous devices pertaining to concentration by flotation, more specifically it might be described as a process whereby the production of an alkaline organic sulphide obtained by the action of an alkali upon the numerous organic compounds, such as the hydro-carbon and carbo-hydrates, and the more specific groups of alcohols, carbon disulphide, carbon tetrachloride, and other carbon compounds, such as methanes, carbon monoxide, etc. In the presence of an organic sulphide, or an alkali sulphide is admitted into the ore at the time when the same is being finely divided by the numerous methods of crushing, or may be admitted at any time thereafter-wards by the ordinary specific methods where it would be feasible in many cases to especially provide for the introduction of such a reagent.

This invention which has to do with the separation of sulphides, carbonates, hydrates, oxides and silicates from other minerals, depends upon the fact that when a suitable alkali organic sulphide is brought into a soluble state in contact with valuable minerals, associated with less valuable ores. The effect of producing within a few minutes under the stress of agitation as applied to flotation a homogeneous aeriated flocculent froth when an examination is found to contain valuable products consisting of sulphides, hydrates, carbonates, oxides and silicates. The froth so produced resembles in its physical nature, texture and general behaviour the resemblance of similar froth that has been obtained with a small quantity of some appropriate oil flotation agent. The principle of this coincidence makes possible the concentration of such minerals as not acted upon by other less suitable oily reagents. The silica and inertia matter mixed with other less valuable minerals not so acted upon by this process can be separated in the ordinary methods of flotation concentration where water is employed. The mineral remaining behind after the separation is due to the specific flotation existing or a governed factor in compounding the desired nature of the alkaline organic sulphide solution. The temperature of the water or the ore under treatment has no effect in the process, though it is recommended that the ordinary temperature be preferable. In any event, variation may be practised in the process of manufacturing the alkali organic sulphide by varying and selecting the raw organic material. The essential feature is that the raw material must consist of a

Exhibit—W-5.

purchasable or commercial obtainable substance, that will combine with an alkali or an alkali sulphide and an organic sulphide in producing the necessary alkali organic sulphide. The presence of water and other impurities are essential in promoting the formation of the desired compound. The reaction that takes place is complicated and many compounds that may be classed as impurities are formed, of which the mercaptans, alkynes and esters, are hereby classed as beneficial to the reaction and necessary in the application of compounding the alkali organic
10 sulphide and the ultimate employment of this agent in promoting flotation. The essential feature in the application of this artificial compound to flotation is that the ore must be finely divided and carrying silica, alumina, magnesium, lime, iron, sulphur and other impurities, consisting of the salts of other metals, suspended in water. The amount preferable is from a trace to a higher percentage, though it is possible to obtain the best results with the various ores by adding a commercial percentage, whereby at once on in a few minutes' agitation the affected constituents become
20 unaffected matter, due to the laws of buoyancy and the change of specific gravity.

In preparing the alkali organic sulphide, I prefer the employment of sodium hydrate, as the alkali, and alcohol and water as the organic base, and carbon disulphide as the organic sulphide, though in practise it is feasible to employ other analogous combination effect the same results.

Many localities containing supplies of similar reagents would commercially change the adoption of this formula, and should similar reagents be employed, the effect would be the same.

30 The reaction of combining the several reagents in forming the alkali sulpho organic compound is much excelerated by a gentle heat, though not absolutely necessary, as an exothermic reaction takes place on the addition of the caustic alkali.

The trouble sought to be remedied in flotation concentration by eliminating the practice of employing oil, is hereby preferably substituted by the adoption of the use of an analogous synthetical compound whose properties can be governed as to the final results, which may be further described by effecting certain chemical
40 changes in the various substances before they are compounded in forming the subsequent reagent.

Having thus described my invention, what I claim and desire to secure by *Letters Patent* is:

- 1st. The production of an organic compound, that may be employed to replace oil in flotation.

Exhibit—W-5.

- 2nd. The employment of an organic compound, in preference to oil to influence flotation.
- 3rd. The employment of an organic compound in connection with oil to promote flotation.
- 4th. The employment of an organic compound in connection with an alkali sulpho hydrate to accelerate flotation.
- 5th. The manufacture of an organic alkali alkyne sulphide from the hydrocarbon and carbon hydrate, in the presence of sulphur and sulphur compounds.
- 10 6th. The manufacture and production of an organic alkali organic sulphide that is amenable in producing recoveries through the medium of flotation.
- 7th. The process of producing an alkali organic compound in the presence of water.
- 8th. The process of production of an alkali organic sulphide by having present a suitable reagent containing sulphur.
- 9th. The process of production of an alkali organic sulphide by having present the constituents of water in the presence of a compound containing sulphur.
- 20 10th. The process of production of an alkali organic sulphide by having present an alkali salt, more preferably the hydrate of an alkali.
- 11th. The process of production of an alkali organic sulphide at the ordinary atmospheric temperature.
- 12th. The process of production of an alkali organic sulphide at an elevated temperature.
- 13th. The process of production of an alkali organic sulphide by the exothermic reaction of combining the several constituents.
- 30 14th. The process of production of an alkali organic sulphide to be used in preference of oil in promoting flotation.
- 15th. The herein described process of producing a compound not classed as an oil to be employed in flotation with or in preference to an oil, for the purpose of producing similar results now obtained by the application of oil in the flotation process.
- 40 16th. The process and application of treating silicious and calcareous substances containing valuable sulphides, oxides, hydrates, silicates and carbonates with an alkali organic sulphide for the purpose of receiving through the medium of flotation the valuable constituents.
- 17th. The process of treating the commercial ores which comprises the employment of an alkali organic sulphide through the ordinary means of flotation.

Exhibit—W-5.

- 18th. The process of treating commercial metalliferous ores, consisting in adding a small or large quantity of an alkali organic sulphide to change the character of the surface of the sulphides, oxides, hydrates, silicates and carbonates, and produce a physical alternation by means of flotation in annexing atmosphere gases to the altered surfaces of such constituents that it is desired to recover.
- 10 19th. The process of treating ores combined with silica, which contain impurities, said process consisting in adding to such ores in a finely divided state, an alkali organic sulphide in commercial proportion, by which the valuable constituents are converted by agitation in the presence of air into a spontaneous buoyant froth which can be removed from the unaffected matter by the ordinary means of mechanical contrivance as set forth in the numerous applications of flotation machines.
- 20th. The herein described process of treating ores with a soluble alkali organic sulphide, both agents preferably suspended and dissolved in water.
- 20 21st. The herein described process of attaching air bubbles to valuable constituents of ores by employing an alkali organic sulphide, in the ordinary flotation machine.
- 22nd. The herein described process of producing and applying an organic alkali sulphide as a frothing nunstrum.
- 23rd. The herein described process of producing and applying an organic sulphide alkali to collect valuable constituents of ores into a magma and the subsequent buoyancy elimination of the same by attaching air through the stress of vigorous agitation.
- 30 24th. The herein described process of producing and applying an alkali organic sulphide in order to produce differential flotation of the comprised constituents of valuable ores.
- 40 25th. The claims herein described for this process are based on the reaction of combining an alkali to an organic compound in the presence of an organic sulphide, which brings about reaction and the production of compounds so requisite for flotation concentration, that said reaction can be brought about by a number of other reagents having the same action, but owing to the cheapness and available hydrocarbon and carbon hydrates, it is preferable to employ these agents.

Exhibit—W-5.

MINOLA
RECONSTRUCTED OIL

covering mineral oils, aromatic oils, pine oils, partly acid oils.

The invention relates to the novel use of employing sulphur for the purpose of rearranging the molecular structure of oils, in view of increasing their electro static properties, whereby a more selective extraction between the treated oil, the natural occurring or the artificial sulphide is produced than otherwise possible to obtain by employing the untreated oil.

10 A feature of this invention consists in the employment of commercial oils, either mineral or organic, and by subjecting such oils to the process of distillation, in the presence of sulphur and superheated steam. A valuable oil is recovered (through the proper condensing apparatus connected to the distilling vessel) much superior to the original oil, when employed as an agent to promote flotation.

The apparatus most preferably suggested as a medium of negotiating the problem of combining elementary sulphur to an oily or hydrocarbon consists of the usual distilling apparatus now
20 in vogue among the numerous oil refineries.

The specific novelty of this invention is solely based upon the application of elementary sulphur and oil to any suitable distilling apparatus, now employed in the segregation of the several distillates into their commercial component fraction.

According to this invention the relative percentage of elementary sulphur to the oil cannot be defined as a definite quantity, owing to the fact that 0.25% of sulphur to many oils influences the final flotation properties equally as well as 30%. The smaller proportion of sulphur employed, the smaller will be the percentage of carboniza-
30 tion, and greater will be the yield of the recovered oil.

The employment of a larger percentage of sulphur will affect the production of a greater quantity of residium with a corresponding relative higher percentage of annoying, troublesome gases, which will be the direct influence of limiting the recovery of the desired distillate.

In practice about five percent of sulphur will give credible results to the reconstructed oil.

No claims are specified in regard to the temperature at which the oil adopted for reconstruction may boil.

40 A most promising feature of this invention is based upon the fact that the lighter constituents of oils boiling up to 300°C. lends more preferable advantages of combining with sulphur than the higher boiling fraction. The physical properties of an oil, including the varying homologues as well as the differential boiling points, are

Exhibit—W-5.

influenced by small or large quantities of sulphur to proportionately form waste bi-products that correspond; namely, to the increased percentage of carbon atoms existing in the molecules approaching the higher fraction of oils.

Other processes of combining sulphur with oils have been tried. The nearest to approach with any degree of success to that of heating an oil with sulphur under pressure whereby the identical reaction takes place and the resulting oil approaches a degree of comparison to the ore that has been obtained by distillation.

- 10 The main object of preparing a reconstructed sulphonated oil has been instigated by the development of the flotation process in the concentration of valuable minerals from their less valuable constituents.

The employment of a reconstructed sulphonated oil in flotation presents the sole advantage of making available the numerous inexpensive minerals, and organic oils that do not lend special enchantment to flotation in their original state of existence.

- 20 Another important fact in connection with flotation, relates to the advantage of employing small quantities of mineral oils, as the active agent of gathering sulphides, or causing the sulphides to become attached through the medium of some frothing agents to air bubbles.

However, reconstruction by sulphonation permits the employment of small quantities of petroleum oils as an active agent.

Pine oils that pull a dirty froth in flotation are materially benefited by sulphonation. The resulting product retains the original bubbling effect without incurring the flotation of large quantities of gangue matter.

- 30 A rather striking coincidence in connection with the adoption of reconstructed sulphonated oil namely involves the option of employing a diminished quantity in preference to the large quantity of the original oil to envoke the same effect.

The technics of the reaction that actually takes place when an oil is heated with sulphur and vapourization is based on the rearrangements of the atoms in the molecule, such rearrangement causing the molecule to stay hold and maintain a potential influence of electro static energy, which causes the oil so treated to more effectively coat the sulphides than is feasible to obtain without such treatment.

- 40 In the treatment of colloidal slimes that accomplish metalliferous ores, reconstructed sulphonated oils in connection with Kotrix play an important part of preventing the flotation of large quantities of gangue matter. More specifically the mode controlling the preparation and application of reconstructed sulphonated oil in connection with flotation are itemized in the following claim.

Exhibit—W-5.

What I claim is:

- 1st. The conversion of oils not amenable to flotation into valuable flotation oils by the process of distilling such oils over elementary sulphur and collecting the volatile distillate through suitable cooling means.
- 2nd. The distillation of oils over sulphur by the application of external heat.
- 3rd. The distillation of oils over sulphur in suitable apparatus that has heretofore been applied for such distillation.
- 10 4th. The distillation of oils over sulphur by conducting superheated steam through the distilling apparatus at the time of distillation for the purpose of assisting the vapour to ascend up and through the condenser.
- 5th. The practice of admitting superheated steam to the still, while the process of distillation is involved, is an old invention. However, if sulphur is present a chemical reaction takes place, and the oil so obtained has undergone a change that will not take place providing sulphur was not present.
- 20 6th. Specific claims are hereby set forth, that either a small or large percentage of sulphur will equally effect the desired transformation of valueless flotation oil to valuable flotation oil by distilling.
- 7th. The production of reconstructed oil by distilling oil over elementary sulphur in the presence of steam, produces certain organic sulphide compounds that lend special attachment when brought into an aqueous ore pulp of coating sulphides, carbonates, hydrates, oxides, silicates with a thin film of the sulphonated oil.
- 30 8th. The process of treating slimes or other finely divided metalliferous ores by flotation for the recovery of valuable constituents with an oil that has been rendered efficient through the medium of reconstruction by distilling over sulphur.
- 9th. The process of treating slimes or other finely divided metalliferous ores by flotation for the recovery of valuable constituents by employing a reconstructed sulphonated oil in connection with an alkaline sulpho hydrate solution.
- 40 10th. The process of preparing an oil whose active constituents are based when applied to flotation upon qualifying activity of elementary sulphur imparting certain reaction to the oil through the instrumentality of distillation.
- 11th. The process of employing a reconstructed sulphonated oil in connection with an alkali sulpho hydrate solution in

Exhibit—W-5.

order to promote commercial flotation among complex ores.

12th. The process of employing a reconstructed sulphonated oil for the purpose of promoting selective flotation.

GRABANOL

The invention relates to the novel use of employing reconstructed sulphonated oil, resin and an alkaline hydrate in the presence of water and by heating to produce a saponified colloidal solution which is much superior to the original agents in producing a
10 frothing reagent.

The object of this invention is based upon the behaviour of a colloidal saponified solution promoting certain valuable influences to the flotation cells under the stress of agitation.

A feature of this invention involves the employment in practice of a diminished percentage of active reagents with the corresponding increase in results than is possible to obtain by employing the reconstructed oil and resin mixed in the same proportion.

A very successful hydrolyzed solution prepared was obtained from

20	reconstructed pine oil	25 parts
	“ mineral oil	15 “
	resin (pine)	5 “
	sodium hydrate	5 “
	boiled with water	50 “

and diluted with 5 parts of water for the final solution.

I do not in any sense limit myself to this particular formula which I only give as a concrete example to convey that the larger portion holding in solution the active material consists of water.

An important feature of this invention solely depends upon
30 the solubility of some resinous compound into a menstrum which will permit through the application of an alkaline agent a colloidal saponification more preferably the soluble mixture should be selected from a source that will produce a colloidal solution in preference to the fatty acids that only produce saponification.

In practice the substitution of aromatic hydrocarbon for the purpose of dissolving the resinous compounds, are looked upon with much favor.

The nucleus of the hypothesis governing the formation of a colloidal hydrolysis in this particular case is based upon the solvent
40 power of some selective oil upon resin and the treatment of oily resin solution with an organic sulphide previous to saponification

Exhibit—W-5.

with an alkali. The presence of water and the application of heat is necessary to complete the reaction.

After the oil containing the beneficial presence of such agents and applied treatment that is necessary to furtherance its application; a dilution should be made with water previous to the introduction to the flotation cell. In order to provide the full benefit of any selective action it may have on differential flotation.

10 The colloidal hydrolysis of an oil solution imparts to the flotation charge a bubbling effect, and in many cases the froth so produced will permit by governing the percentage admitted, the selective flotation of certain valuable minerals in preference to other minerals that are associated in a more numerous percentage in the same charge.

Another important coincidence in connection with an oily compound that has been subjected to colloidal hydrolysis is that the employment of a small percentage will effect certain minerals to float, and on removing the so effected minerals by agitation. The remaining minerals can be floated by the application of other flotation agents.

20 What I claim is :

- 1st. The production of a colloidal hydrolysis.
- 2nd. The employment of such agents that will commercially produce colloidal hydrolysis.
- 3rd. The employment of aromatic minerals and pine oils with resin, sulphur, and an alkali to produce colloidal hydrolysis.
- 4th. The employment of special treated and sulphonated oils in connection with alkalis to produce certain effects that are amenable in producing a colloidal hydrolysis.
- 30 5th. The production of colloidal hydrolysis among oils that will produce a spontaneous froth when agitated with water.
- 6th. The production of colloidal hydrolysis among oils whose equivalent properties will be amenable to governing the frothing qualities that it may be desired to institute as a fixed basis in promoting controllable results in flotation.
- 7th. The application of a hydrolized colloidal oil to effect flotation.
- 8th. The application of a hydrolized colloidal oil to effect differential flotation.
- 40 9th. The application of a hydrolized colloidal solution to produce a voluminous froth.
- 10th. The application of a hydrolized colloidal oil to antagonized sulphides, carbonates, hydrates, oxides and silicates to become entrapped in ratio to their electro static

Exhibit—W-5.

- influenced in regard to the quantity fo the hydrolized colloidal oil present.
- 11th. The application of a hydrolized colloidal oil to promote sulphides, oxides, hydrates, carbonates and silicates to float through the medium of employing less preferable oils.
- 12th. The application of a hydrolized colloidal oil to influence less desirable oils to combine with valuable constituents of ores, thereby effecting the use of oils otherwise inefficient in regard to meritorious value of promoting flotation.

PYROX

My invention pertains to the manufacture and the employment of the following reagents to be used in connection with ore concentration by flotation.

The invention relates to the novel use of certain reagents either employed alone or in combination with other agents, to effect namely:

- 1st. The production of colloidal hydrolysis.
- 20 2nd. The production and maintenance of a spontaneous voluminous froth.
- 3rd. The fixation of atmospheric air to the valuable constituents of ores through the medium of agitation.
- 4th. The fixation of artificial sulphur compounds to valuable constituents of ores by forming an unctious film of amorphous sulphides on such constituents of ores that it is desired to recover.
- 5th. The production of an astatic equilibrium that will unbalance the seclusive electro static effect of minerals and cause these minerals to combine with buoyant and froth agents thereby effecting a surface tension and make possible concentration through the laws of flotation.
- 30 6th. The adoption of the several herein described reagents to fashion the final reaction of accelerating the flotation of the valuable constituents of ores.

It is the intention of the domains of this specification to itemize the employment of such agent or reagents, that will fashion or accelerate specific reaction of producing a spontaneous mineral bearing froth, and at the same time to influence such valuable constituents of ores to combine with such agents or as to cause the desired constituents of ores to combine with other agents that will effect their recovery when submitted to the process of flotation.

40

Exhibit—W-5.

The invested issue of great importance nuczalized on the hypothesis of the introduction of such reagents that will hydrolize in water and a colloidal solution, and by means of agitation in the presence of air deposit a film of amorphous sulphide upon the surface of certain constituents of these ores in preference to other accomplished constituents, thereby influencing certain portions of ores to mechanically separate by definite flotation laws brought on and accelerated by the edition of the foregoing reagents.

Inorganic compounds.

10	ammonium arsenate
	“ arsenite
	“ per-sulphate
	“ sulphide
	“ sulpho hydrate
	“ “ cyanate
	barium sulpho-hydrate
	cadmium sulphide (colloidal)
	calcium sulpho carbonate
	“ “ hydrate
20	carbon monosulphide
	hydrazine sulphide
	hydrogen per sulphide
	magnesium sulphide
	nitrogen sulphide
	potassium monna sulphide
	“ di “
	“ tri “
	“ tetra “
	“ penta “
30	“ sulpho hydrate
	silicic acid
	“ chlorohydrosulphide
	sodium arsenate
	“ arsenite
	“ hydrosulphide
	“ sulpho cyanate
	“ thio arsenate
	“ “ carbonate
	sulphur colloidal
40	Inorganic Oil Substances.
	Antimony penta fluoride
	chlorine hep-oxide
	carbon bisulphide
	hydrozoic acid

Exhibit—W-5.

hydrogen per sulphide
 manganese hept-oxide
 phosphorus-tri-sulpho-tetra-bromide
 potassium thio stannate

Organic Compounds.

	Arsenic-diethyl
	buthyl mercaptan
	“ sulphide
10	cacodyl
	“ sulphide
	carbon disulphide
	chlor naphthaline
	“ phenol
	diethyl sulphite
	“ sulphide
	diemethyl sulphite
	“ sulphide
	di phenyl benzine
	“ phosphine (oil)
	“ sulphide.
20	alcohol-alkolates
	“ arsenate
	“ arsenite
	“ carbonate
	“ chloride
	“ disulphide
	“ hydrazine
	“ hypochlorite
	“ mercaptan
	“ phenol hydrozine
30	“ phosphine
	“ sulphate
	“ sulphide
	alcohol-sulphite
	“ sulphocyanate
	“ sulphone
	“ thio carbonate
	caproic acid
	carvacrol
	citral
40	creosole
	cyclo pentene
	diethyl aniline
	dihydro benzaldehyde
	“ beniyne

Exhibit—W-5.

	diiso amyl ketone
	dimethyl-nitro-amine
	diphemol phosphine
	ethoxy benzoic acid
	ethyl diazoacetate
	“ disulphide
	“ laurate
	“ per chlorate
10	“ phenol hydrozine
	“ propiolate
	<i>ethylene nitrate-nitrite</i>
	euganol
	hept amethylene
	hexenyl ether
	hydrindene
	hydroxy-benzoaldehyde
	<i>Indoxyl</i>
	keto butyric acid
	keto heptamethylene
20	linoleic acid
	methyl furfurol
	“ mercaptan
	“ naphthyl amine
	“ nitro benzol
	“ sulphate
	“ sulphide
	“ sulphuric acid
	nitro anisol
	“ methane
30	“ propane
	“ styrene
	olein
	penta methylene
	phenyl pyridine
	“ quinoline
	phthalyl chloride
	suberone
	thiaphene alchehyde
	tin diethyl
40	tolune-sulphone chloride
	triazobenzene
	trithiocarbonic acid
	vinzy sulphide
	phenyl disulphide
	“ sulphide

Exhibit—W-5.

propyl mercaptan
 “ sulphide
 sodium-ethyl
 “ methyl
 sulpho acetic acid
 sulphonol

Organic Oils.

10 analine
 amino thiaphene
 benzyl ether
 bismuth tri ethyl
 bromo phenol
 “ hydridine
 butyl benzorote

These reagents are to be employed alone or in combination with other flotation agents.

The percentages required to effect a definite result are comprehended to be within the commercial application of similar reagents.

20 The main object of specifying certain groups of chemical reagents to promote or assist flotation has been instituted through the investigation of those groups of reagents that imparts definite and valuable assistance to the flotation of commercial constituents of ores.

It has been discovered that these chemical reagents embrace the alkali sulphides, the alkali organic sulphides and other solutions that produce an astatic colloidal hydrolysis. It also comprehends the influence of the hydrogen salt of silica (SiH_4) Hydrogen Hydroxide ($\text{N}_2\text{H}_4\text{H}_2\text{O}$) Nitrogen Sulphide (N_4S_4), etc.

30 Hydrogen effects flotation by causing air to combine upon the surface of the valuable constituents of ores, especially so if applied in compounds that will undergo a decomposition on coming into contact with atmospheric air and water.

The trouble sought to be remedied in flotation concentration is by eliminating the objectionable and ungoverned influence of oils. This is preferably done by the introduction of a working quantity of some appropriate reagent with or without the addition of oil.

40 The control of the working condition of the froth producing machine is much simplified and a much better extraction can be obtained by the addition of some appropriate chemical agent that has been employed in connection with flotation.

Having thus described my invention. What I claim and desire to secure by letter patent is:

Exhibit—W-5.

- 1st. That the laws of flotation or selective flotation is based upon certain definite astatic influence valuable constituents of mineral water and air under the stress of agitation in the presence of oils. In order to excelerate or govern this force, it has been found that certain classes of chemical reagents belonging to a definite system have beneficial effect on the success of floating the valuable constituents of minerals.
- 10 2nd. The herein described claim that the definite chemical reagents embrace organic alkali sulphides and their combination, the alcohol alkolates and their combination, the arsenates and their combination, sulphur and the combination of sulphur compounds,
- 3rd. Nitrogen and the combination of nitrogen compounds.
- 4th. The employment of the above reagents alone or in combination with other agents to effect flotation.
- 5th. There herein described process of applying the above reagents as named in preference to other chemical agents to produce flotation.

20

UNNAMED SPECIFICATION

My invention relates to a process for bringing about a condition in sulphides, oxides, carbonates, hydrates, silicates, or their combination, to facilitate separation or reorganization of the constituent parts, and pertains especially to a process of producing and for subsequently eliminating by means of concentration through the medium of flotation, valuable metallic constituents from their inertia bases.

30 More specifically it comprehends the addition of a solution or a suitable agent of an alkaline sulpho-hydrate nature into the ore at the time when the same is being finely divided by the numerous methods of crushing; or at any time where it would be feasible for the immediate purpose of facilitating the ultimate recovery of the values by rendering the metallic constituents thereto more amenable to further treatment by ordinary methods of flotation than has been possible heretofore, by causing an alteration on the surface of the sulphides, oxides, carbonates, hydrates, silicates, or their combination, which will result in a complete recovery of these complex ores, by employing a reconstructed oil, in connection with flotation.

40 This invention as it has to do with the separation of metallic constituents from their associated impurities, depends upon the fact that when a suitable alkali sulpho-hydrate or alkaline sulpho-carbonate is brought into a soluble state in contact with sulphides, oxides, hydrates, carbonates, silicates and their seclusive combination, in the presence of water and air, the vehicle of effect is to produce within a few minutes a coating which on examination is found to be a

Exhibit—W-5.

minute amorphous sulphide skum, surrounding as the case may be, the sulphides, oxides, hydrates, carbonates, silicates and their seclusive combination, these minerals are altered by the attachment of the surface tension of the newly formed amorphous sulphides, thereby displaying selective surface tension towards oils, and especially reconstructed oils, making possible the mechanical concentration by means of flotation of such minerals that at the present procedure of the application of flotation do not respond to a profitable separation.

10 The terms, alkali sulpho-hydrate, alkali sulpho-carbonate and alkali sulpho morcaptan, reagents, as I use the same in this specification and claims, refer to such an agent which has the quality and properties either alone or mixed together as the case of the ultimate separation of the valuable constituents of an ore may direct to separate from their impurities. The net results of employing these three solutions, alone, or in such combination of percentages as may give the best results for a given ore, all ores do not respond to the same solution, therefore it becomes necessary to change the proportion of the several solutions, or at times on certain ores to employ a single solution alone in preference to the combination of any two, or the
 20 combination of the three solutions. As the alkali sulpho-hydrates and alkali sulpho-carbonates are very ably described in the Watts chemical dictionary, prevents me from entering their mode of manufacturing here, though the discussion on the alkali mercaptans has not been so fully discussed, suggests that for brevity a description of the manufacture of the alkali mercaptans as applied by me in the treatment of floatable minerals should be discussed. The method I employ in producing an alkali mercaptan, is to act upon carbon disulphide alcohol and water in the presence of an alkali the more preferable the saturated solution of sodium hydrate. The alcohol percentage may be
 30 reduced or entirely dispensed with by employing glucose, glycerine, plenol, benzol, or the gases of mathane carbon monoxide, acetylene or other hydrocarbon gases produced in producer gas manufacture practice. The mixtures of the several reagents are proportioned according to the desired results which it is wished to obtain, though for commercial reasons it is always necessary to limit the proportion of the organic agent in this compound. The union of this solution should always take place in the presence of sulphur, or the presence of an alkali sulpho-hydrate or carbonate in order to prevent the cost of such a solution from being too high. The solution can be prepared either in
 40 the cold, or by gentle heat, or even boiling under pressure. Any of the modes of preparation will give on cooling a highly crystalline mass, for commercial application in promoting flotation either alone or intimately mix with sulpho-hydrate sulpho carbonate the crystalization should be dissipated by dissolving in water.

The term reconstructed oil as applied in connection with the use of alkali sulpho-hydrate, sulpho-carbonate and mercaptan, is best

Exhibit—W-5.

prepared by distilling an oil, which may include petroleum, pine oil, coal tar oils, or any cheap oil over sulphur, for the purpose of investigation I have carried out experiments directing the re-construction in the presence of diversified proportion of sulphur, in order to determine what percentage is most suitable, the conclusion derived was that commercially it was more feasible to employ a small percentage of sulphur, as a larger amount caused carbonization and resulted in a smaller proportion of oil recovered. The procedure may be carried out in the absence or the presence of steam. The oil so prepared is much
10 superior in its application to flotation than the same oil without such a treatment.

The reconstructed oil works well alone, though not as well as it does in conjunction with the alkali sulpho-hydrates sulpho carbonates and the alkali mercaptans.

While the temperature of the water or the ore pulp under treatment generally has no effect on the process, the ordinary temperature is preferable.

The means for bringing the sulphonating reagent in contact with the ore may be accomplished by any of the numerous devices now
20 employed in ore agitation, such as mechanical stirrers, tumbling machines, air or steam agitators, etc.

In using the alkali sulphonating solution, I prefer to employ as much as possible the calcium oxide and hydrates as they are much cheaper to obtain, these salts may be converted into calcium mon-sulphide by passing the sulphur dioxide secured from the roasting of metallic sulphides over incondescent coke in the presence of water as hydrogen sulphide, and subsequently acting upon this compound with elementary sulphur in the presence of water and more preferable the mixture should be heated to the boiling point of water for its complete
30 assimilation, and the reddish brown solution drawn off, probably the most feasible way is to mix according to Ca (SH) 2 slack lime with its ratio of elementary sulphur and boil the mass in the presence of water until the water has dissolved the soluble contents of the lime and sulphur as a calcium sulpho-hydrate, which has the same properties as the solution constructed from the mono-calcium sulphid. I prefer that the calcium should have present as impurities magnesium, silica and iron.

The reaction of combining the above alkali salts with sulphur in the presence of water, seem to accelerate the flotation value by an
40 additional amount of the stronger alkalies such as sodium or potassium hydrate, sodium carbonate, or calcium carbonate.

The trouble sought to be remedied in flotation concentration is by eliminating the objectionable impurities such as ore gangue from the more valuable constituents, this is preferably done by producing such a change on the surface of the valuable particles, as to cause them to readily combine with the oil present, which in the above case the re-

Exhibit—W-5.

constructed oil is more preferable. The whole process may briefly be described as the one now in vogue, with the exception of employing a more active combination of oils and reagents to produce more profitable results with all kinds of ores, than has been possible under the present system. The present system of flotation is not sufficiently flexible to embrace the recovery of rebellious sulphides, or does it include the flotation of carbonates, hydrates, silicates.

The reaction of the sulphonating solution upon sulphides, may be summarized as a great advantage when it is taken into consideration
10 that the extraction of sulphides are limited to the astatic extraction of the more preferable sulphides, thereby leaving the more feeble sulphides in the tailings as a complete loss.

The surface attraction (?) of sulphides, as well as the coating of the oxides, hydrates, carbonates and silicates by employing the alkali sulphonated mixture is greatly an influence towards increasing its surface tension in combining with oil and air, to form a suitable froth, by increasing the ratio of the alkaline carbonate in the mixture, the bubbling effect of the slimy clays is reduced. By increasing the amount
20 of the free caustic alkali the fine sulphides are more proficiently influenced to combine with the bubbles of the froth, there is of course a limit in which these combinations can be applied to commercial practice.

Profitable results have been obtained in many cases with certain combination of the variation of these solutions that was not possible to obtain in any other way.

This process is based on the reaction of influencing the alkali sulpho-hydrates sulpho-carbonates and sulpho alkali mercaptan to combine for the purpose of separating from their seclusive combination sulphides and other valuable minerals in the presence of water from
30 their less valuable constituents by being brought into contact with air bubbles produced by the mechanical stress of agitation.

In most instances, the reagents used are added in minute quantities to bring about the desired reaction which is the freeing of the contaminating matter from the more valuable material, or the application of causing the more valuable constituents to agglomerate into a magma whereby its removal is possible with some appropriate oil either plain or more preferable the reconstructed, sulphonated oil, more fully described in a separate application. An oil so produced
40 seems to have the properties of lending its sulphur annexation to the combining force of the sulphur of the alkali-sulphonated aqueous solution causing a deposition amenable to forming amorphous sulphides on the surface of the metallic salts, which combine more readily with an oil that has been sulphonated than an oil that has not been so treated.

Having thus described my invention, what I claim and desire to secure by letters patent is:

Exhibit—W-5.

- 1st. A process for the manufacture of certain voluble solution, comprehending more fully the alkali sulpho hydrates, alkali sulpho carbonates and the alkali organic sulphides, and reconstructed sulphonated oil.
- 2nd. A process for the application in connection with flotation the employment of the alkali sulpho hydrates, alkali sulpho carbonates and the alkali organic sulphides and reconstructed sulphonated oil, the latter more fully described in a separate application for patent.
- 10 3rd. A process for the employment in flotation of the combined reagents consisting of the alkali sulpho hydrates and carbonates, and the alkali organic sulphides and reconstructed sulphonated oil, or the employment of such combination or single groups of these reagents that will render the most expedient results when applied to the numerous complex ores.
- 40 4th. The employment of such quantities of these reagents that will render the most specific results.
- 20 5th. The employment of these reagents in varying quantities, so as to affect a given mixture, that will harmonize as to the requirements of the final mixture, in influencing certain desired reaction to be imparted, so as to direct the influence of the final mixture in governing the flotation of any single species of mineral or the combination of such species, that it is desired to recover by flotation.
- 30 6th. The process of treating sulphides, oxides, hydrates, carbonates and silicates of the metallic metals salts by an alkali sulpho-hydrate sulpho-carbonate alkali mercaptan agent to produce a film of amorphous artificial sulphide on the surface of these minerals.
- 7th. The herein described process of treating sulphides, oxides, hydrates, carbonates and silicates to produce an amorphous artificial sulphide on the surface of these minerals, which is more amenable to separation by the ordinary flotation methods of concentration.
- 40 8th. The herin described process of treating ores containing the compounds of sulphides, hydrates, carbonates, silicates or other metallic salts either soluble or in soluble by a suitable agent to produce a coating of amorphous artificial sulphide.
- 9th. The process of treating complex ores containing sulphides, hydrates, oxides, carbonates and silicates, which comprise the employment of an alkali sulpho-hydrate sulpho-carbonate alkali mercaptan reconstructed sulphonated oil for the purpose of acting upon the sulphides hydrates, oxides, carbonates and silicates in order to produce an external

Exhibit—W-5.
Exhibit—G-2.

- influence on the surface of the valuable mineral for the sole purpose of rejecting or collecting through the medium of flotation any part thereof.
- 10th. The process of employing in conjunction with an alkali sulpho-hydrate sulpho-carbonate alkali mercaptan a reconstructed oil for the single purpose of producing, promoting and facilitating flotation that otherwise is not possible.
- 11th. The process of employing alone or in combination an alkali sulpho-hydrate sulpho-carbonate alkali mercaptan, reconstructed oil, in order to facilitate flotation.
- 10 12th. The process of employing an alkali sulpho-hydrate sulpho-carbonate alkali mercaptan, reconstructed oil to change the surface character of the natural occurring sulphides, hydrates, carbonates, oxides and valuable silicates into an amorphous artificial sulphide envelope which will more efficiently combine with air and separate from the gangue matter when under the stress of agitation as is employed in flotation.

EXHIBIT—G 2

20 MINERALS SEPARATION AMERICAN SYNDICATE,
(1913), LTD.
RBM/MJB

New York, May 6th, 1915.

Dr. S. Gregory, Director,
Minerals Separation American Syndicate, (1913), Ltd.,
Dupont Hotel, Wilmington, Del.

Dear Sir:—

I beg to request the permission to have executed by some competent draughtsman the transmutation of several rough sketches
30 which I have prepared into a working drawing. They consist namely:

First — One apparatus for the reconstruction of petroleum oil which will be very necessary in harmonizing petroleum oil to flotation. This apparatus should be designed of sufficient capacity in order to produce at least 500 gallons of reconstructed oil per day and probably will have to be installed in the vicinity of the concentrating plant in which it is desired to use the oil in connection with flotation. The drawing should not cost more than \$10., the apparatus will cost about \$500.

Exhibit—G-2.

Second — One apparatus for producing Kotrix. Kotrix (Calura) is the alkaline Hydro Sulphide used so extensively at the Utah Copper Co. The drawing of this apparatus will not cost more than \$5. The installed apparatus per 1000 gallon capacity will cost approximately \$500. We should have prepared in advance the working drawing of these apparatus so as to be in a position when called upon to make a quick installation.

10 *Third* — The design of a flotation cell which represents a new departure in agitation. The construction of this cell will necessarily have to be based upon certain experimental facts obtained from a working model. The value of this cell chiefly depends for agitation in the consumption of less power than is required in the ordinary flotation cell, which fact should be of sufficient importance to warrant the construction of a model.

Another item of importance is the subject of obtaining one of the Janney's laboratory glass side flotation machines. The several sources in which we have tried to obtain this machine have failed, and in my opinion our only resort would be in the construction of one of these machines.

20 In view of considering the many diversified ways of negotiating flotation, I think you will agree with me that the flotation problem in the future will largely depend upon certain fundamental facts based upon the construction of a flotation cell. In order to be successful, it must consume less power than is represented by the cells now in use. Upon the construction of the cells largely depends the possibilities of using the numerous inexpensive oils with the additional advantage of the consumption of a small quantity of oil per ton of ore treated.

30 The distant future in my judgment will not tax the consumption of expensive organic oil, but will wholly depend upon the admixtures of chemicals and specially prepared synthetical substances with petroleum oils in producing flotation.

Therefore, we should install the laboratory with sufficient equipment in the quietude of some suburban building, free from the possibilities of annoying a congested district with the single purpose of taking advantage of the opportunities of working out the available compounds that will minimize the cost of flotation.

40 The cost of rental for a laboratory in a suburban vicinity will not be as expensive as we might expect in some office building. The opportunities of carrying on our work will not be hampered by such an isolation.

Installing the laboratory in the vicinity of New York City would give the director's office a close touch with the progress and development that will take place from time to time as the work is carried on. The processes developed in the laboratory in working

Exhibit—G-2.
Exhibit—P-102.

out new methods could as well be installed in the numerous mines from this point as from any other place.

Yours truly,

(Sgd.) R. B. MARTIN,
Research Chemical Engineer.

EXHIBIT—P 102

MINERALS SEPARATION AMERICAN SYNDICATE,
(1913), LTD.

RBM/MJB

10

New York, May 25, 1915.

Dr. Gregory,
Office.

Dear Sir:—

The equipment of the contemplated laboratory will require in its entirety samples of the numerous crude petroleum oils. These samples should be secured in occurring districts adjacent to the mining properties in which later on it may be desired to employ the local oil in effecting flotation.

20 I am quite anxious to secure for immediate experimental purposes samples consisting of at least five gallons of the crude petroleum oil obtained from the several producing districts namely: Mexico, Texas, Oklahoma, California, Wyoming, Ohio and Penn., also a five gallon sample from each district, representing the cheapest commercial cut that the several refineries produce, approaching a 30 Be. distillate.

30 These samples will indicate by tests the responsive selection of securing a particular occurring oil in preference to one secured from a different district, in relation to the value obtained by reconstruction or the economy incurred by the adoption of a single oil in comparison to another for a given class of ores.

The importance of possessing these various samples from widely situated districts is justified by the supposition that reconstructed oils impart beneficial results to flotation and that all oils reconstructed do not have this essential feature. Reconstruction benefits many oils for flotation in rather a peculiar way, some oils of the Mongrel type when reconstructed are far more affected in gathering up a certain species of sulphides than is possible to obtain by the reconstruction of the asphaltum of paraffine base oils. While

Exhibit—P-102.
Exhibit—G-17.

with other types of sulphides the reverse is the condition, and the employment of the opposite oil bases for reconstruction is very necessary.

Yours very truly,

(Sgd.) R. B. MARTIN.

EXHIBIT—G 17

MINERALS SEPARATION AMERICAN SYNDICATE (1913),
LIMITED.

62, London Wall,
LONDON, E.C., 6th July, 1915.

10

Dictated to M.E.P.
by J.B.

Dr. S. Gregory,
c/o Henry D. Williams, Esq.,
61, Broadway,
NEW YORK.

My dear Doctor:

Mr. Martin sent me a copy of his memo. styled "Permissible Oils employed in Flotation". I have looked at it and certainly
20 tried to read it; but I must frankly confess that I have not been able to make much of it. Whether it is due to my dull understanding, I don't know — I am therefore anxious that you should read it yourself, and discuss it with Mr. Martin, and perhaps also with Mr. Higgins, with a view to getting at the real meaning of the fundamental principles he referred to, and if possible transcribing it into language which can be understood by ordinary people employed in our business. I should also like to have copies of the particulars of his inventions which he gave Mr. Williams soon after we
30 accepted him.

Yours truly,

(Sgd.) JOHN BALLOT,
Managing Director.

Exhibit—G-18.

EXHIBIT—G 18

MINERALS SEPARATION AMERICAN SYNDICATE (1913),
LIMITED.

62, London Wall,
LONDON, E.C., 20th July, 1915.

Dictated to M.E.P.
by J.B.

10 Dr. S. Gregory,
c/o Henry D. Williams, Esq.,
61, Broadway,
NEW YORK, U.S.A.

My dear Doctor:

Thanks for yours of 6th July with enclosures.

* * * *

20 *R. B. MARTIN.* I am looking forward with interest to hear from you, or from Higgins through you, as to the progress of his investigations. The extraordinary style adopted by Martin in the effusion which I wrote you about, has shaken my confidence very much, to say the least I am very sceptical. I am therefore anxious to hear how you size him up and especially also what Higgins thinks of him and his alleged inventions.

Yours very truly,
(Sgd.) JOHN BALLOT.

Exhibit—G-19.

EXHIBIT—G 19

12th St., near Vernon Ave.,
Long Island City,
21st July, 1915.

Dr. S. Gregory,
61 Broadway,
New York City.

Dear Dr. Gregory:—

10 With regard to your communication of the 19th inst. I may
say that the new method for the treatment of carbonates and
oxides is not yet developed, being in reality only in its infancy.
One reason for this is the lack of ores containing substantial quan-
tities of carbonates or oxides. Mr. Nutter's letter is doubly welcome
in offering material for the examination of this process, with possi-
bly commercial application of the latter, Mr. Nutter should,
therefore, either send or direct the Engels Copper Mining Co. to
send me a sample of about 200 lbs. of their carbonate ore at the
earliest possible moment. Further, if he can supply me with any
20 and Zinc carbonates are necessary to me, I shall be much obliged if
he will cause them to be forwarded at his earliest convenience.

I may say that I have spoken with Mr. Williams on the
subject of handing Mr. Nutter such information as I have, and
that he entirely agrees with me, that it is much preferable for me
to first have the ores for examination.

Yours very truly,

(Signed) A. HOWARD HIGGINS.

MINERALS SEPARATION AMERICAN SYNDICATE
(1913), LTD.

30 ss. New York (7.24.15)

SG/EAD

NEW YORK, July 22nd, 1915.

My dear B:

I have yours of the 6th July.

MARTIN — PERMISSIBLE OILS. I will take this matter
up at the earliest opportunity and communicate with you. Enclosed
please find copy of a letter from Higgins, dated July 21st, regarding
carbonate ores. I have also asked Higgins to go into the matter
of patents, etc. with Williams. I think Higgins' idea of carrying on
40 tests here is very desirable.

Yours very truly,

(Sgd.) S. GREGORY.

Exhibit—G-5.

EXHIBIT—G 5

MINERALS SEPARATION AMERICAN SYNDICATE
 (1913), LTD.
 ss. Philadelphia (7/31/15)

SG/EAD

NEW YORK, July 28th, 1915.

My dear B:—

Reverting to the first page of your letter 6th July, I have had a long discussion with Higgins, and send you herewith his report up to date which I think will satisfy your requirements. I am also sending you copies of Martin's inventions, which he gave Williams.

* * * *

MARTIN INVENTIONS—I find that I shall not be able to send you copies of these as promised earlier in my letter by this mail.

* * * *

R. B. MARTIN—Higgins' report is enclosed which brings this matter up to date. We shall have a conference next week with Williams and you will hear from me.

Yours very truly,

(Sgd.) S. GREGORY.

Exhibit—G-6.

EXHIBIT—G 6

12th, St. near Vernon Ave.,
 Long Island City.
 30-7-1915.

Dr. S. Gregory,
 61 Broadway,
 New York City.

Dear Dr. Gregory,

10 Re *Mr. Martin's Classification of Permissible Oils Employed in Flotation*. It is very difficult to regard this document seriously since it is based partially on wrong statements, and partially on conjectures. Further, it is not expressed in plain language and leaves much for the imagination of the reader to supply in order to make sense of the text. The paragraph on the History of the Flotation Process is perhaps a sufficient example, notably the second condition which is cited as being one of the chief evils in retarding the general adoption of, presumably, the Everson process:—"It was assumed in the early experiments that certain fatty acids played the part, with the addition of some mineral acid of generating Hydrogen gas on
 20 the sulphide particles" The document is a conglomeration of generalities in which no trust can be placed because of the glaring mis-statements with which it is permeated. Under these circumstances it does not appear worth while re-writing it, especially as any alleged fact or statement with which we are not already familiar must be corroborated by experiment.

The classification of oils has not been made with regard to different classes or ores, nor does it help one to select an oil for a particular ore.

30 Part II is easily disposed of, being frankly admitted as a compilation from Lunge's "Coal Tar and Ammonia".

Part III consists of results of fractional distillation of oils and gives tests on some of the fractions. It should be noted that only in one test was a quantity of 2.lb of oil per ton or less used. Many of them run onto hundreds of pounds. The case in point is one of the fractions from the distillation of Utah Coal Tar. This material we also distilled in London with results that showed that equivalent fractions to Mr. Martin's could be used profitably in smaller quantities.

40 As regards the inventions made by Mr. Martin very little is found in the document about them, neither is any method of preparation given. Such information as is there is not found to be correct. For instance Kotrix is stated to be Calcium hydro-sulphide, this compound as is seen from the method of preparation given in the patent specification to be Calcium polysulphide.

Mr. Martin's Patent Specifications. I hope to be able to have shortly a further conference with Mr. Williams respecting these.

Exhibit—G-6.

They are, however, imperfect and will require considerable modification before filing.

Work at Long Island City Laboratory. Mr. Martin has made several of his preparations notably Kotrix, Stanol, Grabanol, and Minola. As it is essential that the processes suggested by him should be properly protected by the widest patents, I have spent much time in the careful examination of some of these compounds.

10 *Kotrix.* Mr. Martin gives a method of preparation of this compound in a proposed patent specification viz:—by boiling flowers of sulphur with calcium hydrate or lime. This results not in the formation of calcium hydro-sulphide but in calcium polysulphide, the former compound cannot be prepared by that method. Several solutions have been prepared and tests made, but no assay returns have yet been made. It is certain, however, that the carbonates are floated. Experiments are in hand to find the cause, of which two are possible, first, the action of the sulphide itself, and second, the action of a precipitate which forms on diluting the solution. As yet, I have arrived at no definite conclusion, although the sodium sulphide and sodium hydro-sulphide solutions which do not give any
20 precipitate on dilution also float copper carbonates. One thing is certain—i.e. the metallic sulphides, carbonates, hydrates, oxides, and silicates are not coated with any visible film of amorphous sulphide, but remain unaltered in colour. Otherwise, the invention is the same as Sulman and Picard British, 26,019 of 1909. The Kotrix is effective if used in small quantities, an excess reducing the quantity of froth, which it eventually destroys.

I am trying to obtain samples of "Alkali-waste" and "Gas Lime" which are also calcium sulphides, to find if these act in the same manner.

30 A promising result was obtained on Anaconda feed to flotation plant with Kotrix and Grabanol. Further work is necessary on this material before conditions are established for commercial work. It is interesting that this result was obtained in the absence of heat and acid.

Stanol. Two or three specimens have been prepared from alcohol (denatured) and from wood spirit (methyl alcohol), but as yet, Mr. Martin has been unable to prepare a specimen from a carbo-hydrate, or from a hydrocarbon which gives any result.

40 *Janney Machine.* This was unsatisfactory as a testing machine for dealing with slimes, in that the spitzkasten bottom was not steep enough to return the whole of the tailings to the mixer, the pipe connecting the spitz and the mixer was also deemed to be unsatisfactory. The machine has, however, been altered to suit Mr. Martin's requirements.

Yours very truly,

(Sgd.) A. HOWARD HIGGINS

Exhibit—G-7.
Exhibit—G-20.

EXHIBIT—G 7

Sixty-one Broadway
New York.

3rd Aug. 1915

My dear B.

Many thanks for yours of the 22nd July.

(page 2) You will note from Higgin's report that he takes your views as to Martin. Nevertheless he is of opinion that we can get value out of him. I have told Higgins it is up to him to get all he
10 can out of Martin. I feel very hopeful of the solution of the carbonate and oxide copper ore problem; so is Higgins. Nutter is sending us samples of one for testing. For the present I think Higgins cannot spare waiting but I will bear it in mind.

EXHIBIT—G 20

MINERALS SEPARATION AMERICAN SYNDICATE (1913),
LIMITED

62, London Wall,

LONDON, E.C. 10th Aug. 1915.

Dictated to M. F. P.
by J. B.

20

Dr. S. Gregory,
c/o Henry D. Williams, Esq.,
61 Broadway,
New York.

My dear Doctor,

Thanks for yours of the 28th ult. which came to hand this morning.

Martin's Inventions. Thanks very much for copy of Higgins' letter of the 30th. I quite agree with him, in one sense, "that it is
30 very difficult seriously to regard "Martin's screed entitled "Classification of Permissible Oils etc." I feel sure the man has a screw loose in his brain machinery, or some other unexplainable "kink". The names he gives to his various reagents are quite enough to make one suspicious, and if a practical process to float carbonates and oxides is really evolved from his chaos of words and material, we shall have to thank Higgins for that and not Martin, but how will that effect the patent question?

With kind regards, dear Doctor,

Yours very truly,

(Sgd.) John Ballot,
Managing Director.

40

Exhibit—G-9.

EXHIBIT—G 9

12th St. near Vernon Ave.,
 Long Island City, N.Y., U.S.A.
 14 Aug. 1915.

Dr. Gregory,
 61 Broadway, N.Y.

Dear Dr. Gregory,

Re Mr. Martin's Inventions. The investigations of the application of these inventions to the treatment of carbonate ores continues. Some work has been done on a sample of Miami ore, which was the only available sample containing an appreciable amount of copper carbonate. In this case the best results have been obtained by the addition of the "Kotrix" after the removal of the bulk of the sulphides.

Samples of mineral carbonates have been obtained and mixed with sand for further work. With this material very good indications have been obtained with soluble sulphide added to the pulp, both before and after the addition of, and agitation with, the oil; with "Reconstructed" oil; and with "Grabanol" (Emulsion of "reconstructed" oil). These results suggest that the effect is due to combined sulphur in the oil. "Kotrix" may, therefore, be a method of modifying the frothing agent. If the invention can be placed on this basis Mr. Williams assures me that the patent position will be both simplified and strengthened. The work in hand has, therefore, this object. It should be pointed out that, to the present, results have only been obtained with Pine oil or similar material—Crude wood turpentine, for instance.

Anaconda Feed to Flotation Plant. Mr. Martin is investigating the application of his inventions to this material. The results, as yet, are not, however, so good as those obtained by the conditions already in use at the mine.

Janney Machine. I have already stated that this machine required alterations. For the benefit of future users I may point out:—The spout or lip which deflects the stream of pulp is the spitzkasten is movable, it should be adjusted to leave the aperture of the circular launder fully open. The spitzkasten bottom is not sufficiently steep for use with finely ground ore, ours was increased in slope by about one-half inch in its length. This was done by first cutting away the spitzkasten, than after the adjustment had been made, again welding to the mixer. Whilst the spitz was away, the hole between the spitz and the mixer was filled with aluminum by welding, and a new hole bored at an angle to prevent the sands settling. Mr. Martin informs me that it is highly important that the position of entrance of this hole in the mixer should not be altered; numerous experiments had to be made to determine the best position. The overflow lip was also too high. The proper position was found

Exhibit—G-9.
Exhibit—G-3.

by filling the machine gradually with water containing a soluble frothing agent and marking the level where the biggest froth was obtained. This position is at the horizon of the lower edge of the discharge opening of the circular launder, in other words no banking up of pulp should occur in the latter. The alteration was made by sawing and filing the discharge lip until lowered to the selected position. A brass apron was then fitted to make a discharge spout.

Yours very truly,

(Sgd.) EDWARD HIGGINS.

10 EXHIBIT—G 3

12th St. near Vernon Ave.,
Long Island City,
New York, U.S.A.
August 15th, 1915.

PREPARATION OF FLOTATION REAGENTS

Kotrix, Minola, Cinol, Arenol, Grabanol,
Stanol.

No. 2

PREPARATION OF KOTRIX

20 On the basis of observing that certain porphery copper ores require varying admixtures of alkali sulphides to the aqueous ore pulp, in order to facilitate and effect the combining power of the administered oil to the metallic sulphides, leads to a series of experiments that eventually culminated in the adoption of a solution prepared by boiling calcium oxides or hydrated lime with sulphur in the presence of water.

The most successful solution so far prepared was made by the intimately mixing finely divided lime secured by slacking the oxide in the vessel previous to the introduction of the sulphur in the following proportions:

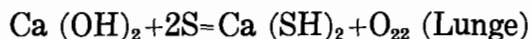
KOTRIX A.

Calcium Oxide.....	47.0%	7.83
Sulphur (Flowers).....	53.0	8.80
Water.....	6 liters	1 liter

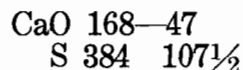
Boil 10 minutes, allow to settle, decanter or filter into a well stoppered bottle.

Exhibit—G-3.

Note this is calculated from the formula.



Should be $3\text{Ca (OH)}_2 + 12\text{S} = \text{CaS}_2\text{O}_3 + 2\text{CaS}_5 \text{ (Lunge)}$



H₂O dissolves 1.3—1.5 gm p 100 cc ::6000 dissolve 78—80 grms leaving
22—20 grms res.

Exp with these propns showed 64 gms residue from 224 CaO+S in
5060cc = 3.16% solubility.

- 10 Variation may be practiced in the performance of producing the Kotrix solution, by such procedures of deviating, to a limited extent, the proportions of lime to that of sulphur. The solubility is not effected. The only marked results being that of a large insoluble residue which should be avoided for economical purposes.

- 20 However, to secure maximum efficiency, the predominance of free sulphur in the final residue should be a mark of distinction in calculating the required quantity of sulphur necessary for the charge. In addition to an excess of sulphur it will be noticed that the boiling solution of Kotrix will show evidence of uncombined sulphur in the froth upon the surface of the vessel containing the solution. This evidence is usually called by the attendants in charge "sulphur bloom" which they consider a very important criterior in governing the quantity of sulphur required to effect the solubility of the lime present.

PREPARATION OF KOTRIX

- 30 The subject of the use of steam versus the boiling of Kotrix in direct fired vessels offers much advantage. Steam boiling permits the preparation of Kotrix in inexpensive wooden tanks while that of boiling in direct fired vessels required metal containers which are subjected to the erosive action of the solution besides the incrustation of the residue upon the bottom of the vessel, which has a tendency to absorb large quantities of heat.

KOTRIX B.

Many ores containing colloidal matter, such as mill slimes and over-flow tank feed, require a different Kotrix. The procedure of preparing the Kotrix remains the same in all cases. The only deviation being the addition of sodium hydrate and lime carbonate. The soda and lime in many cases replacing part of the sulphur and lime which would give the following formulas as an example:

Exhibit—G-3.

Calcium oxide	44.6%	7.45
Sulphur (flowers).....	50.4	8.40
Sodium hydrate.....	1.0	.17
Calcium carbonate.....	4.0	.67
	100.0	16.67
Water six liters.....		1 liter

Long and tedious experiments consisting of varying the percentage of lime to that of the sulphur, in proportion to the quantity of water employed has not involved sufficient data to qualify other experiments to enter here as a competitor of A & B Kotrix. not done here anyway.

Kotrix is only known in solution. Water usually dissolves about 1.3 to 1.5 grams per 100 c.c. of water.

ADMINISTRATION OF KOTRIX

The usual way of introducing Kotrix to the ore charge is previous to the application of the oil, many of the perphary plants now using this solution prefers a mingling device similar to the one employed for forming the oil emulsion before the ore is introduced to the flotation battery.

20 Unfortunately the best results are obtained with Kotrix when Minola is employed in conjunction with Cinol. Though corresponding good results may be obtained by the use of Kotrix with any neutral oil.

It should be borne in mind that the application of Kotrix with acid sludge does not give encouraging results, mostly due to the neutralization of the alkali Kotrix, and the precipitation of the active constituents.

PREPARATION OF MINOLA, CINOL, ARENOL.

30 The process of distilling oils in the presence of sulphur was developed by data secured from the action of Kotrix on colloidal slimes. It was highly desirable to secure an oil whose physical affinity was of sufficient magnitude to conglomerate and quote every available particle of the metallic sulphide present in the ore in order to offer a responsive surface to the atmospheric action.

By distilling an oil in the presence of sulphur, certain selective qualities for embracing the sulphides of ores, was noticed to be more prevalent after the oil was so distilled than was possible to obtain previous to such a treatment.

40 Varying quantities of sulphur were tried with no marked evidence of divulging any data that would be suitable as a standard for defining the percentage that is actually required from 0.25% uo to 20.09 was tried. Five percent has been adopted as a maximum quantity.

Exhibit—G-3.

In treating Petroleum distillates, it is always desirable to secure a mongrel oil (asphaltum paraffine base) of about 30 Baume specific gravity, distilling between 200 and 300 c. or the fraction that is obtained immediately after the fraction representing the Kerosene groups has been removed by the process of distillation. For example:

MINOLA

Petroleum distillate 30 Baume.....	100 Pounds.
Sulphur (Flowers).....	5 “
Distill.	

10 The yield is about 80% when there is not too much of the higher boiling homologues present.

Incidentally it may be mentioned that simillence of improvement is manifested when the distillation has been performed in the presence of superheated steam, similar to the procedure now instituted among the oil refineries for obtaining their distillates.

CINOL

Cinol is produced from the inexpensive commercial pine oils by the same procedure that is now in vogue for producing Minola.

A working example would be

20 Pine Oil (any specified gravity).....	100 Pounds.
Sulphur (Flowers).....	5 “
Distill	

Yield about 95%.

ARENOL

Arenol is the distillation of coal tar oils in the presence of sulphur.

Usually the production of Arenol is chiefly confined to such by products of coal tar distillation that will not command high market values for synthetical commodities.

30 The coal tar practice in the United States has resolved itself into the disposal of such coal tar constituents that has had Benzol, Phenol and Napthaline removed as a flotation oil.

GRABANOL

Grabanol was named after Dr. Gregory and Jr. Ballot and most specific it represents a resin soap.

The method of producing Grabanol is confined to the saponification of cheap resin by caustic soda. It is always the custom to determine in a boiling alcoholic solution the quantity of caustic soda required for complete saponification.

40 In practice, after the quantity of caustic soda required has been determined for the complete saponification of the resin, the correct

Exhibit—G-3.

quantity of caustic soda is added to the melted resin with sufficient water and boil until a sample will give evidence of solubility in water.

For example:

GRABANOL A.

Resin.....100 grams
 Caustic soda.....
 Water.....(not legible)

It is illustrated by the following example that Canol acts very advantageously in producing Grabanol.

10

GRABANOL B.

Resin.....100 grams
 Canol..... 50 c.c.
 Caustic soda..... 10 grams
 Water..... 50 c.c.
 Boil

The addition of Canol seems to present a much better Grabanol than the straight saponification.

GRABANOL C.

20

Resin.....100 grams
 Caustic soda..... 10 c.c.
 Water..... 50 c.c.

Boil. Allow to cool, add Denatured alcohol 50 c.c.

GRABANOL D.

Resin.....100 grams
 Caustic soda..... 10
 Water..... 50

Boil, Allow to cool, add 25 c c Stanol.

In saponifying the resin, the caustic soda should always be dissolved in water previous to its admission as a saponifying agent.

30

After the stock solution has been made up, it can be diluted with water to any desired dilution that it is found expedient for commercial purposes.

Grabanol possesses special virtues when administered in dilute quantities to copper ores containing molybdenum. The molybdenum will float in preference to the copper sulphides and can be removed from the first cell of the flotation battery. The copper can be removed in the subsequent cell by the addition of Minola, Cinol or any other suitable oil.

40 Another form of Grabanol is the saponification of the resin at ordinary atmospheric temperatures which probably will find favor

Exhibit—G-3.

in practice much more so than some elaborate method of boiling. The Grabanol prepared either in the cold or by heat does not seem to materially effect the final solution. Several samples have been prepared by saponifying the resin with sodium carbonate, for example:

Resin.....	100 grams
Sodium carbonate.....	15 "
Water.....	15 c.c.

STANOL

PREPARATION OF STANOL.

10	Measure out	100 c c of Denatured alcohol
	" "	10 c c of Carbon sulphides
	Weigh "	1 gram of Caustic soda

Shake until dissolved and digest under a reflux condenser until the caustic soda has disappeared.

Several Stanols have been made up varying the proportion of carbon disulphides and caustic soda to meet the condition of the ore under treatment. The following proportion will serve as a guidance:

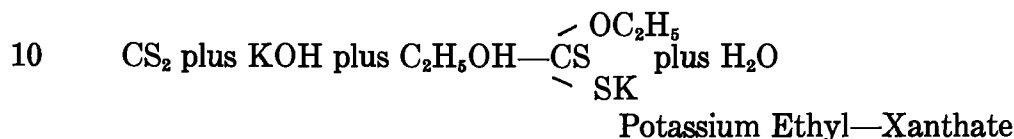
20	A Stanol	Denatured Alcohol.....	100 c c
		Carbon Disulphide.....	10 c c
		Caustic Soda (NaOH).....	1 gram
	B Stanol	Denatured Alcohol.....	90 c c
		Carbon Disulphide.....	20 c c
		Caustic soda (NaOH).....	1 gram
C		Denatured Alcohol.....	80
		Carbon Disulphide.....	30
		Caustic Soda.....	1 gram
30	D	Denatured Alcohol.....	100 c c
		Carbon Disulphide.....	10 c c
		Caustic Soda.....	5 grams
E		Denatured Alcohol.....	90
		Carbon Disulphide.....	20
		Caustic Soda.....	5 grams
F		Denatured Alcohol.....	100 c c
		Carbon Disulphide.....	10 c c
		Resin.....	10 grams
		Caustic Soda.....	1 "
40	G	Denatured Alcohol.....	90 c c
		Carbon Disulphide....	20 c c
		Caustic Soda.....	5 grams
		Resin.....	20 grams

Boil under reflux condenser until the resin is saponified. Dilute with 500 c c of water.

Exhibit—G-3.

These formulas illustrate that in preparing Stanol variation can be practised by the addition of resin, the alcohol can be diminished and the caustic soda should always be governed as to only have present sufficient quantity to produce the reaction sought for. With some ores an excess of caustic soda to neutralize the acidity seems to impart specific results over the neutral Stanol.

The theory of forming flotation compounds from alcoholic caustic Potash and carbon disulphide may be expressed as follows:—



This equation illustrates that if we digest under reflux condenser

Carbon Disulphide.....	57.6%
Caustic Potash.....	26.8
Denatured Alcohol.....	15.6
	100.0

we should upon the completion of the reaction obtained crystalline Potassium xanthate which, however, is soluble in alcohol and can be employed at any strength to effect flotation of copper salts.

Potassium xanthate is not a frothing agent and therefore it must be mixed with some appropriate agents that will give a voluminous froth. Alcohol, Resin and pine oil seem to be the most suitable agents for this purpose.

It might be conjectured that some arrangements of combining Potassium xanthate with alcohol and Resin and then mixing this compound with mineral oil, would be the initial step of using such a commercial mixture for the flotation of copper carbonates.

A substituted product may be formed by using caustic soda

30 Carbon Disulphide.....	70.8%
Caustic Soda.....	10.0
Denatured Alcohol.....	19.2
	100.0

The soda compound does not seem to produce the correspondent good results as is produced with the potassium xanthate. The high cost of potassium salt will prevent this compound from entering as a competitor of the much cheaper sodium salt.

All the experiments conducted so far have been by the use of sodium hydrate and denatured alcohol.

40 A very good compound is made up for alkali ores by using 20% sodium ethyl xanthate and 80% Denatured Alcohol.

Exhibit—G-8.

Exhibit—G-21.

EXHIBIT—G 8

MINERALS SEPARATION AMERICAN SYNDICATE, (1913)
LTD.,

ss Philadelphia (8/28/15)

New York, August 26th, 1915

SG/EAD

My dear B:—

Thanks for yours of the 10th August.

Martin's Invention. I am sending you Higgins reports bringing this matter up to date.

10 With best regards,

Yours very truly,

(Sgd.) S. GREGORY

encls.

(Higgins reports addressed HDW Aug. 21 and 24)

(copy of W. M. Grosvenor's letter Aug. 25)

(copy of Mr. Salinger's letter to Mr. BE Aug 23)

EXHIBIT—G 21

MINERALS SEPARATION AMERICAN SYNDICATE (1913)
LIMITED

20

LONDON, E. C. 31st Aug. 1915.

Dictated to E.N.P.

by J. B.

Dr. Gregory,

c/o Henry D. Williams Esq.,

61, Broadway, NEW YORK, U.S.A.

My dear Doctor,

30 *MARTIN'S INVENTIONS.*—I have read Higgins report of the 14th Aug. with interest though it would seem that he is not yet clear as to exactly what Martin's invention is, but it is important to note that if his Kotrix or modified oil — whatever they may finally decide to call it — can be definitely proved to be an ingredient for modifying the frothing agent in such a way as to ensure useful results, that Mr. Williams believes the patent position could be both simplified and strengthened. I am glad therefore that Higgins is at work with that object in view. At the same time it again emphasizes the question, to my mind, as to what about Martin being the true and only inventor, and how is Higgins to be brought into the matter? or how can Higgins' name be kept out?

Yours truly,

40

(Sgd.) JOHN BALLOT,

Enclo. English form of licence

Managing Director.

Exhibit—G-10
Exhibit—P-105.

EXHIBIT—G 10

per S/S "ST. LOUIS" (sailing 9/18/15).

MINERALS SEPARATION AMERICAN SYNDICATE,
(1913), LTD.,

SG/CF

NEW YORK, September 17th, 1915

My Dear B.:

MARTINS' INVENTION.—This matter is receiving very careful consideration. In view of our past experience, you may be sure the subject will be considered from every angle and the best will be
10 done in the circumstances.

With kind regards,

Yours very truly,
(Sgd.) S. GREGORY

Encls.

EXHIBIT—P 105

REPORT ON THE
FLOTATION VALUE
OF THE
ANACONDA COPPER MINING COMPANY'S
FEED TO FLOTATION PLANT.

20 September 20th, 1915.

12th St. near Vernon Ave.,

Long Island City, NEW YORK, U.S.A.

On June 17th, 1915, a sample of 200 pounds of mill seed to flotation plant, from the Anaconda Mining Company was received by the Mineral Separation Syndicate, Long Island City's laboratory, for the purpose of determining the value of recent acquired patents pertaining to the flotation of porphoretic copper sulphides.

The sample was received in a semi-plastic condition containing
30 13.33% of moisture. On drying the sample and submitting it without further crushing to the following screens, it was observed that the sample was rather coarse for test in the small laboratory machine.

SCREEN ANALYSIS

on 20.....	Mesh Screen.....	0.6%
" 30.....	" ".....	2.0
" 60.....	" ".....	11.2
" 80.....	" ".....	12.0
" 100.....	" ".....	12.4
Through 100	" ".....	61.2
Loss in Screening.....		0.6

Exhibit—P-105.

No assays were performed in ascertaining what percentage of copper value the several screens retained in each segregation. The total flotation results indicate that further crushing would not be profitable in comparison to a supposed additional extracted value plus the cost for fine pulverization.

The flotation value was performed by employing 500 grams sample of the ore without further crushing, with 2000 c.c. of Long Island City (N.Y.) artesian well water, at the ordinary atmospheric temperature, in a Janney circulating laboratory flotation machine,
10 driven at 2,100 R.P.M. for a period of five minutes.

ORIGIN OF THE ORE

No date was received with the sample of the ore, to suggest from what formation it occurred or whether it was obtained from deep mining (below the water level of the mining district) or shallow mining (above the water level of the district).

The sample of Anaconda Heads submitted shows that this particular sample belongs to the acid classification of ores, especially so when it is compared with the alkaline porpheries. Therefore it requires an addition of acid to suspend its colloidal matter and clean
20 up the rusty particles of partly oxidized sulphides. The introduction of alkalides to such an ore would complicate the tendency of precipitating the suspended colloidal matter of the ore charge, which would have a direct influence of prohibiting the buoyant properties from rising to the surface through the flocculent matter, produced by the presence of the alkali.

The quantity of acid required largely depends upon, 1st, the nature of the ore, 2nd, the nature of the flotation agent employed.

It is not commercially expedient to employ a large quantity of a depolarizing agent (acid or alkali) to effect the combining powers of the
30 flotation agent to that of the constituents of the ore that it is desired to recover by flotation. The compromising minimum can always be adjusted by the employment of a harmonizing flotation agent that will give concordant results.

Exhibit—P-106.

EXHIBIT—P 106

NOTES OF EXPERIMENTS

Conducted on the Anaconda Mining Company's
Feed to Flotation Plant.

Sept. 20th, 1915.

12th St. near Vernon Ave.,
Long Island City, NEW YORK, U.S.A.

20/9/15.

Other numerous reagents were tried on the feed to the Anaconda
10 Flotation Plant, which give the following negative results:

Sodium Chloride	1, 2 and 5% solution	No results.
Sodium Chloride	7.8%	
Sulphuric Acid	14.7	No encouraging results.
H ₂ O	77.5	
Grabanol		Not as effective as Minola and Cinol.
Stanol		Not satisfactory for this ore.

10.

20/9/15.

PROMISING FORMULAS

20 Mexican Fuel sp. gr. 21 B 50% Very promising when used with a
frothing agent.

Minola 50%

This experiment shows the possibility of employing California
Crude Oil compounded with California 30 B distillate. Owing to not
having a sample of the California petroleum oils at hand, no experi-
ments were conducted in deriving results that might be obtained by
the use of this oil.

Minola 95%

Crude Carbolic Acid 5% Most excellent results.

30 All formulas containing tar oils, and especially crude carbolic
acid, give most satisfactory results, especially when used in connection
with sulphuric acid.

The integrity of these experiments are more forcibly expressed in
regards to satisfactory results in the special report covering details of
reagents requires; and their cost rated per ton of ore treated. No
satisfactory results were obtained by the use of Kotrix or any other
alkaline reagents. The best results being attained by the use of
sulphuric acid as a depolarizer, Minola as a collector, and Cinol in
combination with crude carbolic acid oils as a frother and collector.

Exhibit—G-11.

Exhibit—G-12.

EXHIBIT—G 11

MINERALS SEPARATION AMERICAN SYNDICATE (1913)
LIMITED62, London Hall,
LONDON, E.C. 1st Oct., 1915.Dictated to M.E.P.
by J.B.

Dr. S. Gregory,
c/o Henry D. Williams, Esq.,
10 61, Broadway, NEW YORK, U.S.A.

My dear Doctor,

re your letters of 10th and 17th September—

MARTIN'S INVENTION.—The copy of his report dated 15th August, reparation of flotation reagents, kotrix, etc. which you enclosed with yours of the 17th is worded in that same pedantic, and to me, objectionable language, because I have not the time and patience to disentangle his meaning, and must therefore live in hopes that with Mr. Higgins' assistance, Mr. Williams and yourself will eventually get to know what it means and embody it in a patent
20 application if deemed necessary.

Yours very truly,
(Sgd.) JOHN BALLOT,
Managing Director.

EXHIBIT—G 12

MINERALS SEPARATION AMERICAN SYNDICATE,
(1913 LTD.

ss St. Louis (10/16/15)

SG/EAD

NEW YORK, October 15th, 1915.

My dear B.:

30 I have yours of October 1st.

MARTIN'S INVENTIONS.—This matter is having all the attention possible in the circumstances and I hope to report to you shortly.

With kindest regards

Yours very truly,
(Sgd.) S. GREGORY

Exhibit—G-13.
Exhibit—P-107.

EXHIBIT—G 13

MINERALS SEPARATION AMERICAN SYNDICATE, (1913),
LTD.

ss Finland (10/24/15)

SG/EAD

New York, October 22nd 1915

My dear B:—

I have yours of the 8th October enclosing copies of letters to Nutter to acknowledge. I have carefully read contents of Nutter's letters. I have been very busy this week with the brief and am still
10 busy with the reply brief. We have a conference on Monday next to finally settle our reply brief, which has to go in by the end of the month. I am glad to say there is absolutely nothing new in Scott's brief. His arguments are very thin in specious and we shall have no difficulty in our reply. We have only three copies so far of Scott's brief and they are all in use but I shall forward you one at the earliest opportunity.

Martin's Inventions. This matter has to wait for the moment.

With kindest regards

Yours very truly

(Sgd.) S. GREGORY

20 EXHIBIT—P 107

12th St., near Vernon Ave.,
Long Island City, N.Y.
11th Dec. 1915

H. D. Williams Esq.,
61 Broadway,
N.Y.C.

Dear Mr. Williams,

I am enclosing you herewith rough drafts of two of Mr. Martins Inventions.

30

Yours very truly,

A. HOWARD HIGGINS

These were draft specifications for Kotrix and Reconstructed Oil.

Exhibit—W-8.
Exhibit—W-11
Exhibit—W-12.

EXHIBIT—W 8

12th St. & Vernon Ave.,
Long Island City, N.Y.,
February Twenty-Third
Nineteen Seventeen.

D. S. Gregory, Esq.,
Minerals Separation, North American Corp.,
61 Broadway,
New York City.

Dear Sir:—

- 10 Mr. Williams informed me some time back that you contemplated purchasing my patents. All the papers have been signed in this connection, and I am wondering if it is convenient for you to let me have a check, in order to complete the transaction.

Yours very truly,

RBM.EH

(Sgd.) R. B. MARTIN

EXHIBIT—W 11

March 21, 1917.

Minerals Separation North American Corporation,
61 Broadway,
20 New York City.

Dear Sirs:

Referring to the agreement between Minerals Separation, Limited, and Rutherford B. Martin dated March 6, 1915, Mr. Martin has now executed all the necessary documents, patent application and assignments covered thereby and is therefore entitled to the sum of \$5,000., the consideration named in the above contract. I am sending you herewith a final receipt upon the signing of which, by Mr. Martin, please deliver to him your check for \$5,000.

Faithfully yours,

30 1 enc.

"H. D. WILLIAMS"

EXHIBIT—W 12

Vol. 54

402
March 21, 1917.

- 40 I Rutherford B. Martin, hereby acknowledge receipt of the sum of Five Thousand Dollars (\$5000.) received from Minerals Separation North American Corporation, in full payment for certain inventions, improvements, patent applications and patents mentioned in a contract dated the 6th day of March 1915, between Minerals Separation Limited and me, the above Minerals Separation North American Corporation having succeeded to the rights of Minerals Limited in this contract.

Exhibit—P-108.

EXHIBIT P-108.

INDEX

to

Reports

of

R. B. MARTIN

BULLETINS NOS. 1 - 72 incl.

Research Laboratory

of

10 MINERALS SEPARATION — NORTH AMERICAN
CORPORATION, LTD.

Long Island City, N.Y., U.S.A.

From 1915 to JANUARY 1923

BULLETINS

- No. 1 Classification of Permissible Oils employed in Flotation.
pp. 116. (1915).
- No. 2 Preparation of Flotation Reagents; Kotrix, Minola, Cinol,
Arrenol, Grobanol, Slanol. pp. 14. (Aug. 15, 1915).
- 20 No. 3 Flotation Value of the Anaconda Mining Company.
pp. 11. (Sept. 20, 1915).
- No. 4 Notes of Experiments conducted on the Anaconda Mining
Company Feed to Flotation Plant. pp. 11. (Sept. 20,
1915).
- No. 5 Report on the Flotation Value of the Anaconda Mining
Company Feed to Leaching Plant. pp. 7. (Sept. 20,
1915).
- No. 6 Notes of Experiments conducted on the Anaconda Mining
Company Feed to Leaching Plant. pp. 4. (Sept. 20,
1915).
- 30 No. 7 Determination of Petroleum Hydrocarbons in Pine Oil.
pp. 2. (No. 11, 1915).
- No. 8 Caluso-Parrot Slimes. pp. 3. (Oct. 11, 1915).
- No. 9 Notes of Experiments — Caluso-Parrot Mining Company
Slimes. pp. 3. (Oct. 11, 1915).
- No. 10 Report on the Flotation Value of Zinc Carbonates
National Zinc Company, Bottesville, Okla. pp. 3.
(Oct. 14, 1915).

Exhibit—P-108.

- No. 11 Report on Oils received from the United States Naval Stores Company. pp. 14. (Nov. 11, 1915).
- No. 12 Report on the Flotation Value of United States Naval Stores Company's Eight Samples of Flotation Oil. pp. 11. (Nov. 11, 1915).
- No. 13 Addendum to Report on United States Naval Stores Company. pp. 3. (Oct. 14, 1915).
- No. 14 United Naval Stores Company. Eight Samples of Oil. pp. 4. (Nov. 11, 1915).
- 10 No. 15 Report on Oils received from General Naval Stores Company. pp. 6. (Nov. 16, 1915).
- No. 16 Georgia Light Pine Oils from Georgia Turpentine & Rosin Company. pp. 8. (Dec. 29, 1915).
- No. 17 Cuban Copper Leasing Company. pp. 12. (Mar. 6, 1916).
- No. 18 Flocculation of Clays. Oil Absorbing Clays. pp. 19. (Mar. 24, 1916).
- No. 19 Martin Centrifugal Flotation Machine. pp. 9. (June 21, 1916).
- 20 No. 20 Interaction of Reagents upon Sulphide Ores and the Application of Colloids in Flotation. pp. 29. (July 31, 1916).
- No. 21 Report on Tar No. 44. Distillation Test and Application in Flotation. pp. 5. (Aug. 22, 1916).
- No. 22 Report on Nutter's Water Gas Tar No. XVIII. Distillation Test and Application in Flotation. pp. 11. (Aug. 30, 1916).
- No. 23 Report on Pine Oils employed in Flotation. pp. 14. (Sept. 29, 1916).
- 30 No. 24 Grasselli Chemical Company Flotation Flour. Examination and Properties. pp. 5. (Oct. 7, 1916).
- No. 25 American Turpentine & Tar Co., New Orleans, La., U.S.A., No. 104. Distillation Test and Application in Flotation. Hardwood Tar. pp. 4. (Oct. 10, 1916).
- No. 26 American Turpentine & Tar Co., New Orleans, La., U.S.A. No. 52 Light Hardwood Oil. pp. 5. (Oct. 10, 1916).
- No. 27 Report on George A. Chapman's Flotation Oil—Distillation Test and Analysis. pp. 2. (Oct. 2, 1916).
- 40 No. 28 Report on Barrett's Special Coal Tar Oil. Distillation Tests. Conversion by Synthesis into Naphthalene Sulphonic Acid, Calcium Sulphonic Acid, Sodium Sulphonic Acid, and Application in Flotation. pp. 12. Nov. 17, 1916).

Exhibit—P-108.

- No. 29 Report on American Tar Products Company's Water Gas Tar Oil — Distillation and Application in Flotation. pp. 4. (Nov. 29, 1916).
- No. 30 Report on American Tar Products Company's Water Gas Tar — Distillation Tests and Application in Flotation. pp. 5. (Dec. 4, 1916).
- No. 31 The Impossibility of Employing Excess of Oil in Flotation. pp. 14. (Dec. 15, 1916).
- 10 No. 32 Evasive Oil Mixtures and the Possibilities of Defeating the Flotation Patent. pp. 9. (Dec. 28, 1916).
- No. 33 Methods for the Determination of Flotation Oils including Petroleum Oils, Pine Oils (Terpene Hydrocarbons), Oleic Acid (Monocarboxylic Hydrocarbons), and Coal Tar Oils (Aromatic Hydrocarbons). pp. 15. (Feb. 22, 1917).
- No. 34 Soluble Cutting Oils from the Vacuum Oil Company, Rochester, N.Y., U.S.A. pp. 4. (July 1, 1917).
- No. 35 Selective Flotation of Lead Sulphides from Zinc Sulphides by using "Kotrix". pp. 4. (June 29, 1917).
- 20 No. 36 Report on Dr. J. H. Bank's Vanadium Ore. No. 45107. pp. 4. (Sept. 6, 1917).
- No. 37 Properties and Determination of Pine Oils. pp. 18. (Jan. 17, 1918).
- No. 38 Laboratory Notes — Melco Chemical Company's Products, including seven samples. Chemical Tests and Application in Flotation. pp. 10. (May 6, 1918).
- No. 39 Report on Melco Chemical Company's Products. No. 211-A Light Oils. Analysis. pp. 5. (May 7, 1918).
- No. 40 Laboratory Notes on the Cracking of Petroleum Oils in Presence of Air. pp. 118. (May 8, 1918).
- 30 No. 41 Report on the Oxidation of Petroleum Oils with Atmospheric Oxygen, compiled from notes of experiments conducted between May 8th and Aug. 19th, 1918. pp. 21.
- No. 42 Laboratory Notes—Ultra Violet Light and the Oxidizing Effect upon Petroleum Oils. pp. 52. (Feb. 26, 1919).
- No. 43 Patent Specifications—Oxidation of Petroleum Oils. pp. 14. (Dec. 11, 1919).
- No. 44 Experimental Notes on Bethlehem Steel Company's Lebanon Iron Ore. pp. 5. (Dec. 17, 1919 to Jan. 24, 1920).
- 40 No. 45 Experimental Flotation Results — Bethlehem Steel Company. pp. 52. (Feb. 2nd to Apr. 9th, 1920).
- No. 46 Results of Investigation and Recommendations — Conclusive Report — Bethlehem Steel Company. pp. 19. (Apr. 15, 1920).

Exhibit—P-108.

- No. 47 Petroleum Frothers and Their Properties. Wilkinson Pot Oil, James and R.U. V. pp. 14. (Sept. 29, 1920).
- No. 48 Synthesis of Metallurgical Frothers from Petroleum Oils by Molten Metals. pp. 21. (Nov. 19, 1920).
- No. 49 Estimated Cost of Proposed Plant for Oxidizing Petroleum Oils, using Molten Metals. pp. 4. (Dec. 31, 1920).
- No. 50 Synthesis of Metallurgical Frothers from Petroleum Oil, using Heated Metal Salts in the Presence of Air. pp. 10. (Jan. 31, 1921).
- 10 No. 51 Extracting Oil from Shale by Employing Limited Combustion in Shaft Furnaces. pp. 16. (Feb. 9, 1921).
- No. 52 Preparation of Hydrogen Sulphide from Sulphur. pp. 11. (Mar. 15, 1921).
- ADDENDUM pp. 3. (May 25, 1921).
- No. 53 Working of Miniature Shale Oil Plant. pp. 26. (Mar. 31, 1921).
- No. 54 Flotation of Soda Springs Phosphate. pp. 3. (May 23, 1921).
- 20 No. 55 Flotation Results of Compagnie De Boleo. pp. 4. (May 31, 1921).
- No. 56 Cost of Producing Hydrogen Sulphide. pp. 3. (June 21, 1921).
- No. 57 Flotation of Copper Carbonates with Modified Kotrix. pp. 5. (June 21, 1921).
- No. 58 Wilkinson Oxidized Pot Oil. pp. 5. (Oct. 13, 1921).
- No. 59 Tom Jenney's Reconstructed Oil Process. pp. 5. (Sept. 14, 1921).
- No. 60 Producing Gasolene from Petroleum Oils by Oxidation. pp. 4. (Dec. 28, 1921).
- 30 No. 61 Flotation Results on Federal Lead Company Oxidized Copper Ore. pp. 6. (Jan. 17, 1922).
- No. 62 Flotation Results on Santa Francesca Dump Ore. (Asientos Unit) pp. 5. (Jan. 10, 1922).
- No. 63 Mercuric Sulphate Test for Flotation Oils. pp. 4. (Feb. 7, 1922).
- No. 64 Preparation of Kotrix and its Use upon Oxidized Copper Ores. pp. 3. (Apr. 17, 1922).
- No. 65 Specification and Estimated Cost of Producing Oxidized Petroleum Oils. pp. 5. (May 17, 1922).
- 40 No. 66 Flotation Results on Inspiration Ore Feed to Leaching Plant and Tailings from Leaching Plant. pp. 4. (May 11, 1922).

Exhibit—P-108.

Exhibit—W-13.

- No. 67 Oxidation of Petroleum Oil over Sodium Chloride, Sodium Carbonate and Sodium Bicarbonate. pp. 4. (May 25, 1922).
- No. 68 Flotation Notes on General Chemical Company Phosphate Rock. pp. 17. (July 24, 1922).
- No. 69 Laboratory Notes of Flotation Results on American Zinc, Lead & Smelting Co. Ore. pp. 16. (Aug. 22, 1922).
- No. 70 Flotation Tests on American Zinc, Lead & Smelting Company, Mascot, Tenn. Flotation Tailings. pp. 2. (Aug. 22, 1922).
- 10 No. 71 Poisonous Effect of Hydrogen Sulphide Gas. pp. 9. (Sept. 18, 1922).
- No. 72 Abstract of Flotation Notes on General Chemical Company Phosphate Rock (Bulletin No. 68). pp. 5. (Jan. 15, 1923).
- NOTE: Bulletins Nos. 1 to 32 will be found not to be numbered Laboratory copies have been so numbered for convenience.

PLAINTIFF'S EXHIBIT 13 ON WILLIAMS DEPOSITION

MARTIN APPLICATIONS

20	SER. No.	FILED	PATENT ISSUED	ABANDONED
	74,921	Jan. 28, 1916	1,236,856, Aug. 14, 1917	
	74,922	Jan. 28, 1916	1,236,857, Aug. 14, 1917	
	420,806	Oct. 30, 1920	<i>Oxidized Oils.</i>	Dec. 8, 1921
	420,807	Oct. 30, 1920	<i>Oil catalyzer Ultra Violet Rays</i>	Sept. 27, 1924
	420,808	Oct. 30, 1920	1,678,403, July 24, 1928	
	420,809	Oct. 30, 1920	<i>Same as flotation agent</i>	Sept. 27, 1924
	458,758	April 5, 1921	<i>Oxidized compound over molten metal.</i>	Jan. 20, 1927
30	458,759	April 5, 1921	<i>Use of same as flotation agent</i>	June 15, 1923
	474,810	June 3, 1921	1,510,150, Sept. 30, 1924	<i>Carbon catalyzer</i>
	475,467	June 6, 1921	<i>Carbon catalyzer</i>	Sept. 16, 1923
	154,309	Dec. 11, 1926	1,686,529, Oct. 9, 1928	<i>Copper Anilide</i>

Exhibit—W-10

PLAINTIFF'S EXHIBIT 10 ON WILLIAMS DEPOSITION

UNITED STATES PATENT OFFICE

RHETHERFORD B. MARTIN, OF NEW YORK, N.Y.,
 ASSIGNOR TO MINERALS SEPARATION NORTH
 AMERICAN CORPORATION, OF NEW YORK,
 N.Y., A CORPORATION OF MARYLAND.

ORE CONCENTRATION

1,236,857. Specification of Letters Patent.

Patented Aug. 14, 1917.

- 10 No Drawing. Application filed January 28, 1916. Serial No. 74,922.
To all whom it may concern:

Be it known that I, RHETHERFORD B. MARTIN, a citizen of the United States, residing at the borough of Manhattan, in the county of New York and State of New York, have invented certain new and useful Improvements in Ore Concentration, of which the following is a specification.

- This invention consists in improvements in processes of ore concentration and has particular reference to the concentration of ores by flotation. In these processes the powdered ore is usually
 20 mixed with water, such mixture being termed the pulp, and to this pulp is added the frothing agent, consisting of an oil or oils or a soluble frothing agent or agents, or both, and the pulp is then subjected to agitation and aeration, for example, supro-aeration by air introduced from above by a suitable agitation of the pulp, or subaeration by air introduced from below through a porous medium or otherwise, as well known in the art, the result of this aeration and agitation being that the air bubbles coursing through the pulp gather up the metalliferous particles and carry them to the surface where they form a
 30 froth. This froth may be readily floated over a lip or dam and will thus carry the metalliferous particles away from the body of the liquid. My present invention relates to the frothing agents to be used in such processes. I have found that if a frothing agent consisting of or containing an oil is prepared for use in such process by heating it in the presence of sulfur, its effectiveness is very greatly increased so that, for example, when used in smaller proportions than are required of the raw frothing agent, it produces improved results.

- The frothing agent may be treated in an autoclave or in a vessel connected with a reflux condenser or in an ordinary still. An auto-
 40 clave may be used where the material treated is very volatile and has to be heated to a temperature above its normal boiling point to obtain the desired modification. A reflux condenser may be used where it is desired to mingle the distillate, with the undistilled material

while under treatment. When an ordinary still is used, the mixture is boiled and allowed to distil until the volatile constituents have passed over, leaving only a tarry residue which may be rejected or added to a new charge.

The proportion of sulfur to frothing agent is capable of wide variation, but since the tarry residue varies as the proportion of sulfur, it is preferable to use small quantities of sulfur. Usually a proportion of about 5 parts by weight of sulfur to 100 parts by weight of the frothing agent is sufficient for the purpose.

10 The materials that have been successfully treated with sulfur in this manner are pine oils, pine tars, turpentine, resin oils, coal tar oils and creosotes, petroleum distillates and mixtures of these oils.

The following are examples of methods of modifying oils in accordance with my invention.

Example 1: A pine oil having a specific gravity of 1.03 was distilled in an ordinary still after mixing with 5 grams of sulfur to 100 cubic centimeters of oil. The distillation temperature ranged from 88° to 350° C., the bulk distilling between 200° and 350° C. The distillate formed a frothing agent with enhanced air-emulsifica-
20 tion properties.

Example 2: The same oil was digested with sulfur in the same proportions for thirty minutes at a temperature of 250° C. in an open vessel fitted with a reflux condenser, with the result that the condensable distillates were returned to the mixture during the treatment. The resulting oil, somewhat darkened in color, had a specific gravity of 1.06 and formed a frothing agent with enhanced air-emulsification properties.

Modified oil produced according to either of the above examples appears to maintain its modified condition and can be made in
30 quantities and stored and transported to the place of use and apparently will keep for an indefinite period.

As an example of the use of oil thus modified, I have found that for the treatment of a porphyry copper ore containing 2.5% of copper as sulfids, 2.06 pounds per ton of the raw oil used in the above examples was required for satisfactory treatment, whereas after this oil had been modified according to Example 2, 0.82 pounds per ton yielded with the same ore richer concentrates and cleaner tailings.

In the application of this invention to various ores and with the use of various oils and under various conditions, the examples
40 of the process above described may be modified without departing from this invention.

I claim:

1. A process of concentrating ores which consists in agitating and aerating an ore pulp with a frothing agent which has been modi-

Exhibit—W-10.

fied by heating it with sulfur so as by such agitation and aeration to obtain a froth of concentrates.

2. A process of concentrating ores containing copper which consists in agitating and aerating a pulp of such an ore with a frothing agent which has been modified by heating it with sulfur so as by such agitation and aeration to obtain a froth of concentrates.

3. A process of concentrating ores which consists in agitating an aerating an ore pulp with a frothing agent comprising an oil which has been modified by heating it with sulfur so as by such
10 agitation and aeration to obtain a froth of concentrates.

4. A process of concentrating ores containing copper which consists in agitating and aerating a pulp of such an ore with a frothing agent comprising an oil which has been modified by heating it with sulfur so as by such agitation and aeration to obtain a froth of concentrates.

5. A process of concentrating ores which consists in agitating and aerating an ore pulp with a frothing agent comprising an oil which has been modified by heating it with sulfur to a temperature in the neighborhood of 250° centigrade, and returning condensable
30 distillates to the mixture during the treatment, so as by such agitation and aeration to obtain a froth of concentrates.

6. A process of concentrating ores containing copper which consists in agitating and aerating a pulp of such an ore with a frothing agent comprising an oil which has been modified by heating it with sulfur to a temperature in the neighborhood of 250° centigrade, and returning condensable distillates to the mixture during the treatment, so as by such agitation and aeration to obtain a froth of concentrates.

In testimony whereof I have affixed my signature in presence of two witnesses,

30 RHETHERFORD B. MARTIN.

Witnesses:

HENRY D. WILLIAMS,
HARRY C. LEWIS.

PLAINTIFF'S EXHIBIT 9 ON WILLIAMS DEPOSITION

UNITED STATES PATENT OFFICE

RHETHERFORD B. MARTIN, OF NEW YORK, N.Y.,
ASSIGNOR TO MINERALS SEPARATION NORTH
AMERICAN CORPORATION, OF NEW YORK, N.Y.,
A CORPORATION OF MARYLAND.

ORE CONCENTRATION

1,236,856. Specification of Letters Patent. Patented Aug. 14, 1917.
No Drawing. Application filed January 28, 1916. Serial No. 74,921.

10 *To whom it may concern:*

Be it known that I, RUTHERFORD B. MARTIN, a citizen of the United States, residing at the borough of Manhattan, in the county of New York and State of New York, have invented certain new and useful Improvements in Ore Concentration, of which the following is a specification.

This invention consists in improvements in processes of ore concentration, and has particular reference to the concentration of ores by flotation. In these processes the powdered ore is usually mixed with water, such mixture being termed the pulp, and to this
20 pulp is added the frothing agent, consisting of an oil or oils or a soluble frothing agent or agents, or both, and the pulp is then subjected to agitation and aeration, for example, supra-aeration by air introduced from above by beating it into the pulp, or subaeration by air introduced from below through a porous medium or otherwise, as well known in the art, the result of this aeration and agitation being that the air bubbles coursing through the pulp gather up the metalliferous particles and carry them to the surface where they form a froth. This froth may be readily floated over a lip or dam, and will thus carry the metalliferous particles away from the body
30 of the liquid. In these processes a mineral acid, such as sulfuric acid, is in some instances used in addition to the frothing agent, and in other instances an alkali is used in addition to the frothing agent, and these additional substances, acid or alkaline, act in some manner not clearly understood to modify the properties of the liquid and to assist the flotation of the metalliferous mineral and to retard or prevent the flotation of the gangue.

I have found that soluble sulfids, such as mono-sulfids, poly-sulfids or hydrosulfids of sodium, potassium and calcium, are capable of use for this purpose, that is to say, to modify the properties of the
40 liquid forming the pulp so that the selective action of the frothing agent and air bubbles is enhanced or usefully influenced, and that by their use in the proportions and under the conditions hereinafter stated, not only may improved concentration be effected of the

Exhibit—W-9.

metalliferous sulfid particles, such as have hitherto been concentrated in flotation processes, but also concentration may be effected of oxidized metalliferous particles, such as oxids and carbonates of copper.

It has hitherto been proposed to treat oxidized metalliferous particles with soluble sulfids under such conditions that the oxidized metalliferous particles are converted into or become superficially coated with metallic sulfids and are thereby rendered suitable for concentration by flotation, as in British Patent No. 26,019 of 1909, to Sulman and Picard. The procedure according to my invention is wholly different from that heretofore proposed, in that the oxidized metallic particles are not converted into or coated with sulfid particles, but are selected and concentrated as oxidized particles. For example, if the ore contains particles of carbonates of copper, of the malachite variety, green in color, these copper carbonate particles remain particles of copper carbonates throughout the process and are clearly recognizable as particles of green copper carbonates in the concentrates. I have further found that if the amount of soluble sulfid is increased above the minute quantities which I have successfully used, or if the duration of its contact with the copper carbonates is extended, or if heat is used, so as in any one or all of these instances to coat the green copper carbonate particles with the sulfid of copper produced by such conversion, which is black, that the process is seriously impaired and that as the condition of conversion into copper sulfid is approached the impairment of the process increases and in some instances useful froth formation is prevented.

The sulfid solution may be prepared by dissolving one or more of the commercial sulfids in water in suitable quantities, or it may be prepared, for example, by boiling in water a mixture of sulfur and one or more alkali hydrates or alkaline earth hydrates. The following are examples of the methods last referred to of preparing sulfid solutions for use in this process.

Example 1: Boil together for a period of about ten minutes the following substances:

52 parts of caustic soda
48 parts of flowers of sulfur
6000 parts of water.

Example 2: Boil together for a period of about ten minutes—
47 parts of lime

53 parts of flowers of sulfur
6000 parts of water.

Example 3: Boil together for a period of about fifteen minutes—
44.6 parts of lime
50.4 parts of flowers of sulfur
1 part of caustic soda
6000 parts of water.

Exhibit—W-9.

The resulting liquid is usually of a dark golden color when hot, and a golden yellow when cold. Some residue may be left, especially when the materials used are impure, which should be allowed to settle and the clear liquid decanted; or the solution may be cleared by filtration.

In the use of this invention a small quantity of the solution, depending on the strength of the solution and on the nature of the ore, is added to the pulp either before or with or after the addition of the frothing agent, and before or at the beginning of the agitation and
10 aeration. The best results have been generally obtained by the addition of the solution before the frothing agent. Further, an excess of the sulfid solution may cause coagulation of the ore slimes and may prevent the formation of a froth and is to be avoided.

I have found that for the treatment of 500 grams of finely powdered porphyry copper ore, containing about 2% of copper as sulfids, 10 cubic centimeters of the solution made according to Example 1 (sodium poly-sulfid 0.034% of the ore) in 1500 grams of water at normal temperature, may be used with a frothing agent in the proportion of one pound to the ton of ore, such frothing agent
20 consisting of 90% gas tar creosote and 10% yellow pine oil. Agitation and aeration produce a mineral froth containing the copper sulfids of the ore, and the froth is floated off or separated in any suitable manner. With such treatment any copper carbonates present in the ore are concentrated with the copper sulfid and appear in the concentrates as copper carbonates; and an ore or tailings containing the copper in the form of carbonates without any copper sulfids may be efficiently concentrated and the carbonates of copper will appear in the froth and will be separated as concentrates in the form of copper carbonates.

30 In place of the solution made according to Example 1, a sulfid solution may be prepared by dissolving sodium polysulfid, commercially known as fused sodium sulfid, in the proportion of 40 grams per liter of water; and 10 cubic centimeters of this solution may be used with 1500 grams of water at normal temperature for the treatment of 500 grams of the ore above referred to with the frothing agent above referred to. The proportion of the polysulfid in the ore pulp will be 0.027% of the water of the ore pulp and 0.08% of the ore.

The following is an example of carrying out my process on a large scale, the material treated being mill slimes of a porphyry
40 copper ore in a pulp containing four parts of water to one of the slimes. The average assay value of the slimes was about 0.9% copper, of which 0.3% was oxidized. A solution made according to Example 3 was added to the pulp in the proportion of six gallons of the solution per ton of slimes treated, and thereafter the frothing agent was added to the solution. This frothing agent contained oils modified in accordance with the process described and claimed in my application for Letters Patent filed simultaneously herewith, Serial No. 74,932, and not claimed herein, that is to say, these oils

Exhibit—W-9.

had been mixed with sulfur and subjected to high temperatures, in the neighborhood of 200° to 350° centigrade, in an ordinary still, and the distillates collected for use. Prior to the distillation treatment, the pine oil had been mixed in the proportion of 100 cc. of the oil and 5 grams of sulfur, and the petroleum distillate and sulfur, had been mixed in like proportions. This frothing agent contained 10% of light coal tar oil, 10% of pine oil modified as above described, and 80% of a petroleum distillate of 30° Baumé modified as above described, and was used in the proportion of 1.7 to 2 pounds per ton of slimes treated. Agitation and aeration produced a froth of concentrates which assayed about 10% copper and tailings which assayed about 0.1% copper.

In the application of this invention to various ores and under various conditions, the examples of the process above described may be modified without departing from this invention.

I claim:

1. A process of concentrating ores which consists in submitting an ore pulp to flotation separation with agitation and aeration in the presence of a soluble sulfid dissolved in the pulp in an amount and under conditions of treatment so regulated as not to coat oxidized particles with a visible layer of sulfid during such treatment, and by such treatment to obtain a froth of concentrates.
2. A process of concentrating ores containing oxidized metalliferous particles which consists in submitting an ore pulp of such an ore to flotation separation with agitation and aeration in the presence of a soluble sulfid dissolved in the pulp in an amount and under conditions of treatment so regulated as not to coat oxidized particles with a visible layer of sulfid during such treatment, and by such treatment to obtain a froth of concentrates.
3. A process of concentrating ores containing copper which consists in submitting an ore pulp of such an ore to flotation separation with agitation and aeration in the presence of a soluble sulfid dissolved in the pulp in an amount and under conditions of treatment so regulated as not to coat oxidized particles with a visible layer of sulfid during such treatment, and by such treatment to obtain a froth of concentrates.
4. A process of concentrating ores containing oxidized copper which consists in submitting an ore pulp of such an ore to flotation separation with agitation and aeration in the presence of a soluble sulfid dissolved in the pulp in an amount and under conditions of treatment so regulated as not to coat oxidized particles with a visible layer of sulfid during such treatment, and by such treatment to obtain a froth of concentrates.

In testimony whereof I have affixed my signature.

RHETHERFORD B. MARTIN.

Witnesses:

HENRY D. WILLIAMS,
HARRY C. LEWIS.

EXHIBIT G-4

PREPARATION OF FLOTATION REAGENTS

Kotrix A.	Kotrix B.	Kotrix C.
Minola,	Cinol,	Arenol,
Grabanol,	Stanol,	Avenol.

KOTRIX A.

Calcium oxide	47.0%	7.83
Sulphur (flowers)	53.0	8.80
Water	6 liters	1 liter

10 Boil 10 minutes, allow to settle, decanter or filter into a well stoppered bottle.

KOTRIX B.

Calcium oxide	44.6%	7.43
Sulphur (flowers)	50.4	8.40
Sodium hydrate	1.0	0.17
Water	6 liters	1

Boil 10 minutes, allow to settle, decanter or filter into a well stoppered bottle.

KOTRIX C.

20	Sodium tetra sulphide		
	$N_2 S_4 + 6H_2O$		
	Sodium hydrate	52.0	8.7
	Sulphur (flowers)	48.0	8.0
	Water	6. liters	1. liter

Boil until the sulphur goes into solution.

MINOLA

Petroleum distillate 30B.	100 pounds	200 cc.
Sulphur (flowers)	5 "	10 gr.

Distill.

30

CINOL

Pine oil (any specific gravity)	100 pounds	200 cc.
Sulphur (flowers)	5 "	10 gr.

Distill.

ARENOL

Coal tar oil	100 pounds	200 cc.
Sulphur (flowers)	5 "	10 gr.

Distill.

Exhibit—G-4.

GRABANOL O

Resin	100 pounds	100 gr.
Caustic soda	10 "	10 "
		20 cc.

Soda should be dissolved in 20 of water and added to the resin.
Boil or digest in the cold until saponification takes place.

GRABANOL OO

Resin	100 pounds	100 gr.
Sodium carbonate	15 "	15 "

Boil until complete saponification takes place.

10

STANOL

Denatured alcohol	100 c.c.
Carbon disulphide	100 "
Caustic soda (NaOH)	100 gr.

Digest under reflux condenser.

ABENOL No. 308

21/10/15	200 gr. Rosin ½c.	
	2000 cc. Petroleum Distillate 30° Bé (Fuel Oil)	4c. p. gal.

N.Y. in Tank Cars.

GAS PARAFFINE OIL

20 Possession 5 gal.	Price	.05 cts. per gal.
Laboratory No. 503	Sp. gr. 92.	0.860 March 21—1916
	Oil No. XVII	
	obtained	

Texas Oil Co.

Properties

Petroleum distillate

Distilled over 5 grams of sulphur.

Distilling temp

30	210 to 250 c	21.0%	Heavy illuminating
	250 — 300	37.5	Light Lubricating
	300 — 350	26.5	Heavy Lubricating
	Last and Residue	15.0	
		<hr/>	
		100.0	
	Total oil recovered	85.0%	
	" residue	15.0	
		<hr/>	
		100.0	

Exhibit—G-4.

GEORGE LINE

100 cc. American Pine Products Co.
Light Pine Oil No. XCIX

Treated at ordinary temperature
with 25cc. of 10% solution.

Kotrix C. by agitation one minute.

Drawing off bottom solution.

Using top oil strata for flotation.

Yield 98%

10

No. 15—13

Feb. 14—1919.

ALBERTINE

100 cc. Pensacola Pen Oil No. 350

Treated same as Georgeline.

No. 14—13.

Feb. 14—1919.

May 20 — 1920

Mr. Thomas — Smoler and myself.

Notice the oil vapour of about 200 temperature — when pass
over asbestos — or reaching (?) in presence of asbestos —
causes the asbestos to glow.

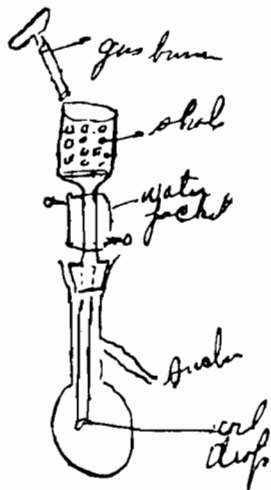
20

R. B. MARTIN.

January 28, 1921.
Morning.

Mr. George A. Chapman — Wilkins — Trotier and Smoler.

Saw oil shale mix with a small quantity of carbon like (?)
and sulphuric acid, and agitated give a separation of sand and
oil conglomerate.



30

Date

Afternoon 4.05 P.M.

By placing granulated shale in the
sketch herein shown and sucking air
through while the flame was playing
on the surface of the shale a froth
was noticed at the filter pump, also
oil was dropping from the bottom of
the tube extending in the flask this
oil gave a froth with the B & S ore.

R. B. MARTIN.

EXHIBIT D-99

UNITED STATES PATENT OFFICE

CLEMENT L. PERKINS, OF PITTSBURGH, PENNSYLVANIA,
ASSIGNOR TO METALS RECOVERY COMPANY, OF
NEW YORK, N.Y., A CORPORATION OF MAINE.

FLOTATION OF MINERALS

1,364,304. Specification of Letters Patent. Patented Jan. 4, 1921.
No Drawing. Application filed July 21, 1919. Serial No. 312,247.

To all whom it may concern:

10 Be it known that I, CLEMENT L. PERKINS, a citizen of the United States, residing at Pittsburg, in the county of Allegheny, State of Pennsylvania, have invented certain new and useful Improvements in Flotation of Minerals; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

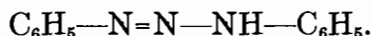
This invention relates to the concentration of minerals, such as sulfid ores and the like, by flotation, and is based upon the discovery that improved results can be obtained by carrying out the flotation
20 operation with the addition, to the ore or mineral pulp, of certain non-oleaginous solid organic compounds, which themselves have substantially no frothing properties, but which have valuable properties as collecting agents for the mineral. Such collecting agents, although substantially non-frothing, nevertheless so act upon the minerals that they very materially assist in the recovery of the minerals when used in connection with a suitable frothing agent.

Among the agents which are of particular value as collecting agents, and which are substantially non-frothing, are reduced compounds which are relatively easily oxidizable, such as, for example,
30 diazo-amino-benzene. It may be that these compounds are absorbed at the surface of the sulfid or mineral particles, and that they so modify the sulfid or mineral surfaces that these surfaces are less easily wet by water or made more readily susceptible to attachment by the air bubbles. In some cases the reduced compound is apparently further reduced by the action of the sulfid particles, so that the action may partake of both a chemical and physical nature. These agents also appear to be absorbed to some extent by certain of the gangue particles, but in this case oxidation of the collecting agent apparently takes place at the surface of the gangue particles, so that
40 the actual surface presented is an oxidizing or oxidized surface, while in the case of the sulfid particles it appears to be a reduced surface. However, I do not desire to limit myself by any theoretical explanation of the mechanism of the action of these collecting agents.

Although the collecting agents are substantially insoluble, and are commonly referred to as insoluble, nevertheless, they are soluble to a very small degree, and it appears to be by reason of this solubility that they are able to act upon the minerals and exert their marked selective and collecting properties.

10 These collecting agents are, as above noted, non-frothing, and it is, therefore, important to supplement their action by an agent which will provide an appropriate amount of froth. The frothing agent, however, may be entirely inadequate to perform the flotation process by itself, inasmuch as its function appears to be simply to provide bubbles in sufficient quantity and of sufficient persistency to overflow into the launder of the flotation machine, thereby mechanically facilitating the removal of the mineral concentrate.

Among the collecting agents of the character above referred to, that is, which are substantially non-frothing, are included certain of the aromatic thio-ureas, and many of the azo and diazo compounds. A collecting agent which I have found of particular value is diazo-amino-benzene,



20 The collecting agents can be incorporated with the ore or mineral pulp in any suitable manner which will insure their uniform incorporation and dissemination through out the pulp. They may thus be incorporated by grinding in the tube mill in which the ore itself is ground, or they may be dissolved in an organic solvent therefor, and the resulting solution added to the ore pulp. When a solvent of the collecting agent is employed, it may advantageously itself be an agent with good frothing properties, so that its action will supplement the collecting action of the collecting agent. After the collecting agent and a suitable frothing agent have been thoroughly and uni-
30 formly distributed throughout the ore or mineral pulp, the resulting mixture is subjected to a flotation operation, as in an ordinary Callow cell, or in apparatus where the air is incorporated in the ore pulp by mechanical agitation.

It is of advantage in many cases to add to the ore pulp a small amount of alkali, which may be sufficient in amount to give to the ore pulp a distinct alkaline reaction. When alkalis are used, it is desirable to give them an appreciable time of contact with the ore pulp before subjecting the latter to flotation. When possible, it appears preferable to add the alkalis in the grinding mill, so that they
40 will be present during the grinding operation. The alkali may be caustic alkali, such as sodium hydrate, or an alkaline salt, such as sodium carbonate, or even lime or calcium oxid or hydrate can be used.

The amount of the collecting agent added may vary somewhat, and may be as little as one-fifth or one-fourth of a pound per ton of ore (that is, calculated on the weight of the ore and not of the ore

pulp). I have obtained good results with the use of as little as one-thirtieth of a pound per ton of ore. Larger amounts may, however, be used.

The invention will be further illustrated by the following specific examples of tests made upon a copper sulfid ore having a silicious gangue containing its floatable or sulfid copper in the form of a mixture of chalcocite and chalcopyrite, that is, so-called porphyry ores, such as those of the Utah Copper Company. In general, treatment of the ore with alkalis, as above noted, gives improved results, the alkalis themselves apparently having a valuable effect upon the ores in improving the floatation thereof.

1. *Diazo-amino-benzene and terpeneol*.—500 parts by weight of Utah Copper Company's ore was ground in the pebble mill with 250 parts of water, sodium hydroxid equivalent to two pounds per ton of dry ore, and diazo-amino-benzene equivalent to one-fifth pound per ton. The ground pulp was transferred to a Callow rougher cell, diluted, and floated with the addition of terpeneol equivalent to about one-tenth pound per ton. The heads for this test contained 1.42 per cent. copper, the concentrates produced contained about 22.4 per cent. copper, and the recovery was about 91.4 per cent.

2. *Diazo-amino-benzene and terpeneol*.—The details of the test were the same as those of No. 1, except that the proportion of diazo-amino-benzene was reduced to one-thirtieth pound per ton. The heads for test No. 2 contained 1.38 per cent. copper, the concentrate produced contained about 17.3 per cent. copper, and the recovery was about 87.7 per cent.

3. *Diazo-amino-benzene and pine oil*.—The details of this test were the same as those of No. 1, except that steam-distilled pine oil was substituted for terpeneol. The proportion of pine oil used was about one-fourth pound per ton of dry ore. The heads for test No. 3 contained 1.39 per cent. copper, the concentrates produced contained about 17.9 per cent. copper, and the recovery was about 88.6 per cent.

4. *Diazo-amino-benzene dissolved in xylidin*.—Five hundred parts by weight of Utah Copper Company's ore were ground with 250 parts of water and sodium hydroxid equivalent to two pounds per ton of dry ore. The ground ore pulp was diluted, and to the ground and diuted pulp was then added a solution of diazo-amino-benzene in xylidin. The proportion of the compound reagent used was one-half pound per ton of dry ore. It contained about 80 per cent. xylidin and 20 per cent. diazo-amino-benzene. The pulp was transferred immediately to a Callow rougher cell and floated. The heads for this test contained 1.39 per cent. copper, the concentrates contained about 21.9 per cent. copper, and the recovery was about 91.2 per cent.

As appears from these examples, different frothing agents may be used, and the collecting agents may be itself dissolved in a solvent

Exhibit—D-99.

which has good frothing qualities. Xylidin is a good example of a valuable flotation agent which serves as a solvent for the diazo-amino-benzene and supplies the necessary froth for the flotation operation. Among other solvents may be mentioned anilin, dimethyl anilin, toluidin and other organic liquids in which the particular collecting agent is soluble, such as cresol, alcohol, etc. The solution of diazo-amino-benzene in xylidin can be promoted by heating, but prolonged heating may result in chemical changes which reduces the effectiveness of the resulting product.

- 10 The collecting agent can with advantage be used in addition to many of the usual oil mixtures which are commonly employed in flotation, with resulting improvements in the flotation operation. Flotation oils or oil mixtures which are good frothing agents, such as pine oil and other wood distillation oils, are of particular value, inasmuch as they supplement the action of the collecting agent, and are themselves supplemented in their ore floating properties by the collecting action of the collecting agent.

- 20 In the flotation of zinc-lead ores, it has been found of advantage to grind the collecting agent with the neutral ore pulp; and still better results have been obtained when copper sulfate and sulfuric acid were also used in the proportion of about one pound of each per ton of dry ore. A small amount of a suitable frothing agent, as above noted should also be used.

- 30 It is characteristic of the invention that the froth obtained is readily broken up, while a relatively clean concentrate and high recovery can, nevertheless, be obtained. A part of the collecting agent, together with a part of the frothing agent, can sometimes be recovered so that they are available for use in the treatment of further amounts of ore; for example, by recovering the circuit water from the concentrates, or even that from the tailings, and returning the same to the process, with resulting economy in the amount to be added for subsequent operations. The amount of the collecting agent can, however, in many cases be reduced to such a small amount that it is feasible to practise the invention without such recovery of the agents employed.

- 40 As above noted, the action of the collecting agent appears to be a selective action upon the mineral surfaces, rather than any action of importance upon the water itself, inasmuch as the removal of all water from the ore pulp, after the collecting agent has been incorporated therewith, and the subsequent washing of the ore, does not deprive it of its improved flotation properties when made into an ore pulp with fresh water and subjected to flotation. Investigations of this kind indicate that the collecting agents have a selective modifying effect upon the mineral surfaces, and that any modifying effect upon the water is incidental. The modifying action of the collecting agents upon the mineral particles is sufficiently marked

Exhibit—D-99.

so that it is possible to raise larger mineral particles, and hence finer grinding becomes of less importance, or unnecessary.

The process in which organic nitrogen compounds, including azo and diazo compounds, many of which are non-frothing collecting agents, are employed, is claimed in my companion application, Serial No. 312,248 filed of even date herewith; the more specific claims of said application being directed to the process in which diazo-amino-compounds are employed, and specifically to the process in which diazo-amino-toluene is employed.

10 The flotation process in which organic thio-urea and other nitrogen-sulfur compounds, such as thio-carbanilid, many of which are likewise non-frothing collecting agents, are employed, is claimed in two joint applications, of the present applicant with Ralph Sayre Serial No. 284,981, filed March 25, 1919, and Serial No. 337,322, filed November 11, 1919.

The more comprehensive claims of the present case are intended to be of a comprehensive and generic character for the process in which such substantially non-frothing collecting agents are employed; while the specific claims of the present case are directed to the process
20 in which diazo-amino-benzene is employed.

I claim:

1. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a small amount of a substantially non-oleaginous organic mineral collecting agent which is substantially non-frothing, and subjecting the resulting mixture to a froth flotation operation: substantially as described.

2. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a substantially non-frothing, non-oleaginous organic mineral collecting agent which
30 is substantially non-frothing, together with an agent having good frothing properties, and subjecting the resulting mixture to a froth flotation operation; substantially as described.

3. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a small amount of a reduced and easily oxidizable organic mineral collecting agent which is substantially non-frothing, and subjecting the resulting mixture to a froth flotation operation; substantially as described.

4. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a small amount
40 of a reduced and easily oxidizable organic mineral collecting agent which is substantially non-frothing, together with an agent having good frothing properties, and subjecting the resulting mixture to a froth flotation operation; substantially as described.

5. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a solution of a

Exhibit—D-99.

substantially non-frothing non-oleaginous organic collecting agent in an organic solvent therefor which has good frothing properties, and subjecting the resulting mixture to a froth flotation operation; substantially as described.

6. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a small amount of a reduced and readily oxidizable organic mineral collecting agent which is substantially non-frothing and which is dissolved in a solvent therefor which is a good frothing agent, and subjecting the resulting
10 mixture to a froth flotation operation; substantially as described.

7. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a small amount of diazo-amino-benzene, and subjecting the resulting mixture to a froth flotation operation; substantially as described.

8. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a small amount of diazo-amino-benzene, together with an agent having good frothing properties, and subjecting the resulting mixture to a froth flotation operation; substantially described.

20 9. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a small amount of diazo-amino-benzene, together with a small amount of a frothing oil, such as pine oil, and subjecting the resulting mixture to a froth flotation operation; substantially as described.

10. The method of effecting the concentration of minerals by flotation, which comprises adding to the mineral pulp a small amount of diazo-amino-benzene, dissolved in an organic solvent therefor, and subjecting the resulting mixture to a froth flotation operation; substantially as described.

30 In testimony whereof I affix my signature.

CLEMENT L. PERKINS.

EXHIBIT D-91

MINERALS SEPARATION NORTH AMERICAN CORPORATION
Office of the Chief Engineer
220 Battery Street, San Francisco, Cal.

San Francisco, California, April 28th, 1921.
JVC—LM

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N.A. Corp.,
220 Battery St., San Francisco, Calif.

10 Dear Sir:

The following items were offered by the various members of the staff at our meeting this morning, April 28th, as subjects suitable for extensive research work worthy of our best efforts, and they present some problems that have so far met with little success towards a commercial solution.

We intend to extend ourselves on these various subjects when laboratory work is slack and sincerely hope to be able to report some interesting progress at a future date. The items offered were as follows:—

- 20 1. The recovery of high silver values from zinc concentrates, either by mechanical or chemical treatment.
2. The differential separation of copper, lead, zinc and iron in these complex ores.
3. The chemical treatment of zinc concentrates to prepare them for a better precipitation by electrolysis.
4. To determine the effect of colloidal material of an amenable ore to flotation on another ore that is only partially amenable to flotation and vice versa.
- 30 5. The application of the Harvey Smith method to ores containing copper silicate, carbonates and oxides, endeavoring to prepare the silicates and oxides to be more amenable to sulphidization.
6. The recovery of flour gold from dredges and sand bars in a fairly high grade concentration by flotation.
7. Mechanical adjustments in the W and L machine, in order to obtain the best possible metallurgical results at the lowest operating cost.
8. To try out the practicability of a pneumatic cell employing a vibrating air mat.
- 40 9. The differential separation of arsenic gold ores by flotation.

Exhibit—D-91.

Exhibit—K-13.

10. Extensive research on the Compagnie De Belso ore, using a combination of leaching and flotation.
11. The alteration of copper silicate by chemical means, thereby preparing the surfaces to make them amenable to sulphidization.
12. The effect of differential separation on various ores when treated under pressure and heat. In this work it is not intended that any chemical or physical changes shall occur within the mineral particles, but the effect, if any, will be on the exposed surfaces.

Any progress made on these subjects will be reported from time to time as the results become complete.

Yours very truly,

(Sgd.) J. V. QUIGLEY.

EXHIBIT K-13

C O P Y

San Francisco, California, Sept. 7, 1922.

CHK—M

Edward H. Nutter, Esq., Chief Engineer,
 Minerals Separation N. A. Corp.,
 20 220 Battery St.,
 San Francisco, Calif.

Dear Sir:

In the treatment of oxide ores, sodium sulphide or hydrogen sulphide are often resorted to in order to make them amenable to flotation. However, this treatment is open to a number of serious objections, both from a physico-chemical and operating standpoint.

In endeavoring to devise a more satisfactory method it occurred to me to employ a sulphide of Mercury. At first an attempt was made to use mercury sulphide precipitated in the pulp while
 30 grinding. Another attempt was made, introducing a Mercury salt in the flotation machine with the pulp, adding the necessary precipitant. Both of these experiments resulted abortive. Consequently the operation finally decided on and adhered to in subjoined tests, consists in mixing the ore, ground to 8 or 10 mesh, with 1 - 2% sulphur and .5 - 1.5% mercury, according to quantity of mineral to be floated, and pulverizing dry to flotation size in a rod mill. The sulphide of mercury thus produced has a strong affinity for

Exhibit—K-13.

oxide ores, such as carbonates, sulphates, silicates, etc., gives a very tenacious coating (uninfluenced by weak alkalies or acids) a voluminous froth in the flotation machine with ordinary reagents, (acting as a good collector) and can be recovered almost entirely by distillation.

However, at an early stage of these investigations it was found that the recovery of oxide particles from an ore depended largely on the metal content of each individual particle. It seems, unfortunately, a fact that metal oxides are largely dissolved in the gangue material. Upon comminution the ore is not separated into almost pure metal oxide particles and almost pure gangue, as in many sulphide carrying materials, for instance, but different gradations varying in metal content are brought forth. It was found that the particles with a low metal content were not at all or only insufficiently coated. To prove my contention I carried out some tests on synthetic ores, made up by grinding sand with various pure oxide minerals, of more or less metal oxide content. By employing high grade chrysocolla a recovery by mercury sulphide coating and flotation of 84% was obtained, whereas a low grade chrysocolla gave only an extraction of 43%. While in the former case hardly any green particles could be detected by the lens, in the latter case a large number of partly translucent green particles could be made out with the unassisted eye.

A test made on very low grade material from South America, served as an additional proof. Ore ground dry with Hg and S, made up with water to thin pulp, ores added and floated. Lab. sample No. 17511.

TEST No. 1.

	WEIGHT	DESCRIPTION	CU.	EXTRACTION
30	36 gr.	Concentrate	1.04	17.3
	556 gr.	Tailings	0.32	82.7
				100.0%

Another test, made on Engels carbonate and silicate of somewhat higher grade, gave the following results after the same treatment.

TEST No. 2. Lab. Sample No. 10552.

	WEIGHT	DESCRIPTION	CU.	EXTRACTION
	280 gr.	Concentrate	8.56	79.3
	540 gr.	Tailings	1.16	20.7
				100.0%

40

The same difficulty was encountered with oxide Pb. ores. Synthetic ore made up of high anglesite, corrusite and pyromorphite

Exhibit—K-13.

and sand, gave an extraction of 88.5%; low grade material gave an extraction of 31% only. As there was no higher grade ore available I used some Corro Gordo flotation tailings, with the following result:

TEST No. 3.

WEIGHT	DESCRIPTION	CU.	EXTRACTION
90 gr.	Concentrate	8.8	27.0
505 gr.	Tailings	4.2	73.0
			100.0%

A low grade Adams ore gave a similar result:

10 TEST No. 4. Lab. Sample No. 18570.

WEIGHT	DESCRIPTION	PB.	EXTRACTION
100 gr.	Concentrate	6.6	31.8
488 gr.	Tailings	2.9	68.2
			100.0%

Synthetic zinc ore, made from high grade calamine and sand, gave an extraction of 79.5%; of low grade material 29.8% extraction. Tests on zinc ore had to be deferred until some suitable material could be procured.

In testing for extraction of precious metals by the mercury sulphide flotation method, I had a larger choice of material to
20 work on and consequently its scope could be defined more readily.

TEST No. 5. Ore from Big Chief Mine. Lab. Sample No. 20900.

WEIGHT	DESCRIPTION	AU.	EXTRACTION	AG.	EXTRACTION
50 gr.	Concentrate	17.56	83.0	104.0	76.6%
546 gr.	Tailings	0.33	17.0	2.9	23.4
			100.0	100.0%	

Another test on the same sample — No. 20900.

TEST No. 6.

WEIGHT	DESCRIPTION	AU.	EXTRACTION	AG.	EXTRACTION
85 gr.	Concentrate	11.22	86.8	69.8	81.5%
30 518 gr.	Tailings	0.28	23.2	2.6	18.5
			100.0	100.0%	

TEST No. 7.

A third test on Sample No. 20900 resulted in an extraction of 91.4 of the gold and 81.5 of the silver, after regrinding and

Exhibit—K-13.

retreating primary tails. The final tailings ran 0.19 Au. and 2.8 in Ag.

TEST No. 8. Big Chief Final Table Tails — Treated, reground and floated. Lab. Sample No. 20958.

WEIGHT	DESCRIPTION	AU.	EXTRACTION	AG.	EXTRACTION
155 gr.	Concentrate	0.74	81.6	9.6	63.
515 gr.	Tailings	0.05	18.4	1.7	37.
			100.0%	100.0%	

TEST No. 9. Flotation Tails Big Chief. Sample No. 20912.

10 WEIGHT	DESCRIPTION	AU.	EXTRACTION	AG.	EXTRACTION
73 gr.	Concentrate	1.74	60.5	11.6	44.5
555 gr.	Tailings	0.15	29.5	1.9	55.5
			100.0%	100.0%	

TEST No. 10. Big Chief Silver ore. Lab. Sample No. 20901.

WEIGHT	DESCRIPTION	AU.	EXTRACTION	AG.	EXTRACTION
89 gr.	Concentrate	1.0	64.5	275.6	79.8
615 gr.	Tailings	0.08	35.5	10.1	20.2
			100.0%	100.0%	

TEST No. 11. Big Chief Silver ore. Lab. Sample No. 20902.

20 WEIGHT	DESCRIPTION	AU.	EXTRACTION	AG.	EXTRACTION
160 gr.	Concentrate	0.82	66.2	276.2	90.3
670 gr.	Tailing	0.10	33.8	6.9	9.7
			100.0%	100.0%	

TEST No. 12. Engels Carbonate & Silicate Cu ore. Lab. Sample No. 10552.

WEIGHT	DESCRIPTION	AG.	EXTRACTION
187 gr.	Concentrate	4.42	87.1
612 gr.	Tailings	0.20	12.9
		100.0%	

30 TEST No. 13. Adams low grade Pb. ore. Lab. Sample No. 18575.

WEIGHT	DESCRIPTION	AG.	EXTRACTION
100 gr.	Concentrate	51.7	73.1
488 gr.	Tailings	3.9	26.9
		100.0%	

Exhibit—K-13.
Exhibit—K-18.

In conclusion I wish to state that results obtained by above method compare favorably with Na_2S or H_2S coating and with amalgamation, floating and tabling. With exception of Test No. 7, where two treatments were resorted to, all other results were gotten in one operation without recleaning of concentrates. The recovery of the reagents should not present any difficulty, as much as 95% of the mercury having been distilled off on small scale test.

Yours truly,

(Sgd.) C. H. KELLER.

10

EXHIBIT K-18

MINERALS SEPARATION NORTH AMERICAN CORPORATION
OFFICE OF THE CHIEF ENGINEER

220 BATTERY STREET, SAN FRANCISCO, CAL.

San Francisco, California, Dec. 11th, 1922.

CHK-M

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Cal.

Dear Sir:

20 In re Mr. Chester B. Allen's letter dated Nov. 9th, 1922, concerning the use of locally manufactured Thiocarbanilide on Anaconda slimes, it appeared advisable to carry out a number of experiments to elucidate some of its qualities and functions.

Thiocarbanilide $\text{CS}(\text{NH}_2)_2$, described in most chemical handbooks as being a water insoluble compound, was found to be distinctly soluble, in distilled water, tap water; in pine oil, coal tar creosote; in water containing pine oil or coal tar creosote in emulsion, or water containing certain electrolytes. The solubility varies with the purity of the material under examination.

30 1. The locally manufactured, of a sliding M.P. 148-150 containing a number of possibly more soluble contaminating sulfosalts, due to the excess of carbon disulfide necessary to preparation, showed the greater solubility.

2. Next to it the "Practical" (Technical) salt of Eastman Kodak laboratory manufacture, M.P. 150-152, containing also some possibility more soluble contaminants, gave a fair solubility.

3. The "Pure" salt of Eastman Kodak laboratory manufacture M.P. 154, having been refined, showed the least solubility of the three

Exhibit—K-18.

examined. Similarly the action in the flotation machine demonstrated that the locally manufactured No. 1, as well as the "Practical" No. 2 salt gave results superior to No. 3 "Pure."

In accordance with your indication, water extracts were made of the 3 salts, carefully filtered and the liquid employed in the flotation machine with a diminished yet fairly pronounced result. At the rate of dilution of 1 part to a million parts of water it could be detected on preparations 1 and 2, whereas a more concentrated solution was required on No. 3. As 1 mgr. of the salt agitated with one liter of
 10 water for a prolonged period showed the crystals apparently undiminished in size and only a slight rounding of the crystal edges noticeable at the bottom of the vessel, only V.S.S. in water is shown as per Atack's "Chemists Year Book" Vol. 1 for 1922, which by the way was the only handbook of a large number consulted that attributed any solubility to the stuff at all. The solubility in cold pine oil or cold coal tar creosote was greater for the impure salts than for the "Pure". In hot pine oil all 3 dissolved with greater ease. As the material employed at Anaconda is no doubt manufactured in larger quantities ipso facto its content of impurities will be relatively
 20 greater, with a probable enhancement in flotation action.

Another factor contributing to the favorable results obtained by means of Thiocarbanilide on the Anaconda Slimes sample sent to us from Butte, is the relatively large amount of pine oil in the supernatant liquid. This pine oil is no doubt introduced through the accidental contact with pine wood chips, mine timber, etc. This may also explain some of the erratic results obtained at Anaconda, with ordinary flotation reagents, whose compatibility with pine oil is doubtful.

Subjoined flotation tests made by Mr. C. P. Lewis do not
 30 require any explanation further than that Walker ore was selected for its quick response to flotation reagents.

The remark colloidal silver reaction positive or negative refers to the detection of the Thiocarbanilide and its contaminants in the filtered circuit water.

TEST No. 1.

Anaconda Slime — Tap water Colloidal silver reaction positive.

Treat — 30 min.

With .5 gr. carb. prepared locally — Thio. No. 1.

.15 No. pine oil

40 Good frothing conditions.

	Cu.
Concentrates	29.52
Tails	0.52

Exhibit—K-18

TEST No. 2

Walker Ore — Tap water — Reaction positive.

Thio. No. 2. Preag.— 4 min. with .125 gr. "practical Carb."

Treat — 6 min. No reagents.

Fair frothing. Needs frother to clean up.

Cu.

Concentrates	29.52
Tails	0.52

TEST No. 3.

10 Walker Ore — Anaconda slime water — Reaction strongly positive.

Thio. No. 2. Preag.— 4 min. with .125 gr. "Practical Carb"

Treat — no reagents 6 minutes.

Abundant froth — good conditions.

Cu.

Concentrates	19.04
Tails	0.22

TEST No. 4.

20 Walker Ore — Tap water — Reaction positive.

Preag. — Pine wood extract 200 cc.

.125 grams "Practical Carb." Thio. No. 2.

Treat.— No reagents 6 minutes.

Fair frothing. Requires little frother to clean up.

Cu.

Concentrates	25.68
Tails	0.58

TEST No. 5.

30 Walker ore — Tap water — Reaction negative.

Preag. — .125 gr. Pure Carbanalide Thio. No. 3.

4 minutes.

Treat.— No reagents. 6 minutes.

Very little frothing.

Cu.

Concentrates	28.64
Tails	2.80

TEST No. 6.

40 Walker Ore — Tap water — Reaction slightly positive.

Preag. — Pure Carbanalide Thio. No. 3. .125 gr.

Pine Oil 0.1 No.

4 minutes.

Exhibit—K-18.

Treat. — No reagents. 6 minutes.
 Good frothing conditions. Rapid float.

	Cu.
	—————
Concentrates	25.2
Tails	0.38

TEST No. 7.

Anaconda Slimes — Tap water — Reaction negative.

Preag. — .125 gr. Pure Carbanilide Tio. No. 3.
 4 minutes.

10 Treat C₁ No reagents. 5 minutes
 Very little selection. Poor conditions.
 Treat C₂ 21V. O.1 No.
 20 Minutes.

Little better on selection and more light froth.

	Cu.
	—————
Conc. 1	9.44
Conc. 2	6.44
Tails	2.80

20 While it appears from the foregoing that the different impurities
 in the manufactured and technical thiocarbanilide play a most
 important role in its flotation action, the question "Is thiocarbanilide
 proper a soluble organic flotation reagent" can be answered in the
 affirmative.

Yours very truly,

CORNELIUS H. KELLER.

EXHIBIT K-24

MINERALS SEPARATION NORTH AMERICAN CORPORATION
OFFICE OF THE CHIEF ENGINEER

220 BATTERY STREET, SAN FRANCISCO

ASSAY CERTIFICATE

Date 3/15 1923.

		ASSAYS						
		GOLD	SILVER					
		OZ.	OZ.	COPPER	LEAD	ZINC	IRON	INSOLS
10	SAMPLE	PER TON	PER TON	%	%	%	%	%
	T 933			0.40	1.7	2.1	10.8	
	T 937				0.8	0.4	24.6	
	T 903			1.24				
	T 927			0.74				
	C 936				4.1	2.5	22.7	
	C 935				13.2	26.0	17.2	
	C 939			4.56	15.2	29.2	14.8	
	C 942			7.68	13.6	24.0	18.6	
	C 944			1.44	4.5	11.0	36.0	
20	C 934				58.4	7.2	7.5	
	C 938			90.4	36.4	14.0	14.0	
	C 941			4.88		5.0	4.7	
	C 931			6.48				
	C 925			8.32				
	C 932			3.68				
	C 926			4.64				
	C 923			2.32				
	T 940			0.82	2.4	1.5	11.5	
	T 945			0.52	1.6	2.0	6.8	
30	T 949			1.80				
	C 943			2.88	0.3	31.4	18.0	
	C 951		10.1		22.0	24.8	12.6	
	C 950		24.0		57.6	6.4	9.2	
	C 941				63.6			
	C 948			10.16				
	C 957			24.56				
	890	0.04	1.6					
	889	0.6	6.4					
	888	0.05	5.3					
40	887	0.18	20.2					
	886	0.5	29.5					
	919		18.9					
	920		5.6					

MINERALS SEPARATION NORTH AMERICAN CORPORATION
 OFFICE OF THE CHIEF ENGINEER
 220 BATTERY STREET, SAN FRANCISCO

ASSAY CERTIFICATE

Date 3/7 1923.

		ASSAYS						
SAMPLE		GOLD	SILVER	COPPER	LEAD	ZINC	IRON	INSOLS
		OZ.	OZ.					
		PER TON	PER TON	%	%	%	%	%
10	937		0.44					
	936		1.44					
	935		6.0					
	934		24.6					
	T 955	0.87	3.5					
	C 954	1.88	6.6					
	T 918	0.025	2.8					
	T 945	0.02	1.9					
	C 941	0.10	38.1					
	C 916	0.14	28.9					
20	C 942	0.66	17.3					
	C 917	0.26	8.8					
	C 944	1.62	6.6					
	C 943	0.08	5.1					

Exhibit—W-15 (also Exhibit—K-2)

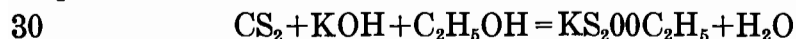
EXHIBIT W-15 (also K-2)

March 28, 1923.
CHK—CPLEdward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

Dear Sir:

Subjoined tests are the results of investigations carried out inter-
mittently for a number of months on organic-inorganic sulfidising
10 reagents. The sulfidising chemicals commonly employed in differ-
ential flotation are either costly, hard to apply or difficult to control,
consequently the endeavor was made to find a substance free from
these objections.

As an indication for the direction the investigations should have
to take, we started with the Mercaptans. Tests 1 and 2 gave results
obtained with Ethyl-Mercaptan. This substance, while giving good
results, is impracticable, owing to price and offensive odor. The
Xanthates or Xanthogenates, alkali salts of Ethyl-oxydi-thiocarbonic
20 acid were finally adopted as being the most feasible. As the potas-
sium salt can be prepared with ease and its raw material cost does
not exceed 10 cents per lb. it was adhered to in most of the subsequent
tests. Potassium Xanthate is a white crystalline substance, which
turns yellowish but does not decompose on exposure to air; it is
non-deliquescent, soluble in all proportions in water, soluble in
Ethyl-Alcohol and slightly soluble in some oils. It decomposes at
200 C. on heating dry and by prolonged boiling of its water solution.
The method of preparing it consists in adding carbondisulfide to an
alcoholic solution of Potassium hydrate. While the reaction takes
place according to the formula—



calling for 46 parts of Alcohol; the actual manufacturing may require
a larger amount. Allowance has been made for this in computing
the approximate cost upon the basis of the New York quotations,
trebling the amount of alcohol theoretically necessary. This excess
alcohol may be recovered by distillation in large scale manufacturing
operations.

A few tests to show the possibilities of compounds, such as
Potassium Xanthate with incorporated Pine oil, were also carried out.
In compliance with your indications we put the results obtained by
40 our method and those employing the usual reagents, in juxtaposition
to clearer indicate advantages to be gained by the use of the Xantho-
genates.

The first tests, made on Anaconda slimes, show a marked decrease
in the time of treatment with a higher grade of final concentrate than
has heretofore been possible in a neutral circuit on this material.

Exhibit—W-15 (also Exhibit—K-2)

Similarly the test quoted on Inspiration gave a greater recovery of the sulfide copper than any previous test had shown. It was found that altho a great excess of Xanthogenate was used in treating oxidized copper ores no sulphidizing action was obtained and consequently our testing was confined to complex ores and those sulfide ores that had proven refractory to ordinary methods of treatment by flotation where only a limited sulphidization was required to effect the desired recovery.

10 It was also found that a small amount of the Xanthogenate used with a minimum of frothing oil such as pine oil or coal tar creosote in combination on gold-silver ores or complex gold-silver bearing ores gave a greater recovery of the precious metals than was found possible without the use of the Xanthogenate, e.g., California Rand and Park Utah Silver ore. In the case of complex ores where the silver and/or gold was known to be present as distinct minerals the recovery of gold and/or silver in all cases was greater in the first (lead) concentrate when treated with a small amount of Xanthogenate. As for example tests on Daly Judge, S.F. del Ore and Cash ore.

20 Lead zinc ores treated by flotation using from .1 to .2 lbs. per ton Xanthogenate with a minimum of coal tar creosote gave a higher grade concentrate with as high or higher recovery of the lead than was found possible without its use. The effect of the Xanthogenate when added to the pulp is instantaneous and changes appearance of the lead bearing froth to heavily coated sparkling froth that resembles a graphite froth in most cases. The lead contained therein is intensely flocculated and as a rule is recovered in from $\frac{1}{2}$ to $\frac{2}{3}$ of the time ordinarily required.

The following points in favor of these reagents as compared to those now in use are:—

- 30 1. Low cost.
2. Unlimited supply.
3. Complete solubility.
4. Rapidity of action—requires no emulsification or preagitation.
5. Reduction of time of flotation treatment.
6. Greater recovery of lead from complex ores in a higher grade product.
7. Increased recovery of silver and gold, in a more marketable product, from complex ores.
8. Adaptable to all type machines.
- 40 9. Oils can be incorporated to make a unit reagent. (The amount and kind of oil would have to be determined for each ore.)
10. Can be used in alkaline, neutral or acid circuits without decomposition or hindering the value of Xanthogenate as a flotation reagent.

Exhibit—W-15 (also Exhibit—K-2)

11. Its use in lead-zinc separations does not hinder or retard the subsequent zinc float as is the case in using the polysulfides of sodium or when cyanide is used.

It is recommended that the above points be covered by patent should they prove of sufficient merit.

Very truly yours,

ANACONDA SLIME.

	% WT.	Cu.	Cu.
10	Heads 100.0	3.48	100.0
	Conc. 27.1	12.32	96.2
	Tails 72.9	.18	3.8

TREATMENT

Mercaptan 2.5#
Pens. Lt. Pine Oil 0.05#
20 Min.

	% WT.	Cu.	Cu.
	Heads 100.0	3.58	100.0
	Conc. 29.4	11.52	94.9
	Tails 70.6	.26	5.1

20

TREATMENT

Mercaptan 0.9#
Water Gas Tar 0.4
20 Min.

New sample (lower grade) quoted for comparison.

	% WT.	Cu.	Cu.
	Heads 100.0	2.95	100.0
	Conc. 25.1	10.40	88.7
	Midds. 7.9	2.44	6.5
	Tails 67.0	.22	4.8

30

TREATMENT

Sodium Silicate 1.5#
Sodium Pentasulfide 2.0
Coal Tar 2.0
Pine Oil 0.3
30 Minutes

Exhibit—W-15 (also Exhibit—K-2)

ANACONDA SLIME (Contd.)

		% WT.	CU.	CU.
	Heads	100.0	2.95	100.0
	Conc.	16.5	15.84	88.9
	Midd.	13.3	1.04	4.7
	Tail.	70.2	.28	6.4
		TREATMENT		RETREAT.
	Potassium Xanthogenate		0.2#	No reagents
	Pine Oil		0.2	5 minutes
10		20 Minutes		
		% WT.	CU.	CU.
	Heads	100.0	2.92	100.0
	Conc.	27.9	9.92	93.7
	Tail.	72.1	.26	6.3
		TREATMENT		
	Potassium Xanthogenate		0.4#	
	Pine Oil		0.25	
		10 Minutes		
20		% WT.	CU.	CU.
	Heads	100.0	2.81	100.0
	Conc.	18.5	14.16	93.2
	Midd.	17.7	.36	2.3
	Tails	3.8	.20	4.5
		TREATMENT		
	Potassium Xanthogenate into which	} 1.0#		
	is incorporated 33% by volume of			
	pine oil before precipitation by CS ₂ .			
		12 Minutes.		
30		% WT.	CU.	CU.
	Heads	100.0	2.72	100.0
	Conc.	18.0	13.20	87.7
	Midd.	20.9	1.04	7.9
	Tail.	61.1	.20	4.4
		TREATMENT		RETREAT.
	Potassium Xanthogenate mixed with	} 1.0#		No reagents
	33% by volume of pine oil after			
	precipitation CS ₂ .			
		12 Minutes.		3 Minutes.

Exhibit—W-15 (also Exhibit—K-2)

ANACONDA SLIMES (Continued)

ACID CIRCUIT.

	% WT.	Cu.	Cu.
Heads	100.0	2.83	100.0
Conc.	31.9	8.64	97.1
Tails.	68.1	.12	2.9

TREATMENT

10 H₂SO₄ 25.0#
 Potassium Xanthogenate into which
 is incorporated 33% by volume of } 0.4#
 pine oil before precipitation by CS.₂
 20 Minutes.

Acid circuit did not give us quick action or as intensive flocculation as in neutral circuit.

BENZENE SERIES OILS IN ACID CIRCUIT.

	% WT.	Cu.	Cu.
Heads	100.0	3.00	100.0
Conc.	15.2	16.88	85.7
Midd.	10.6	3.00	10.6
20 Tails.	74.2	.15	3.7

TREATMENT

H₂SO₄ 25.0#
 Crude Solvent Naptha 3.2
 30 Minutes.

INSPIRATION CRUDE ORE.

	% WT.	Cu.	Ox. Cu.	Cu.	Ox. Cu.
Heads	100.0	1.46	.62	100.0	100.0
Conc.	5.6	14.08	1.24	54.2	11.2
Midd.	10.5	1.40	.76	10.1	12.9
30 Tail.	83.9	.62	.56	35.7	75.9

TREATMENT

Potassium Xanthogenate 0.3#
 Light Pine Oil 0.15
 Coal Tar Creosote 2.0
 Sodium Silicate 1.0
 20 Minutes.

RETREAT.

No reagents
 5 Minutes.

Best previous test showed a tailing of .90 Cu. and .5 Oxide Cu. using Kotrix as a modifying agent with a similar oil combination as was used in the above test.

Exhibit—W-15 (also Exhibit—K-2)

CALIFORNIA RAND. 15 min. grind.

	% WT.	AU.	AG.	AU.	AG.
Heads	100.0	.089	24.93	100.0	100.0
Conc.	14.5	.52	168.7	84.6	98.3
Tail.	85.5	.016	0.5	15.4	1.7

TREATMENT

Potassium Xanthogenate 0.2#
Pine Oil 0.15#

20 Minutes.

10 25 MIN. GRIND.

	% WT.	AU.	AG.	AU.	AG.
Heads	100.0	.15	27.9	100.0	100.0
Conc.	7.4	1.42	353.4	68.2	93.3
Midd.	8.2	.30	18.0	15.6	5.3
Tail.	84.4	.03	.48	16.2	1.4

PREAGITATION

Soda Ash 3.2#
Xylene & Petrolastic
Cement 1.2#
20 Heavy Fuel Oil 0.4

TREAT.

Xylene & Petrolastic
Cement 1.2#
Kerosene .3
Cresylic Acid .2

20 Minutes.

RETREAT.

Cresylic Acid 0.1#

PARK UTAH AG. ORE.

	% WT.	AU.	AG.	AU.	AG.
Heads	100.0	.05	20.7	100.0	100.0
Conc.	14.6	.24	122.2	71.3	85.9
Midd.	9.1	.03	17.0	5.5	7.5
Tails.	76.3	.015	1.8	23.2	6.6

TREATMENT

30 Potassium Xanthogenate into which
is incorporated 33% by volume of
pine oil before precipitation by CS₂. } 0.3#
10 Minutes.

RETREAT.

No reagents.
3 Minutes.

	% WT.	AU.	AG.	AU.	AG.
Heads	100.0	.054	21.0	100.0	100.0
Conc.	14.3	.22	116.3	58.0	79.3
Midd.	7.2	.10	40.7	13.2	14.0
Tails.	78.5	.02	1.8	28.8	6.7

Exhibit—W-15 (also Exhibit—K-2)

PARK UTAH AG. ORE (Continued)

TREATMENT			RETREAT.	
Soda Ash			4.0#	No reagents
Mexican Fuel Oil	30%		1.3#	4 Minutes.
Water White Solvent Naphtha	70%			
15 Minutes.				

PARK UTAH PB - ZN ORE.

	% WT.	AG.	PB.	ZN.	AG.	PB.	ZN.
10 Heads	100.0	5.39	8.24	12.55	100.0	100.0	100.0
Pb.	11.7	31.3	60.0	10.4	68.0	85.2	9.7
Zn. Conc.	29.3	4.2	1.7	32.0	22.8	6.1	75.3
Zn. Midd.	3.0	3.6	3.5	12.4	2.0	1.3	4.6
Tails	56.0	0.7	1.1	2.3	7.2	7.4	10.4

TREAT PB.		TREAT ZN.	
Potassium Xanthogenate	0.15#	Soda Ash	1.5#
Barrett No. 2	0.15#	CuSO ₄	0.5
10 Minutes.		Water Gas Tar	1.0
		Pine Oil	0.05
		15 Minutes.	

20 RETREAT ZN.
No reagents
5 Minutes

	% WT.	AG.	PB.	ZN.	AG.	PB.	ZN.
Heads	100.0	6.63	8.8	12.0	100.0	100.0	100.0
Pb. Conc.	11.9	27.8	55.2	10.2	50.0	77.8	10.2
Zn. Conc.	13.4	7.3	5.0	35.6	26.7	7.9	40.1
Zn. Midd.	18.3	4.5	2.6	25.7	12.3	5.7	39.7
Tail.	56.4	1.3	1.3	2.1	11.0	8.6	10.0

TREAT PB.		TREAT ZN.	
30 Soda Ash	4.0#	CuSO ₄	0.5#
Saturated solution	} 0.7	Water Gas Tar	1.2
Naphthaline in Xylene		Light Pine Oil	0.05
Cresylic Acid	0.1	15 Minutes.	
10 Minutes.			

RETREAT ZN.
No reagents
5 Minutes.

Exhibit—W-15 (also Exhibit—K-2)

DALY JUDGE N. & S. CO. Table Zn. Concts.

	% WT.	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.
Heads	100.0	17.68	4.56	39.0	7.7	100.0	100.0	100.0	100.0
Pb. Conc.	6.5	171.6	40.0	19.0	2.6	63.0	57.2	3.1	2.2
Zn. Conc.	67.6	7.7	2.1	53.0	5.6	29.4	31.0	91.8	49.2
Tails.	25.9	5.2	5.9	7.6	14.4	7.6	11.8	5.0	48.6

	TREAT PB.	TREAT ZN.
10	Potassium Xanthogenate 0.2#	CuSO ₄ 0:5#
	Coal Tar Creosote 0.1	Water Gas Tar 1.8
	10 Minutes	Steam Distilled Pine Oil 0.05
		10 Minutes.

	% WT.	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.
Heads	100.0	15.65	5.9	40.8	7.3	100.0	100.0	100.0	100.0
Pb. Conc.	23.1	24.4	17.5	11.5	21.0	36.0	68.5	6.5	66.9
Zn. Conc.	62.0	15.16	2.6	59.2	2.5	60.0	27.4	89.9	21.4
Tails.	14.9	4.14	1.6	9.5	5.7	4.0	4.1	3.6	11.7

	TREAT PB.	TREAT ZN.
20	Na ₂ S 5.0#	Soda Ash 1.0#
	Mexican Fuel Oil 30% } 1.0#	CuSO ₄ 1.0
	Water White So. } 1.0#	Water Gas Tar 2.0
	Naphtha 70% } 0.1#	Heavy Naphtha 0.2
	Cresylic Acid 0.1#	Light Pine Oil 0.1
	12 Minutes.	10 Minutes.

CASH MINE ORE.

	% WT.	AU.	AG.	CU.	PB.	ZN.	FE.	AU.	AG.	CU.	PB.	ZN.	FE.
Heads	100.0	.129	8.3	1.87	9.85	11.3	10.7	100.0	100.0	100.0	100.0	100.0	100.0
Conc. Pb.	15.0	.50	39.5	6.96	54.0	9.6	8.6	58.2	71.4	56.0	82.4	12.7	12.0
Conc. Fe.	3.1	.84	6.7	1.0	5.0	10.4	36.8	20.2	2.5	1.7	1.5	2.8	10.6
Conc. An.	21.2	.06	5.4	2.32	3.2	36.0	18.4	9.9	13.7	26.3	6.9	67.5	36.4
Tails.	60.7	.025	1.7	0.5	1.5	3.2	7.2	11.7	12.4	16.0	9.2	17.0	41.0

	TREAT PB.	TREAT ZN.
30	Potassium Xanthogenate 0.1#	CuSO ₄ 0.7#
	Coal Tar Creosote 0.4	Water Gas Tar 0.9
	10 Minutes.	Steam Distilled Pine Oil .05

Zn. Conc. tabled for Fe. Conc.

Exhibit—W-15 (also Exhibit—K-2)

CASH MINE ORE (Continued)

	% WT.	AU.	AG.	CU.	PB.	ZN.	FE.	AU.	AG.	CU.	PB.	ZN.	FE.
Heads	100.0	.086	8.91	2.05	11.02	11.4	11.3	100.0	100.0	100.0	100.0	100.0	100.0
Pb. Conc.	19.6	.14	28.9	5.44	46.4	17.2	6.6	31.8	63.6	52.3	81.8	29.6	11.5
Zn. Conc.	16.5	.26	8.8	3.52	6.5	38.0	12.0	49.7	16.3	28.3	9.6	55.2	17.5
Tails.	63.9	.025	2.8	.62	1.5	2.7	12.6	18.5	20.1	19.4	8.6	15.2	71.0

TREAT PB.

	Soda Ash	2.0#	CuSO ₄	0.7#
	Tetralene	0.6#	Water Gas Tar	0.6
10	Cresylic Acid	0.05	Light Pine Oil	0.1
	10 Minutes.		15 Minutes.	

TREAT ZN.

This ore spotty as to gold values.

SAN FRANCISCO DEL ORO

	% WT.	AG.	PB.	ZN.	AG.	PB.	ZN.
Heads	100.0	16.5	9.4	16.1	100.0	100.0	100.0
Pb. Conc.	12.2	67.0	64.0	12.0	49.4	83.2	9.1
Zn. Conc.	28.9	23.6	3.6	43.4	41.2	11.1	78.0
Tails.	58.9	4.5	1.5	6.0	9.4	5.7	12.9

TREAT. PB.

20	Potassium Xanthogenate	0.15#	CuSO ₄	0.2#
	Coal Tar Creosote	0.15	Water Gas Tar	1.2
	10 Minutes.		Steam Distilled Pine Oil	0.05#
			15 Minutes.	

TREAT. ZN.

	% WT.	AG.	PB.	ZN.	AG.	PB.	ZN.
Heads	100.0	17.5	8.1	17.4	100.0	100.0	100.0
Pb. Conc.	13.4	50.0	49.0	14.8	38.2	81.1	11.4
Zn. Conc.	27.0	30.6	3.02	45.2	47.2	10.0	70.2
Zn. Midd.	2.5	15.5	4.11	14.0	2.2	1.3	2.0
Zn. Tails.	57.1	3.8	1.08	5.0	12.4	7.6	16.4

30 TREAT. PB.

	Soda Ash	2.0#	Heavy Naphtha	0.8#
	Saturated sol.	} 1.0	15 Minutes.	
	Naphthalene in Xylene			
	15 Minutes.			

RETREAT. ZN.

No reagents.

8 Minutes.

Assay shows this sample to contain 1.85% of Pb. and 3.1% of Zn. as non-sulfides.

40 Silver recovery increased in cases where Zn. content of Pb. Conc. was raised above that shown in 2nd test.

Exhibit—W-15 (also Exhibit—K-2)

SULLIVAN ORE.

	% WT.	AG.	PB.	ZN.	FE.	PB.	ZN.	FE.
Heads	100.0	12.06	15.3	30.6	100.0	100.0	100.0
Pb. Conc.	15.0	13.1	60.4	9.2	8.2	75.2	9.0	4.0
Pb. Midd.	5.3	3.6	15.6	12.0	35.5	6.8	4.2	6.0
Zn. Conc.	29.3	2.1	6.0	43.0	13.3	14.4	82.4	12.6
Zn. Midd.	4.6	1.1	3.6	6.4	45.2	1.4	2.0	6.8
Tails.	45.8	0.4	.6	.8	47.2	2.2	2.4	70.6

TREAT. PB.		
10	Potassium Xanthogenate	0.1#
	Coal Tar Creosote	0.1
	7 Minutes.	

RETREAT PB.	
	No reagents.
	5 Minutes.

TREAT. ZN.		
	Soda Ash	2.0#
	CuSO ₄	0.5
	Water Gas Tar	1.3
	15 Minutes.	

RETREAT. ZN.	
	No reagents.
	5 Minutes.

No comparative test on this sample of ore. However, previous samples from the same mine gave results of the following order:—

	% WT.	PB.	ZN.	FE.	PB.	ZN.	FE.	
20	Heads	100.0	9.2	15.1	33.2	100.0	100.0	100.0
	Pb. Conc.	10.5	61.2	12.6	5.6	69.9	8.9	1.7
	Pb. Midd.	3.2	26.6	18.0	21.7	9.3	3.5	2.0
	Zn. Conc.	27.5	5.4	42.0	15.1	16.1	76.4	12.5
	Zn. Midd.	14.7	2.4	10.5	46.4	3.8	10.1	20.5
	Tails.	44.1	0.2	.4	47.7	.9	1.1	63.3

TREAT. PB.			RETREAT. PB.		
	Soda Ash	10.0#		Light Pine Oil	0.05#
	Heavy Naphtha	0.6		7 Minutes.	
30	Saturated solution	} 0.5#			
	Naphthalene in Xylene				
	10 Minutes.				

TREAT. ZN.			RETREAT. ZN.	
	Kerosene	0.7#		No reagents.
	Heavy Naphtha	1.0		8 Minutes.
	Saturated Solution	} .4		
	Naphthalene in Xylene			
	18 Minutes.			

Exhibit—W-15 (also Exhibit—K-2)

NORTH STAR MINE (FEDERAL M. & S. CO.)

	% WT.	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.
Heads	100.0	10.24	7.2	20.8	11.0	100.0	100.0	100.0	100.0
Pb. Conc.	4.85	71.4	55.4	10.0	8.9	34.0	37.4	2.4	3.9
Pb. Midd.	2.67	21.0	16.8	17.2	10.7	5.5	6.2	2.2	2.6
Zn. Conc.	55.5	9.0	5.4	34.0	11.6	48.7	41.7	90.7	58.5
Zn. Midd.	10.8	4.2	3.5	4.6	12.5	4.4	5.3	2.4	12.3
Tails.	26.18	2.9	2.6	1.8	9.5	7.4	9.4	2.3	22.7

10 TREAT. PB.
Potassium Xanthogenate 0.25#
Coal Tar Creosote 0.2
10 Minutes.

RETREAT PB.
No reagents.
5 Minutes.

TREAT. ZN.
CuSO₄ 0.5#
Water Gas Tar 1.5
15 Minutes.

RETREAT. ZN.
No reagents.
5 Minutes.

Best previous test on this material showed the following assays:—

	% WT.	AG.	PB.	ZN.	FE.
Heads	100.0	11.0	7.9	19.5	12.8
20 Pb. Conc.	31.0	13.0	6.8	13.6	10.5
Zn. Conc.	45.0	11.6	5.8	26.0	11.8
Tails.	24.0	5.8	3.75	7.2	

TREAT. PB.

95 parts Barrett No. 4
5 " Steam Distilled Pine Oil
5 " flowers of sulphur distilled at 235° F.

} 0.5#

TREAT. ZN.

30 CuSO₄ 1.0#
Coal Tar Creosote 1.0
Heavy Naphtha 0.5
15 Minutes

EXHIBIT W-14

MINERALS SEPARATION NORTH AMERICA CORPORATION
OFFICE OF THE CHIEF ENGINEER

220 BATTERY STREET, SAN FRANCISCO, CAL.

Confidential
CC NY

San Francisco, Calif.,
March 30, 1923.

EHNm

Minerals Corporation N. A. Corporation,
61 Broadway,
10 New York.

Gentlemen:

Herewith I am sending you a report to me signed by Messrs. Keller and Lewis covering work which they have been doing in the laboratory for some months. Their report sets forth with reasonable clearness the objects aimed at, the results obtained, and the advantages derived from the use of the new reagent which they have developed. It remains, perhaps, for me to summarize the matter to some extent and point out certain salient features of the possibilities pertaining to the use of this new reagent.

20 In general it can be said that it shortens the time factor in the treatment of the ores to which it is applicable, as excellent coagulation or "clean-up" is obtained quickly when using this reagent, and the froth obtained has a distinctly superior appearance to that ordinarily obtained. All things considered, better results have been had with it on a number of important ores than we have been able to get in any other way. This is notably true with the Anaconda slime, Park Utah silver, and silver-lead-zinc ores, the Daly Judge table zinc concentrates, Cash Mine ore, the San Francisco Del Oro ore, and the Sullivan Mine ore. We have also obtained a result which is
30 very nearly a commercial result, if not quite so, on the North Star Mine ore which has hitherto been the unsolvable ore of the Coeur d'Alene, and it is an important mine. We are waiting for the further samples of Utah Copper ore to try its application to that, and we will doubtless find an increasingly wider application for this reagent as we make more and more tests with it on different ores.

One of its important effects is to put into the lead concentrate the precious metals which accompany an ore, in a larger measure than anything we have so far tried. This, of course, is quite important. In general it appears to be a cheaper and a more efficient
40 reagent than the alphabetical reagents of Callow, though it is yet to be demonstrated how it compares therewith in the broadness of its application, and it can be made in unlimited quantities, which is not true of the alphabetical reagents.

Exhibit--W-14.

Our search of the patent files which we have here does not indicate that it has so far been covered in any way by any of the claims of similar patents. I think, therefore, by all means it should be made either the subject for a patent application, or else we might well consider keeping it a secret reagent and manufacturing and selling it ourselves, as it lends itself to such exploitation.

While xanthogenate itself could of course be identified if it was furnished as such, it would probably defy detection if mixed in with an oily flotation reagent and sold in that way. Thus, by making
10 a paste of it with pine oil or other oily reagent, depending upon the treatment characteristics of the ore, and furnishing it to our licensees, we could probably keep the composition of it to ourselves indefinitely. The disadvantage, however, from the standpoint of income in doing this, assuming that it came into general use, is this: It is effective in such small quantity that we would have to charge a pretty stiff price for it in order for the income therefrom to approach the royalty rate on ores, and of course, such procedure would probably not be a very popular one with our licensees. It may be the wiser course, therefore, to patent it, instead of exploiting it as a secret preparation,
20 although I must confess that the latter procedure has attractive possibilities. In any event we should take steps to patent it, or exploit it, shortly, so that our important licensees can begin the use of it during the present year.

It might be useful later, when we are ready to let our licensees have this information or furnish them the reagents themselves, to pass on the San Francisco Del Oro results to the well known metallurgical firm of Knox & Allen for contemplation. Should they re-act with an inquiry as to how we did it we would of course want them to settle for the past and take out a license for the future before
30 disclosing anything to them. It might develop that we could clear up that situation in this way.

If the new reagent is patented Messrs. Williams & Pritchard will doubtless have a good deal of further work they would want done on it, so we will await your further word.

Yours very truly,

(Sgd.) EDWARD H. NUTTER
Chief Engineer.

EXHIBIT W-16

Received May 1, 1923

WILLIAMS & PRITCHARD
COUNSELLORS AT LAW, PATENT ATTORNEYS
61 BROADWAY, ADAMS BUILDING
NEW YORK

Apr. 30, 1923

Minerals Separation North American Corporation,
61 Broadway, New York City.

10 Dear Sirs:—

Keller and Lewis, Xanthate Flotation Agent

In order to be on safe ground in drafting the patent specification and claims for this invention it will be necessary to be absolutely certain whether or not potassium xanthate is a frothing agent. If it is not a frothing agent we should know whether it has an effect parallel to some of the known modifying agents. For example, is its effect parallel to that of silicate of soda or to some one of the Kotrix compositions? If it is not parallel to either of these, we ought to know what substance it most nearly resembles in its action in a
20 pulp. Perhaps you might obtain further light on the action of xanthate in pulps if its effect were tried upon a coal pulp which was being concentrated by the froth flotation process. If you approve of this suggestion, we shall be pleased to have the data even if the result is wholly negative.

We enclose a copy of this letter, which you may find convenient to forward to Mr. Nutter.

Faithfully yours,

WILLIAMS & PRITCHARD

ET

30 1 enc.

EXHIBIT K-3

May 3rd, 1923.
CPL-CHK

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.
220 Battery St., San Francisco, Calif.

Dear Sir,

Supplementing our report of March 28th on Xanthogenates, we have to report additional tests made with sodium and potassium
10 Xanthogenates as well as various oil Xanthate mixtures.

Tests 1, 2 and 3 were made on Anaconda slime using Sodium Xanthogenate and the frothing agents as noted. Test 3 was made in hot circuit showed the expected increase in rapidity of float with approximately the same recovery in a shorter time with a reduction in the reagent consumption.

Test 4 was made to confirm our previous test on San Francisco del Oro ore using Sodium Xanthogenate rather than the potassium salt.

Test 5 on Cash mine ore using a Xanthate oil mixture shows the possibilities of complex ores treated by this method where the proper
20 oil has been incorporated with the Xanthate. You will note in this case a recovery of 95% of the lead, 85% of the copper and 87% of the silver in a product carrying only 5% of zinc. It is to be expected that in most ores of this type each ore should have its own reagent combination worked out to give the best possible separation.

Tests 6, 7, 8, 9, 10 and 11 were made on Utah Copper Ore using the reagent combinations noted in each case and are included to bring out the possibility of substituting resin for pine or other frothing oils in combination with the Xanthogenates.

Tests 12 and 13 on ore from the Silver King Coalition Mine
30 are cited to show the characteristic increase in silver recovery in the lead product where Xanthogenate was used as compared to the usual method and reagent combination on this class of ore.

Test 14 was made on the North Star Mine ore in confirmation of the test reported on the 25th. In this case the rougher concentrates were not retreated for grade but the test shows a separation checking the previous test on this ore.

Tests 15 and 16 on Barron Mine ore again show the increase in lead and silver recoveries in the lead product when treated with Xanthogenate as compared to the usual oil treatment for such ores.

40 A further series of tests on Anaconda Copper ores under all conditions are in hand and will be reported when completed.

Very truly yours,

Exhibit—K-3.

ANACONDA SLIME.

		% WT.	CU.	CU.
	Heads	100.0	2.93	100.0
	Conc.	17.5	15.44	92.3
	Midd.	17.8	.80	4.9
	Tails.	64.7	.13	2.8
		TREAT.		RETREAT.
		Sodium Xanthogenate	0.7	No reagents.
10		G.N.S. No. 5	0.25	3 Minutes.
		12 Minutes.		
		% WT.	CU.	CU.
	Heads	100.0	2.89	100.0
	Conc.	20.3	13.20	92.7
	Midd.	21.5	.60	4.5
	Tails	58.2	.14	2.8
		TREAT.		RETREAT.
		Sodium Xanthogenate	0.6	No reagents.
		Sludge	0.8	4 Minutes.
20		Hardwood Creosote	0.2	
		13 Minutes.		
		% WT.	CU.	CU.
	Heads	100.0	2.91	100.0
	Conc.	18.1	14.56	90.6
	Midd.	17.3	.84	5.0
	Tails	64.6	.20	4.4
		TREAT.		RETREAT.
		Pulp temp. 45° C.		No reagents.
		Sodium Xanthogenate	0.5	3 Minutes.
30		G.N.S. No. 5	0.2	
		10 Minutes.		
		Tails looked clean in 6 Minutes.		

SAN FRANCISCO DEL ORO.

	% WT.	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.
Heads	100.0	17.5	9.9	17.1	5.4	100.0	100.0	100.0	100.0
Conc. Pb.	14.2	61.8	56.0	16.6	3.2	50.2	80.2	13.8	8.4
Zn. Conc.	28.5	22.0	4.3	42.0	9.7	35.8	12.2	70.4	51.3
Tails.	57.3	4.3	1.3	4.7	3.8	14.0	7.6	15.8	40.3

Exhibit—K-3.

SAN FRANCISCO DEL ORO. (Continued)

TREAT PB.		TREAT ZN.	
Sodium Xanthogenate	0.15	CuSO ₄	0.3
Barrett No. 2	0.15	Barrett No. 4	1.3
5 Minutes.		G.N.S. No. 5	0.1
		10 Minutes.	

CASH MINE ORE.

	% WT.	AU.	AG.	CU.	PB.	ZN.	FE.	AU.	AG.	CU.	PB.	ZN.	FE.
Heads	100.0	.154	10.7	3.18	14.9	7.7	11.9	100.0	100.0	100.0	100.0	100.0	100.0
Conc. Pb.	38.0	.24	24.6	7.12	37.4	5.0	21.4	59.4	87.1	85.2	95.3	24.7	60.4
Zn. Conc.	15.4	.36	6.3	2.56	3.0	35.2	12.0	36.0	9.0	11.0	3.1	70.5	15.5
Tails.	46.6	.015	0.9	.26	0.5	0.8	4.2	4.6	3.9	3.8	1.6	4.8	16.4

10	TREAT PB.	TREAT ZN.
	Potassium Xanthate with 10% of Saturated solution of Naphthalene in Xylene	CuSO ₄
	10 Minutes.	Barrett No. 4
		G.N.S. No. 5
		10 Minutes.
	.3	0.2
		1.0
		0.1

UTAH COPPER ORE.

	% WT.	CU.	CU.
Heads	100.0	1.02	100.0
Conc.	6.5	12.80	81.7
Tail.	93.5	.20	18.3

20	TREAT.	
	Potassium Xanthogenate with 3% of Rosin	} 0.3
	G.N.S. No. 5	
	10 Minutes	} 0.05

	% WT.	CU.	CU.
Heads	100.0	1.08	100.0
Conc.	6.2	14.08	80.9
Tail.	93.8	.22	19.1

30	TREAT.	
	Potassium Xanthogenate with 25% of Rosin	} 0.3
	10 Minutes.	

	% WT.	CU.	CU.
Heads	100.0	1.04	100.0
Conc.	8.1	10.80	84.0
Tail.	91.9	.18	16.0

Exhibit—K-3.

UTAH COPPER ORE. (Continued)

TREAT.

Potassium Xanthogenate	}	0.3
with 3% of Rosin		
G.N.S. No. 5		0.05

12 Minutes.

	% Wt.	Cu.	Cu.
10	Heads 100.0	1.04	100.0
	Conc. 8.0	10.72	82.4
	Tail. 92.0	.20	17.6

TREAT.

Potassium Xanthogenate	}	0.3
with 5% of Rosin		
G.N.S. No. 5		0.05

10 Minutes.

	% Wt.	Cu.	Cu.
20	Heads 100.0	.96	100.0
	Conc. 5.2	13.75	74.7
	Midd. 15.8	.74	12.2
	Tail. 79.0	.16	13.1

TREAT.

Potassium Xanthogenate	0.4
Coal Tar (Barrett)	1.5
G.H.S. No. 5	0.3

20 Minutes.

RETREAT.

No reagents.
5 Minutes.

	% Wt.	Cu.	Cu.
30	Heads 100.0	1.05	100.0
	Conc. 5.7	14.96	80.8
	Midd. 10.7	.88	8.9
	Tail. 83.6	.13	10.3

TREAT.

Potassium Xanthogenate	}	0.6
with 33% pine oil		
Int. C. Prod. Co.		0.7

12 Minutes.

RETREAT.

No reagents.
4 Minutes.

Exhibit—K-3.

SILVER KING COALITION MINE ORE.

	% WT.	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.
Heads	100.0	9.11	3.9	21.6	13.3	100.0	100.0	100.0	100.0
Pb. Conc.	27.4	30.0	9.6	10.0	32.7	90.2	91.1	12.6	67.3
Zn. Conc.	34.4	1.8	0.4	53.2	6.2	6.8	4.8	84.6	16.1
Zn. Midd.	2.7	2.3	0.5	7.6	16.2	.7	.3	1.0	3.2
Tailings	35.5	0.6	0.3	1.1	5.0	2.3	3.8	1.8	13.4

	TREAT PB.	TREAT ZN.	TREAT ZN.
	Potassium Xanthogenate	0.2	CuSO ₄ 0.5
10	Barrett No. 2	0.2	Barrett No. 4 1.3
	Hunter Johnson 206	0.1	G.N.S. No. 5 0.1
	10 Minutes.		5 Minutes
			10 Minutes.

	% WT.	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.
Heads	100.0	7.73	2.7	20.3	11.3	100.0	100.0	100.0	100.0
Pb. Conc.	24.2	23.6	9.0	7.0	34.4	73.9	88.2	8.3	73.6
Zn. Conc.	34.5	5.1	1.4	52.6	5.7	22.3	17.6	89.1	17.3
Tails.	41.3	0.7	.15	1.3	2.5	3.8	2.2	2.6	9.1

	TREAT PB.	TREAT. ZN.
20	H ₂ S	Barrett No. 4 1.0
	H ₂ SO ₄	G.N.S. No. 5 0.1
	Barrett No. 4	10 Minutes.
	Barrett No. 2	
	10 Minutes.	

NORTH STAR MINE (FEDERAL N. & S. CO.)

	% WT.	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.
Heads	100.0	10.1	7.9	18.4	14.8	100.0	100.0	100.0	100.0
Pb. Conc.	13.7	37.4	31.2	10.8	13.2	50.7	54.9	8.0	12.2
Zn. Conc.	57.7	7.9	5.0	27.0	17.0	45.0	36.9	84.9	66.2
Tails.	28.6	1.5	2.5	4.6	11.2	4.3	8.2	7.1	21.7

	TREAT PB.	TREAT ZN.
30	Potassium Xanthogenate with	CuSO ₄ 0.7
	10% of Barrett No. 2 Creosote	Soda Ash 1.5
	Barrett No. 2	Barrett No. 4 1.4
	10 Minutes.	15 Minutes.

BARRON MINE ORE.

	% WT.	AU.	AG.	PB.	ZN.	FE.	AU.	AG.	PB.	ZN.	FE.
Heads	100.0	2.47	157.1	7.2	7.2	3.5	100.0	100.0	100.0	100.0	100.0
Pb. Conc.	15.8	9.0	925.0	44.0	12.4	8.5	57.5	92.9	96.4	27.1	38.
Zn. Conc.	14.8	3.08	41.0	0.8	33.6	7.0	18.4	3.9	1.7	69.4	29.
Au. Conc.	1.1	46.16	70.0	20.5	.5
Tails.	68.3	.13	6.2	0.2	0.4	1.7	3.6	2.7	1.9	3.5	32.

Exhibit—K-3.

BARRON MINE ORE (*Continued*)

TREAT PB.		TREAT ZN.	
Potassium Xanthogenate	0.2	Soda Ash	1.5
Barrett No. 2	0.2	CuSO ₄	0.5
5 Minutes.		Barrett No. 4	1.1
		G.H.S. No. 5	0.1
		10 Minutes.	

Zinc. Con. tabled for An.

	% Wt.	AU.	AG.	PB.	ZN.	FE.	AU.	AG.	PB.	ZN.	FE.
Heads	100.0	1.57	165.1	6.2	7.5	3.8	100.0	100.0	100.0	100.0	100.0
Pb. Conc.	10.2	6.36	1312.0	51.0	8.4	2.4	41.4	81.2	84.2	11.4	6.4
Zn. Conc.	16.4	4.72	104.0	4.0	37.6	7.8	49.3	10.1	10.5	82.6	33.5
Fe. Conc.	5.6	1.16	17.1	2.4	3.2	21.6	4.3	5.8	2.1	2.4	31.6
Tails.	67.8	.13	7.1	.3	0.4	1.6	5.1	2.9	3.2	3.6	38.5

TREAT. PB.		TREAT. ZN.		TREAT FE.	
Soda Ash	2.0	CuSO ₄	0.5	Na ₂ S	3.0
Barrett No. 2	0.8	Barrett No. 4	1.0	Int. C. Prod. Co.	0.3
Hunter Johnson		G.N.S. No. 5	0.1	G.N.S. No. 5	0.1
206	0.15				
10 Minutes.		12 Minutes.		5 Minutes.	

20

San Francisco, Calif.,
May 3, 1923.
EHNm

Minerals Separation N. A. Corporation,
61 Broadway,
New York.

Gentlemen:

Herewith is supplementary report by Messrs. Keller and Lewis on further tests with the Xanthogenates on sundry ores, together with the details of the tests.

30 This report covers work which was done chiefly with the view of determining the effects of reagents made up of mixtures of Xanthogenates and different oils, although other tests are included.

We are finding that there is practically no difference in the action of the sodium as compared with the potassium Xanthogenates, and the sodium Xanthogenate will probably be cheaper to prepare.

More recent tests on Utah Copper ore show improved results over those reported herewith, but they will be included in later reports.

I am sending an extra copy of this report for our patent agents, as it doubtless has matter therein in which they will be interested.

Very truly yours,

Chief Engineer.

40

enc 2

Exhibit—K-26.

EXHIBIT K-26

San Francisco, Calif.,
May 7, 1923.
EHNm

Minerals Separation N. A. Corporation,
61 Broadway,
New York.

Gentlemen:

I have your letter of the 1st enclosing copy of a letter from
10 Messrs. Williams & Pritchard in regard to the Xanthates as flotation
agents. Messrs. Keller and Lewis have read this over, and in reply
thereto state:

We have found potassium and sodium Xanthate to have
only very slight frothing qualities. The froth produced is
similar to that made with soda ash. However, it was found
that the quantity of frothing oil required was materially less
when used with Xanthates than without on the same ore."

Soda ash makes a slight scum when used alone without frothing
reagents. The Xanthates appear to work well with most of the
20 ordinary frothing oils, and good results are obtained with almost
any of them, though of course some are better than others. In brief,
it appears to be a reagent which when added in small quantity to
an ore pulp, makes the valuable minerals therein amenable to the
action of almost any of the ordinarily used flotation reagents without
the trickiness of action which is characteristic of so many of them
in the treatment of rather difficult ores.

So far the two best tests we have made on the Anaconda slime
with the Xanthates is one using hardwood creosote, such as the Ana-
conda Company use, wherein 94.2% recovery was made in a con-
30 centrate assaying 14.72% copper, together with a small low grade
middling, and a tailing assaying .12% copper. The other test was
using Xanthate and kerosene acid sludge and in it we made a con-
centrate assaying 13.52% copper, with a small middling, and with a
.12% tailing, and 92.2% recovery in the concentrate. The con-
ditions of this last test were repeated, except that we used a spitz
machine instead of a subaeration machine, and we got therewith
a .15% tailing. These tests were made at ordinary temperature in
neutral circuit, in fifteen minutes.

The Xanthate appears to be of broad scope and application
40 with all of the frothing oils, and makes an ore amenable to almost
any of them, so that a frothing oil can be chosen which gives the best
conditions for either machine.

Exhibit—K-26.

Exhibit—K-17.

We have so far used the Xanthate with various pine oils, with sludge, and with hardwood creosote, alone and in combination, with resin in combination, with coal tar products, and with fuel oil and turpentine, and it works well with all of them. We will extend this list as fast as we can.

Very truly yours,

Chief Engineer.

EXHIBIT K-17

MINERALS SEPARATION NORTH AMERICAN CORPORATION

10

OFFICE OF THE CHIEF ENGINEER

220 BATTERY STREET, SAN FRANCISCO, CAL.

San Francisco, Calif., May 7, 1923.

E. H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A., Corporation,
220 Battery St., San Francisco, Calif.

Dear Sir:

In order to strengthen our position regarding soluble sulfidizing reagents, I beg to point out to you the advisability of taking measures at once for the protection of Thiocarbonic Acid H_2CS_3 and some of its
20 salts such as—

Sodium Sulfocarbonate	Na_2CS_3
Potassium " "	K_2CS_2

Indicative tests made with these materials do not show as strong an action as soluble organic sulfidizing reagents, yet their action for differential work stamps them with being useful, moreover their low raw material cost, and ease and mode of preparation might well be used to our disadvantage by a competitor.

As soon as the pressure of work in hand subsides, systematic work ought to be carried out with these reagents. As far as I am
30 aware no patent has been taken out on them, nor are they covered by Mr. Martin's patent 1236856.

Incidentally, I may add that it may be well to investigate the action of alkali di-Thionates such as $Na_2S_2O_6$, Tri-Thionates $K_2S_3O_6$, Tetra-Thionates $K_2S_4O_6$ and Penta-Thionates $K_2S_5O_6$, which by reason of their chemical structure appear pregnant with possibilities.

Yours very truly,

(Sgd.) CORNELIUS H. KELLER

Exhibit—D-62.

EXHIBIT D-62

San Francisco, Calif.,
May 10, 1923.

Memorandum for Mr. E. H. Nutter:

The samples of sodium Xanthates labeled "A" and "B", were prepared as follows: Sample "A" was separated from the alcoholic solution by refrigeration, filtered off, and then fused at a temperature just slightly above its melting point, and kept at that temperature until freed from its alcohol. It was then allowed to solidify and was
10 subsequently pulverized. Sample "B" was obtained by taking the residual alcoholic solution, filtered off from the crystals of Sample "A", putting it into a still and distilling the crystals of Sample "A", putting it into a still and distilling the alcohol until the solid residue had left in it only a small amount. This solid residue was then carefully fused, poured out of the still and put into a hot closet, where it was kept just above its melting point until the last trace of alcohol was gone.

If the observations are correct that sample "B" did not work the same as Sample "A", it would mean that in the distillation of
20 the alcohol at ordinary pressure the temperature that is reached as the solution concentrates is sufficient to cause decomposition of the sodium xanthate, and this may be due to a combination of an increase in temperature, together with a small amount of water which is necessarily present as the result of the reaction. To prevent decomposition, it may be necessary to recover the alcohol by distilling at a lower temperature and under reduced pressure, or to use some other means of alcoholic recovery which will not result in decomposition of the sodium xanthates.

Further samples will be submitted.

30

Dr. Rosenstein

Exhibit—K-4.

EXHIBIT K-4

MINERALS SEPARATION NORTH AMERICAN CORPORATION
OFFICE OF THE CHIEF ENGINEER
220 BATTERY STREET, SAN FRANCISCO, CAL.

San Francisco, Calif.,
May 11, 1923.
CHK-CPLm

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corporation,
10 220 Battery Street,
San Francisco, Cal.

Dear Sir:

Re Anaconda Copper Mining Co.—Xanthogenates

The Xanthogenate or Xanthate used in the subjoined tests on Anaconda slimes and sands is an alkali salt of Ethyl-Oxydithiocarbonic acid. It is easily prepared by mixing an alcoholic (denatured) solution of an alkali hydrate such as potassium hydrate or sodium hydrate with carbondisulfide.

20 These ingredients can be obtained in unlimited quantities at an approximate cost of 10 cents per pound, in large scale operations, for the final reagent. As the manufacture does not entail expensive machinery nor difficult manipulation, cost should be low. Any excess of alcohol required for the solution of the alkali hydrate and not entering into chemical combination, can be recovered during manufacture.

30 Potassium and sodium Xanthogenate in the pure state are yellowish crystalline substances which turn darker when exposed to light and air without decomposing. They are non-inflammable, non-deliquescent, have an aromatic odor, are neutral in reaction, soluble in all portions in both cold and hot water, soluble to a less degree in alcohol (20), and slightly soluble in some oils. Their concentrated water solution decomposes slightly at 50°C. without seriously impairing their action as a flotation modifying agent. However, under special climatic conditions it may be advisable to feed the reagent to the ore pulp in solid form. There is no apparent difference in the effect of the Xanthates being fed to the ore pulp as a dry salt or solution respectively, so that the question of mode of use will depend on local conditions. However, it would appear to be most advantageously used in a concentrated aqueous solution to insure a uniform
40 feed at all times.

Exhibit—K-4.

The tests reported in ours of the 3rd, specifying pine and other oils, or resin, incorporated with the Xanthates, were made with the object of demonstrating the possibility of making mixtures of this type without impairing the action of the reagent. The method of manufacture of these mixtures differs only in that the oil or resin is either incorporated with the alcoholic hydrate solution or the carbondisulfide before mixing, to effect the precipitation of the final salt. The choice of solvent for the oil or resin depends on the solubility of the oils or resin in either the alcoholic hydrate solution or the carbondisulfide. Our tests have shown no apparent difference in action between the sodium and the potassium Xanthate on the Anaconda ores. The use of either will be dependent on the cost rather than any difference in metallurgical results, as shown by the subjoined tests.

The Xanthogenates or Xanthates are primarily modifying agents, although their use does result in a slight frothing effect. The physical effect of their use on Anaconda ores is a brightening of the sulphides and the increase in flocculation of the mineral after the addition of oils. This flocculation is apparently not dependent on the kind of oil used and makes it possible to employ practically any type of oil desired for the proper frothing effect.

Tests were made to determine the exact amount of Xanthogenate required on the sands and slimes, and also to note the effect of adding a large excess of this reagent. In the case of the sands it was found that .15 lbs. gave the desired effect and no apparent difference was noted when this quantity was increased to 2.0 lbs. per ton. The slimes showed a slightly higher rate of consumption; the initial effect became apparent with .2 lbs., with no advantage to be gained by any great excess over .3 or .4 lbs. per ton. In testing these ores an arbitrary amount of .5 to .7 lbs., was used on the slimes, and .3 to .5 lbs. on the sands to be sure that sufficient of the reagent was used. It is quite probable that their use in practice will show a maximum requirement nearer the smaller figure quoted in each case.

The brightening and flocculating effect is instantaneous, requiring no preagitation, and does not diminish with prolonged agitation of the pulp so treated.

The Xanthates were found to be effective in acid, alkaline, and neutral circuits. No advantage was gained by the use of acid or alkali. Their use did not apparently affect the action of the Xanthates, except for the expected deflocculation by the acid making for higher oil consumption, longer treatment period, and high soluble copper loss in tailing filtrate.

The oily reagents employed with Xanthates will in a large measure be dependent on the type of froth desired to meet the type of machine and operating conditions. The attached test show a slight variation in recoveries with different combinations of oils. The

Exhibit—K-4.

following types of oils in the order named appear to be most effective disregarding the quality and type of froth produced by the oils:—

1. Tar acids
2. High acid coal tar creosote
3. Kerosene acid sludge
4. Hardwood creosotes
5. Neutral coal tar creosotes
6. Pine Oils
7. Coal tar
- 10 8. Petroleum oils.

The choice of oils or oily reagents will of course be dependent on the balance between grade of concentrate and recovery, as well as the character of froth desired, and reagent cost. From our tests, the most promising reagents for use at Anaconda in the spitz type machines would appear to be either the high acid coal tar creosotes, sludge, hardwood creosotes, or crude pine products. The oily reagent consumption in any case would be materially lower than at present, by reason of the neutral circuit and the use of Xanthates. The mineral is in a more readily floated condition, readily flocculated,
20 and rapidly floated in a very clean product.

Heating the pulp has the effect of increasing the rapidity of float, increasing the intensity of flocculation of the mineral, and aiding in emulsification of the oily reagents. However, the advantage to be gained is apparently not sufficient to warrant the extra cost.

Our tests have shown the time of treatment necessary to effect a high recovery on Anaconda ores by use of the Xanthates to be less by approximately one half of the time required in their present practice.

The slime sample tested showed an appreciable amount of over-
30 size material that on examination was found to have some unfreed sulfides. The following screen test on tailings from Test No. 7B bears out the fact that a complete recovery could not be effected without regrinding of the oversize material:—

	Mesh	% Weight	% Cu.
	100.0	.21
	+65	2.0	.45
	+80	2.3	.42
	+100	2.3	.24
	+150	3.7	.18
40	+200	16.7	.13
	-200	73.0	.22

Exhibit—K-4.

Similarly a screen test on the reground table tailings (Test No. 3-4) showed the +65 mesh material to have the highest copper content:

	Mesh	% Weight	% Cu.
		100.0	.09
	+65	1.3	.20
	+80	3.3	.08
	+100	6.6	.06
	+150	13.2	.08
10	+200	30.6	.06
	-200	45.0	.12

Table 1, covering our tests on Anaconda slimes under a variety of conditions, oil combinations and machines, does not show a wide variation in the metallurgical results where the Xanthates were employed, except in the case of Test No. 9-H where an acid circuit was used, showing a loss of soluble copper of .40%.

Table II, covers tests made on sand products in confirmation of our tests on slimes, and shows on the whole results similar to them. The test made on the D. C. O. do not equal those on the reground
20 Table Tailings, possibly due to the fact that they were dried and have been on hand for over a year.

Briefly our tests, using Xanthogenates of sodium and potassium, have shown the following advantages to be gained by their use on Anaconda ores:—

1. Low reagent cost.
2. Neutral circuit with no loss of soluble copper.
3. Cheaper mill upkeep costs with substitution of iron for bronze parts in flotation units.
4. Shortened time of treatment with greater capacity in present
30 installation.
5. Adaptability to machines as installed.
6. Minimum of frothing oil required.
7. Unlimited number of oil combinations possible.
8. Unlimited supply of reagents that can be made locally.

Very truly yours,

CORNELIUS H. KELLER. C. P. LEWIS.

ANACONDA SLIMES ASSAYING 2.95% CU.

Test No.	Wt Tested	Temp °C	Type Machine	R Conc. %		Midd. Tail %		RECOVERIES		REAGENTS		Xan	Oils	Treatment Time Minn	REMARKS
				C	Cu	% Cu	% Cu	Conc	Midd Tail	Acid	Alk				
1A	1000	17°	SUB-A	6.1	15.84	1.04	.28	88.9	4.7	6.4	...	K-O.2	Steam Dist. Pine	0.2	20
1B	900	17°	SUB-A	3.6	9.9226	93.7	6.3	K-O.4	"	0.25	10
1C	900	17°	SUB-A	5.4	14.16	.36	.20	93.2	2.3	4.5	...	K-O.7	"	0.30	12
1D	950	17°	SUB-A	5.6	13.20	1.04	.20	87.7	7.9	4.4	...	K-O.7	"	0.30	12
1E	1000	17°	SUB-A	3.1	8.6412	97.1	...	2.9	25.0	K-O.3	"	0.10	20
1F	1150	17°	SUB-A	5.7	15.44	.80	.13	92.3	4.9	2.8	...	Na-O.7	"	0.25	12
1G	1200	45°	SUB-A	5.5	14.56	.84	.20	90.6	5.0	4.4	...	Na-O.5	"	0.20	10
1H	1200	17°	SUB-A	5.2	13.68	.68	.20	91.5	4.1	4.4	...	Ash	"	0.20	15
1J	1000	17°	SUB-A	4.1	9.2098	74.5	...	25.5	"	0.25	25
2A	800	17°	SUB-A	4.0	10.8822	94.3	...	5.7	...	K-O.7	Barrett No. 2 Creosote	0.4	20
3A	1000	17°	SUB-A	4.1	11.4426	93.4	...	5.6	...	K-O.6	Steam Dist. Pine	0.15	20
4A	1000	17°	SPITZ	4.6	12.48	.96	.22	90.5	5.0	4.5	...	K-O.7	Crude Turps.	0.4	15
5A	1350	17°	SUB-A	5.6	15.60	.48	.082	95.3	2.2	2.5	...	K-O.5	Cresylic Acid	0.7	15
6A	1100	17°	SUB-A	5.6	15.76	1.16	.18	92.0	3.8	1.2	...	K-O.5	Crude Carbolic	0.8	15
7A	1350	17°	SUB-A	5.3	14.72	.68	.12	94.2	3.1	2.7	...	K-O.7	Hardwood Creosote	0.5	15
7B	1300	17°	SPITZ	5.3	13.76	1.68	.20	87.1	8.5	4.4	...	K-O.7	"	0.8	15
7C	1000	17°	SUB-A	4.5	12.72	.60	.13	93.8	3.7	2.5	...	Na-O.7	"	0.5	15
8A	1250	17°	SUB-A	4.9	13.20	.60	.14	92.7	4.5	2.8	...	Na-O.6	Sludge 0.8	0.2	15
9A	1400	17°	SUB-A	4.9	13.52	.72	.12	92.7	5.0	2.3	...	K-O.7	Hardwood Creosote	0.5	15
9B	1400	17°	SPITZ	5.3	13.52	1.40	.15	88.3	8.3	3.4	...	K-O.7	Sludge	1.5	15
9D	1350	17°	SPITZ	5.2	13.84	.92	.20	91.3	4.2	4.5	...	K-O.7	"	1.0	25
9E	1800	17°	SPITZ	5.4	15.12	1.52	.18	89.5	6.5	4.0	18.0	K-O.7	"	0.9	20
9F	1700	41°	SPITZ	5.6	15.12	1.88	.22	86.5	8.8	4.7	...	K-O.7	"	1.3	20
9G	1650	42°	SPITZ	5.0	13.76	.84	.20	87.1	8.8	4.1	...	K-O.7	"	1.2	20
9H	1750	43°	SPITZ	4.8	13.76	.76	.60	85.4	2.3	12.3	20.0	K-O.7	"	1.8	25
9I	1250	68°	SPITZ	4.3	11.28	1.28	.26	88.2	6.8	5.2	...	K-O.7	"	1.5	22

Sol Cu Loss not included in tailing assay.

Rosin incorporated in Xanthate.

C.P. Sodium Xanthate used.

Filtrate from tails show sol Cu loss of .40% Cu.

ANACONDA SANDS ASSAYING 1.30 CU.

Exhibit K-4.

Test No.	Wt Tested	Temp ° C	Type Machine	ASSAYS			RECOVERIES			REAGENTS LBS./TON			Treatment Time Minn	REMARKS	
				R Conc %	Midd Tail %	Conc Midd Tail %	Alk	Acid	Xan	Oils	Alk	Acid			Xan
1A	1250	45°	SUB-A	8.1	9.76	.22	83.1	16.9	...	Na-O.5	Steam Dist. Pine	0.1	10	D.C.O. As Received	
1B	1200	17°	SUB-A	6.7	8.24	.20	87.9	12.1	...	Na-O.5	"	0.2	10	"	
1C	1000	17°	SUB-A	7.4	8.48	1.12	86.8	3.9	9.3	...	K-O.5	Crude Turps.	0.25	7	"
1D	800	17°	SUB-A	7.1	6.80	.22	87.0	13.0	...	K-O.5	Sludge	0.5	10	"	
1E	1000	17°	SUB-A	6.8	8.16	.22	86.7	13.3	...	K-O.5	Hardwood Creosote	0.4	10	"	
2A	1000	17°	SUB-A	8.7	9.36	1.28	84.8	8.1	7.1	...	K-O.3	Crude Turps.	0.3	10	Table Tailings Reground
2B	1000	17°	SPITZ	6.7	7.20	.16	83.6	7.2	9.2	...	K-O.4	Calif. Fuel	1.2	10	"
2C	1000	17°	SUB-A	7.5	8.24	1.08	86.4	8.5	5.1	...	K-O.3	Crude Turps.	0.5	10	"
2D	1000	17°	SUB-A	8.0	7.76	.76	85.3	8.1	6.6	...	K-O.3	Calif. Fuel	1.2	10	"
3A	1000	17°	SUB-A	8.0	8.64	.96	88.0	5.3	6.7	...	K-O.3	Crude Turps.	0.3	10	"
4A	1000	17°	SUB-A	8.3	10.0	1.16	87.7	5.8	6.5	...	K-O.3	Sod. Silicate	0.4	10	"
4B	1000	17°	SUB-A	7.2	8.40	.60	90.3	4.5	5.2	...	K-O.5	Barrett No. 635	0.6	10	"
5A	1000	17°	SUB-A	8.6	9.68	1.12	87.5	5.5	7.0	...	K-O.3	Steam Dist. Pine	1.0	10	"
5B	1000	17°	SUB-A	20.4	16.88	3.96	63.3	16.1	20.7	Hardwood Creosote	0.1	10	"
6A	1000	17°	SUB-A	6.7	8.56	.80	90.1	5.5	4.4	...	K-O.3	Creosote	0.5	10	"
6B	1000	17°	SPITZ	7.3	7.52	.72	84.3	9.3	6.4	...	K-O.4	Sludge	0.4	10	"
6C	1000	17°	SUB-A	7.7	8.56	.76	86.8	6.6	6.6	...	K-O.4	"	1.4	10	"
6D	4000	17°	SUB-A	7.2	8.16	1.36	88.4	5.2	6.4	...	K-O.3	"	1.4	10	"
										...		1.2	10	"	
										...		0.5	10	"	
										...		0.5	10	"	
										...		0.4	10	"	
										...		0.4	10	"	
										...		0.4	10	"	
										...		0.4	10	"	
										...		1.4	10	"	
										...		1.4	10	"	
										...		1.2	10	"	
										...		0.5	10	"	

"C.P. Potassium Xanthate

"with Sludge

"4Tests in Cycle

Exhibit—K-27.

EXHIBIT K-27

C O P Y

M.S.N.A.C.

San Francisco

San Francisco, Calif.,

May 15, 1923.

CHKm

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N.A., Corporation,
10 220 Battery Street,
San Francisco.

Dear Sir:—

Referring to our recent conversation regarding Xanthates and substances belonging to the same class of salts of sulfo carbonic Ethers, and supplementing my personal letter to you dated the 7th inst., I herewith beg to submit an incomplete list of substances, which partly by reason of chemical structure, partly by reason of indicative tests, belong within the scope of our first report as being patentable.

Mr. C. P. Lewis not being sufficiently conversant with the
20 chemistry involved, I take it upon myself to write on our joint behalf.

As the time at my disposal is not sufficient to carry out investigations of this type, prepare pure reagents and carry on routine assaying, I took the liberty of asking some Eastern firms through Braun-Knecht-Heimann Co. of this City by telegraph for some of these substances, but as most all of them are rarely employed, the wires elicited only negative replies. The last answer was received today, hence the delay in writing this Memo.

As most important representatives of the family of Etheral sulfo salts, alkali Xanthogenate deserves first place. The tests carried out
30 satisfactorily with this substance you are familiar with. The next in importance is the Mono Ethyl tri-Thiocarbonate of Potass $C_2H_5SCS_2K$. This salt is not sufficiently stable to give uniform results under all conditions, as evidently some of it decomposes, the mode of preparation being a little more complex and consequently more expensive, it was abandoned in favor of the alkali salts of Xanthogenic acid, only a few tests having been made with the material before our first report.

Regarding the action of the Xanthates proper in the flotation circuit, I beg to state that it is a brightening of the metal surfaces
40 through formation of an insoluble metal. Xanthate most likely, thus making them amenable to flotation.

Whenever the statement "positive" appears after the adjoined list of chemicals, a bottle test was made with particles of mineral, full flotation tests were carried out, or their action in a physico-chemical sense ascertained.

Exhibit—K-27.

	Ethylene dithiocarbonate	$C_2H_4COS_2$	tried positive
	“ tri “	$C_2H_4CS_3$	tried positive
	Ethylene Sulfide	C_2H_4S	tried positive
	Di-Ethylene disulfide	$C_2H_4S_2C_2H_4$	not tried
	Di-Ethyl Trisulphide	$C_4H_{10}S_3$	} A mixture of them obtained, apparently positive.
	“ “ Tetrasulfide	$C_4H_{10}S_4$	
	“ “ Penta sulfide	$C_4H_{10}S_5$	
	Salts of sulfo carbonic Ethers.		
	Potass. Mono Ethyl Thiocarbonate	C_2H_5OCO SK	tried positive
10	Di-Ethyl	$(C_2H_5)_2OCO$ S	not tried
	Salts of Di-Ethyl Ethylene di-Thiocarbonate		
	“ “ Ethyl Isobutyl Thiocarbonate	$(C_2H_5OCO S)_{12}C_2H_4$	not tried
	“ “ Isobutyl Ethyl “	$C_2H_5OCO S C_4H_9$	“ “
	“ “ Di-Ethyl Thiocarbonate	$C_4H_9OCO S C_2H_5$	“ “
	“ “ Mono Ethyl Di-thiocarbonate (Xanthogenic acid)	$(C_2H_5)_2 OCS O$	Not tried
	“ “ Methyl Ethyl Dithiocarbonate	$C_2H_5OCS_2 H$	used positive
20	“ “ Di-Ethyl Dithiocarbonate (Xanthic Ether)	$C_2H_5C S_2M$	used from denatured alcohol positive
	“ “ Ethelene Ethyl di-thiocarbonate	$(C_2H_5)_2 OCS_2$	
	“ “ Ethyl Propyl dithiocarbonate	$(C_2H_5OCS_2C_2H_2$	not tried as such
	“ “ Mono “ “	$C_2H_5OCS_2P$	not tried
		$P. O C S_2$	impure stuff obtained positive
30	“ “ Di Ethyl di Thiocarbonate	$Co (SC_5H_2)_2$	tried positive
	“ “ Mono Ethyl-Tri-Thiocarbonate	$C_2H_5SCS_2H$	
	Tried and found positive but discarded in favor of di salt		
	“ “ Di Ethyl tri thiocarbonate	$(C_2H_5)_2 SCS_2$	not tried
	“ “ Ethyl Ortho thiocarbonate	$C (SC_2 H_5)_4$	not tried

This list can no doubt be extended. For the present the substances enumerated cover the essentials necessary for a blanket patent claim.

As it may be necessary, however, to make actual working tests,
40 I beg to ask you to inquire from Eastman laboratory if a supply of c.p. substances could be manufactured.

Yours faithfully,

(Sgd.) CORNELIUS H. KELLER

Exhibit—K-28.

EXHIBIT K-28

San Francisco, California, May 16th, 1923
CHK-CPL-M

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

Dear Sir:

Re: ETHEREAL SULFO SALTS.

Supplementing report re Xanthogenates, we beg to inform you
10 that an alkali salt of Mono Ethyl Tri Thiocarbonic $\text{HC}_2\text{H}_5\text{SCS}_2$ was
made by us March 3rd, 1923. As it was not found to be very stable
and consequently not expected to give uniform results under all
conditions, it was discarded in favor of the more stable Xanthates.

A test carried out by Mr. C. P. Lewis on March 6th, 1923, using
above in treating Bunker Hill & Sullivan ore, gave the following
results:—

	% Wt.	Ag.	Pb.	Zn.	Fe.	Ag.	Pb.	Zn.	Fe.
Heads.....	100.0	3.4	8.2	2.34	22.2	100.0	100.0	100.0	100.0
Pb Conc....	13.1	21.5	53.0	9.2	9.2	82.8	84.8	51.3	5.4
Zn Conc....	5.6	5.4	13.4	16.0	16.2	8.9	9.2	38.5	4.1
Tails.....	81.3	.34	.6	.3	24.7	8.3	6.0	10.2	90.5

20

This test using .2% of the Potassium Mono Ethyl Tri Thio-
carbonate in place of the soda ash used in previous tests showed a
similar recovery in the same grade of concentrates with the same oil
combination. The lead froth in this case had the characteristic bright
sheen and appeared to carry the lead sulfides in a more flocculated
form than the previous tests on the same ore.

Very truly yours,

CORNELIUS H. KELLER
C. P. LEWIS

Exhibit—D-92.

EXHIBIT D-92

POSTAL TELEGRAPH — COMMERCIAL CABLES
TELEGRAM

B120cba 941 am 97
Po Newyork NY May 25 23
Edward H Nutter
220 Battery St Sanfrancisco

After conference Laist agreed give use of New Reagents an exhaustive
trial he will provide all necessary assistance an cooperation stop
10 arrange send soon as possible staff men necessary and requisite
supplies to anaconda to show what can be done chapman cannot be
spared from here stop wire us so we can advise laist here when he may
expect your man stop our understanding with laist is that demon-
stration will cost him nothing stop for your information laist expressed
disappointment and regret that results our last demonstration
proved so unsatisfactory after strong claims made on our part.

MINSEPNAC

EXHIBIT D-63

20

Postal Straight.
EHNm,
San Francisco, Calif.,
May 25, 1923.

Minerals Separation North American Corporation,
61 Broadway,
New York.

Lewis will leave for Anaconda twenty-ninth with samples of
material now being made and conduct preliminary laboratory tests
therewith after first testing it out here Stop If nothing unforeseen
occurs shipment of two hundred fifty pounds each kind will be made
30 by express about June first and Littleford and myself will leave
to arrive Anaconda about June fifth to conduct tests therewith.

CHARGE E H NUTTER

E. H. NUTTER

Exhibit—D-64.

EXHIBIT D-64

Office of the Chief Engineer
220 Battery Street, San Francisco, Cal.
San Francisco, Calif.,
May 29, 1923.
EHNm,

Minerals Separation N. A. Corporation,
61 Broadway,
New York.

10 Gentlemen:

In accordance with my telegram of the 25th, I am arranging for Lewis, Littleford and myself to go to Anaconda to conduct tests there with the Xanthates. Lewis left this morning with a sample of the Xanthate we have been using in the laboratory, and also a new sample from the Great Western Electro-Chemical Co. of material which as they are making to fill our order for 250 lbs. of potassium and sodium Xanthate. They so far have made only the sodium Xanthate and expect to have both ready for express shipment on Friday of this week. We will sample each lot before it goes out.

20 Mr. Littleford is leaving on the 3rd and will go directly to Anaconda and I am leaving on the 4th. I will stop over that the Betty O'Neal Mines at Battle Mountain, Nevada, as the addition to their mill is nearly ready and Mr. Getchell asked me if I would do this. I will be there on the 5th, then go on to Salt Lake and Anaconda, spending the morning of the 6th in Salt Lake City, and arrive at the Montana Hotel, Anaconda, the morning of the 7th, where I will doubtless be for a week or ten days. I may run over to the Coeur d'Alenes while in Montana, and will doubtless return home via Salt Lake City
30 instructions for me.

I will of course wire you from time to time as to how the tests are coming along.

Very truly yours,

(Sgd.) EDWARD H. NUTTER,
Chief Engineer.

Exhibit—K-5.

EXHIBIT K-5

WESTERN UNION
TELEGRAM

RECEIVED AT

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ANACONDA MONT 14

MINERALS SEPARATION NORTH AMERICAN CORPN

61 BROADWAY NEW YORK NY

- 10 REPLYING YOUR BLUE FOURTEENTH WILL BE GUIDED THEREBY REGARD-
ING DISCLOSURES STOP TAILINGS ASSAY YOU REFER TO INCLUDES COPPER
LOST WITH TAILINGS WATER AND CALCULATED AGAINST TONNAGE
STOP ASSAYS JUST OUT FOR PAST TWO DAYS FOR ONE MACHINE SAME
CONDITIONS AS THE EIGHTH EXCEPT TEMPERATURE OF EIGHTY
DEGREES AND MACHINE OPERATED BY REGULAR CREW TAILINGS
SULPHIDE COPPER DECIMAL NAUGHT NINE TOTAL INCLUDING EVERY-
THING DECIMAL TWENTY NINE STOP SAME MACHINE RUN TODAY
FOR CONFIRMATION UNDER IDENTICAL CONDITION WITH APPARENTLY
IDENTICAL RESULTS STOP ANOTHER MACHINE EQUIPPED WITH AIR
20 BASKETS IN SPITZES OPERATED BY US PREAGITATING IN PACHUCA
WITH FIVE POUNDS ONLY OF ACID TAILINGS TOTAL COPPER DECIMAL
THIRTY EIGHT AND DECIMAL THIRTY THREE WITH DIFFERENT OIL
COMBINATIONS STOP SAME MACHINE OPERATED BY US TODAY WITHOUT
ACID PREAGITATING WITH KELLER LEWIS REAGENT IN PACHUCA GAVE
APPARENTLY SOMEWHAT SIMILAR OR BETTER RESULTS STOP LAST
HOUR TODAY WITH SAME CONDITIONS EXCEPT THAT TEN POUNDS ACID
ADDED TO HEAD OF MACHINE GAVE APPARENTLY BEST RESULTS YET
OBTAINED AND CREATED QUITE A FURORE AND WE WILL MAKE
CONFIRMATION
- 30 RUN TOMORROW STOP SATURDAY SUNDAY AND MONDAY WILL OPERATE
TWO MACHINES IN SAND PLANT ONE ON CURRENT SANDS AND THE OTHER
ON OLD SANDS THIS WILL ABOUT FINISH OUR QUANTITY OF REAGENT
STOP TUESDAY I PLAN LEAVING FOR SALT LAKE AND SEND LEWIS AND
LITTLEFORD SAN FRANCISCO STOP EXPECT TO MEET MR LAIST THURSDAY
AT OGDEN AND RIDE BACK WITH HIM TO POCATELLO AND DISCUSS
RESULTS AND POSSIBLE FULL MILL SCALE TESTS AND THEN RETURN TO
SAN FRANCISCO TO MEET CRAMER RESEARCH ENGINEER FOR PHELPS
DODGE WHO WILL BE THERE TO OBSERVE TESTS ON THEIR SAMPLES
PLEASE WIRE IF I SHALL NOT DISCLOSE TO HIM STOP FIFTEENTH I GO
40 TO BUTTE TO SEE RECEIVERS BOSTON AND MONTANA PLEASE WIRE IF
MATTER IS SUFFICIENTLY IMPORTANT FOR ME TO DISARRANGE ABOVE
SCHEDULE IF NECESSARY INFORMATION CANNOT BE OBTAINED EASILY
AND QUICKLY

E H NUTTER

Exhibit—D-65.

EXHIBIT D-65

Anaconda, June 18th, 1923.

Minerals Separation N. A. Corporation
61 Broadway
New York

Gentlemen:

We finished up our schedule of tests today, and I feel pretty well satisfied that the Anaconda officials here think we have done pretty well. There are some cans of xanthate left, and I have outlined work
10 for them to use this in, that will elaborate our results.

The next stage of testing however, is with at least a full section of the mill, and there is not enough xanthate left to do that usefully.

Assays were out today on two days testing in the sand plants.

On the current sands assaying about .15% cu. practically no improvement was made in recovery, but we do not know yet about the concentrates. It may be that using more xanthate would show an improvement but Mr. Laist once told me that they recovered practically all the free mineral in the sands, which of course could not be improved upon.

20 They may find, however that xanthate can be substituted at a saving for part of their present reagents. The tailing looked lighter colored today when using a larger quantity, but we have no assays on the run yet.

In the mill which is retreating old sand tailings which are reground and floated, the assays were tailings.

Saturday the 16th .13% cu

Sunday " 17th .17% "

On Sunday the launders got blocked and conditions were bad for a time which may be the reason for the higher tailing. The tailing in
30 this plant varies from about .22% Cu. to .30% Cu. and averages about .24% Cu.

We increased the recovery therefore about 2# of copper per ton, with say 20c. per ton net at an increased cost of say 4c. per ton, which will doubtless be balanced by increased silver recovery.

This should increase their profits about \$25000 per month on the tonnage they are now treating of these sands. On the slimes the financial improvement should amount to about as much, thus making the use of xanthate worth about \$50000 per month to the Anaconda Co. with their present tonnage. This does not figure in the pond
40 slimes, which are about exhausted nor the zinc ore on which the silver recovery can doubtless be improved.

Exhibit—D-65.

This estimate is of course tentative, but nevertheless is based on work with full size machines, and I think is well within the mark. The action of the xanthate is very definite and reliable, and when uniform conditions are maintained uniform and consistent results are obtained. They need to make no changes in their plant to get the major part of the improvements except to add the xanthate and cut down somewhat the oils. To get the full benefit on the slimes they should add air baskets to the spitzer and preagitate the xanthate in pachucas. This should increase their recovery about another pound of
10 copper per ton, and if they run with a 10°F. hotter circuit, they should get still another pound of copper.

The callow representatives have been getting .38% to .42% Cu. tailings in the remodeled M.S. machines in the slime plant, as compared with our .26% to .38% cu. in the same machine. We used about half the air they do.

In the standard machine without air we got with the regular crew .34% to .35% Cu. at the usual temperature of 70 F. and .29% Cu. at 80 ° F.

I will go over the results with Mr. Laist in Salt Lake the 20th
20 and be home the 22d. Lewis and Littleford will be home the 21st.

Very truly yours,

(Sgd.) EDWARD H. NUTTER

Exhibit—D-66.

EXHIBIR D-66

ANACONDA COPPER MINING COMPANY
ANACONDA REDUCTION DEPARTMENTRESEARCH-TESTING DEPARTMENT
6—19—23.R—T—D F.N. 600.1166
S. N. 78

Machine: No. 10 M. S. Slime Plant.

Feed: Current Slime

Reagents: Sludge, H₂SO₄, Creosote, Z—Oil.

Preliminary Test to Determine Best Reagent & Oper. Cost.

10	Date.....1923.	6—8	6—9	6—11	6—12				
	Shift								
	Tons/24 hrs.	118.9	122	121.9	116				
	% Solids	12.35	12.72	12.66	12.07				
	Hrs. Run:	4 2	3.5 3	5 ½	3½ 2½				
	Temp., °F.	62	61	75	61				
	Reagents, #s./Ton:								
	Sludge	6.5 by hand	3.1	3.1	3.01	3.01		
	H ₂ SO ₄ Acid	22.6 22.6	21.4 	5.73	5.73	15.3	8.22		
20	Sod. Silicate								
	50% H ₂ Oby hand		2.19		
	Creosote	2.68	2.04	2.04	2.16	2.16		
	Z—Oil; 80% H ₂ O	1.65 1.65	2.03 2.03	2.21	2.21		
	Genasco	1.22		
	Assays:								
	Feed:								
	% Cu	2.47	3.28	3.03	
	% Ox Cu28	.4434	
	Conc. : †								
	% Cu	6.31 7.94	7.47	8.12	13.16	11.20			
30	% Insol.	58.2 36.1	51.0	29.7	38.4	43.2			
	Tails: (Z Reagent)								
	% Cu	.38 1.24	.43 .44	.88	.96	.64	1.01		
	% Ox Cu	.23 .24	.23 .21	.39	.46	.28	.31		
	Slime Plant Tails:								
	% Cu	.61	.58	.78	.75				
	% Ox Cu	.37	.34	.41	.41				

† This concentrate is from middle of machine and is sent to Cleaners.
Tailings hand sampled.

Exhibit—D-66.

ANACONDA COPPER MINING COMPANY
ANACONDA REDUCTION DEPARTMENT

RESEARCH-TESTING DEPARTMENT
6—19—23

R—T—D F.N. 600.1166
S. N. 78

Machine: No. 10 M. S. Slime Plant.

Feed: Current Slime.

Reagents: Sludge, H₂SO₄, Creosote, Z-Oil.

Regular Reagents Plus Z Reagent

10	Date.....1923.	Shift			Averages			
		6—13	6—14 *	6—15*	13, 14 & 15th			
	Shift							
	Tons / 24 hrs.	120	111	114	115			
	% Solids	12.95	13.70	13.55	13.40			
	Hrs. Run	6.5	7.5	7.5	21.5			
	Temp., °F.	78	82	71	77			
	Reagents, lbs./Ton:							
	Sludge	6.45	7.89	6.1	6.81			
	H ₂ SO ₄ Acid	19.7	21.6	17.3	19.5			
	Sod. Silicate,							
	50% H ₂ O							
	Creosote	2.2	2.36	3.06	2.54			
	Z-Oil, 80% H ₂ O	2.18	2.92	2.66	2.59			
	Genasco							
	Assays:							
	Feed:							
	% Cu	3.09	3.28	3.10	3.16			
	% Ox Cu	.31	.26	.33	.30			
	Conc. : †				Sample			
	% Cu	9.57	6.55	11.43	7.60	lost 7.42	8:51	
30	% Insol.	45.8	57.6	36.9	5.41	34.8	50.6	46.63
	Tails: (Z Reagent)							
	% Cu	.34	.29	.35	.326			
	% Ox Cu	.20	.20	.21	.20			
	Slime Plant Tails:							
	% Cu	.58	.44	.60	.54			
	% Ox Cu	.36	.26	.28	.30			

* Run made on Sodium Zanthate with regular slime plant reagents & Zanthate.

† 1st Concentrate shown is finished concentrate.

2nd " " " regular " which was being delivered.

to cleaners.

40

No Automatic Sampler on Tailings — hand sampled.

Exhibit—D-66.

ANACONDA COPPER MINING COMPANY
ANACONDA REDUCTION DEPARTMENT

RESEARCH-TESTING DEPARTMENT
6-19-23.

R—T—D F.N. 600.1166
S. N. 78

Machine: Remodelled M. S. No. 1 Slime Plant.

Feed: Current Slime.

Reagents: H₂SO₄
Sludge
Genasco
Z - Oil

10

Date	1923. 6-13		6-14*			6-15	
Shift	1	1	1	1	1	1	1
Tons / 24 hrs.	120		102			127	
% Solids	12.95		13.70			13.55	
Temperature, °F.	84		81			83	
Manifold Pressure	9		5.5			5.0	
Air—Cu. Ft./Min.	120		175			195	

Time:

Operating	5½		6½			5	
20 Sampling	3	2½	1½	1½	1½	2	2

Reagents, lbs./Ton:

H ₂ SO ₄	7.07	7.07	10.9	9.71
Sludge	3.37	3.84	2.32	2.32	2.32	3.48
Creosote						
Genasco	.92	1.34	0.81	0.81	0.81	0.58
Z - Oil, 70% H ₂ O	.83	.83	1.95	1.95	1.95	1.3

Assays: Feed:

% Cu	3.09		3.38			3.10	
% Ox Cu	.26		0.26			0.33	

30

Conc. :

% Cu	11.35		12.37	11.90	12.00
% Insol.	43.8		32.1	38.1	34.1	31.8

Tails:

% Cu	.38	.33	.54	.48	.26	.39	.37
% Ox Cu	.18	.21	.18	.17	.16	.20	.21

Slime Plant Tails:

% Cu	0.58		0.44			0.60	
% Ox Cu	0.36		0.26			0.28	

*Neutral

Automatic Sampler on Tailings

Exhibit—D-66.

ANACONDA COPPER MINING COMPANY
ANACONDA REDUCTION DEPARTMENT

RESEARCH-TESTING DEPARTMENT
6—19—23.

R—T—D F.N. 600.1166
S. N. 78

Machine: 4—1 M. S.
Feed: Table Tails
Reagents: Z - Oil
Sludge
H₂SO₄

10 Date 1923.	6—16	6—17	6—18		Average
Shift	1	1	1	1	
Tons / 24 hrs.	475	820	795		697
Temperature, °F.	52	51	53		52
Time, hrs. :					
Operating	4 ³ / ₄	7	1 ¹ / ₂	3 ¹ / ₄	16 ¹ / ₂
Sampling	2 ³ / ₄	5 ¹ / ₄	1	2 ³ / ₄	11 ³ / ₄
Reagents, lbs./Ton:					
Na-Z-Oil, 80% H ₂ O	.99	.41	1.03	.63	.765
Sludge	2.50	1.50	4.64	4.64	3.32
20 H ₂ SO ₄	7.08	4.99	4.28	4.28	4.93
Assays:					
Feed:					
% Cu	1.48	2.39	1.21	1.47	1.64
% Ox Cu	.09	.06	0.06	0.09	.075
Conc. :					
% Cu	5.17	7.18	7.15	8.42	6.98
% Insol.	51.7	22.3	(Not Out)		
Tails:					
30 % Cu	.13	.16	.09	.13	.128
% Ox Cu	.04	.04	.02	.01	.028
Section Tails:					
% Cu	.14	.16	(Not Out)		
% Ox Cu	.05	.04	(" " " ")		
Tailings hand sampled.					

Exhibit—D-66.

ANACONDA COPPER MINING COMPANY
ANACONDA REDUCTION DEPARTMENT

RESEARCH-TESTING DEPARTMENT
6—19—23.

R—T—D F.N. 600.1166
S. N. 78

Machine: Remodelled 5-1 M. S.
Feed: New Works Tails
Reagents: Z - Oil
Sludge
H₂SO₄

10 Date1923.	6—16	6—17	6—18	Average
Shift	1	1	1	
Tons / 24 hrs.	411	433	437	427
% Solids
Temperature	50	51	53	51
Alkalinity
Manifold Pressure	1.5	1.75	1.75	1.67
Time, Hrs. :				
Operating	5¼	3	4½	12¾
Sampling	3¼	2½	3½	9¼
20 Reagents, lbs./Ton:				
Z-Oil, 70% H ₂ O	.67	.595	.74	.668
Sludge	3.96	3.98	3.92	3.95
H ₂ SO ₄	11.45	10.9	10.8	11.05
Assays:				
Feed:				
% Cu	.52	.62	0.63	.59
% Ox Cu	.12	.13	0.11	.12
Conc. :				
% Cu	5.72	5.42	5.09	5.41
30 % Insol.	48.0	48.4	53.9	50.1
Tails:				
% Cu	.13	.17	0.14	.147
% Ox Cu	.06	.06	0.03	.05
Section Tails:				
% Cu	.21	.24	(Not Out)	
% Ox Cu	.09	.12		

Date on Sludge, Acid & Tonnages based on mill figures.
Automatic Sampler on Tailings.

Exhibit—D-67.

EXHIBIT D-67

MINERALS SEPARATION NORTH AMERICAN CORPORATION
 Office of the Chief Engineer
 220 Battery Street, San Francisco, Cal.

Salt Lake, June 20th, 1923

Minerals Separation N.A. Corporation,
 61 Broadway,
 New York.

Gentlemen:

10 I met Mr. Laist today here by appointment, and went over the test results we got at Anaconda, and he seemed impressed by them, but was non-committal. He expressed a wish that we had done alkaline circuit work in the large machines, but we had concentrated on the acid circuit work as being more economical. Thus the 20# per ton of acid required in the slime plant costs them less than the 10# per ton of lime required with the T.T. mixture by about a cent a ton treated. Soda ash would cost still more. In the laboratory, tests using lime had not been especially good, but we will do more work with lime.

20 He asked if they would have the right to use xanthate under their contract, without additional royalty, and I said "certainly".

Mr. Laist asked me to get quotations and deliveries on xanthate for the quantities they would want. This affects 5400 tons per day.

Since my last letter to you we got assays on our last day's tests, and we succeeded in reducing the current table sands from .15% Cu. to .09% Cu. total, by increasing the xanthate to .25 pounds per ton.

30 At Mr. Laist's suggestion I told Mr. Elton, manager of the International Co. of the xanthate, and specified that the information is confidential. Mr. Elton is going to send us samples of Walker Nine ore, and of the complex lead-zinc-silver ores tributary to the Salt Lake district.

I also called on the Silver King Coalition people, and on Mr. Lambourne of the Judge and Park Utah companies, but there was nothing of especial note came up in our talks.

Very truly yours,

(Sgd.) EDWARD H. NUTTER.

Exhibit—P-100.

EXHIBIT P-100

Machine: No. 10 Slime Plant

Feed: Current Slime

Reagents: Regular + "Z"

Date.....1923.	JUNE 25			JUNE 26			Z AVG.	REG. AVG.
Shift	1	2*	3*	1	2*	3*		
Tons per 24 hrs	142	App	120	115	App	120	129	App 120
Hours Operating	6	8	8	6.5	8	8	12.5	16
“ Sampling	5	8	8	5.5	8	8	10.5	16
10 Tailing Sampler	Automatic							
% Solids	14.77			12.66				
Reagents — lbs. per ton:	**			**				**
H ₂ SO ₄	16.0	24.0	21.3	24.0	18.6	24.0		24.0
Sludge	5.2	5.3	6.4	5.3	5.8	5.3		5.3
Creosote	1.8	2.0	2.7	2.0	2.3	2.0		2.0
“ Z ”	.34	0.42	0.38
Assays: Feed:								
% Cu.	3.19	3.09	3.14
% Ox. Cu.	0.28	0.30	0.29
20 Concentrate:								
% Insol.	35.7	37.0	50.8	25.0	39.7	30.3	32.5
Tails. :								
% Cu.	0.33	0.46	0.43	0.34	0.40	0.39	0.335	0.42
% Ox. Cu.	0.20	0.24	0.32	0.19	0.31	0.27	0.195	0.285
Slime Plant Tails. :								
% Cu.	0.49	0.68	0.70	0.77	0.82	0.6068
% Ox. Cu.	0.27	0.34	0.36	0.46	0.42	0.3236

*No. 10 machine with regular reagents and no "Z" on afternoon and night shifts.

**Normal average reagents.

Exhibit—P-100.

		Machine: 5-3 (No. 3 Machine Sec. 5)							
		Feed: New Works Tailings							
		Reagents: Regular + "Z"							
Date.....1923.		6/25	6/26	6/28	6/29	7/2	7/3	7/5	Avg.
	Shift	1	1	1	1	1	1	1	
	Tons per 24 hrs.	412	358	378	425	342	464	422	413
	Temp. ° F.	53	53	54	55	62	56	56	55.6
	Tails. Sampler	Automatic							
	Hours Operating	8	5	6.5	6.5	6.5	5.5	6.0	44.0
10	" Sampling	6	4	5.5	5.5	4.5	5.5	5.0	36.0
	Reagents — lbs. per ton:								
	Acid	11.45	13.15	12.45	11.09	10.09	10.12	11.1	11.46
	Sludge	4.15	4.93	4.43	3.94	3.77	3.69	4.16	4.15
	"Z"	.196	.224	.213	.187	.192	.213	.226	.207
	Assays — Feed* :								
	% Cu.	.71	.54	.50	.62	.72	.74	.70
	% Ox. Cu.	.11	.12	.09	.10	.12	.15	.16
	Conct. :								
	% Insol.	43.8	27.0	49.9	49.9	44.1	59.0	40.0	44.8
20	Tails. :								
	% Cu.	.26	.15	.16	.19	.21	.25	.30	.217
	% Ox. Cu.	.11	.05	.08	.07	.08	.09	.11	.08
	Sec. 5 Tails. :	.21	.21	.21	.21	.23	.2722
	Excluding 5—3	.12	.12	.12	.11	.14	.13	.16	.13

* Feed assays represent N. W. Tails. treated that day (24 hours).

SAMPLING INVESTIGATION — SLIME PLANT

Time: Three day shifts.										Avg.	
Machines:	1	2	3	4	5	7	8	9	1-9	No. 10	
30	Hose Sample										
	% Cu.	.63	.70	.67	.61	.63	.63	.63	.58	.64	.60
	% Ox. Cu.	.38	.36	.35	.35	.35	.36	.33	.34	.35	.35
	West Automatic:										
	% Cu.									.60	
	% Ox. Cu.									.33	
	No. 10 Automatic										.49
											.30

Hose samples taken by drawing a continuous stream of pulp through a one-inch hose placed in wall of No. 15 cell of each machine midway between wall and center of cell and opposite to forward movement of center of impeller blade.

40

Since this test the launder leading to automatic sampler has been rebuilt and stream to cutter made to flow more uniformly. No test on remodelled stream has been made.

Exhibit—D-68.

EXHIBIT D-68

MINERALS SEPARATION NORTH AMERICAN CORPORATION
61 BROADWAY,
NEW YORK

June 26, 1923.
AR:mm

Mr. Edward H. Nutter, Chief Engineer,
Minerals Separation North American Corp.,
220 Battery Street,
10 San Francisco, California.

Dear Sir:

We have followed the course of the recent experimental work at Anaconda with the Xanthate reagents, as set forth in your various letters, with keenest interest. However, we have made no effort to reply until receiving final reports of the work.

We have your letter of the 18th in regard to the sand plant tests, in which you sum up the results of the work, and have just received your letter of the 20th on your interview with Mr. Laist.

We are gratified to note your favorable opinion in the summary of
20 the work. So far as we could judge at this distance, and with only partial information, the results of some of the tests seemed to be extremely good and the tailings assays so low that they were approximately equal in this respect to the much vaunted results of the Thio-Carbanilid mixture. However, there is apparently considerable to be learned in regard to the use of these reagents, and also the full economic effects of their use in various combinations are not clear to us.

It seems clear enough, however, that the use of Xanthates,
20 in addition to their regular reagent mixture, on the slimes should be well worth their while, even though they are not able to give up the use of acid and sludge, as the laboratory tests indicated.

From the information in your last letter, the same seems to apply to the use of Xanthate on the current table sands. It will be very interesting to see what can be done with further work along the lines of preagitating with Xanthates, then floating with acid in the circuit, and also there may be possibilities in the way of using Xanthate and other reagents in place of the kerosene acid sludge and acid, as well as in alkaline or neutral circuits. Certainly it would seem that the results of the tests must challenge their attention,
40 and they can hardly disregard doing further and more exhaustive work with them.

We note Mr. Laist's expression of disappointment that you did not carry out tests in an alkaline circuit. However, in view of

Exhibit—D-68.
Exhibit—D-93.

the fact that you found on arrival that the fresh slimes worked out so differently than the samples after standing, and also that you had only a limited quantity of the reagents with you and immediately available, it would seem to us you had followed a very logical course in not going too far afield at the start.

It seems hardly likely that Mr. Laist's attitude will be to disregard results of these tests and not to do further work with the Xanthates at Anaconda. No doubt he will first want to assure himself to a reasonable degree of certainty as to what the reagent
10 will cost in large quantities and that an ample supply can be had. It may take a little time to get him these assurances from the Great Western Company.

Your last two letters arrived after Dr. Gregory left for Bangor. He has not seen them, and I cannot speak for him; but it seems to me, that if, after receiving assurances on these points, Mr. Laist still hesitates to do more test work, then we ought to offer to carry on further tests at Anaconda at our expense to thrash the question out thoroughly.

I think if you will send us a full tabulation and report of the
20 tests to supplement the data given in your letters and telegrams we will have a clearer picture of the work and results to date.

Yours very truly,

(Sgd.) ALBERT ROBERTS
Secretary.

EXHIBIT D-93

M.S.N.A.C.
San Francisco

JU DL
EHNm

CC NY

San Francisco, Calif.,
July 5, 1923.

30

Bayard S. Morrow,
Anaconda Copper Mining Company,
Anaconda, Montana.

Great Western Electro Chemical Company can begin delivery potassium salt at rate of one hundred to one hundred fifty pounds daily in four days after receipt of order Stop This will be the uncentrifuged salt which works all right technically but would require somewhat larger quantity per ton of ore Stop The price on one thousand pounds of this will be twenty five cents per pound as it
40 has to be hand made Stop Will have quotations about July eleventh on larger quantities we discussed.

E. H. NUTTER

Charge E H Nutter.

Exhibit—D-69.

EXHIBIT D-69

ANACONDA COPPER MINING COMPANY
ANACONDA REDUCTION DEPARTMENT

Anaconda, Montana
July 6th, 1923

Mr. E. H. Nutter, Chief Engineer,
Minerals Corporation Company, Ltd.,
220 Battery Street,
San Francisco, California.

10 Dear Sir:—

I have placed a requisition with our Purchasing Department for 1000 pounds of Z reagent. I have asked them to order this reagent through your Company and to have shipments made by express in lots of 250 pounds each.

Later developments in our flotation work indicate that the No. 10 machine at the Slime Plant gives a tailing lower than the normal tailing without the Z reagent. We therefore require an additional amount of this reagent in order to secure complete data as to the difference that will exist between its use and not using it.

20 I note by your wire of July 5th, that you will have quotations, about July 11th, for the larger quantities of Z discussed by yourself, Mr. Frick and I just before you left Anaconda.

All of our test work indicates that it is necessary to operate one unit and one-half of the Slime Plant with special reagents in order to definitely demonstrate their effectiveness. One machine naturally receives more personal attention than can be given to a complete unit. This attention has some effect on the tailings produced.

No doubt you will hear from our Purchasing Department within a very few days.

30 I will appreciate it if this order is delivered promptly.

Yours truly,

CC.Mr. Frick
Mr. Rodgers
Mr. Klepetko

(Sgd.) BAYARD MORROW

Supt. of Concentration.

Exhibit—D-70.

Exhibit—D-71.

EXHIBIT D-70

CC NY

San Francisco, Calif.,
July 19, 1923.Bayard S. Morrow, Esq.,
Anaconda Copper Mining Company,
Anaconda, Montana.

Dear Mr. Morrow:—

It seemed to me that it might be useful for us to have a man at Anaconda experienced in the use of "Z" reagent while you are conducting the tests with the 1000 lbs. which you have ordered, as some point might come up regarding which he can make useful suggestions. I am, therefore, having Mr. C. P. Lewis go to Anaconda for this purpose and he will arrive there on or about the 23rd and will be our representative while there. I trust that this is agreeable to you, and I am,

Very truly yours,

EDWARD H. NUTTER

Chief Engineer.

EXHIBIT D-71

20 M.S.N.A.C.

San Francisco

CC NY

Mr. Allen

Mr. Lewis

San Francisco, Calif.,

July 21, 1923

EHNm

Bayard S. Morrow, Esq., Supt. of Concentration,
Anaconda Copper Mining Company,
Anaconda, Montana.

Dear Sir:—

I have your letter of the 17th in reply to mine of the 11th and have read it carefully, and I thank you for the tabulation of further results with the "Z" reagent.

In regard to the quotation made by the Great Western Company, I think that perhaps my previous letters were not exactly clear, to the effect that they agree to refund to you on both the minimum carload and the 1000 ton lot, provided you should order either or both from them, all profit they may make in excess of twenty percent. This would be after the orders were completed and they were able to figure exactly the cost of the material to them. They agree also in

Exhibit—D-71.

connection with this to open up their books to either your representative or ourselves, so that we can check their statements; but in any event the price would not exceed 25 cents per pound on the minimum carload, nor 19 cents per pound on the 1000 ton lot.

As soon as I received your letter I took up with them the matter of getting a freight rate, and their Vice President is now at work on this. I seemed to me that they would be able to get quicker action on a freight rate than we could, and they are shipping their chemicals right along, and of course we are not. I will advise you
10 as soon as we get any figure from them.

I enclose a sheet giving the best method of assaying this material which has so far been worked out by our chemist, Mr. Keller. The quantity of the CS_2 radical in the finished salt is the essential thing to determine.

The 1000 pounds of "Z" reagent has now been made and shipped to you. While as I stated it was the uncentrifuged salt, it had nevertheless been dried at a low temperature and did not contain much mother liquid.

The tabulated figures which you have sent me are very interesting
20 and I thank you for them. The lower tailing which the No. 10 machine seems to be able to make so consistently, as compared with the rest of the Slime Plant, is a bit puzzling. I am wondering, in view of the amount of special work which was done on this machine, it has since been running with higher levels in the spitzkasten, thus giving a longer time of treatment to the pulp. It may be that the valves got set in it so that this result would follow. Should this
30 be the case I think it might be well worth your consideration to set up one machine with the weir overflows, such as are shown on the drawing I gave to Mr. Rogers while at Anaconda in June, as that type of weir will give very satisfactory running conditions and a
40 uniformly high level can be maintained in the spitzkasten without overflow of pulp.

The quantities of "Z" reagent shown in the tabulations I presume refer to the dried salt. If they refer to the amount of solution the amounts would be much smaller than we were able to get good results with.

The higher tailing obtained with the "Z" reagent in the 5-3 machine, as compared with the tailing got in the 5-1 machine while we were there, would appear to indicate that the air fingers were
40 assisting substantially in the recovery.

Very truly yours,

EDWARD H. NUTTER
Chief engineer.

Exhibit—K-29.

EXHIBIT K-29

GREAT WESTERN ELECTRO-CHEMICAL COMPANY

Executive and Sales Offices
9 Main St., San Francisco, California
July 21, 1923

Minerals Separation North American Corp.,
220 Battery St.,
San Francisco, Calif.

ATTN: MR. NUTTER

10 Gentlemen:

In compliance with the request of your New York office, I am transmitting to you herewith a detailed description of the process for the manufacture of XANTHATES, as worked out by myself. In writing this letter I am assuming that the information contained therein will be treated as confidential by the Minerals Separation North American Corp; and will, more particularly, not be revealed to any one outside of your own organization, nor used in any way detrimental to the interests of the Gt. Western Electro-Chemical Co. nor myself, the inventor. My date of conception was May 7-8, 1923.

20 You are probably aware of the fact that the method described in the books for the preparation of sodium or potassium Xanthate require that an alcoholic solution of caustic potash or soda be first made and that to this alcoholic solution carbon bisulfide be added. It is also usually stated that water must be entirely absent. This method requires, since the alkalis have only a limited solubility, the use of considerable excess alcohol, and involves a decrease in the yield of xanthates due to their large solubility in the excess alcohol. It also involves the recovery of the xanthate out of solution.

30 My method consists in an exactly reverse procedure. I have found that if a concentrated solution of alkali in water is first prepared, and to this solution alcohol added, the alkali is largely precipitated in a very fine and active condition. If then, to this mixture of alcohol with alkali and water, carbon bisulfide is added, the reaction proceeds smoothly to give a solid material, which on drying, becomes Xanthate. This method makes it possible to work with practically the theoretical quantities of reagents and to obtain a nearly theoretical yield of solid, crystalline material which can be readily dried to Xanthate.

To give an example:

40 Fifty-two pounds of commercial potassium hydroxide, containing 88.2% K O H were dissolved in 12 liters of water. To this solution, while hot, were added 22 liters of denatured ethyl alcohol, containing 95% alcohol by volume and having a specific gravity of .825. This mixture was then cooled to about 20°C. and to it was added, with

Exhibit—K-29.

constant stirring and careful cooling, 24 liters of refined carbon bisulfide. After all the ingredients were mixed and the mass thoroughly cooled there was obtained about 185 lbs. of semi-solid material, which when put on trays readily dried in the air and gave commercial potassium xanthate in nearly theoretical yield.

In case it is the desire of the Minerals Separation N. A. Corp. to use this letter for the purpose of making a patent application in my name, it must be understood that I am working with the Gt. Western Electro-Chemical Co. under an agreement to assign to them, for
 10 agreed compensation, additional to my regular salary, patents and processes which would be considered of value to the company. The assignment of my patent rights to the Minerals Separation N. A. Corp. could therefore not be made by me, but necessarily by the Gt. Western Electro-Chemical Co. after assignment to them.

Very truly yours,
 (Sgd.) LUDWIG ROSENSTEIN

R/F

GREAT WESTERN ELECTRO-CHEMICAL COMPANY
 Executive and Sales Offices
 20 9 Main St., San Francisco, California
 July 21, 1923

Minerals Separation North American Corp.,
 220 Battery St.,
 San Francisco, Calif.

ATTN: MR. NUTTER

Gentlemen:

I am sending you herewith Example No. 2 which describes the method used for the preparation of Sodium Xanthate.

This is in pursuance with our telephone conversation of Saturday,
 30 July 21.

Very truly yours,
 (Sgd.) LUDWIG ROSENSTEIN

R/F

M. SN. A. Corp.

EXAMPLE No. 2.

Forty-two pounds of commercial sodium hydroxide, containing 94% NaOH, were dissolved in 13.5 liters of water. To this solution, while hot, were added 28 liters of denatured ethyl alcohol containing 95% by volume of alcohol and having a specific gravity of .825. This

Exhibit—K-29.

Exhibit—K-35.

mixture was then cooled to about 20°C. and to it was added, with constant stirring and careful cooling, 28.5 liters of refined carbon bisulfide.

After all the ingredients were mixed and the mass thoroughly cooled there was obtained about 192 lbs. of semi-solid material. This was dried carefully and at a low temperature in a shelf dryer by a current of warm air, and gave commercial sodium xanthate in nearly theoretical quantity.

L.R.

10 EXHIBIT K-35

MINERALS SEPARATION NORTH AMERICAN CORPORATION

Office of the Chief Engineer

220 Battery Street, San Francisco, Cal.

San Francisco, California, July 23, 1923.

CHK-M

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

Dear Sir:

20 Referring to my memo dated July 20th, 1923, subject — "Assay of Alkali Xanthates," I beg to state that the formula covering the reaction between Cu and CS₂ radical is, of course, not the final one, as the cupric Xanthate decomposes into cuprous Xanthate and Ethyldioxythiocarbonate. Thus:



The formula given in my memo suffices to show the basic reaction involved.

30 Regarding the shipping of Alkali Xanthates in concentrated solution to Anaconda, wish to say that at least 120% by weight of water will be required to dissolve a material similar to the one submitted and retain it in solution at 20° above zero. While freezing at 20° below zero for a period of 6 hours does not seem to affect either the solution or the moist salt, raising the temperature above the critical point of 50° seriously impairs its efficiency. As the material would no doubt be subjected to the extremes of temperature while in transit or at Anaconda I cannot recommend the shipping of larger quantities in that form. Smaller quantities may be shipped in drums for a test. In any event the freight differential will have to be the determining factor.

40 As to Ludwig Rosenstein's conception, contained in his letter to M.S. dated July 21, 1923, I beg to ask you to keep it in abeyance for the present.

Yours respectfully,
(Sgd.) CORNELIUS H. KELLER

Exhibit—K-30.

EXHIBIT K-30

C O P Y

San Francisco, Calif.,
July 25, 1923.
CHKm

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corporation,
220 Battery Street, San Francisco, Cal.

Dear Sir:

10 Regarding Ludwig Rosenstein's method of alkali xanthate manufacture contained in his letter to Minerals Separation N. A. Corp., dated July 21, 1923.

 He claims that a precipitate of alkali in a very fine and active condition is formed by mixing the alkali water solution with the alcohol. Such seems not to be the case, as my own observation in performing the operation outlined is that no precipitate is formed, but a suspension of the alkali water solution in the alcohol alkali solution. This suspension being turbid until the water phase settles out, may be what he refers to as a precipitate. Approximately 35% of the total
20 alkali given in his examples goes into the water phase, and as such is not available for xanthogenation. His final product has a low Xanthate tenor, a small amount of Thio carbonate (Na_2CS_3) and a relatively large amount of free alkali. These impurities not only detract from the effectiveness of the Xanthate but promote its decomposition. Drying it is also made difficult, as driving off the excess water involves raising the material to a temperature much above the critical temperature of 50°C ., unless air drying is resorted to by means of a fan, which ipso facto is slow and expensive. The excess alkali, too, may exert an undesired influence in the flotation circuit.

30 The method suggested by you in our conversation of this morning appears to have considerable merit. As a matter of fact it combines the advantageous point of Rosenstein's dissolving of the alkali in water with the purity of the end product, as obtained by my method. If my memory serves me right it consisted in decanting the alcohol alkali solution to be used for the Xanthate manufacture from the alkali water phase, this later to be evaporated for its alkali recovery which can be returned to the circuit. The method employed by me in the laboratory consisted in dissolving the alkali in an excess of
40 with CS_2 . In both these methods the yield should be almost theoretical, and the Xanthate should be almost pure and free from contaminants.

Very truly yours,

CORNELIUS H. KELLER

Exhibit—D-72.

EXHIBIT D-72

ANACONDA COPPER MINING COMPANY
 Anaconda Reduction Department
 ANACONDA, MONTANA

July 26, 1923

Mr. E. H. Nutter, Chief Engineer,
 Minerals Separation North American Corp.,
 220 Battery Street,
 San Francisco, California.

10 Dear Sir:—

I have your letters of July 19th and July 21st, and am taking this opportunity to answer them.

Lewis arrived Monday afternoon.

Monday morning I went into the mill with "Z" reagent and operated one machine at the Concentrator; On Tuesday operated one machine at the Slime Plant; on Wednesday operated one machine at the Concentrator and one at the Slime Plant. I gave these machines my personal attention and thought that I was making a very satisfactory tailing. The assays did not bear out my observations.
 20 Monday, treating New Works Tailings, a tailings containing .2% copper was made, using normal acid and sludge plus approximately one-half pound of "Z" per ton. The section tailing assayed .24% copper. At the Slime Plant on Tuesday, using approximately $\frac{3}{4}$ pound of "Z" per ton, a tailing containing .49% copper was made. The assays on the other machines are in error and are being repeated today. They will probably run about .6% copper.

Lewis and I are disappointed in the results obtained so far. Nothing is being done today. As soon as yesterday's assays are available we will go over the data and see what is the best method to
 30 try for tomorrow. I will keep you advised of our results.

The quantities of "Z" used per ton, in the tabulation mailed you recently, referred to the "Z" salt as taken from the drums.

As near as I am able to determine, the special machine at the Slime Plant is operated at practically the same water level as are the other machines. We carry a high water level at all times. With reference to weir overflow, my experience has been that they cause more trouble and confusion than the valve control. Recently, while in the Timber Butte Mill, I noticed that the weir overflows on their lead machines were badly choked and that the water level control depended
 40 almost entirely on the valves. Their Superintendent admitted that this was poor operating. This fact bears out our observations here, and I am exceedingly doubtful about securing better results with a

Exhibit—D-72.

Exhibit—K-36.

weir overflow. No doubt skilled men would keep a constant level machine operating very satisfactorily. The ordinary workman, however, assumes that the constant level devices were placed there and will work satisfactorily without any attention.

We have found that the air finger machine is not satisfactory. The air fingers become clogged in about ten days or two weeks. After that the air distribution is very poor and results obtained deteriorate rapidly. It is almost impossible to clean the fingers sufficiently to get a good air distribution, consequently, the fingers would have to be
 10 renewed in not over two or three weeks. We have abandoned this machine.

Yours truly,

(Sgd.) BAYARD S. MORROW,
Supt. of Construction.

BSM-RL
 CC-Mr. Frick.

EXHIBIT K-36

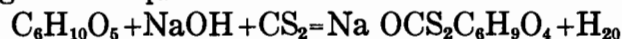
MINERALS SEPARATION NORTH AMERICAN CORPORATION
 OFFICE OF THE CHIEF ENGINEER
 220 BATTERY STREET, SAN FRANCISCO, CAL.

20 San Francisco, California, July 27, 1923.

Edward H. Nutter, Esq., Chief Engineer,
 Minerals Separation N. A. Corp.,
 229 Battery St., San Francisco, Cal.

Dear Sir:

Obviously the $C_2H_5O_4$ radical in alkali Xanthates can be replaced by certain carbohydrates such as Cellulose or Starch according to the equation—



A number of these alkali Cellulose Thiocarbonates or Alkali Cellulose
 30 Xanthates were made by me in March, 1923. All of these substances appear to be of a colloidal nature and on contact with metal salt solutions break up into Cellulose Hydrate, alkali Hydrate and Carbondisulphide. Free cellulose hydrate cannot be considered advantageous in the flotation circuit, Alkali Hydrate can be added in a less expensive and more effective form and the power of free carbondisulphide as a modifying agent is doubtful.

As the patent situation in regard to Viscose is rather involved (several covering Starch Xanthate alone) and they are not very stable

Exhibit—K-36.

Exhibit—D-73.

and of very complex composition, I did not pursue the investigation further than turning the material over to Mr. Lewis for flotation tests during May, 1923. These tests, not being promising, were only recorded in Mr. Lewis' personal notebook, indicating that the ground had been covered. These results have been duplicated by Mr. Littleford in accord with instructions.

10 The Cellulose employed by me was normal cellulose according to Cross and Bevan, Mr. Littleford's tests were made using both this material and the Viscose, made as per New York wire. As it is *difficult* to make a material free from Thiocarbonates or Alkali Hydrate a difference in the flotation results using the two materials was to be expected. Hence telegram to New York for pure material. The action of the stuff to the flotation is similar to that of the Alkali Thiocarbonates.

As it seems to me that an invasion of the Xanthate patent from the direction of the Thiocarbonates rather than from that of the Cellulose is to be apprehended, I beg to refer you to mine dated May 7th, 1923, re Thiocarbonates.

Yours very truly,

20

(Sgd.) CORNELIUS H. KELLER

EXHIBIT D-73

M.S.N.A.C.

San Francisco

Confidential

San Francisco, Calif.,

August 2, 1923.

EHN.m

Anaconda Copper Mining Company,
Anaconda, Montana.

Gentlemen:

30

ATTENTION: MR. EDWARD S. MORROW

In accordance with my letter of July 24th, wherein I stated that we were investigating the matter of the manufacture of the Xanthate, I now beg to advise you that there are two methods of manufacture which we have developed in the laboratory by means of which we have made Xanthates that are both of apparent promise.

I may say here that the method used by the Great Western Electro-Chemical Co., was to first put the potassium hydrate in solution with water before adding the alcohol, but we have deter-

Exhibit—D-73.

mined that this makes a salt containing only about 65% of available Xanthate, as the water keeps a considerable portion of the potassium hydrate in solution as such.

The first method which we have devised is to put the potassium hydrate in solution in alcohol, using an excess of alcohol, and then evaporating off the excess of alcohol for recovery before adding the carbon bi-sulphide. The carbon bi-sulphide is then added and the Xanthates precipitates in almost theoretical quantity. The merit of this method is that it saves heating and drying the salt after
 10 precipitation and thus avoids any danger of decomposition, and also saves wasting the alcohol, as stated. As you of course know, commercial alcohol contains approximately 5% of water. This water of course takes up and holds a certain amount of potassium hydrate, and it can be separated by standing, as it is considerably heavier than alkali alcohol. The refinement of this method, therefore, would be to separate the alkali water phase by gravity and decantation and evaporating it to dryness and reusing the hydrate. If this is done Xanthate of almost complete purity is obtained.

The second method is to grind the potassium hydrate with
 20 alcohol, using not more than the theoretical amount required, and preferably less. When this has been ground into a paste add it to a mixture of the theoretical amount of carbon bi-sulphide and the remainder of the alcohol if the full theoretical amount was not used in the first operation.

The alkali alcohol should be cooled to not over 45°C., and the carbon bi-sulphide with or without alcohol mixed with it, as the case may be, should be under agitation when the alkali alcohol paste is added to it. This precipitates the Xanthate also in practically theoretical quantities and no evaporation of alcohol is required.
 30 This second method possibly would be the most economical to use, and as stated we have made Xanthate in our laboratory hereby both methods.

The Great Western Electro-Chemical Co. have been revising their estimates of the cost of manufacture of the Xanthate, and now state that they can make and sell it here at a price of fifteen cents per pound of 100% Xanthate, that is the price would be based on 100% purity. As this most recent estimate is not based on either of the methods I have outlined herein, it is possible that they can still further shade this figure.

40 Mr. Henderson, their Vice President, offers to go to Anaconda to discuss the matter with you and lay all of his cards on the table, if you would be interested in having him do this. If you care to discuss the matter with him, therefore, I suggest that you let me know and I will so advise him.

Very truly yours,
 EDWARD H. NUTTER,
 Chief Engineer.

CC Mr. Frick.

Exhibit—K-37.

EXHIBIT K-37

MINERALS SEPARATION NORTH AMERICAN CORPORATION
 OFFICE OF THE CHIEF ENGINEER
 220 BATTERY STREET, SAN FRANCISCO, CAL.

San Francisco, California, Aug. 2, 1923
 CHK—M

Edward H. Nutter, Esq., Chief Engineer,
 Minerals Separation N. A. Corp.,
 220 Battery St., San Francisco, Calif.

10 Dear Sir:

RE: MANUFACTURE OF ALKALI XANTHATES

An alternative method of preparing these salts consists in grinding the alkali-hydrate with a small amount of alcohol not to exceed the amount necessary for subsequent Xanthate formation, in a suitable grinding apparatus. The finely ground alkali-alcohol mush is discharged into a closed vessel containing the remainder of the alcohol to make up to the theoretical if that amount has not been used in the first place and the carbon disulfide. This vessel must be provided with a stirring arrangement to keep the alcohol CS₂ mixture in agitation, a condensing arrangement to prevent escape of volatilized CS₂ or alcohol and a water jacket to prevent overheating or allow of the admittance of steam for heating if necessary.

The advantage for this method consists in working with theoretical quantities only.

Yours very truly,
 (Sgd.) CORNELIUS H. KELLER

MINERALS SEPARATION NORTH AMERICAN CORPORATION
 OFFICE OF THE CHIEF ENGINEER
 220 BATTERY STREET, SAN FRANCISCO, CAL.

30 San Francisco, California, Aug. 2, 1923.
 CHK—M

Edward H. Nutter, Esq., Chief Engineer,
 Minerals Separation N. A. Corp.,
 220 Battery St., San Francisco, Calif.

Dear Sir:

RE: MANUFACTURE OF ALKLI XANTHATES.

A low-grade alkali Xanthate can be prepared by using an alkali carbonate instead of alkali hydrate. A long continued digestion of the alkali carbonate with the alcohol-carbon disulfide mixture is essential, as the Xanthate strength of the resultant product is proportional to the length of exposure to the Xanthogenating reagents.

Yours very truly,
 (Sgd.) CORNELIUS H. KELLER

Exhibit—K-38.

EXHIBIT K-38

MINERALS SEPARATION NORTH AMERICAN CORPORATION
OFFICE OF THE CHIEF ENGINEER

220 BATTERY STREET, SAN FRANCISCO, CAL.

San Francisco, California, August, 2, 1923.

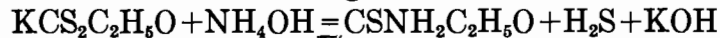
CHK—M

Edward H. Nutter, Esq., Chief Engineer,
Mineral Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

10 Dear Sir:—

XANPHOGENAMIDE OR XANTHANIDE

Above substance made according to the reaction:—



was investigated chiefly for the strategical position it occupies. As it holds a similar place in regard to the Alkali Xanthates as the Compound $\text{C}_{14}\text{H}_{18}\text{NH}_4\text{S}_3$, mentioned in my letter of June 24th, does to Thiocarbanilid, it may be advisable to cover it by a separate patent. Flotation tests made with the material in corroboration of indicative work done with it a few months ago, seem to show up
20 fairly well. It evinces good selective power between Ag, Pb and Zn and on Phelps Dodge table concentrates a rather difficult Cu. ore, a very clean tail and fairly good grade concentrate was obtained. In both cases the froth appeared of a good quality.

Yours very truly,

(Sgd.) CORNELIUS H. KELLER

MINERALS SEPARATION NORTH AMERICAN CORPORATION
OFFICE OF THE CHIEF ENGINEER

220 BATTERY STREET, SAN FRANCISCO, CAL.

San Francisco, California, August 2, 1923.

CHK—M

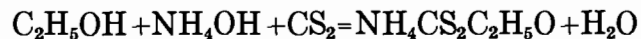
30

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

Dear Sir:

RE: AMMONIUM XANTHATE.

Completing the subject of Alkali Xanthates an Ammonium Xanthate was made in accordance with the reaction:—



This substance is not very stable. Flotation tests made with it
40 corroborative of former work indicate that it gives a good selection between Pb and Zn. Its action on Ag though is not as favourable as Potassium or Sodium Xanthate.

Exhibit—K-38.

On Phelps Dodge Cu. table concentrates it did not evince any selective power. In both cases of Ag. Pb, Zn ore and Cu. ore the froth was fragile.

Yours very truly,
(Sgd.) CORNELIUS H. KELLER

MINERALS SEPARATION NORTH AMERICAN CORPORATION
Office of the Chief Engineer
220 Battery Street, San Francisco, Cal.
San Francisco, California, August 2, 1923.
CHK-M

10

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

Dear Sir:

RE: XANTHIC ACID.

Xanthogenic acid ($CS_2C_2H_5OH$) is best prepared from Alkali Xanthate by addition of dilute HCl. It is a heavy pungent smelling oil, which decomposes on exposure to air at 25° and also but more slowly, in contact with water, it is but slightly soluble in the latter
20 menstrum. The inflamability, strong odor and difficulty of application preclude its practical application as a flotation reagent. Being the basis of the Alkali Xanthates, however, it may be considered advisable to have it covered specifically. A couple of flotation tests were made with the material, which demonstrated its selectivity, without showing any stronger action than Alkali Xanthates.

I am attempting to produce the substance by direct action between Alcohol and Carbondisulfide, so far without great success, chiefly for the reason that I am greatly pressed for time. While the material does not at present seem to offer much possibility, I shall
30 continue the investigation, which may yet bring to light some interesting facts.

Yours very truly,
(Sgd.) CORNELIUS H. KELLER

Exhibit—K-31.

Exhibit—D-80.

EXHIBIT K-31

xx
CC NYW U N L
EHNmSan Francisco, Calif.,
August 3, 1923.C. P. Lewis,
c/o Montana Hotel,
Anaconda, Montana.

- 10 Keller has determined that reagent made by Great Western method without centrifuging which was method used in making shipment under test contains other soluble sulphides which in acid circuit may tend to weaken action of major substance by evolution of hydrogen sulphide.

E. H. NUTTER.

Charge E. H. Nutter.

EXHIBIT D-80

Anaconda, Montana,
August 4, 1923.

- 20 E. H. Nutter, Esq., Chief Engineer,
Minerals Separation N.A. Corporation,
220 Battery Street, San Francisco.

Dear Sir:—

- Received your wire of yesterday this noon after I had spent the morning checking up on the reagent now being used. My finds confirm those of Mr. Keller in regard to the liberation of H_2S with the material on hand and no H_2S from the material made here, using the excess alcohol method of manufacture. I also find that the small amount of H_2S has a decided deadening effect on the copper mineral (chal-
30 cocite) and the opposite effect on the pyrite and chalcopyrite.

As I remember the tests, made in the San Francisco office, using thio-carbonate as a reagent, showed a non-selective action on the same minerals, while it has been proven that the Xanthate has the opposite effect, e.g. the selection of chalcocite over pyrite. An aqueous solution of the material now being used shows a decided orange colour, the distinctive colour of thiocarbonate, whereas properly made Xanthate shows a decided lemon yellow.

- 40 There is no doubt an excess of Xanthate in the material on hand, but in my opinion the amount of thio-carbonate present is sufficient to partially mask the Xanthate effect and cause the tailing in the present tests to run from .1 to .2 higher than what was within reason to expect. Microscopic examination of the tailing has shown that the

Exhibit—K-80.

Exhibit—D-94.

small amount of sulphide left was almost entirely chalcocite rather than pyrite, as was the case in the former tests. The total amount of sulphides present has been on the whole *less* than before, which fact naturally led to a sampling and assaying investigation.

In view of the above, I would recommend that the Great Western replace this lot of Xanthate with Xanthate manufactured according to our original outline as soon as possible.

On my arrival Mr. Morrow said he had ordered this lot through the N.S. Co. to insure getting the proper material and was dependent
 10 on us to insure him against substitution. In view of his present attitude of co-operation and his personal wish to see the Z reagent in use here I can see no other course open but to explain the situation to him in detail and, on account of the limited time, to rush a 1000 pound lot of known quality Xanthate here to make the Section tests they had outlined, to prove the value of this as a flotation reagent.

In the meantime I would suggest that the present tests be discontinued and that I go to Wallace until the other material arrives.

The results from yesterday's test on No. 10 machine gave a
 20 tailing of .39 total Cu. against a .79 for the Section. This, however, was only by use of an excessive amount of pine oil that would be charged against us in balancing any benefits.

Kindly advise me as soon as possible what action you wish me to take.

Yours very truly,

C. P. LEWIS

EXHIBIT D-94

POSTAL TELEGRAPH : COMMERCIAL CABLES

T E L E G R A M

30

1923 AUG 6 AM 1 20

A12 CH 159 COLL NL

ANACONDA MONT 5

E H NUTTER

220 BATTERY ST SANFRANCISCO CAL

REPLYING YOUR WIRE OF THIRD FIND EVOLUTION OF HYDROGEN SULFIDE FROM MATERIAL USED EFFECTS RECOVERY OF CHALCOCITE STOP COLOR OF SOLUTION ALSO INDICATES PRESENCE OF CARBONATE IN QUANTITY SUFFICIENT TO HINDER COMPLETE RECOVERY STOP ALL TESTS SHOW TAILORING ONE TO TWO TENTHS HIGHER THAN REASONABLY

Exhibit—D-94.
Exhibit—D-95.

EXPECTED WITH ALL OTHER FACTORS ELIMINATED STOP STRONGLY
RECOMMEND THAT GREAT WESTERN REPLACE DEFECTIVE MATERIAL
WITH LIKE AMOUNT MADE ACCORDING TO ORIGINAL PLAN OF MANU-
FACTURE THAT PRESENT

BESTS BE DISCONTINUED AND THAT I RETURN HERE FROM WALLACE
ON ARRIVAL OF NEW MATERIAL AND COMPLETE TESTS AS ORIGINALLY
OUTLINED STOP FEEL IT ADVISABLE TO MAINTAIN VESENT SPIRIT OF
COOPERATION BY EXPLAINING IN DETAIL DEFECTS AND REQUEST
PERMISSION TO DO SO STOP WILL BE NECESSARY TO RUSH FRESH SHIPMENT
10 AS DEFINITE DECISION WILL BE MADE SOON STOP TAILINGS FOR FRIDAY
POINT THREE NINE AGAINST POINT SEVEN NINE REGULAR HOWEVER
OIL CONSUMPTION EXCESSIVE STOP KINDLY SOON AS POSSIBLE ON
ABOVE AND WIRE ONE HUNDRED EXPENSE ADVANCE

C. P. LEWIS.

EXHIBIT D-95

Postal Nite
EHNm

CC NY

San Francisco, Calif.,
August 6, 1923.

20

C. P. Lewis,
c/o Montana Hotel,
Anaconda, Montana

Replying your wire please discuss matter frankly with Anaconda
officials and suggest that tests be stopped until supply of new material
arrives which is now being manufactured and will be shipped by
express in two hundred pound lots as made Stop Be sure and see
McCarthy of Hecla at Wallace extend our sympathy and proffer our
assistance and advice in designing or working out flow sheet for new
30 plant Stop At Morning mine and elsewhere keep new reagent out of
your conversation.

E. H. NUTTER

Exhibit—P-101.

EXHIBIT P-101

Charge E. H. Nutter.

Anaconda, Montana,
August 7, 1923.E. H. Nutter, Esq.; Chief Engineer,
Minerals Separation N. A. Corporation,
220 Battery Street, San Francisco.

Dear Sir:

Received your wire of yesterday this morning and explained in
10 detail the situation and cause of high tailings to Mr. Morrow. He
agreed that, while the tests just made, show a saving over their
present operations, they had not come up to the standard set by the
previous tests, and as he explained it, "now is a better time to find
out about any defects in manufacturing than later when it might
mean a car load loss." This would indicate his still favorable attitude,
in spite of the present tests.

I have an appointment with Mr. Laist for this evening and will
go over the matter with him and make a few necessary suggestions
regarding and sampling, so that our tests on the new lot will show
20 accurate results.

I have found quite an error in the assays here and as a check I
managed to get half the tailing samples from the last four runs that
I would like to have Mr. Keller assay for total Cu and Ox Cu. These
were mailed from here today.

Mr. Broan of Stearns-Roger is back on the job to test out his
machine with carbanalid. The 5 - 1 has been converted into a Brown
machine, so arranged to give any required number of agitators for
pre-agitation. He will first try the New Works tailings, but is rather
dubious of success on them, due to the poor showing made by Mr.
30 Bragg on this material. Bragg's tailings still average .30 total Cu.

Brown says that thio has fallen flat at Neihart; their recovery is
now about 60%. It might be well to get in touch with Mr. Young, as
they are evidently contemplating changes in their present mill. He
also says that the U.S. Smelting at Midvale are or would be active
prospects provided they could arrange a royalty on the tonnage
treated by flotation rather than total mill tonnage.

I will leave tomorrow for Wallace and have requested Mr.
Morrow to wire me as soon as a sufficient quantity of the new ship-
ment of Xanthate has arrived to start testing.

40

Very truly yours,

C. P. LEWIS

Exhibit—D-96.

EXHIBIT D-96

ANACONDA COPPER MINING COPPER
Anaconda Reduction Department
ANACONDA, MONTANA,
August 8, 1923.

Mr. E. H. Nutter, Chief Engineer,
Minerals Separation North American Corp.,
220 Battery Street,
San Francisco, Cal.

10 My dear Mr. Nutter:

I am in receipt of your letter of August 2nd addressed to the Anaconda Copper Mining Company, attention myself.

You have, no doubt, heard often from Mr. Lewis and are aware of the results he and I have been able to obtain from the shipment of Z. recently ordered through you. Mr. Lewis now believes that the Z. furnished by the Great Western Electro Chemical Company is not a pure potassium xanthate. Mr. Lewis further believes that some potassium thio-carbanate has been formed and that this salt in the dilute acid solution produces small amounts of hydrogen sulphide gas and that
20 this gas will hinder the flotation of chalcocite.

Mr. Lewis was in the office this morning and told me that you were making arrangements to replace the 1000 pounds of Z. recently ordered with another 1000 pounds of Z. made according to your original formula. This Z., as I understand it, would be the same material as that used in the tests when you were in Anaconda about a month ago. We feel that the sampling methods used in the test recently conducted are accurate, that the assay is quite reliable and that the machine operation was uniform at all times. The sulphide content of the tailing, as panned, was low. The copper content of the
30 tailing was high, as the assay shows. It is entirely possible that this is due to chalcocite; we apparently cleaned up practically all of the iron, although no iron determinations were made on the tailing. I showed your letter to Mr. Laist and he said that we had better await results obtained from the 1000 pounds of Z. that will be furnished to replace the 1000 pounds that Mr. Lewis thinks is faulty.

With regard to Mr. Henderson coming to Anaconda, I believe that a letter from you to Mr. Laist reviewing briefly the status of Z. at Anaconda and suggesting to him that Mr. Henderson is willing to come to Anaconda and discuss the matter with him will be the proper
40 method of procedure.

I believe that in matters of confidential import it would be just as well for you to address such letters to me personally or to Mr. Laist, instead of to the Anaconda Copper Mining Company, because your

Exhibit—D-96.

Exhibit—K-34.

letter of August 2 was opened by somebody in the Purchasing Department and then sent to me.

Mr. Lewis left today for Wallace and will be back in Anaconda by the time the first shipment of Z. arrives. We will then make further tests at the Slime Plant and later on at the Concentrator on new work's tailings.

Yours very truly,

(Sgd.) BAYARD S. MORROW

Supt. of Concentration.

10 BSM: IS

EXHIBIT K-34

MINERALS SEPARATION NORTH AMERICAN CORPORATION

OFFICE OF THE CHIEF ENGINEER

220 BATTERY STREET, SAN FRANCISCO, CAL.

San Francisco, Calif.,

August 9, 1923

EHNm

Minerals Separation N. A. Corporation,
61 Broadway,

20 New York.

Gentlemen:

You have doubtless noted in the copies of letters and telegrams from Lewis that I have sent on to you that the last lot of 1000 pounds of xanthate has not given as good results as it should have given at Anaconda. The results are better than they could get otherwise, but still they are not good enough.

This last lot was made by the same method as was used in making the original lot, except that it was not centrifuged. Our tests in the laboratory indicated that the centrifuging made no difference and we so advised the Great Western Company, as they did not have a centrifuge of their own and it would have delayed matters some time for them to have obtained one.

It appears from chemical investigation made that the xanthate made by Dr. Rosenstein's method contains some thiocarbonate. This is reduced by centrifuging, but not eliminated. When this is added into an acid circuit some hydrogen sulphide is evolved which tends to weaken the effect of the xanthate itself.

I am now having the Great Western Company make up a 500 pound lot of pure xanthate, made by different method which I rather hope will give the best results yet obtained.

Exhibit—K-34.

Exhibit—K-32.

I have also advised the Anaconda Company of a method of purifying the remainder of the 1000 pounds, so that it can be used and give good results.

Very truly yours,
 (Sgd.) EDWARD H. NUTTER
 Chief Engineer

EXHIBIT K-32

San Francisco, Calif.,
 August 9, 1923

10

EHNm

Great Western Electro-Chemical Co.,
 9 Main Street,
 San Francisco, Calif.

Gentlemen:

ATTENTION DR. L. ROSENSTEIN

I beg to confirm my conversation this morning over the telephone with Dr. Rosenstein, as follows: The method which we wish to have used in making up the new lot of 500 lbs. of potassium xanthate is as follows:

20 Dissolve the proper amount of potassium hydroxide for the batch that is being made up in an excess of alcohol. The alcohol should be in sufficient excess so that the mixture will be liquid at the temperature resulting. This is then to be allowed to stand until the solid impurities and such of the water as is present and will do so have a chance to settle out. The supernatant liquid, consisting of the alkali alcohol phase is then to be syphoned or ladled off and mixed with the proper amount of carbon bi-sulphide. The water phase following the mixture of potassium with alcohol, which of course will consist almost entirely of a solution of the hydrate in
 30 water, should not be reused in making this particular shipment nor should the mother liquor resulting from centrifuged xanthate crystals, although it will consist mainly of alcohol.

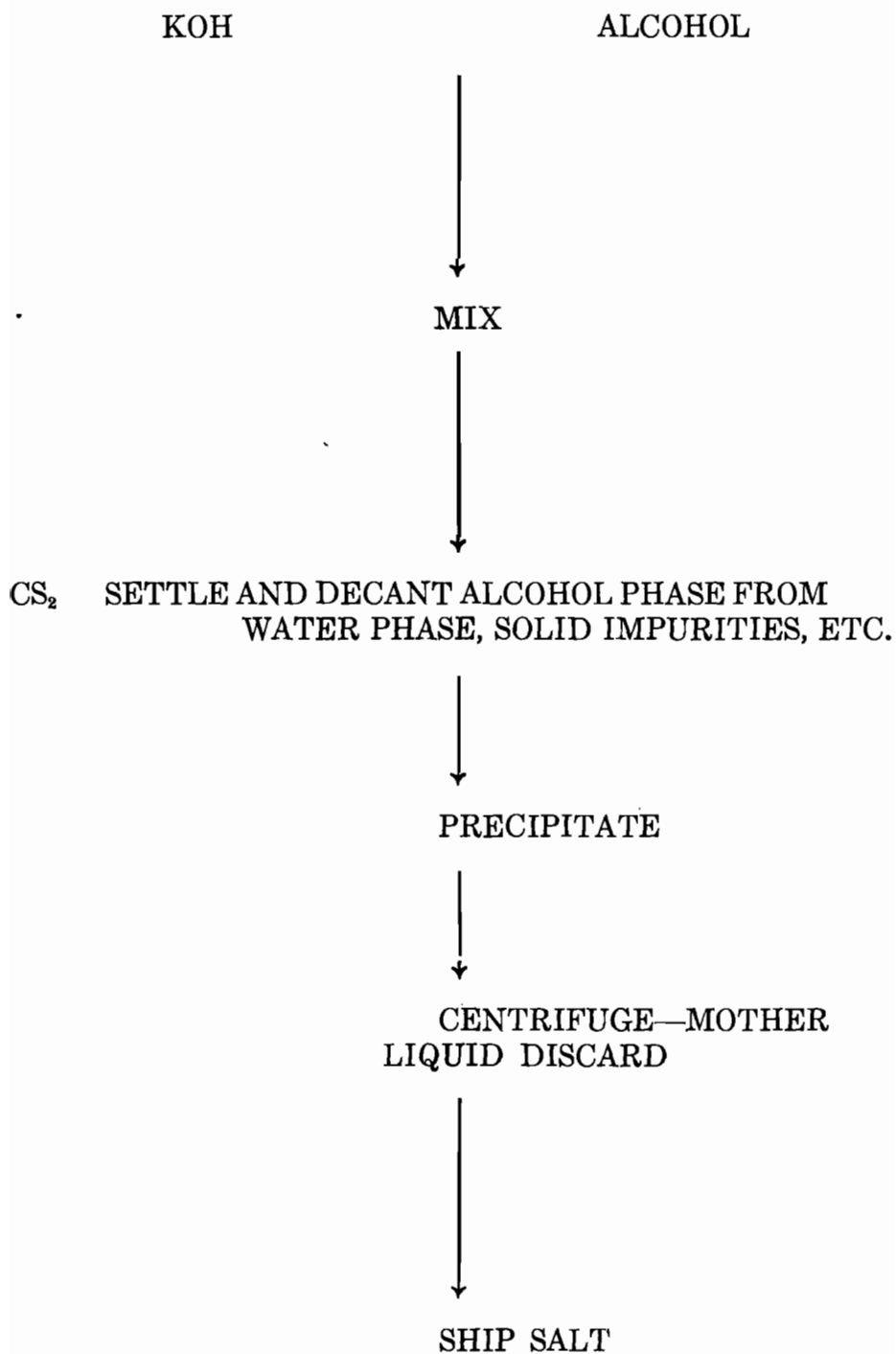
The indications of the test work at Anaconda are that the purer the xanthate the better the results, so for this particular shipment we want to give them material of extra good quality, and I think that the same will result from the method indicated.

For further clearness I outline the method in the form of a flowsheet which I enclose herewith.

40 You will please send us as soon as possible a sample of each batch made.

Very truly yours,
 EDWARD H. NUTTER
 Chief Engineer

Exhibit—K-32.

CC Great Western Electro-Chemical Co.,
Pittsburg, California

Exhibit—K-33.

EXHIBIT K-33

San Francisco, Calif.,
August 9, 1923
EHNm

Anaconda Copper Mining Company,
Anaconda, Montana

Gentlemen:

ATTENTION MR. BAYARD S. MORROW

10 In regard to the 1000 pound shipment of xanthate which did not give as good results as expected, you have doubtless discussed the matter with Mr. Lewis, and he has advised you that we are having a further lot made up which will be shipped to you.

The first lot shipped was made by the same method (which was a method devised by the Chief Chemist of the Great Western Electro-Chemical Co.) as the 1000 pound lot which you ordered, except that the first lot was centrifuged and the second lot was not. The Great Western Company had only borrowed a centrifuge for making the first lot and were unable to get it quickly when you ordered the second lot.

20 We made tests here on your slimes with the uncentrifuged salt and found no difference in effect as compared with the salt which had been centrifuged, so I advised the Great Western Company that they could ship you the uncentrifuged salt. It appears, however, that by the method which they used a certain amount of thio-carbonate is formed. This is reduced by that which goes out in the mother liquid in centrifuging. The thio-carbonate in acid circuit of course forms a certain amount of hydrogen sulphide which tends to weaken the effect of the xanthate.

30 We are now having the Great Western Company make 500 pounds of Xanthate by the method which we described to you under date of August 2nd, in which the water in the alcohol is eliminated by gravity after adding the potassium hydrate and before xanthate is precipitated with carbon bi-sulphide. Xanthate made in this way will be centrifuged, and will be shipped to you and should prove I think superior to the lot with which we ran tests.

40 The material which you already have can be purified by the following method. Dissolve in alcohol, using one-half to three-quarters, by weight, of alcohol as compared with the xanthate, and warming to about 40°C. Keep at this temperature until all superfluous water, carbonates, thio-carbonates and alkalies have settled out, and decant off the supernatant liquid. This should be a solution of xanthate chiefly in alcohol and can be used directly in your flotation operations. If you do not want to do this, I suggest that you ship that material back to the Great Western Electro-Chemical Co. and

Exhibit—K-33.
Exhibit—D-97.

I will have them do it and re-crystalize the xanthate and ship it back to you. I may add that there is a progressive double decomposition which takes place in the salt which you have which results in the formation of thio-carbonate, and therefore the sooner this material is purified the better it will be.

The Great Western Company will not be able to have a centrifuge installed until the 13th, but they should be ready to ship the additional 500 pounds by the 15th or the 16th of August, which I trust will be satisfactory.

10

Very truly yours,

EDWARD H. NUTTER
Chief Engineer.

CC Mr. Frick.

EXHIBIT D-97

MINERALS SEPARATION NORTH AMERICAN CORPORATION
OFFICE OF THE CHIEF ENGINEER
220 BATTERY STREET, SAN FRANCISCO, CAL.
San Francisco, Calif.,
August 30, 1923.

20

EHNm

Minerals Separation N. A. Corporation,
61 Broadway,
New York,

Gentlemen:

RE: ANACONDA

Yesterday after receiving the wire from Lewis giving the very good results that were being obtained, I received another wire from him, as follows:

30

“Recent developments indicate your presence here soon advisable.”

to which I replied:

“Please wire me indication of reasons making my presence there soon advisable and whether you think Allen who is at Spokane could substitute.”

At the same time I wired Allen, as follows:

“Please keep me closely advised of your movements as it may be advisable for you to make early visit to Anaconda.”

Exhibit—D-97.
Exhibit—D-74.

This morning I received Lewis' reply, as follows:

“Reason for request was to bring certain pertinent facts before management before decision was made: Allen satisfactory”

which indicates that there were certain facts which he did not want to put on the wire at Anaconda. I therefore wired Allen as follows:

10 “Developments at Anaconda make it advisable for you to proceed there forthwith, consult with Lewis, and lay before management certain facts of which Lewis will inform you, completing your Washington and Idaho itinerary afterwards. Please wire reply.”

and have his reply, as follows:

“Your two telegrams received. Have wired New York for instructions, and if they agree with you I will proceed immediately Stop In Absence of further word from me you may take it for granted that I leave here tonight.”

20 I haven't the least idea as to what it is that Lewis refers to, but it will of course develop soon. He has apparently learned something that is going on up there which he feels hardly strong enough to himself present to the management, but which he considers important, else he would not suggest that I make the trip from here to Anaconda for the purpose. As Allen is so near at hand it seemed quite the logical thing for him to run over there for the purpose instead of for me to go from here.

Very truly yours,

EDWARD H. NUTTER
Chief Engineer.

EXHIBIT D-74

30 MINERALS SEPARATION NORTH AMERICAN CORPORATION
OFFICE OF THE CHIEF ENGINEER
220 BATTERY STREET, SAN FRANCISCO, CAL.

Anaconda, Montana
September 4, 1923.

Dr. S. Gregory, President,
Minerals Separation N. A. Corp.,
61 Broadway,
New York City.

Dear Dr. Gregory:—

40 I arrived here this morning and went over the situation with Allan and Lewis. It appears certain that Callow left here last Thursday with it practically agreed upon that the Anaconda Com-

Exhibit—D-74.

pany would install his machines and use his process on the slimes. It appears that this conclusion had been reached with a very little consideration given to our figures and a decided ignorance of them on the part of Mr. Laist.

Allen had an interview Saturday with Laist and pointed out to him the results that had been obtained using Xanthate, and there appears to have developed since then a renewed interest on the part of Messrs. Morrow and Frick in doing further testing with Xanthate.

10 This afternoon, Allen, Lewis and myself had a long interview with Messrs. Morrow, Frick and Klepetko, and later we all had a long talk with Mr. Laist. Messrs. Damon and Brown, of the Stearns-Roger Company also had a long session with Laist before ours. The upshot of the interview is that there will be two weeks more of testing as soon as we can get the necessary additional Xanthate. These tests will be strictly competitive under rigidly parallel conditions. Mr Morrow exhibited considerable temper when we were discussing the conditions of the tests, and we gathered that he was displeased at there being any continuation of the tests.

20 Mr. Laist suggested during our talk that he would be willing to give us an order for Xanthate at 25c a pound to run the slime plant for six months, this being the period he estimated that it would take to install the Callow apparatus. I asked him if they would forget the Callow apparatus if our results were superior in the two weeks' tests. He said they would like to forget it. Later in the conversation, he said they would not want to be obligated to use this supply of Xanthate after the expiration of our first patent. Mr. Laist launched upon something of a tirade against our Kerosene Acid Sludge patent and also later against Chapman's Grinding-In-Oil patent, and said it would be absurd and unethical for us to attempt
30 to collect royalties from the Anaconda Company for the use of those patents, or any detail patents, and that he would fight it to the last ditch if it took a million dollars. He said further that our Solution Patent ought to be dominated as to its termination by our first patent, although he admitted that you had denied this in the original negotiations. He is familiar in its entirety, I think, with the agreement with the Jackling and Lewisohn companies, and quite appreciates the strategic advantages thereby inuring to the Anaconda Company in negotiations with us.

40 I suggested that we might arrange by guaranteeing the amortization of a plant to manufacture their requirements of Xanthate, as Mr. Laist had stated that this would greatly simplify the proposition and make them consider much more favorably the use of it. All this may mean, however, is that it will enable them to cut their losses on the slimes before they can install the Callow plant. At the same time, if the Xanthate results are enough better than the Callow results, the question may be settled then and there.

Exhibit—D-74.

Exhibit—D-98.

I therefore wired you recommending that we make such a guarantee to the Great Western Company, because I think that enough of our licensees will use Xanthate to amortize the plant, and we will be out little or nothing at the end of the three-year period that I asked the Great Western Company to use in estimates. It is possible, of course, that the Great Western Company may not have the ready cash to use in building the plant immediately, and ask us to advance it, which I think we might do.

10 There was, of course, much in our talk to-day that I cannot well put into a letter, as it would expand it unduly, and I think this gives the leading facts.

Very truly yours,

EDWARD H. NUTTER.

EXHIBIT D-98

SAN FRANCISCO, CAL., SEPT 5, 1923

E. H. NUTTER
HOTEL MONTANA
ANACONDA

20 ON THE BASIS OF CONTRACT FOR QUANTITIES MENTIONED OVER A PERIOD OF AT LEAST ONE YEAR CAN SUPPLY EIGHT HUNDRED POUNDS WATER FREE Z AT EIGHTEEN CENTS PER POUND FOR PITTSBURGH DITTO FIFTEEN HUNDRED POUNDS DAILY FIFTEEN CENTS PER POUND DITTO TWENTY FIVE HUNDRED POUNDS DAILY FOURTEEN CENTS STOP LIMITED TIME AVAILABLE DOES NOT PERMIT US TO INVESTIGATE DELIVERY NECESSARY EQUIPMENT SUFFICIENTLY TO GUARANTEE DELIVERY AT RATE OF EIGHT HUNDRED POUNDS DAILY BEGINNING OCTOBER FIRST BUT WOULD OF COURSE MAKE EVERY EFFORT TO DO SO AND BELIEVE IT COULD BE ACCOMPLISHED STOP IN ANY EVENT COULD START SHIP-
30 MENT AT LESSER RATE OCTOBER FIRST STOP ALL ABOVE PRICES INCLUDE THREE QUARTERS CENTS PER POUND FOR AMORTIZATION INVESTMENT OF FIFTEEN THOUSAND DOLLARS WHICH MEANS INVESTMENT WILL BE AMORTIZED COMPLETELY WHEN ONE THOUSAND TONS HAVE BEEN SHIPPED THEREAFTER PRICE WOULD BE REDUCED THREE QUARTER CENT PER POUND STOP ALL ABOVE PRICES BASED ON NON GUARANTEED PRICES FOR TWO INGREDIENTS BUT BELIEVE WE CAN ACTUALLY BUY AT LOWER THAN PRICE ESTIMATED AND WILL OF COURSE GIVE YOU OR YOUR CUSTOMER BENEFIT OF SUCH SAVING IF MADE

GREAT WESTERN ELECTRO CHEMICAL CO.

Exhibit—D-76.

EXHIBIT D-76

ANACONDA, MONT., SEPT. 6, 1923

GREAT WESTERN ELECTRO CHEMICAL CO
 9 MAIN STREET,
 SAN FRANCISCO

MR HENDERSON I HAVE ASSUMED IN MY TELEGRAMS THAT MATERIAL REFERRED TO IN QUOTATIONS AND OTHERWISE WILL BE SUBSTANTIALLY LIKE MATERIAL NOW BEING RECEIVED EXCEPT THAT Z IN MOTHER LIQUOR WILL BE RECOVERED STOP I WISH YOU PERSONALLY WOULD
 10 GO OVER PROPOSED METHOD OF MANUFACTURE WITH OUR CHEMIST MR KELLER AND HAVE HIM APPROVE SAME AND THEN OF COURSE YOU WILL NOT CHANGE METHOD WITHOUT OUR KNOWLEDGE AND APPROVAL STOP THIS REFERS TO FIFTEEN HUNDRED POUND LOT AND LARGER QUANTITY JUST ORDERED STOP AS FIFTEEN HUNDRED POUND LOT IS FOR CRITICAL COMPETITIVE TEST PURITY OF THIS MATERIAL FROM INTERFERING SUBSTANCES IS ESSENTIAL STOP PLEASE SHOW THIS TELEGRAM TO KELLER

E. H. NUTTER

EXHIBIT D-75

20

ANACONDA, MONT., SEPT. 6, 1923

GREAT WESTERN ELECTRO CHEMICAL CO
 9 MAIN STREET
 SAN FRANCISCO

I NOW HAVE FIRM ORDER FOR ONE HUNDRED TWENTY FIVE THOUSAND POUNDS Z DELIVERIES TO BEGIN NOT LATER THAN OCTOBER FIRST AT RATE OF NOT LESS THAN EIGHT HUNDRED POUNDS DAILY WITH RIGHT RESERVED FOR PURCHASE TO REDUCE AMOUNT EIGHT HUNDRED POUNDS FOR EACH DAYS DELAY IN BEGINNING DELIVERIES STOP FIRST SHIPMENTS WILL BE MADE BY EXPRESS STOP PRICE TO BE ARRANGED
 30 BETWEEN YOU AND ME BUT TO BE IN HARMONY WITH QUOTATIONS WIRED ME STOP YOU HAD BETTER ACCUMULATE STOCK OF HAND MADE MATERIAL AGAINST OCTOBER FIRST DELIVERY AND PLAN TO MANUFACTURE AT MAXIMUM RATE POSSIBLE IN PROPOSED PLANT AS THIS IS SIMPLY A TRIAL ORDER WIRE ANSWER

E. H. NUTTER

Exhibit—D-77.

EXHIBIT D-77

GREAT WESTERN ELECTRO-CHEMICAL COMPANY
9 MAIN STREET, SAN FRANCISCO, CALIFORNIA

Minerals Separation Company,
San Francisco, Calif.,

Sept. 15, 1923.

Gentlemen:—

ATTENTION MR. KELLER

In response to your inquiry as to the manner in which we manufacture the Potassium Xanthate now being shipped to Anaconda, 10 we are pleased to give you the following outline of method followed.

Into a steam jacketed vessel with an agitator are placed 60 gallons of alcohol and a sufficient quantity of Potassium Hydrate so that when equilibrium is reached there will be some solid Potassium Hydroxide left. The mixture is then heated and agitated, the alcoholic vapors coming off being condensed in a reflux condenser and returned to the vessel. When saturation has been reached the entire contents are drawn off in a settling tank and allowed to settle for 24 hours. The alcoholic layer is now carefully separated from the aqueous layer below and transferred to another vessel. A sample 20 of the alcoholic layer is sent to the laboratory for analysis. From the analysis of the alcoholic layer in the laboratory a calculation is made of the amount of carbon bisulphide necessary for complete reaction. This quantity of carbon bisulphide is then added slowly and with continuous agitation. When all of the carbon bisulphide has been introduced the mixture is transferred to crystalizing vessels where it is allowed to cool and crystalize for 24 hours. Crystals are then centrifuged and after being dried in this manner are placed on trays where they are kept for 12 hours, drying, before packed and shipped.

30 The mother liquor produced in the process is not being recovered but dumped down the sewer.

Very truly yours,

GT. WESTERN ELECTRO CHEMICAL CO.

C. T. HENDERSON
Sales Manager.

OTH:J

Exhibit—D-78.
Exhibit—K-39.

EXHIBIT D-78

POSTAL TELEGRAPH—COMMERCIAL CABLES
TELEGRAM

1923 SEP. 27 PM 4 24

21280H 26

ANACONDA MONT 27

DR S GREGORY

SIXTY ONE BROADWAY NEWYORK NY

MANAGER TOLD ME TODAY WE HAD WON BY SEVEN TO ELEVEN CENTS
10 PER TON ACID CIRCUIT AND FIVE CENTS LIME CIRCUIT NO NEW CON-
STRUCTION NOW CONTEMPLATED

E. H. NUTTER

EXHIBIT K-39

San Francisco, California, Oct. 15, 1923.
CHK—M

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Cal.

Dear Sir:—

20 In reply to Mr. B. Morrow's letter dated 11th inst., regarding ap-
parent quality variations of Xanthate shipped to Anaconda.

I beg to state that the alcohol insoluble is due to formation of
oxidation products by decomposition of the Xanthate due to over-
heating in the dryer. This troublesome phase may be overcome in
two ways, by washing with carbon tetrachloride previous to drying
or by desicating in a vacuum dryer. As it will be some time before
a vacuum dryer can be installed, resort will be had to the carbon
tetrachloride wash. The oxidation products formed lower the grade
of the material but are apparently inert, metallurgically speaking.

30 Canary yellow appearance denotes insufficient drying. This
will of course be done away with as soon as the dryer troubles have
been eliminated. The uncombined alcohol discolored by mother
liquor causing this yellow appearance tends likewise to lower grade
but does not interfere in the flotation circuit.

The gray discoloration is due to new reaction vessels and to above
decomposition products.

So far samples corresponding to shipments under large contract
have come up to specifications as regards being free from deleterious
substances. Grade of course has varied to a considerable extent.

40

Yours very truly,

CORNELIUS H. KELLER

Exhibit—K-39.

EXHIBIT K-39

San Francisco, California, Oct. 17, 1923.

CHK—M

Edward H. Nutter, Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Cal.

Dear Sir:

RE: ALKALI XANTHATE ANALYSIS.

Aside from determining CS_2 equivalent, which has been covered
10 in detail by letter, alcohol insoluble determination which Messrs.
Anaconda appear likewise to be familiar with, there remains the
qualitative determination of H_2S likely to be evolved in an acid
flotation circuit from the action of the acid upon Xanthate impuri-
ties, such as thiocarbonates or alkali sulfides. Exhaustive tests
carried out in this laboratory have shown that it is practically
impossible to determine the small quantities of thiocarbonates,
alkali, sulfides, etc., contained in the Xanthate quantitatively.

As the qualitative test for these substances is comparatively
simple, moreover as the material giving strong positive indications
20 of thiocarbonates, etc., does not give good results in an acid
flotation circuit and conversely, the material giving only slight
indication of impurities gives good results in an acid flotation
circuit, the following test was adopted:—

Dissolve 10 gr. Xanthate in 50 cc. dewatered denatured
cold alcohol, shake occasionally, let stand for 1 hour and
filter through coarse filter paper. To solution add 100 cc.
cold distilled water and 30 cc. cold H_2SO_4 solution (25%
by volume) insert test paper moistened with Pb. acetate
solution, taking care not to touch liquid or sides of flask.

30 Only a slight discoloration, if any, should occur. If black-
ened at once a sufficient amount of impurities is indicated to make
material unsuited for acid circuit work.

Yours very truly,

CORNELIUS H. KELLER.

Exhibit—K-39.

EXHIBIT K-39

C O P Y

San Francisco, California, Oct. 29, 1923.

CHK—M

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

Dear Sir:

10 Complying with instructions, I arranged with the Chief Chemist of the G. W. E. Ch. Co., for a method of analysis of alkali xanthates of commercial grade manufactured with excess alcohol.

The samples taken at G. W. and submitted to this laboratory are fairly representative of the shipments made to Anaconda. Differences in results between Anaconda and ourselves are due to the details of analytical procedure rather than to the sampling. As these differences are not large enough to affect seriously either price or working efficiency of the material I beg to suggest that in the preliminary settlement the average between Anaconda and San Francisco results be accepted. Should larger differences occur umpiring should be resorted to. The umpire sample might well be a half and half mixture of Anaconda and Great Western samples.

METHODS OF ANALYSIS

No. 1. The weighed material is subjected to a vacuum over sulphuric acid or Phosphorus pentoxide for 12 hours. Heating to 105° for one hour as suggested by Anaconda, results in slight decomposition, due to oxidation.

No. 2. The alcohol insoluble is determined by extracting a weighed quantity of material in a Soxhlet extractor 6 times with Methyl alcohol. As this substance can be obtained free from water and its solvent action on Xanthate is greater, it was agreed upon in preference to dehydrated denatured alcohol.

No. 3. The total S equivalent can be determined in the dry material from Test No. 1 by any of the conventional methods.

No. 4. The S equivalent of the alcohol insoluble can be determined from the alcohol extracted material from test No. 2 by any of the usual methods.

C O P Y

E.H.N. Re: Methods of Analysis.

10/29/23.

No. 5. Available alkali xanthate equals total S equivalent minus alcohol insoluble S equivalent.

No. 6. H_2S evolved in an acid flotation circuit to be determined qualitatively by dissolving a sample of the material in cold alcohol (either methyl or denatured dehydrated Ethyl alcohol) filtering from insoluble, diluting filtrate with H_2O , acidifying and inserting test paper as per my note dated October 17th, 1923.

10 In this connection I wish to point out that H_2S forming compounds are conspicuous by their absence in Xanthate manufactured by the alcohol excess method.

Regarding Mr. B. S. Morrow's inquiry as to grade of commercial Xanthate, I beg to state that 85% available Xanthate should be obtained with present equipment, *i.e.*, shelf drying at ordinary temperature.

Regarding the yellow color of some of the material shipped no apprehension need be felt, the material having been crystallized and centrifuged from alcoholic solution. As the present dryer
20 capacity allows a drying period of 12 hours per batch at ordinary temperature only, an increase in atmospheric moisture inhibits the total evaporation of the alcohol, hence the yellow color. Fluctuations may consequently be expected upon the advent of the rainy season. By that time it is to be hoped the rotary vacuum dryer will have been procured, cutting down time, and increasing drying efficiency, which undoubtedly will result in a material over 90% available Xanthate, dry, and containing very little alcohol insoluble. This latter is due to overheating and consequent oxidation. While it cuts down grade somewhat it does not
30 seem to affect the flotation properties of the Xanthate adversely as it contains an organic radical aside from a sulphur radical. Some flotation tests will have to be made with the alcohol insoluble under different conditions so as to definitely dispose of this moot point, thus possibly opening again the question of final settlement. With this in mind and in the spirit of fairness only a preliminary settlement should be made at present.

Yours truly,

(Sgd.) C. H. KELLER.

Exhibit—K-39.

EXHIBIT K-39

C O P Y

Dr. Rosenstein.

San Francisco, California, Oct. 29, 1923.

CHK—M

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Cal.

Dear Sir:

10 In compliance with instructions I arranged with Dr. L. Rosenstein, Chief Chemist of Great Western E. C. Co., for a method of analysis of alkali xanthates of commercial grade manufactured by the alcohol method, as follows:—

1. Loss in weight on drying. This to be determined by drying in vacuum over sulfuric acid.
2. Alcohol insoluble by extracting with Methyl alcohol 6 times in a Soxhlet.
3. Total S equivalent iodometrically, titrating back excess.
4. Alcohol insoluble S equivalent by any of the usual methods.
- 20 5. Available Xanthate equals difference between total S equivalent and alcohol insoluble S equivalent.
6. H_2S given off in acid flotation circuit qualitatively by dissolving material in cold dry Methyl alcohol, filtering, diluting with water and inserting test paper. Immediate blackening denotes presence of H_2S making material unfit for acid circuit work.

Yours truly,

CORNELIUS H. KELLER.

Exhibit—K-40.

EXHIBIT K-40

MINERALS SEPARATION NORTH AMERICAN CORPORATION
Office of The Chief Engineer
220 Battery Street, San Francisco, Cal.

San Francisco, California, Nov. 26, 1923.

CHK—M

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

10 Dear Sir:

With a view to the utilization of mother liquor resulting from Xanthate manufacture the following experiments were undertaken. As I had to substitute drying or in some instances, refrigeration, for centrifuging considerable weight discrepancies resulted, consequently it would be unwise to deduce from these laboratory tests quantitative results obtainable in actual practice. However, the tests demonstrated beyond a doubt that the mother liquor can be used and that the resultant Xanthate is of fair grade.

20 Both pregnant liquor running 32.5% Xanthate and liquor exhausted by refrigeration averaging 24.5% Xanthate were employed.

PREGNANT LIQUOR

TEST	LIQUOR	KOH	ALCOHOL	H ₂ O	CS ₂	RESULT		ASSAY
						WET	DRY	
1	100 gr.	28	23	..	40	110 gr.	93 gr.	82.68
2	100 gr.	28	..	20 gr.	40	124 gr.	100 gr.	95.8
3	35 gr.	28	..	20 gr.	40	105 gr. moist		81.12 moist

30 In test No. 1 the KOH and Alcohol were ground in a mortar, resulting in a mush, this was added to the liquor and subsequently under refrigeration the carbon disulfide was stirred in. As the KOH dissolved only partly in the theoretical alcohol taken and very little seemed to be dissolved by the excess alcohol of the mother liquor, moreover, as the resultant Xanthate had to be dried without centrifuging, thiocarbonates and other reaction products formed were retained by the Xanthates, resulting in H₂S reaction upon acidifying.

The main object of test No. 1 was to show the possibility of using the excess alcohol of the mother liquor to aid in dissolving

Exhibit—K-40.

the KOH instead of an excess of fresh alcohol, in other words, endeavoring to circulate mother liquor and adding KOH and fresh alcohol in theoretical amounts. This obviously would result in a considerable saving, though the accumulation of thiocarbonates after circulating the original mother liquor a number of times might act detrimentally.

Test No. 2 carried out by adding KOH in water solution and making use of the excess alcohol of the mother liquor as a reaction medium resulted in a good grade Xanthate giving only a slight
10 H₂S indication.

Test No. 3 demonstrated the fact that mother liquor in approx. theoretical amounts yields a slightly lower grade Xanthate giving some H₂S indication. This seems to point to using a larger amount of mother liquor than the theoretical to obtain a maximum result in actual practice, though the excess might not be as large as in tests No. 1 and No. 2.

EXHAUSTED MOTHER LIQUOR

TEST	LIQUOR	KOH	ALCOHOL	H ₂ O	CS ₂	RESULT		ASSAY	
						WET	DRY		
20	1	80	28	23	..	40	106	79	82.11
	2	80	28	..	20	40	112	87	96.2
	3	26	23	..	18	30	76	63	73.0

As from 8—12% (by weight of mother liquor) good grade Xanthate can be recovered from the pregnant mother liquor by refrigeration, this may be resorted to increase the yield of first crop crystal. However, the pregnant liquor can be employed as shown in first series of tests with a corresponding higher yield. The observations in regard to tests with pregnant mother liquor apply to those with exhausted mother liquor as well.

30 The saving accruing by employing the method of test No. 1 of either series would be the difference in price between excess fresh alcohol and mother liquor as a circulation medium and the price of Xanthate in the original liquor and recovered. In tests No. 2 and No. 3 of either series use being made of the uncombined alcohol for the formation of Xanthate, the saving would amount to the actual cost of alcohol plus the Xanthate recovered from solution in the original liquor.

Yours very truly,

CORNELIUS H. KELLER.

Exhibit—K-41.

EXHIBIT K-41

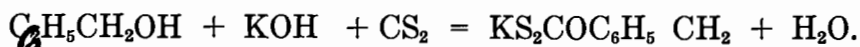
San Francisco, California, Feb. 7, 1924.

CHK—C PL—M

Edward H. Nutter, Esq., Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

Dear Sir:

In consideration of a possible bearing on the Xanthate Patent situation, as well as in view of the facility of preparation in large
10 scale manufacturing operation, a number of flotation tests were carried out jointly on a salt of Benzylalcohol prepared according to the following formula—



As the use of Ethylalcohol, Methylalcohol, Amylalcohol, etc., for Xanthate formation are well known and have been covered by former experimentation, if not in the original Xanthate patent application, Benzylalcohol appeared as another alternative. While this alcohol is at present but little used and consequently high-
20 priced, its preparation on a large scale well balanced operation, should not present much difficulty and consequently its price should be largely determined by that of the raw material, *i.e.*, Toluene.

As Potassium hydrate is only sparingly soluble in Benzylalcohol, the hydrate was dissolved first in a small amount of H₂O, which was stirred into the alcohol and subsequently the CS₂ was added. The finished salt, a Potassium Benzyl Xanthate, is soluble in all proportions in water, slightly hygroscopic and gives the characteristic Xanthate reactions with metal salts. Its action as a modifying agent in the flotation circuit is strong as subjoined experiments attest.

30 L. D. Adams No. 25907.

	% WT.	AG.	PB.	ZN.	AG.	PB.	ZN.
Heads	100.0	5.67	13.02	14.2	100.0	100.0	100.0
Pb. Conc.	17.5	25.7	66.4	12.0	79.4	89.2	14.8
Zn. Conc.	13.4	3.4	2.4	49.0	7.9	2.5	46.2
Zn. Midd.	8.0	2.9	3.5	25.4	4.1	2.2	13.4
Tails.	61.1	.8	1.3	5.7	8.6	6.1	24.7

Exhibit—K-41.

TREAT. PB.		TREAT. ZN.		RETREAT. ZN.	
Z-2	0.15#	CuSO ₂	0.5#	No reagents	
4H	0.4	4J	1.3	5 Minutes	
6 Minutes		20A	0.15		
		Z-2	0.2		
		15 Minutes			

L. D. Adams No. 25907.

	% WT.	AG.	PB.	ZN.	AG.	PB.	ZN.
Heads	100.0	5.45	13.2	14.4	100.0	100.0	100.0
10 Pb. Conc.	17.3	24.6	64.8	16.4	78.2	86.7	19.8
Zn. Conc.	20.0	2.8	4.6	41.0	10.3	6.7	57.2
Tails.	62.7	1.0	1.6	5.3	11.5	7.6	23.0

TREATMENT Pb.

Benzyl Z 0.2#

33A 0.1

10 Minutes

TREATMENT Zn.

4J 1.4#

20A 0.1#

CuSO₄ 0.8

15 Minutes

In case of the above lead-zinc ore its use results in products almost identical with those from the use of Z-1 but owing to its 20 frothing qualities the amount of oily reagents were reduced. In the following test on Anaconda slime it was noted that the bulk of the recovery was made before the addition of any frothing oil and what oil was used in this test served only as a means for a final clean-up of the sulfides. The froth formed after the addition of the pine oil apparently carried an excess of pyrite while the first froth formed with Benzyl Z alone carried an excess of copper sulfides.

Anaconda Slimes.

	% WT.	Cu.	Cu.
30 Heads	100.0	3.60	100.0
Conc.	28.6	11.92	94.8
Tails.	71.4	.26	5.2

TREATMENT

Benzyl Z 0.4 #

20A 0.15#

15 Minutes

This selective action is again shown in the following two tests on Shattuck Arizona Copper-Iron ore. The two tests were given identical treatments with the use of Benzyl Z in the first case and

Exhibit—K-41.

Exhibit—K-6.

Z-1 in the second. You will note a selective concentrate in the first case and little selection in the second.

Shattuck Arizona.

	% WT.	CU.	FE.	CU.	FE.
Heads.	100.0	3.64	39.1	100.0	100.0
Conc.	13.2	17.84	37.7	64.9	12.7
Midd.	15.6	3.56	42.0	15.2	16.8
Tail.	71.2	1.02	38.8	19.9	70.5

	PREAGITATION B. M.	TREATMENT	RETREATMENT
10	CaO 6.0#	Benzyl Z 0.2# 20A 0.15#	No reagents
	20 Minutes	15 Minutes	5 Minutes

Shattuck Arizona.

	% WT.	CU.	FE.	CU.	FE.
Heads.	100.0	3.65	39.8	100.0	100.0
Conc.	12.2	6.56	42.7	21.9	13.1
Midd.	50.1	4.24	43.2	58.1	54.2
Tails.	37.7	1.94	34.5	20.1	32.7

	PREAGITATION B. M.	TREATMENT	RETREATMENT
20	CaO 6.0#	Z-1 0.2# 20A 0.15#	No reagents
	20 Minutes	15 Minutes	5 Minutes

Yours very truly,

EXHIBIT K-6

EXTRACT FROM

ENGINEERING & MINING JOURNAL - PRESS

Feb. 9, 1924.

Experimentation by the Anaconda and Phelps Dodge companies has proved the value of another unusual reagent — thiocarbanalid — which was developed by C. L. Perkins working under the ægis of the Metals Recovery Co. The use of this chemical, in alkaline circuit, is reported to have given better recoveries than ever before at Anaconda, with costs also a trifle lower. At Nacozari and Globe it has also proved its worth in increasing the tenor of copper con-

Exhibit—K-6.
Exhibit—D-79.

centrate by dropping pyrite. The so-called "T - T" mixture is thiocarbanalid and orthotoluidin. The last-named chemical, a toluol derivative, has also been used as a solvent for alpha-naphthylamin, known for some years now as a promising though somewhat expensive reagent. Minerals Separation has also developed an interesting reagent which has given fine results, called "Z", that letter phonetically referring to the potassium xanthate which is here the vital principle. The thiocarbanalid requires a frothing oil, but the "Z" does not. Many of these newer reagents
10 are derivatives of the thio series, CS, or CS groupings, stabilized as cheaply as possible to give a soluble sulphide with high diffusing power in water.

EXHIBIT D-79

220 Battery Street,
San Francisco, Calif.,
February 14th, 1924.

Mr. T. A. Janney, Metallurgist,
Utah Copper Company,
Garfield, Utah.

20 Dear Mr. Janney:—

Doubtless you will recall my request for a copy of the analytical methods used by the Goodrich Rubber Company for determining the quality of Potassium Xanthate, reference to which is contained in the letter from Mr. J. R. Silver, Jr. to yourself, under date of December 18th, 1923. We are interested in comparing the results obtained by their methods of analysis with those obtained by our own methods, and if it would not be too much trouble for you, I should greatly appreciate your sending me the desired information at your early convenience.

30 Xanthate, in its various forms, is finding a remarkably broad application on a great variety of ores, and I have been wondering, since I saw you, whether and to what extent you have found it to be useful for treating the ores of your company. I feel confident that a working-scale test of the reagent would show some very interesting possibilities and I trust that you will not fail to give it a trial in your experimental section before long. If we can assist you in any way, please do not fail to let us know.

Exhibit—D-79.

Exhibit—K-7.

The Great Western Electro-Chemical Company is now supplying our licensees with crystalline potassium-xanthate of 97% purity, the superior quality of their product being due to improved manufacturing methods that we have devised. In addition to the xanthate crystals at 25c. a pound, and the mother-liquor at around 15c. a pound, the manufacturers are prepared to supply xanthate paste at a price of about 16c. a pound, — this latter product being an uncentrifuged mixture of the first two substances. These are the straight alcoholic products, with the water-phase entirely
 10 eliminated in the manufacturing process.

I will arrange to have laboratory samples of each of these substances sent to you at an early date.

I take this opportunity of thanking you for the many courtesies extended to me on my recent visit to your plant, and with kindest personal regards, beg to remain,

Very truly yours,

CCNY—SF
 JD

20 EXHIBIT K-7

EXTRACT FROM

ENGINEERING & MINING JOURNAL - PRESS

Aug. 2, 1924.

“FLOTATION OILS AND REFERENCES”

“One of our engineers has asked me to try to get information with reference to the flotation method as used in the copper industry, especially as to the kinds and methods of using oils. It has occurred to me that your company could assist us in the matter by suggesting textbooks and publishing houses where these these textbooks can be obtained.”

30 In the flotation process, a minute quantity of an oil or other suitable reagent is added to a mixture of finely ground ore and water. The whole is then violently agitated with rapidly revolving paddles (in the “mechanical” process) or led through a trough through the porous bottom of which air is blown (in the “pneumatic” process), so that a thick froth is produced. The mineral content of the ore will largely be found in the bubbles of this froth, which are skimmed off, broken down with a fine spray of water, and the mineral recovered by settling and filtration. The oil and any other reagent that may be added performs two functions: it causes a
 40 proper froth to be formed, and it coats the mineral particles, causing them to attach themselves to the bubbles. Some oils do

Exhibit—K-7.

Exhibit—K-8.

one of these things and some the other; also, some do both. Those which perform the first-named function are termed frothing oils; the others collecting oils. The most popular frothing oils are refined pine oils, and the most commonly used collecting oils are crude coal tar, the various creosotes, and fuel oils.

The oils used may also be classified according to their origin: (1) Petroleum oils, including petroleum residuum, fuel oil, crude kerosene, and sludge-acid kerosene. (2) Coal-tar products, such as crude coal tar, coal-tar oil and coal-tar creosote. (3) Hardwood
10 distillates. (4) Pine distillates, including both crude and refined steam and destructively distilled pine oils, pine tar, pine-tar oil, and crude turpentine. A newer class of reagents, which may be used either with or without those already mentioned, includes alpha-naphthylamine, orthotoluidine, thiocarbonyl, and potassium xanthate. The ore pulp is also frequently made acid or alkaline to assist recovery of the mineral.

The latest important books on flotation include:

“The Flotation Process,” second edition, 1918, by McGraw; McCraw-Hill Book Co., New York. “Concentration by Flotation,”
20 1921, by Rickard; John Wiley & Sons, New York. “A Manual of Flotation Processes,” 1921, by Taggart; John Wiley & Sons.

EXHIBIT K-8

EXTRACT FROM

ENGINEERING & MINING JOURNAL—PRESS

Oct. 18, 1924.

“Flotation Reagent” — No. 1,507,089.

Sept. 2, 1924. Ludwig Rosenstein, San Francisco. A method of producing alkali metal xanthates, consisting in bringing together an alkali metal hydroxide, water, alcohol, and carbon bisulphide.
30 The patentee was until a few months ago connected with the Great Western Electro-chemical Works, which was the first plant to make and sell potassium xanthate in large quantities to flotation operators.

Exhibit—K-10.

Exhibit—K-9.

EXHIBIT K-10

EXTRACT FROM
ENGINEERING & MINING JOURNAL-PRESS

Nov. 1, 1924.

“FLOTATION”

No. 1,512, 139. Oct. 21, 1924. R. E. Sayre, New Rochelle, N.Y.
The patent covers the use, as a flotation agent, of a heavy-metal xanthate in a non-acid pulp.

EXHIBIT K-9

10

EXTRACT FROM
ENGINEERING & MINING JOURNAL-PRESS

Nov. 22, 1924.

“THE FLOTATION SITUATION”.

The EXPIRATION of the famous flotation patent No. 835,120, issued to Sulman, Picard, and Ballot, and covering the use of less than 1 per cent of oil in connection with violent agitation, has apparently not resulted in Minerals Separation losing any of its licensees. Practically all of the important mining companies with the exception of Magma Copper, and most of the smaller operators, continue to pay
20 royalty for the use of the process. Minerals Separation has, however, consented to a 50 per cent reduction in the amount of the royalty, provided its licensees will continue their payments until the expiration of the other important patent, No. 962,678, covering the use of soluble frothing agents, which will be on June 29, 1927. What will happen after that is problematical. It is likely that the royalty fee will again be reduced to such a figure that it will pay operators to continue as licensees so that they may get the benefit of various minor improvements controlled by the patent exploiting company. Some of these
30 are probably of considerable utility. A recent introduction, for which the Minerals Separation research engineers seem to be responsible, is the use of potassium and sodium xanthates as flotation reagents. These salts are now being used by several of the larger companies, with a marked improvement in recoveries; they have proved of value in selective work and in some instances in the flotation of oxidized mineral. The use of xanthate is one of the most important advances made in the technology of the flotation process in recent months.

Unfortunately, flotation litigation is still with us, though in lessened amount. The Magma suit is still before the court, but the judge's decision is overdue; it may come at any time. The Wolf suit
40 has been postponed again, this time until January, we understand.

Exhibit—K-9.
Exhibit—K-11.

And there is some litigation pending with regard to the use of certain reagents.

EXHIBIT K-11

EXTRACT FROM ENGINEERING AND
MINING JOURNAL PRESS

Dec. 20, 1924.

THE ANALYSIS OF XANTHATE

Flotation Operators, Until Now, Have Had No Reliable Method of
Determining the Purity of What They Get

10 BY W. S. CALCOTT, F. L. ENGLISH, AND F. B. DOWNING
Flotation Laboratory, E. I. du Pont de Nemours & Co., Inc.,
Wilmington, Del.

The use of Xanthate in the concentration of sulphide ores by
flotation has now become widespread, the most popular of the
xanthates being the potassium and sodium salts of ethyl xanthate.
Metallurgists have been unable to determine the actual xanthate
content of commercial xanthates, because heretofore no accurate
method of analysis has been published or communicated to them.
It has, therefore, been difficult to evaluate properly the several
20 kinds and grades of xanthate offered for sale, and to determine
the degree of uniformity of the shipments of these products.
Common impurities in commercial products are water, ethyl alcohol,
potassium or sodium carbonates, and complex oily compounds rich
in sulphur.

The most common method for the estimation of xanthate is
the determination of the total sulphur of the sample, and, assum-
ing this sulphur to be the sulphur of the true xanthate, its direct
calculation to xanthate. As other sulphur-containing bodies are
generally present in commercial xanthate, the method gives inac-
30 curate results and its use has led to many erroneous assumptions
by manufacturers and consumers of xanthate. At least one mining
concern has been accustomed to apprise all the material soluble
in alcohol as xanthate. In the presence of other alcohol-soluble
bodies, this method will obviously give high results.

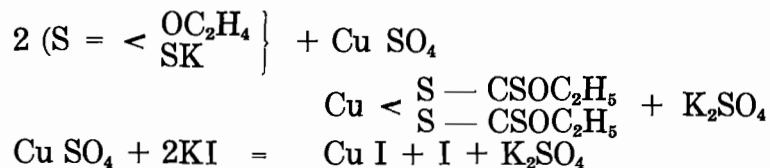
Because of its interest to the mining industry and to its own
manufacturing program, the Du Pont Flotation Laboratory under-
took the necessary research work to develop the method of analysis,
which is here presented by its authors in detail.

PRINCIPLE AND APPLICATION OF THE METHOD

40 The xanthate is precipitated as the copper salt from a slightly
alkaline solution using a copper sulphate-sodium tartrate reagent,

Exhibit—K-11.

filtered off and decomposed with nitric acid. The copper is then determined by titration with 0.1 N. sodium thiosulphate.



The method has been applied to both pure and crude materials. The results are not affected by the presence of xanthyl disulphide, the diethyl xanthate derivative, or such impurities as thiosulphates, sulphites, or carbonates.

The presence of sulphides or potassium thiocarbonate (K_2CS_3) causes high results, but as these impurities have been found absent in properly manufactured commercial products, examination for their presence is limited to qualitative tests. Duplicate determinations of potassium xanthate require approximately four hours and should check within 0.5 per cent of the amount present.

(a) *Copper sulphate-tartrate solution.* Dissolve 25 g. of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 600 cc. of hot distilled water, cool to room temperature, and add 200 g. of c.p. sodium tartrate. After complete solution of the tartrate, slowly add 10 per cent Na_2CO_3 solution until the color of the reagent turns to dark blue and the reagent is just alkaline to litmus, indicating an excess of Na_2CO_3 ; then dilute the solution to a volume of one liter, and filter.

(b) *0.1 N. sodium thiosulphate solution.* The standard sodium thiosulphate solution must be standardized against 0.2 to 0.3 g. samples of pure copper which have been dissolved in 30 cc. of 2:1 HNO_3 (by volume) in 500-cc. Erlenmeyer flasks and treated according to the procedure for the estimation of copper in xanthate described in the procedure below.

Calculation

$$\frac{\text{wt. of sample}}{\text{cc. Na}_2\text{S}_2\text{O}_3 \equiv 0.06357} = \text{Normality of thiosulphate.}$$

PROCEDURE FOR QUALITATIVE TESTS AND FOR XANTHATE DETERMINATION

(a) *Sulphides.* Dissolve a 1-g. sample in 100 cc. of cold water and add a few cc. of a 2 per cent aqueous solution of sodium nitroprusside. The immediate appearance of an intense reddish violet coloration indicates the presence of appreciable quantities of sulphides. Report presence or absence of sulphides.

(b) *Thiocarbonates.* Dissolve a 1-g. sample in 50 cc. of cold distilled water and add 5 cc. of 10 per cent lead acetate solution. The curdy precipitate formed should be cream colored or almost

Exhibit—K-11.

white. If a brown or red precipitate is obtained, the presence of thiocarbonates is indicated. Report presence or absence of thiocarbonates.

(c) *Potassium xanthate*. Dissolve a 1.5-g. sample in 300 cc. of cold distilled water in a 500-cc. Erlenmeyer flask and slowly add with agitation 125 cc. of the copper precipitating reagent. Close the flask with a rubber stopper and immerse it in a bath of ice and water. Allow the mixture to stand with occasional shaking for at least one hour but not more than three hours; then filter off
10 the yellow precipitate upon a large (30 cc.) Gooch crucible containing an asbestos mat, and wash precipitate and flask thoroughly with cold distilled water. Avoid allowing the precipitate to suck dry and cake during washing.

Place the Gooch crucible containing the precipitate in a 400-cc. beaker covered with a watch glass and pour into the Gooch 30 cc. of 2 parts conc. HNO_3 to 1 part water by volume (Hood). Allow the mixture to stand for five minutes; then rinse and remove the Gooch crucible and place in it a thin mat of asbestos fibre. Filter
20 the copper nitrate solution containing the oily products of decomposition of the xanthate and the asbestos fibre through the Gooch crucible and wash with water. Return the filtrate and washings to the original 500-cc. Erlenmeyer flask, add 15 cc. of conc. H_2SO_4 and a dozen small glass beads, then boil the solution in the hood until the nitric acid is completely expelled and dense fumes of SO_3 fill the flask. Allow the flask to cool, then dilute to 100–125 cc. with distilled water and again cool to room temperature. Add conc. NH_4OH to the solution until a slight excess is present, as indicated by the appearance of an intense blue color. Boil off any excess ammonia, then cool the solution to room temperature and
30 acidify with exactly 7 cc. of glacial acetic acid.

Dissolve 5 g. of c.p. potassium iodide in the solution, then titrate the iodine liberated with 0.1 N. sodium thiosulphate using 10 cc. of 0.1 per cent starch solution as indicator.

Calculation

$$\text{cc. Na}_2\text{S}_2\text{O}_3 \times \text{Normality of Na}_2\text{S}_2\text{O}_3 \times 32.06$$

Wt. of sample

= per cent potassium xanthate

$$\text{cc. Na}_2\text{S}_2\text{O}_3 \times \text{Normality of Na}_2\text{S}_2\text{O}_3 \times 28.84$$

Wt. of sample

= per cent sodium xanthate

40

EXPERIMENTAL DATA ON USE OF METHOD

Typical analyses of pure potassium xanthate follow in Table I. The pure potassium xanthate was prepared from absolute alcohol,

Exhibit—K-11.

pure carbon bisulphide and carbonate-free potassium hydroxide. The product was then recrystallized from 95 per cent alcohol, washed with ether, and vacuum dried. A total sulphur analysis indicated a purity of 99.4 per cent.

Application of the method of analysis to commercial products (Table II) revealed the unsatisfactory nature of the present methods of analysis.

Although doubtless the "sulphur method" has been the cause of many false conclusions in the determination of the purity of commercial xanthates, and in the interpretation of flotation efficiencies, we do not wish to minimize its importance, when used in connection with the "copper" method, as an indication of the presence of sulphur-bearing impurities.

An investigation is now in progress to devise a method for the direct determination of these impurities.

TABLE I — ANALYSES OF POTASSIUM XANTHATE BY THE COPPER PRECIPITATED METHOD

Purified material, 99.4 per cent by sulphur analysis. Sodium thio-sulphate normality = 0.1031

No.	Sample, Grains	Thiosulphate Consumed, Cubic Centimeters	Purity Found, Per Cent
1	2.0000	60.17	99.4
2	2.0000	60.25	99.6
3	1.0000	30.00	99.2
4	1.0000	30.10	99.5
5	1.0000	30.05	99.3
6	1.0000	30.11	99.5
7	2.0000	59.85	98.9
8	2.0000	59.92	99.0
9	1.0000	30.10	99.5
10	1.0000	30.12	99.6

TABLE II — COMPARATIVE ANALYSES OF COMMERCIAL POTASSIUM XANTHATE

No.	Sample	Purity Claimed	Purity by Copper Method	Purity by Total Sulphur
1	Solid	95—98	80.5	83.5
2	Solid	90—93	83.9	86.7
3	Liquid	50—55	49.5	(a)
4	Solid	90—92	90.4	92.1
5	Solid	90—92	90.1	92.6
6	Solid	90—92	90.1	93.0
7	Solid	95—97	94.5	96.3
8	Solid	95—97	94.5	94.3
9	Solid	95—97	95.0	96.4

(a) Rich in Sulphides.

Exhibit—K-46.

EXHIBIT K-45

San Francisco, California, Sept. 11, 1925.

CHK—M

Mr. Edward H. Nutter, Chief Engineer,
Minerals Separation N. A. Corp.,
220 Battery St., San Francisco, Calif.

Dear Sir:

10 An extensive survey of the field of organic-inorganic flotation reagents has disclosed so far only one group of substances, which in point of over all efficiency compare with Xanthates and in certain features, exceed them.

These are the Mercaptides or alkali salts of sulphur alcohols or Mercaptans.

20 Some flotation experiments were carried out a considerable time ago with Ethylmercaptan. They were discontinued, however, for the following reasons:—Ethylmercaptan is a liquid with a very low boiling point; it evaporates quickly at ordinary temperatures; it decomposes with ease; it does not dissolve in water in all proportions and its odor is very obnoxious. The alkali salts of this Mercaptan on the other hand, appear to have less objectionable features: They are solids soluble in all proportions in water; they decompose comparatively slowly; their odor is obnoxious but less so than Mercaptan proper. The price of Mercaptides is at present rather high, for the reason that demand for them is limited. This, of course, is likely to change at any time, as soon as manufacturers and metallurgists realize that the Mercaptides are unprotected by patent and are potential competitors of the Xanthates.

30 Chemically, the Mercaptans may be viewed as alcohols in which oxygen has been replaced by sulphur. They readily form salts with metals of the alkali metal groups. These latter on exposure to air for a period of several days gradually decompose, forming salts of sulphonic acids — for instance, Sodium Mercaptide C_2H_5Na forms Sodium Ethylsulphonate $C_2H_5SO_3Na$.

The alkali salts of the mercaptans are easily formed by introducing an alkali metal into the mercaptan, according to the following equation $C_2H_5SH + Na = C_2H_5S Na + H$.

40 For the production of the Mercaptan a number of methods have been devised such as — the action of Potassium hydrogen sulphide on Ethylchloride, or reducing chlorides of sulphonic acids as — Ethylsulphonchloride for instance, with metallic zinc or tin and dilute sulphuric or hydrochloric acid. These processes are, of course, expensive as they involve high priced raw materials.

For large scale manufacture several processes are available at present. One method comprises the interaction between hydrogen

Exhibit—K-45.

sulphide on alcohol vapour at 300° — 350° in presence of anhydrous Thoria (Th O₂) according to the formula $C_2H_5OH + H_2S = C_2H_5SH + H_2O$.

Another operation consists in the reduction of disulphides by dextrose, which might advantageously be introduced as a side line in corn products, paper or beet sugar industries; another consists in the action of Aluminum chloride on a mixture of a Hydrocarbon and Sulphur at 80°, which results in an aromatic mercaptan and corresponding sulphides.

- 10 All these methods are, of course, subject to improvement and will result in a cheapening of the reagent as the demand for this type flotation reagent becomes established.

A number of flotation tests were carried out by Mr. C. F. Williams, using Xanthate and Sodium Ethylmercaptide respectively, to demonstrate their similarity of action in certain instances and the superiority of Sodium Ethylmercaptide over Xanthates in others.

In the subsequent tests NaZ denotes Sodium Xanthate and Super Z — Sodium Mercaptide.

20 TEST 6. Anaconda Copper Ore — Composite Run of Mine.

8/27/25.

		A S S A Y S			% R E C O V E R Y	
		Wt. %	Cu.	Fe.	Cu.	Fe.
	Heads	100.0	3.6	11.9	100.0	100.0
30228	Conc.	27.1) 32.6	12.64) 10.75	34.2) 31.0	94.1) 96.3	78.2) 85.3
30229	Midd.	5.5)	1.44)	15.4)	2.2)	7.1)
30230	Tail.	67.4	.20	2.6	3.7	14.7

1000 Grams

	PREAG. IN BALL MILL	TREATMENT	RETREATMENT
30	CaO 6.0#	NaZ .1#	No reagents
		4H .25	
		20-0 .2	
	15 Minutes	13 Minutes	3 Minutes

TEST 3. Anaconda Copper Ore — Composite Run of Mine

		A S S A Y S			R E C O V E R I E S	
		Wt. %	Cu.	Fe.	Cu.	Fe.
	Heads	100.0	3.64	12.0	100.0	100.0
30237	Conc.	10.1) 21.1	27.12) 14.7	13.4) 13.3	75.4) 85.3	11.2) 23.3
30238	Midd.	11.0)	3.28)	13.2)	9.9)	12.1)
30239	Tail.	78.9	.68	11.7	14.7	76.7

Exhibit—K-45.

		—1000 Grams. 8/27/25.	
PREAG. IN BALL MILL		TREATMENT	RETREATMENT
CaO	6.0#	Super Z .12#	No reagents
	15 Minutes	4H .25	3 Minutes
		20-0 .20	
		13 Minutes	

TESTS No. 6 AND No. 3

As can be seen, the flotation operation was carried out under identical conditions of alkalinity, oils, agitation and time of treatment. Sodium Xanthate in test No. 6 being substituted by Sodium Mercaptide in test No. 3. It is evident that the use of Mercaptide on this ore gives lower extraction but a much higher grade cleaner concentrate.

TEST 15. Utah Copper Ore — 1000 Grams. 8/24/25.

	WT. %		CU.	FE.		CU.	FE.
	Heads	100.0	1.05	3.2		100.0	100.0
30165	Conc.	4.0) 7.1	19.68) 12.5	32.2) 21.6		74.6) 84.2	40.1) 47.8
30166	Midd.	3.1)	3.24)	8.0)		9.6)	7.7)
30167	Tail.	92.9	.18	1.8		15.8	52.2

20	PREAG. IN BALL MILL	TREATMENT	RETREATMENT
	CaO 6.0#/Ton.	NaZ .15#	33C .1#
	5 Minutes	33C .2	3 Minutes
		8 Minutes	

Test No. 15 demonstrates the effect of Xanthate, giving a lower grade concentrate and much less FE rejection. This test was run as a comparison between Xanthate and Mercaptide.

(Compare with Test No. 11)

TEST 11. Utah Copper Ore — 1000 Grams. 8/24/25.

	ASSAYS			RECOVERIES		
	WT. %	CU.	FE.	CU.	FE.	
	Heads	100.0	1.01	2.64	100.0	100.0
30183	Conc.	2.0) 3.6	35.56) 23.3	25.7) 17.3	72.4) 82.9	19.5) 23.6
30184	Midd.	1.6)	6.60)	6.8)	10.5)	4.1)
30185	Tail.	96.4	.18	2.1	17.1	76.4

	PREAG. IN BALL MILL	TREATMENT	RETREATMENT
	CaO 6.0#	Super Z .24#	33C .3#
	5 Minutes	33C .1	3 Minutes
		8 Minutes	

Test No. 11 shows a considerable increase in grade over the Xanthate Test No. 15, with approximately the same extraction and a markedly higher FE rejection.

Exhibit—K-45.

TEST 12. Utah Copper Ore — 1000 Grams. 8/24/25.

	ASSAYS					RECOVERIES				
	Wt. %	Cu.		Fe.		Cu.		Fe.		
Heads	100.0	1.04		2.60		100.0		100.0		
30180 Conc.	1.4) 3.4	39.84)	21.98	24.3)	14.1	53.8)	71.9	13.1)	18.5	
30181 Midd.	2.0)	9.48)		7.0)		18.1)		5.4)		
30182 Tail.	96.6	.30		2.2		28.1		81.5		

10	PREAG. IN BALL MILL		TREATMENT		RETREATMENT	
		CaO	6.0#	Super Z	.04#	33C
		5 Minutes	33C	.2		3 Minutes
				8 Minutes		

Test No. 12 was run to demonstrate that a very small amount of Mercaptide suffices to give a noticeable effect. A considerable grade increases, a lower extraction and a very high FE rejection can be noted.

TEST 13. Utah Copper Ore — 1000 Grams. 8/24/25.

	ASSAYS					RECOVERIES				
	Wt. %	Cu.		Fe.		Cu.		Fe.		
Heads	100.0	1.03		2.64		100.0		100.0		
30177 Conc.	2.3) 4.9	32.96)	17.5	24.5)	15.1	73.5)	83.4	21.4)	28.1	
30178 Midd.	2.6)	3.92)		6.8)		9.9)		6.7)		
30179 Tail.	95.1	.18		2.0		16.6		71.9		

	PREAG. IN BALL MILL		TREATMENT		RETREATMENT	
		CaO	6.0#	Super Z	.15#	33C
		5 Minutes	33C	.2		3 Minutes
				8 Minutes		

TEST 14. Utah Copper Ore — 1000 Grams. 9/2/25.

	ASSAYS					RECOVERIES				
	Wt. %	Cu.		Fe.		Cu.		Fe.		
Heads	100.0	1.06		2.60		100.0		100.0		
30330 Conc.	2.3) 5.3	34.0)	16.7	22.0)	13.3	78.8)	88.8	19.5)	27.1	
30331 Midd.	3.0)	3.52)		6.6)		10.0)		7.6)		
30332 Tail.	94.7	.18		2.0		16.2		72.9		

	PREAG. IN BALL MILL		TREATMENT		RETREATMENT	
		CaO	6.0#	Super Z	.15#	
		5 Minutes	33C	.2		33C
				8 Minutes		3 Minutes

Exhibit—K-45.

Tests 13 and 14 are duplicate tests as to reagents, etc., Mercaptide being used in both instances. The object of these tests was to show that no impairment in the effectiveness of the reagent took place, although test 13 was with freshly made reagent, August 24, 1925, whereas test 14 was made September 2nd, 1925, with the same material, which had been exposed to the air for 9 days. After the addition of 0.1 lb. of Mercaptide a marked brightening of the sulphides took place in both tests.

TEST 10. Engels Copper Ore — 1000 Grams. 8/24/25.

	ASSAYS					RECOVERIES				
	Wt. %	Cu.		Fe.		Cu.		Fe.		
Heads	100.0		100.0		100.0		
30186 Conc.	3.9) 6.7	33.68)	22.4	14.3)	12.6	73.7)	84.3	5.6)	8.5	
30187 Midd.	2.8)	6.76)		10.2)		10.6)		2.9)		
30188 Tail.	93.3	.30		9.7		15.7		91.5		

20

PREAG. IN BALL MILL	TREATMENT	RETREATMENT
15 Minutes	Na ₃ PO ₄ 2.5# (to alk)	33C .3#
	Super Z .12#	3 Minutes
	33C .2	
	10 Minutes	

Test 10 was run with the object of showing that the use of Mercaptide results in a higher grade concentrate than with the use of ordinary reagents, which give ordinarily between 26—28% grade.

TEST 21. San Francisco Mines of Mexico Ore—1000 Grams. 9/3/25.

	ASSAYS						RECOVERIES						
	Wt. %	Ag.	Pb.	Zn.	Fe.		Ag.	Pb.	Zn.	Fe.			
Heads	100.0	22.4	10.1	22.2	6.3		100.0	100.0	100.0	100.0			
30349 Pb. Conc.	12.0	53.3)	41.5	57.6)	31.8	13.2)	26.3	6.7)	9.2	28.5	68.7	7.1	12.7
30350 Pb. Midd.	16.2	32.7)	12.8)	36.0)	11.1)		23.7	20.6	26.3	28.5			
30351 Zn. Conc.	21.5	29.2)	28.4	0.6)	1.5	51.6)	46.2	6.5)	6.8	28.1	1.3	50.0	22.2
30352 Zn. Midd.	5.3	25.6)	5.2)	23.7)	8.1)		6.0	2.7	5.7	6.3			
30353 Tail.	45.0	6.8	1.5	5.4	4.2		13.7	6.7	10.9	29.8			

PREAG. IN BALL MILL	TREATMENT Pb.	RETREATMENT Pb.
Na ₂ S .5#	Soda Ash 3.0#	2B .15#
15 Minutes.	Super Z .04	7 Minutes.
	Barrett 634 .3	
	2B .1	
	5 Minutes.	

870

Exhibit—K-45.

TREATMENT Zn.	RETREATMENT ZN.
CuSO ₄ .5#	No reagents
Super Z .1	3 Minutes.
5S .3	
20-0 .2	
12 Minutes	Na ₂ S completely decomposed.

TEST 22. San Francisco Mines of Mexico—1000 Grams. 9/4/25.

	A S S A Y S						R E C O V E R I E S			
	WT. %	AG.	PB.	ZN.	FE.	AG.PB.	ZN.	FE.		
Heads	100.0	21.9	10.4	22.7	6.7	100.0	100.0	100.0	100.0	
30382 Pb. Conc.	11.0	61.3)	56.8 58.4)	43.0 15.6)	22.9 4.3)	6.3	30.9	61.9	7.6	7.0
30383 Pb. Midd.	8.5	50.8)	22.8)	32.3)	8.8)		19.7	18.7	12.1	11.2
30384 Zn. Conc.	28.1	23.4)	23.3 3.2)	3.7 51.0)	46.2 8.4)	8.7	30.0	8.7	63.2	35.2
30385 Zn. Midd.	6.0	22.8)	6.0)	23.2)	10.4)		6.2	3.5	6.1	9.3
30386 Tail.	46.4	6.2	1.6	5.4	5.4		13.2	7.2	11.0	37.3

PREAG. IN BALL MILL	TREATMENT PB.	RETREATMENT PB.
Na ₂ S .5#	Soda Ash 3.0#	2B .2#
15 Minutes	Super Z (New lot) .04#	5 Minutes
	5S .3	
	2B .15	
	7 Minutes	
	TREATMENT ZN.	RETREATMENT ZN.
	CuSO ₄ .5#	No reagents
	Super Z .1	3 Minutes
	5S .3	
	20-0 .2	
	10 Minutes	

Test No. 22 run as duplicate to No. 21. In the latter test (21) old Mercaptide (1 week old) was used. In test 22 new Mercaptide was employed, better grade AG. and PB. and better recovery can be noted on this test, whereas ZN recovery and grade on both tests is approximately the same. The FE. rejection on test 22 shows an increase. No Sodium Cyanide was used in either of these tests.

TEST 25. San Francisco Mines of Mexico—1000 Grams. 9/3/25.

	A S S A Y S					R E C O V E R I E S				
	WT. %	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.	
Heads	100.0	26.0	11.2	24.6	6.9	100.0	100.0	100.0	100.0	
30354 Pb. Conc.	12.1	72.0)	56.1 59.6)	32.0 13.0)	25.4 5.1)	9.4	33.4	64.5	6.4	8.9
30355 Pb. Midd.	19.5	46.9)	14.9)	33.2)	12.0)		35.1	26.0	26.2	34.0
30356 Zn. Conc.	24.4	18.1)	18.4 0.8)	1.64 50.8)	47.0 8.2)	8.2	17.0	1.7	50.4	29.0
30357 Zn. Midd.	6.1	19.7)	5.0)	31.5)	8.2)		4.6	2.7	7.8	7.2
30358 Tail.	37.9	6.8	1.5	6.0	3.8		9.9	5.1	9.2	20.9

Exhibit—K-45.

	PREAG. IN BALL MILL	TREATMENT PB.	RETREATMENT PB.
	Na ₂ S .5#	Soda Ash 3.0#	2B .15#
	15 Minutes	NaZ .04	3 Minutes
		5S .3	
		2B .1	
		4 Minutes	
		TREATMENT ZN.	RETREATMENT ZN.
		CuSO ₄ .5#	No reagents
		NaZ .1	3 Minutes
10		5S .3	
		20-0 .2	
		10 Minutes	

Test 25 with Sodium Xanthate was made to compare with 21 and 22. It is apparent that Mercaptide gives slightly less silver grade and recovery in test 21. Tests 22 and 25 compare well as to grade and recovery of the same metal. Grade and recovery on PB. and ZN. are approximately the same. A larger FE. rejection in the Mercaptide tests is apparent.

TEST 23. San Francisco Mines of Mexico—1000 Grams. 9/3/25.

		ASSAYS					RECOVERIES			
	WT. %	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.	
	Heads	100.0	21.7	9.8	21.9	6.5	100.0	100.0	100.0	100.0
30365	Pb. Conc.	9.0	68.9)	69.5 72.0)	55.1 8.0)	15.1 1.7)	3.6	28.4	66.0	3.3 2.4
30366	Pb. Midd.	6.8	70.4)	32.7)	24.6)	6.2)		22.0	22.7	7.6 6.5
30367	Zn. Conc.	28.5	23.0)	22.8 0.6)	1.4 52.8)	47.5 7.0)	7.6	30.1	1.7	69.1 30.8
30368	Zn. Midd.	7.2	22.0)	4.4)	25.7)	10.0)		7.3	3.2	8.5 11.1
30369	Tail.	48.5	5.4	1.3	5.2	6.6		12.2	6.4	11.5 49.2

	PREAG. IN BALL MILL	TREATMENT PB.	RETREATMENT PB.
	Na ₂ Z .5#	Soda Ash 3.0#	2B .25#
30	15 Minutes	NaCN .8	4 Minutes
		Super Z .04	
		5S .3	
		2B .1	
		7 Minutes	
		TREATMENT ZN.	RETREATMENT ZN.
		CuSO ₄ 1.0#	No reagents
		Super Z .1	3 Minutes
		5S .3	
		20-0 .2	
40		12 Minutes	

Exhibit—K-45.

TEST 24. San Francisco Mines of Mexico—1000 Grams. 9/4/25.

	ASSAYS					RECOVERIES				
	WT. %	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.	
Heads	100.0	20.9	9.8	22.2	6.1	100.0	100.0	100.0	100.0	
30387 Pb. Conc.	8.8	68.5)	71.7 69.6)	56.8 9.4)	14.7 1.5)	3.2	28.8	62.8	3.7	2.1
30388 Pb. Midd.	5.8	76.6)	37.4)	22.7)	5.8)		21.3	22.2	5.9	5.5
30389 Zn. Conc.	27.6	22.4)	21.8 1.0)	2.0 52.8)	47.5 7.4)	7.8	29.5	2.8	65.9	33.1
30390 Zn. Midd.	8.2	20.0)	5.4)	29.5)	9.4)		7.9	4.6	10.9	12.5
30391 Tail.	49.6	5.3	1.5	6.1	5.8		12.5	7.6	13.6	46.8
Rf. Pb. Conc.							50.1	85.0	9.6	7.6
Rf. Zn. Conc.							37.4	7.4	76.8	45.6

PREAG. IN BALL MILL	TREATMENT PB.	RETREATMENT PB.
Na ₂ S	Soda Ash 3.0#	2B .2#
15 Minutes	NaCN .8	3 Minutes
	Super Z (new lot) .04	
	5S .3	
	2B .15	
	7 Minutes	
	TREATMENT ZN.	RETREATMENT ZN.
	CuSO ₄ 1.0#	No reagents
	Super Z .1	3 Minutes
	5S .3	
	20-0 .2	

Tests 23 and 24 run with old and new Mercaptide respectively. They compare well with each other as to grade and recovery.

TEST 26. San Francisco Mines of Mexico—1000 Grams. 9/3/25.

	ASSAYS					RECOVERIES				
	WT. %	AG.	PB.	ZN.	FE.	AG.	PB.	ZN.	FE.	
Heads	100.0	21.3	10.6	23.0	6.0	100.0	100.0	100.0	100.0	
30377 Pb. Conc.	10.6	78.0)	71.6 70.8)	50.1 9.0)	18.6 1.4)	4.0	38.9	70.8	4.1	2.5
30378 Pb. Midd.	8.1	63.2)	22.8)	31.2)	7.4)		24.1	17.4	11.0	10.0
30379 Zn. Conc.	27.0	16.3)	16.1 1.0)	1.67 55.4)	48.2 6.8)	7.2	20.7	2.5	65.3	30.8
30380 Zn. Midd.	6.9	10.0)	4.6)	27.6)	10.0)		5.8	3.0	8.3	11.5
30381 Tail.	47.4	4.7	1.4	5.5	5.7		10.5	6.3	11.3	45.2

Exhibit—K-45.

	PREAG. IN BALL MILL	TREATMENT PB.	RETREATMENT PB.
	Na ₂ S .5#	Soda Ash 3.0#	2B .15#
	15 Minutes	NaCN .8	4 Minutes
		NaZ .04	
		5S .3	
		2B .1	
		6 Minutes	
		TREATMENT ZN.	RETREATMENT ZN.
		CuSO ₄ 1.0#	No reagents
10		NaZ .1	3 Minutes
		5S .3	
		20-0 .2	
		10 Minutes	

Test 26 with Sodium Xanthate and Sodium Cyanide was made for comparison with tests 23 and 24. It is evident that the use of Mercaptide gives better PB. grade and recovery. ZN. recovery and grade are slightly better with Mercaptide. AG. grade and recovery approximately the same with either of the reagents. FE. rejection is slightly higher with Mercaptide.

20 TEST 30. California Rand Ore—1200 Grams. 8/28/25.

		ASSAYS				RECOVERIES			
Wt. %		AU.	AG.	FE.	AU.	AG.	FE.		
Heads	100.0	.29	36.6	5.5	100.0	100.0	100.0		
30253 Conc.	5.0) 11.0 3.64)	1.96	631.)	303 32.3) 20.8	63.3) 75.2	86.1) 91.3	29.4) 41.6		
30254 Midd.	6.0)	.57)	31.7)	11.2)	11.9)	5.2)	12.2)		
30255 Tail.	89.0	.08	3.6	3.5	24.8	8.7	58.4		

	PREAG. IN BALL MILL	TREATMENT	RETREATMENT
	Trona 3.3#	Super Z .17#	20-0 .04#
	20 Minutes	4H .33	4 Minutes
30		20-0 .17	
		15 Minutes	

TEST 31. California Rand Ore—1200 Grams. 8/28/25.

		ASSAYS				RECOVERIES			
Wt. %		AU.	AG.	FE.	AU.	AG.	FE.		
Heads	100.0	.34	35.9	4.4	100.0	100.0	100.0		
30256 Conc.	7.9) 13.9	2.6) 2.21	383.0) 232	34.0) 22.7	60.0) 89.9	84.4) 90.0	60.2) 71.0		
30257 Midd.	6.0)	1.7)	33.5)	8.0)	29.9)	5.6)	10.8)		
30258 Tail.	86.1	.04	4.2	1.5	10.1	10.0	29.0		

Exhibit—K-46.

PREAG. IN BALL MILL	TREATMENT	RETREATMENT
Trona .33#	Z .17#	20-0 .04#
20 Minutes	4H .33	4 Minutes
	20-0 .17	
	15 Minutes	

Tests 30 and 31 run on California Rand ore show lower grade AU., higher grade AG., and considerably more FE. rejection, with lower over all recovery in the former employing Mercaptide than in test 31 using Xanthate.

10 TEST 32. Betty O'Neal Ore — 1000 Grams 8/28/25.

		ASSAYS			RECOVERIES	
		WT. %	AG.	FE.	AG.	FE.
	Heads	100.0	23.0	2.61	100.0	100.0
30262	Conc.	3.0) 9.4	634.9) 22.0	6.8) 6.6	82.7) 89.9	7.8) 23.7
30263	Midd.	6.4)	25.8)	6.5)	7.2)	15.9)
30264	Tail.	90.6	2.56	2.2	10.1	76.3

PREAG. IN BALL MILL	TREATMENT	RETREATMENT
No reagents.	Super Z .2#	5 Minutes
20 Minutes	4H .3	
20	20-0 .4	
	18 Minutes	

TEST 33.

		ASSAYS			RECOVERIES	
		WT. %	AG.	FE.	AG.	FE.
	Heads	100.0	23.4	2.97	100.0	100.0
30269	Conc.	5.4) 9.5	389.7) 228	22.2) 14.1	90.1) 92.7	40.4) 45.2
30270	Midd.	4.1)	16.2)	3.5)	2.6)	4.8)
30271	Tail.	90.5	1.88	1.8	7.3	54.8

PREAG. IN BALL MILL	TREATMENT	RETREATMENT
No reagents.	NaZ .2#	20-0 .05#
20 Minutes	4H .3	5 Minutes
30	20-0 .4	
	18 Minutes	

Tests 32 and 33 on Betty O'Neal give a much higher grade AG., with larger FE. rejection and slightly less AG. recovery using Mercaptide in test 32 than test 33 employing Xanthate.

Yours faithfully,

Exhibit—P-117.

Exhibit—K-47.

EXHIBIT P-117

CONSUMPTION OF FLOTATION REAGENTS

(From the Colorado School of Mines Quarterly
April, 1926)

DURING THE YEAR 1923

	Total for All Ores	Total for Lead and Lead-Zinc Ore
Pine Oil	5,346,370 lbs.	392,287 lbs.
Sodium Silicate	153,580 "	153,580 "
10 Sulphur	836,816 "	699,066 "
Sulphuric Acid	115,922,434 "	None.

EXHIBIT K-47

DEPARTMENT OF COMMERCE

UNITED STATES PATENT OFFICE

To all persons to whom these presents shall come, Greeting:

THIS IS TO CERTIFY that the annexed is a true copy from the records of this office of the File Wrapper and Contents, in the matter of the

20 Letters Patent of
Cornelius H. Keller, assignor to
Minerals Separation North American Corporation,
Number 1,554,216, Granted September 22, 1925,
for
Improvement in Froth-Flotation Concentration of Ores.

30 IN TESTIMONY WHEREOF I have hereunto set my
hand and caused the seal of the Patent Office
to be affixed at the City of Washington, this
first day of April, in the year of our Lord
one thousand nine hundred and forty - four
and of the Independence of the United States
of America the one hundred and sixty-eighth.

(S E A L)

ATTEST:

D. E. WILSON

Chief of Division.

CONWAY P. COE,

Commissioner of Patents.

NUMBER (Series of 1915), 1923 (EX'R'S BOOK)

670242
Div. 25

PATENT No. 1554216

Name CORNELIUS H. KELLER and KARL PIERCE LEWISAssor to Minerals Separation North American Corporation, of New
York, N.Y., a corp. of Maryland.

of SAN FRANCISCO BURLINGAME

10 *State of* CALIFORNIA*Invention*

FROTH FLOTATION CONCENTRATION OF ORES

ORIGINAL

RENEWED

APPLICATION FILED COMPLETE Oct. 23, 1923

PARTS OF APPLICATION FILED

<i>Petition, Specification,</i>	} Oct. 23, 1923
<i>Oath, First Fee, \$20.</i>	

<i>Examined and passed for Issue</i>	Aug. 19, 1925
20 J. H. Lightfoot	<i>Exr. Div. 25</i>

<i>Notice of Allowance</i>	Aug. 19, 1925
	<i>By Commissioner.</i>
<i>Final Fee \$20</i>	Aug. 21, 1925

Patented , 192

Attorney WILLIAMS & MORSE, 61 BROADWAY, N.Y., N.Y.
(No. of Claims Allowed 11) Print Claim 1 in O.G. (Cl. 83 85)
Title as Allowed Froth-Flotation Concentration of Ores.

Exhibit—K-47.

WILLIAMS & PRITCHARD
 Counsellors at Law, Patent Attorneys
 61 BROADWAY, NEW YORK, N.Y.

P E T I T I O N

TO THE COMMISSIONER OF PATENTS:

Your petitioners, CORNELIUS H. KELLER and CARL PIERCE LEWIS, citizens of the United States and residents respectively of San Francisco, County of San Francisco, State of California, and Burlingame, County of San Mateo, State of California, the post
 10 *office address of both being 220 Battery Street, San Francisco, California, pray that Letters Patent may be granted to them for improvements in*

FROTH FLOTATION CONCENTRATION OF ORES
as set forth in the annexed specification.

And they hereby appoint the firm of WILLIAMS & PRITCHARD, composed of Henry D. Williams and William S. Pritchard, of 61 Broadway, New York, N.Y., whose registry number is 10,918, their attorneys with full power of substitution and revocation, to
 20 *prosecute this application, to make alterations and amendments therein, to receive the patent, to sign the drawings, and to transact all business in the Patent Office connected therewith.*

Inventor's full name CORNELIUS H. KELLER
 CARL PIERCE LEWIS

S P E C I F I C A T I O N

TO ALL WHOM IT MAY CONCERN :

Per C Be it known, that we, CORNELIUS H. KELLER and CARL

PIERCE LEWIS,^a *citizens of the United States, and*^a *residents*
respectively of San Francisco, County of San Francisco, State of
Per C California, and Burlingame, County of San Mateo, State of Cali-
fornia, have invented certain new and useful improvements in

30 Froth-Fotation Concentration of Ores

of which the following is a specification.

This invention relates to the froth-flotation concentration of ores, and is herein described as applied to the concentration of certain ores with mineral-frothing agents in the presence of certain organic compounds containing sulphur.

It has been found that certain sulphur derivatives of carbonic acid greatly increase the efficiency of the froth-flotation process when used in connection with mineral-frothing agents. The increased

Exhibit—K-47.

efficiency shows itself sometimes in markedly better recoveries, sometimes in effecting the usual recoveries with greatly reduced quantities of the usual mineral-frothing agents, and sometimes in greatly reducing the time needed for agitation to produce the desired recoveries.

Insert D' The invention is herein disclosed in some detail as carried out with salts of the ~~alkyl~~ sulphur derivatives of carbonic acid known as xanthates, as the new substance. *These form anions and cations in solution.* Excellent results were also obtained by agitating ore pulps with the complex mixture produced
 10 when 33 $\frac{1}{3}$ % of pine oil was incorporated with an alcoholic solution of potassium hydrate, and xanthates or analogous substances were produced by adding carbon disulphide to this mixture.

The galena-bearing froth obtained with xanthates or analogous substances used at the rate of 0.2 pounds per ton of ore had a characteristic bright sheen, like a plumbago-bearing froth, and seemed to make a more coherent froth than when other minerals were used on the same ore.

In general the substances referred to are not mineral-frothing agents, — producing only a slight scum, and some evanescent frothy
 20 bubbles, when subjected to agitation which would produce mineral-bearing froth on an ore pulp in the presence of a mineral-frothing agent. The substances are effective in enabling a selective flotation of lead and zinc, and cause uncombined silver, if present, to tend to go into the lead concentrate rather than with the zinc, where these are separated in separate concentrates. Usually pre-agitation is unnecessary, the brightening and other effects seeming to be practically instantaneous. The pulps may be either acid, alkaline or neutral according to circumstances.

Insert C1 — For laboratory purposes potassium xanthate was prepared as
 30 follows:

198.4 grams of 88.5% caustic potash was dissolved in 524 grams ethyl alcohol (denatured No. 5 formula) at a temperature of 124°F., in a reflux condenser. The solution was cooled to 58°F. It contained a large excess of alcohol over the theoretical amount needed for the subsequent reactions. To this was added, while stirring, and in a cooling bath, the theoretical amount of carbon disulphide. The reaction was substantially instantaneous, producing a thick pulp of potassium xanthate. The pulp was cooled and centrifuged in a laboratory machine, yielding crystals containing about 20% moisture.
 40 The yield thus obtained was 74.7%. Another 17.5% was obtained by evaporation of the mother liquor. Both the centrifuged crystals and the residue from the mother liquor gave excellent results in flotation. It was found ~~that~~ in cases where ~~the use of~~ sulphuric acid ~~is~~
Per C ~~was used that~~
" C ~~necessary~~ the centrifuged material yielded better results than the uncentrifuged.

Exhibit—K-47.

A pulp of Anaconda slimes which had been stored for several days assaying 2.95% copper (a part being oxidized copper mineral) was treated with cresylic acid as frothing agent and potassium xanthate, the latter used at the rate of half a pound to a ton of the slimes. With no pre-agitation, and fifteen minutes agitation in a laboratory sub-aeration machine these slimes yielded a concentrate containing 15.6% copper, a middling containing 0.48% copper and a tailing containing 0.082% copper. The small proportion of silver present was recovered in about the same proportions. Similar results
10 were obtained with the use of sodium xanthate and General Naval Stores No. 5 flotation oil, a steam distilled pine oil.

A pulp of Cash mine ore subjected first to agitation for ten minutes with a mixture of 0.3 pounds per ton of potassium xanthate with a small proportion of a 10% of a saturated solution of naphthalene in xylene, to yield a lead concentrate, and then subjected to agitation for ten minutes with copper sulphate 0.2 pounds per ton, Barrett No. 4 flotation oil 1.0 pound per ton, and General Naval Stores No. 5 flotation oil 0.1 pounds per ton, yielded the results shown in the following table. Attention is called to the recovery of 95% of the lead
20 in a concentrate containing 87% of the silver but containing only 5% of zinc, while 70% of the zinc was recovered in the zinc concentrate.

Cash Mine Ore, Assays

	% Wt.	Au. (oz.)	Ag. (oz.)	Cu.	Pb.	Zn.	Fe.
Heads	100.0	.154	10.7	3.18	14.9	7.7	11.9
Pb. conc.	38.0	.24	24.6	7.12	37.4	5.0	21.4
Zn. conc.	15.4	.36	6.3	2.56	3.0	35.2	12.0
Tails.	46.6	.015	0.9	.26	0.5	0.8	4.2

% Recoveries

	Au.	Ag.	Cu.	Pb.	Zn.	Fe.
Pb. conc.	59.4	87.1	85.2	95.3	24.7	68.4
Zn. conc.	36.0	9.0	11.0	3.1	70.5	15.5
Tails.	4.6	3.9	3.8	1.6	4.8	16.4

A pulp of San Francisco Mines of Mexico ore was agitated for ten minutes with potassium xanthate 0.15 pounds per ton and the same amount of coal tar creosote to yield a lead concentrate. The remaining pulp was agitated for fifteen minutes with 0.2 pounds per ton of copper sulphate, 1.2 pounds per ton of water-gas tar, and 0.05 pounds per ton of steam distilled pine oil. The results are shown in
40 the following table:

San Francisco Mines of Mexico

	% ASSAYS				% RECOVERIES		
	% Wt.	Ag. (oz.)	Pb.	Zn.	Ag.	Pb.	Zn.
Heads	100.0	16.5	9.4	16.1
Pb. conc.	12.2	67.0	64.0	12.0	49.4	83.2	9.1
Zn. conc.	28.9	23.6	3.6	43.4	41.2	11.1	78.0
Tails.	58.9	4.5	1.5	6.0	9.4	5.7	12.9

Similar results were obtained with this ore by agitating for five
 10 minutes with 0.15 pounds each of sodium xanthate and Barrett No. 2
 flotation oil per ton of ore, to yield a lead concentrate, and then
 agitating for ten minutes with 0.3 pounds copper sulphate, 1.3 pounds
 Barrett No. 4 flotation oil, and General Naval Stores No. 5 flotation
 oil 0.1 pounds, all per ton of ore, to yield a zinc concentrate.

During a seven and one-half hours run on current Anaconda
 slimes at the rate of one hundred and eleven tons per twenty-four
 hours in a minerals separation standard machine from a feed containing
 3.28% copper, 0.26% being acid soluble copper, there was recovered a
 concentrate containing 11.43% Cu. and 36.9% insolubles, — the
 20 latter being a proportion desirable for smelting. The tailings con-
 tained 0.29% copper of which 0.20% was acid soluble copper. This
 test was run at a temperature of 82°F. using for reagents, 7.89 pounds
 of kerosene acid sludge, 21.6 pounds of chamber sulphuric acid, 2.36
 pounds of hard wood creosote, and 2.92 pounds of a twenty per cent
 solution of potassium xanthate in water all quantities being in pounds
 per short ton of ore.

Anaconda old gravity concentration tailings reground for
 flotation were treated at the rate of four hundred and eleven tons per
 twenty-four hours in a minerals separation standard machine pro-
 30 vided with a porous bottom in the spitzkasten through which air was
 admitted. From a feed containing 0.52% copper, of which 0.12%
 was acid soluble copper there was recovered a concentrate containing
 5.72% copper and 48% insolubles. The tailings from this contained
 0.13% copper of which 0.08% was acid soluble. This test was run at
 a temperature of 50°F. using for reagents per ton of solids, 0.67
 pounds of a 30% solution of potassium xanthate in water, 3.96
 pounds of kerosene acid sludge, and 11.45 pounds of chamber
 sulphuric acid.

Anaconda ore slimes containing 3.28% copper, of which 0.45%
 40 was acid soluble, were subjected to froth-flotation concentration at
 70 F. with 7.8 pounds per ton of dry hydrated lime, 0.132 pounds
 per ton of steam distilled pine oil, 0.38 pounds per ton of destructively

Exhibit—K-47.

distilled pine oil (G.N.S. No. 11 flotation), and 0.69 pounds per ton of potassium xanthate in solution, yielding concentrates containing

Per C 13.76% copper and 34.7% insolubles, with a tailing of 0.317⁰/₁₀₀ copper of which 0.173% was acid soluble. This represented a recovery of 92.2%.

The lime was mixed with water and fed as a watery paste with the slimes to the first agitator of a series of nine Standard Minerals Separation agitators or mixing compartments through which the pulp passed in series at the rate of 97 tons in 24 hours. The No. 11 pine
10 oil was added at the seventh agitator, and 0.6 pounds per ton of the xanthate at the ninth. The pulp returned through fourteen spitzkastens, each equipped with a Brown aerator and circulating device. At the sixth spitzkasten the balance of the xanthate was added. At the tenth spitzkasten the steam distilled pine oil was added. The froth from the first nine spitzkastens was collected as a finished concentrate, that from the remaining five was returned to the first as a middling.

It has also been found that for the froth-flotation concentration of certain ores¹ such as Calumet and Hecla, Britannia, Cananea and

Per C 20 Moctezuma¹ mother liquor obtained from centrifuging the above described xanthates in the process of manufacture of said xanthates may be used with about the same proportions of mother liquor as of the xanthate crystals specified with results as good or better than those above given.

Insert C² ————— Having thus described certain embodiments of the invention, what is claimed is:

Per D 30 1. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a sulphur derivative of carbonic acid¹ adapted to form in solution anions and cations and adapted to co-operate with a mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

Per D 40 2. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of a sulphur derivative of carbonic acid¹ adapted to form in solution anions and cations and adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

3. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of an alkyl sulphur derivative of carbonic acid adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of

Exhibit—K-47.

the ore, said agitation being so conducted as to form such a froth, and separating the froth.

4. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of an ethyl-sulphur derivative of carbonic acid adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

10 5. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and an alkali-metal salt of an ethyl-sulphur derivative of carbonic acid adapted to co-operate with the mineral-frothing agent to produce by the actions of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

20 6. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and an alkaline xanthate adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

In testimony whereof, I have affixed my signature to this specification.

Inventor's full name CORNELIUS H. KELLER.
CARL PIERCE LEWIS

O A T H

City and

County of San Francisco } ss.

30 State of California.

CORNELIUS H. KELLER and CARL PIERCE LEWIS

the above named petitioners, being duly sworn depose and say that they are citizens of the United States, and residents respectively of San Francisco, County of San Francisco, State of California, and Burlingame, County of San Mateo, State of California, that they verily believe themselves to be the original, first, and joint inventors of the improvements in

FROTH FLOTATION CONCENTRATION OF ORES

40 *described and claimed in the annexed specification; that they do not know and do not believe that the same was ever known or used before their invention or discovery thereof, or patented or described in any printed*

Exhibit—K-47.

publication in any country before their invention or discovery thereof, or more than two years prior to this application, or in public use or on sale in the United States for more than two years prior to this application; that said invention has not been patented in any country foreign to the United States on an application filed by them or their legal representatives or assigns more than twelve months prior to this application; and that no application for patent on said improvement has been filed by them or their representatives or assigns in any country foreign to the United States.

10 Inventor's full name CORNELIUS H. KELLER
CARL PIERCE LEWIS

Subscribed and sworn to before me this 15th day of October, 1923.

O. A. EGGERS,

(Signature of Notary or Justice)

Notary Public in and for the City and County
of San Francisco, State of California.

(Seal)

(Official Character)

Div. 25 Room 315

Address only

20 "The Commissioner of Patents,
Washington, D.C.,"

and not any official by name.

2-260

Paper No. 2

TEM : GB All communications respecting this
application should give the serial number,
date of filing, title of invention, and
name of the applicant.

DEPARTMENT OF THE INTERIOR

UNITED STATES PATENT OFFICE

30 WASHINGTON October, 31, 1923

Williams & Pritchard,
61 Broadway,
New York, N.Y.

*Pease find below a communication from the EXAMINER in charge
of the application of*

Keller and Lewis, filed Oct. 23, 1923, Ser. No. 670,242

FROTH FLOTATION CONCENTRATION OF ORES

THOMAS E. ROBERTSON,

Commissioner of Patents.

Exhibit—K-47.

This application has been examined.

Claims 1 and 2 are rejected as too broad since it is not evident that all sulfur derivatives of carbonic acid will serve the purpose of froth selectivity modifiers; and as without patentable novelty in view of the disclosure of carbon bisulfide in the prior art. See lines 17—20, page 218, The Flotation Process, Megraw, McGraw-Hill Book Co., Inc., New York, 1916, copy in Room 315, this office.

The remaining claims may be allowed as at present advised.

The following claims are suggested with a view to an inter-
10 fference:

The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate.

The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of an alkaline pulp to a flotation operation in the presence of a xanthate.

The improvement in the concentration of minerals by flotation which comprises subjecting the minerals in the form of a pulp made alkaline by the addition of lime to a flotation operation in the
20 presence of a xanthate.

The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of potassium xanthate.

The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of an alkaline pulp to a flotation operation in the presence of potassium xanthate.

The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a pulp made alkaline by the addition of lime to a flotation operation in the
30 presence of potassium xanthate.

The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate and a frothing agent.

The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of an alkaline pulp to a flotation operation in the presence of potassium xanthate and a frothing agent.

The improvement in the concentration of minerals by flotation
40 which comprises subjecting the mineral in the form of a pulp made alkaline by the addition of lime to a flotation operation in the presence of potassium xanthate.

Applicants are given thirty days from the date hereof within which to adopt said claims. Failure to so adopt will, without

Exhibit—K-47.

further action, be treated as a disclaimer of the subject matter thereof.

J. H. LIGHTFOOT,
Examiner, Div. 25.
M.

Recd Nov. 17/23

UNITED STATES PATENT OFFICE

In the application of Cornelius H. Keller
and Carl Pierce Lewis
Froth Flotation Concentration of Ores,

10 Serial No. 670,242, filed October 23, 1923

Hon. Commissioner of Patents,

Division 25

Room 315.

Sir:—

Please add the following claims:

- a*' 7. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate.
8. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of an alkaline pulp to a flotation operation in the presence of a xanthate.
- 20 9. The improvement in the concentration of minerals by flotation which comprises subjecting the minerals in the form of a pulp made alkaline by the addition of lime to a flotation operation in the presence of a xanthate.
- a*' ~~8~~ 10. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of potassium xanthate.
11. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of an alkaline pulp to a flotation operation in the presence of potassium xanthate.
- 30 12. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a pulp made alkaline by the addition of lime to a flotation operation in the presence of potassium xanthate.
- a*' ~~9~~ 13. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate and a frothing agent.
- 40 14. The improvement in the concentration of minerals by flotation which comprises subjecting the minerals in the form of an

Exhibit—K-47.

alkaline pulp to a flotation operation in the presence of potassium xanthate and a frothing agent.

15. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a pulp made alkaline by the addition of lime to a flotation operation in the presence of potassium xanthate.

REMARKS

The claims are added at the suggestion of the Examiner in his letter dated October 31, 1923, for the purpose of interference.

10

Respectfully,

WILLIAMS & PRITCHARD
Attorneys.

Div. 25 Room 315

Address only

"The Commissioner of Patents,
Washington, D.C.,"
and not any official by name.

Paper No. 4

20

2-960

All communications respecting this
application should give the serial number
date of filing, title of invention, and
name of the applicant.

TFM : GB

DEPARTMENT OF THE INTERIOR

UNITED STATES PATENT OFFICE

WASHINGTON

December 3, 1923

Williams & Pritchard,
61 Broadway,
New York, N.Y.

30 *Please find below a communication from the EXAMINER in charge
of the application of*

Keller and Lewis, filed Oct. 23, 1923, Ser. No. 670,242

FROTH FLOTATION CONCENTRATION OF ORES

THOMAS E. ROBERTSON,
Commissioner of Patents.

Exhibit—K-47.

Responsive to the amendment filed Nov. 17, 1923:

Applicants are requested to cancel claim 15, for the reason that said claim is a duplicate of claim 12, which fact was overlooked when the Office letter of October 31, 1923 was formulated.

Response is requested within a period of ten days.

J. H. LIGHTFOOT,
Examiner, Div. 25.

UNITED STATES PATENT OFFICE

10	In the application of Cornelius H. Keller, and Carl Pierce Lewis, Froth Flotation Concentration of Ores, Filed October 23, 1923, Serial No. 670,242.	} } } }	Division 25 Room 315.
----	---	------------------	------------------------------

Hon. Commissioner of Patents.

Sir:

In response to Official letter of December 3, 1923, applicants herewith cancel claim 15.

Respectfully,

WILLIAMS & PRITCHARD,
Attorneys.

20

2—079

INTERFERENCE.

Interference No. 50394

Paper No. 6

Name, C. H. Keller

Serial No. 670,242

Title, Froth Flotation Concentration of Ores

Filed, October 23, 1923

Interference with Ralph E. Sayre

DECISIONS OF

Ex'r. of Interferences, Favorable

Dated, Aug. 4, 1925

Exhibit—K-47.

Forwarded from 25 Div. to
Examiner of Interferences.

Paper No. 7
[INTERFERENCE.]

TFM:GB

DEPARTMENT OF THE INTERIOR,
UNITED STATES PATENT OFFICE,
WASHINGTON, D. C.

Williams & Pritchard,
61 Broadway,
New York, N.Y.

- 10 *Please find below a copy of a communication from the Examiner concerning the application of Keller and Lewis, filed October 23, 1923, Ser. No. 670,242, for Froth Flotation Concentration of Ores.*

Very respectfully,

Room No. 315

ADDRESS ONLY
THE COMMISSIONER OF PATENTS
WASHINGTON, D. C.

THOMAS E. ROBERTSON,
Commissioner of Patents.

- 20 *The case, above referred to, is adjudged to interfere with others, hereafter specified, and the question of priority will be determined in conformity with the Rules.*

The statement demanded by Rule 110 must be sealed up and filed on or before Mar. 3, 1924, with the subject of the invention, and name of party filing it, indorsed on the envelope. The subject-matter involved in the interference is

1. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of an alkyl sulphur derivative of carbonic acid adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of
30 the ore, said agitation being so conducted as to form such a froth, and separating the froth.
2. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of an ethyl-sulphur derivative of carbonic acid adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.
3. The process of concentrating ores which consists in agitating
40 a suitable pulp of an ore with a mineral-frothing agent and an alkali-metal salt of an ethyl-sulphur derivative of carbonic acid adapted to co-operate with the mineral-frothing agent to produce by the actions of both a mineral-bearing froth containing a large proportion of a

Exhibit—K-47.

mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

4. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and an alkaline xanthate adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

10 5. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate.

6. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of an alkaline pulp to a flotation operation in the presence of a xanthate.

7. The improvement in the concentration of minerals by flotation which comprises subjecting the minerals in the form of a pulp made alkaline by the addition of lime to a flotation operation in the presence of a xanthate.

20 8. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of potassium xanthate.

9. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of an alkaline pulp to a flotation operation in the presence of potassium xanthate.

10. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a pulp made alkaline by the addition of lime to a flotation operation in the presence of potassium xanthate.

30 11. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate and a frothing agent.

12. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of an alkaline pulp to a flotation operation in the presence of potassium xanthate and a frothing agent.

The interference involves your application above identified, and

40 (a) An application for Concentration of Ores by Flotation, by Ralph E. Sayre, of 50 East 41st St., New York, N.Y., whose attorneys are Pennie, Davis, Marvin & Edmonds, of 165 Broadway, New York, N.Y.

(c) The relation of the counts of the interference to the claims of the respective parties is as follows:

Exhibit—K-47.

	Counts	Keller et al.	Sayre
	1	3	9
	2	4	10
	3	5	11
	4	6	12
	5	7	1
	6	8	2
	7	9	3
	8	10	4
10	9	11	5
	10	12	6
	11	13	7
	12	14	8

Counts compared.

J. H. LIGHTFOOT,
Examiner, Div. 25.

Recd Mar. 23/24

UNITED STATES PATENT OFFICE

In application of Keller and Lewis,
Froth-Flotation Concentration of Ores, } Division 25
20 Filed October 23, 1923; Serial No. 670,242 } Room 315.

Hon. Commissioner of Patents,
Washington, D.C.

Sir:

Please amend as follows:

Page 1, line 7 from bottom. Change "we" to - I, and strike out "and Carl Pierce Lewis".

Page 1, line 6 from bottom. Change "citizens" to - a citizen, and "residents respectively" to - a resident.

30 Page 1, lines 5 and 4 from bottom. Erase all words commencing with "and Burlingame" and ending with "California".

40 Page 3, after line 10 insert the following:—Two sticks of caustic potash weighing perhaps 15 grams were partly immersed in about 80 cc. of commercial carbon disulphide and kept for about ten days in a closed bottle containing some air in the warm region of the laboratory where the hot plates used for drying. These eventually yielded a yellow or orange salt which was used with pine oil at the rate of approximately half a pound to a ton of ore in concentrating Hibernia ore from Timber Butte Mining Company. The test was with a neutral pulp, and the concentrates were seen to be clean with brightened lead sulphide particles.

Page 3, line 27. After "found" erase "that"; after "where" erase "the use of".

Exhibit—K-47.

Page 3, line 28. Erase "is necessary" and insert in place thereof - was used that -.

Page 4, line 2 of first table at bottom of page. Change "Conc.Pb." to - Pb. Conc. - .

Page 4, line 1 of second table at bottom of page. Change "Conc Pb" to - Pb. Conc. - .

Page 5, line 9. Erase the comma after "Francisco".

Page 6, line 16. Erase "An".

Page 6, line 17. Change "was" to - were - .

10 Page 6, line 24. Insert - % - after "0.317".

Page 7, line 11. Insert a comma after "ores".

Page 7, line 12. Insert a comma after "Moctezuma".

Page 7, after the last paragraph insert the following:

20 A pulp of 80 mesh Calumet and Hecla slimes containing 0.45% copper, mostly native, with considerable mineral occluded in the coarses particles of gangue, was agitated five minutes with 0.3 pounds potassium xanthate, then was agitated for twenty minutes with 0.4 pounds steam distilled pine oil, all per ton of ore. The concentrate thus obtained was agitated for five minutes and the froth separated to form a finished concentrate, the remainder forming a middling. The results are shown in the following table:

c 2

	% Wt.	% Assays Cu.	% Recoveries Cu.
Heads.....	100.0	0.45	
Conc.....	0.7	41.72	64.5
Midd.....	3.9	1.44	12.3
Tails.....	95.4	0.11	23.2

30 A Chinese graphite ore containing 49% carbon as graphite, chiefly in amorphous state, ground to 80 mesh, was agitated ten minutes with 0.2 pounds potassium xanthate and 0.15 pounds pine oil, both per ton of ore, yielding a froth concentrate and a tailing. The concentrate was reagitated for five minutes to yield a finished concentrate and a middling.

The finished concentrate contained 72.8% carbon as graphite, the middling 36.8%, and the tailing 15.2%. These represented respectively 79.2%, 10.2% and 10.6% of the original graphite content of the ore.

40 In the absence of xanthate the same ore required a larger amount of mineral-frothing agent and longer agitation to produce the same results.

Cancel claims 8, 9, 11, 12, 14, and adjust ordinals of remaining claims.

Exhibit—K-47.

Page 10, line 1. Change “we” to—I, “our”to—my, and “signatures” to—signature.

At the end of the specification cancel the signature “Carl Pierce Lewis”.

File the annexed amended petition and supplemental oaths.

REMARKS

In accordance with the rulings of the Court of Appeals D.C. In re Roberts, 273 O.G. 410, 1920 C.D. 158; 263 Fed. 646; Bijur v. Kennington, 295 O.G. 887, 1922 C.D.; 278 Fed. 313, and Briggs
 10 v. Kaisling, 311 O.G. 3, 1923 C.D.; 288 Fed. at 256, this joint application is above amended so as to become the sole application of Cornelius H. Keller, one of the original joint applicants. Claims 8, 9, 11, 12 and 14 are cancelled herefrom and are concurrently incorporated in a separate application of the other of said joint applicants, Carl Pierce Lewis. The new petition and supplemental oaths of Messrs. Keller and Lewis are annexed hereto.

The Primary Examiner is requested to take jurisdiction of this application for the purpose of considering this amendment. The time for filing preliminary statements has not expired and Messrs.
 20 Keller and Lewis have not filed a preliminary statement.

The amendments to the specification consist merely in adding examples of the practice of the invention. The claims retained have not been altered in any respect. The amendments are believed to be properly admissible at this time. In *Badische A. u. S. F. v. Klipsteim*, 125 Fed. at 554, it was held, quoting *Godfrey v. Eames*, 1 Wall, 324, that an additional example of a method of making a product did not “affect the sufficiency of the original application, or the legal consequences flowing from it.” So also in *Schillinger v. Gunther*, 16 O.G. 905, 1879 C.D. 610, it was held that a reissue
 30 was not void even though it contained the result of experiments conducted after the issue of the original patent.

The Examiner is requested to reconsider his rejection of claims 1 and 2. The reference merely says “Carbon bisulphide and rosin oil (1:1 by weight) gave * * * with an extraction of 50%”. There is no mention of the presence of any reagent in sufficient quantity to react with the carbon bisulphide. The conditions described do not indicate the presence of the alkali necessary to form the “sulphur derivative of carbonic acid” called for by claims 1 and 2.

Even if such might possibly have been formed by unmentioned
 40 ingredients, the reference is fatally defective since it in no way mentions the formation of “a sulphur derivative of carbonic acid”, and moreover is a description of a test which failed. The “extraction of 50% by the carbon-bisulphide rosin-oil mixture was a failure as evidenced by comparison with the “extraction of over 73%” obtained with pine oil on the same ore and described on the preceding page

Exhibit—K-47.

of the Megraw book. In this connection the Examiner's attention is called to *Permutit Co. v. Harvey Laundry Co.*, 279 Fed. at 718, where the Court of Appeals for the Second Circuit, speaking through Manton, C.J., said:

"It is well established that complete failure by an inoperative and abandoned apparatus cannot defeat a later meritorius and successful invention. If the apparatus did not, when constructed, anticipate the inventive thought, a description of it could do no more."

10

Very respectfully,

WILLIAMS & PRITCHARD,
Attorneys.

UNITED STATES PATENT OFFICE.

In application of Keller and Lewis,	}	Division 25
Froth-Flotation Concentration of Ores,		

Filed October 23, 1923; Serial No. 670,242

AMENDED PETITION.

To the Commissioner of Patents:

20 Your Petitioner Cornelius H. Keller, one of the two applicants
 30 Cornelius H. Keller and Carl Pierce Lewis who on October 23,
 1923, filed application for letters patent Serial No. 670,242 for
 improvements in froth-flotation concentration of ores, prays that
 letters patent may be granted upon said application to his
 assignee Minerals Separation North American Corporation for the
 said invention as described and claimed in the specification of
 said application as above amended.

CORNELIUS H. KELLER.

30 Carl Pierce Lewis, one of the two joint applicants Cornelius
 H. Keller and Carl Pierce Lewis who on October 23, 1923, filed
 application for letters patent Serial No. 670,242, for improvements
 in froth-flotation concentration of ores, hereby consents to the
 above amended petition.

CARL PIERCE LEWIS.

Exhibit—K-47.

Minerals Separation North American Corporation, assignee of the above-entitled application, hereby consents to the above amended petition.

(signature illegible)
President.

Attest:

ALBERT ROBERTS,
Secretary.

UNITED STATES PATENT OFFICE.

10	In application of Keller and Lewis, Froth-Flotation Concentration of Ores, Filed October 23, 1923; Serial No. 670,242	}	Division 25
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SUPPLEMENTAL OATH OF CARL PIERCE LEWIS

State of New York County of New York	}	ss.
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Carl Pierce Lewis being duly sworn deposes and says that he is one of the two joint applicants Cornelius H. Keller and Carl Pierce Lewis who on October 23, 1923, filed application for letters
 20 patent Serial No. 670,242 for improvements in froth-flotation concentration of ores; that he joined with said Cornelius H. Keller in executing and filing the said application, including the oath sworn to by said applicants as a part of said application, under the mistaken belief that he and the said Cornelius H. Keller were joint inventors of the invention described and claimed in said application and without fraudulent or deceptive intent; that he has since the declaration of interference No. 50,394 involving the said application and an application of Ralph E. Sayre, as the result
 30 of careful consideration of the facts in relation to the origination and development of the said invention, reached the conclusion that he is not a joint inventor and that he did not in any way participate in the invention described in said application and claimed in claims 1, 2, 3, 4, 5 and 6 thereof, *i.e.* all the claims of said joint application as filed, and claims 7, 10 and 13 of said application which were suggested by the Patent Office and inserted by amendment preliminarily to the declaration of said interference, and that he is the sole inventor of the invention described in said application and claimed in claims 8, 9, 11, 12 and 14 thereof,

Exhibit—K-47.

which were suggested by the Patent Office and inserted by amendment preliminarily to the declaration of said interference.

Subscribed and sworn to before }
me this 26th day of March, 1924 } CARL PIERCE LEWIS.

F. L. REQUA
Notary Public, N. Y. County No. 298

UNITED STATES PATENT OFFICE.

In application of Keller and Lewis,
Froth-Flotation Concentration of Ores, } Division 25
10 Filed October 23, 1923; Serial No. 670,242 }

SUPPLEMENTAL OATH OF CORNELIUS H. KELLER

State of New York }
County of New York. } ss.

Cornelius H. Keller being duly sworn deposes and says that he is one of the two joint applicants Cornelius H. Keller and Carl Pierce Lewis who on October 23, 1923, filed application for letters patent Serial No. 670,242 for improvements in froth-flotation concentration of ores; that he joined with the said Carl
20 Pierce Lewis in executing and filing the said application, including the oath sworn to by said applicants as part of said application, under the mistaken belief that he and the said Carl Pierce Lewis were joint inventors of the invention described and claimed in said application and without fraudulent or deceptive intent; that he has since the declaration of interference No. 50,394 involving said application and an application of Ralph E. Sayre, as the result of careful consideration of the facts in relation to the origination and development of the said invention, reached the conclusion that
30 he is the sole inventor of the invention described in said application and claimed in claims 1, 2, 3, 4, 5 and 6 thereof, *i.e.* all the claims of said application as filed, and claims 7, 10 and 13 of said application, which were suggested by the Patent Office and inserted by amendment preliminarily to the declaration of said interference, and that he is not the inventor and did not participate in the invention described in said application and claimed in claims 8, 9, 11, 12 and 14 thereof, which were suggested by the Patent Office and inserted by amendment preliminarily to the declaration of said interference; that he verily believes himself to be the original, first and sole inventor of the invention described in said

Exhibit—K-47.

application and claimed in said claims 1 to 7 inclusive and 10 and 13 thereof; that he does not know and does not believe that the same was ever known or used before his invention or discovery thereof or more than two years prior to the filing of said joint application or in public use or on sale in the United States for more than two years prior to the filing of said joint application; that said sole invention has not been patented in any country foreign to the United States on an application filed by him or his legal representatives or assigns more than twelve months prior
 10 to the filing of said joint application, and that no application for a patent on said sole invention was filed by him or his legal representatives or assigns in any country foreign to the United States prior to the filing of said joint application, and that said sole invention has not been abandoned.

Subscribed and sworn to before }
 me this 25th day of March, 1924. } CORNELIUS H. KELLER.

F. L. REQUA,
 Notary Public, N. Y. County No. 298

	Div. 25	Room 315	Paper No. 9
20	Address only		All communications respecting this application should give the serial number, date of filing, title of invention, and name of the applicant.
	"The Commissioner of Patents, Washington, D.C.,"		
	and not any official by name.		
			2—260

TFM : GB

DEPARTMENT OF THE INTERIOR

UNITED STATES PATENT OFFICE

WASHINGTON

Williams & Pritchard,
 30 61 Broadway,
 New York, N.Y.

Please find below a communication from the EXAMINER in charge of the application of

Cornelius H. Keller, filed Oct. 23, 1923, Ser. No. 670,242

Froth Flotation Concentration of Ores.

THOMAS E. ROBERTSON,

Commissioner of Patents.

Exhibit—K-47.

You are advised (a) that the joint application of Keller & Lewis, Ser. No. 670,242, filed Oct. 23, 1923, for Froth Flotation Concentration of Ores, involved in Interference No. 50,394, has been changed by amendment to a sole application by you; (b) that said interference No. 50,394, is modified by withdrawal of counts 6, 7, 9, 10 and 12, and continued as to the remaining counts, counts 8 and 11 being renumbered 6 and 7.

The new relation of the claims of the respective parties is as follows:

10	Counts	Keller	Sayre
	1	3	9
	2	4	10
	3	5	11
	4	6	12
	5	7	1
	6	8	4
	7	9	7

The statement, as to the counts of the issue now remaining, demanded by Rule 110, must be sealed up and filed on or before
 20 June 9, 1924, with the subject of the invention, and name of the party filing it, indorsed on the envelope.

J. H. LIGHTFOOT,
 Examiner, Div. 25.

In the matter of the application of C. H. KELLER Froth-Flotation Concentration of Ores. Serial No. 670,242, Filed Oct. 23, 1923.	}	Division 25 Room 315.
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TO THE COMMISSIONER OF PATENTS:

SUBSTITUTE POWER OF ATTORNEY

30 We hereby substitute for ourselves the firm of Williams & Morse, composed of Henry D. Williams and Howard M. Morse, of No. 61 Broadway, New York, N.Y., whose Registry No. is 12,068, as attorneys in the matter of the above application, with full power of substitution and revocation, to present and prosecute this application, to make alterations and amendments therein, to sign the drawings, to receive the patent, to transact all business in the Patent Office connected therewith, and to whom all communications should be addressed.

WILLIAMS & PRITCHARD,
Attorneys of Record.

Exhibit—K-47.

UNITED STATES PATENT OFFICE.

CORNELIUS H. KELLER	}	Interference No. 50,394.
vs.		
RALPH E. SAYRE		

SUBSTITUTE POWER OF ATTORNEY.

We hereby substitute for ourselves the firm of Williams & Morse, composed of Henry D. Williams and Howard M. Morse, of No. 61 Broadway, New York, N.Y., whose Registry Number is 12,068, as attorneys for Carl Pierce Lewis in the above-entitled
10 Interference.

WILLIAMS & PRITCHARD,
Attorneys of Record.

UNITED STATES PATENT OFFICE

In the application of Conrelius H. Keller Froth-Flotation Concentration of Ores, Serial No. 670,242, filed October 23, 1923	}	Division 25 Room 315.

Hon. Commissioner of Patents,

Sir:—

20 Please amend the above-entitled application in the following particulars.

Page 2, line 16. Erase "alkyl".

25 *S* ' | Page 2, line 17. After "acid" insert - containing an organic radical, such as an alkyl radical, and

Page 2, line 17. Insert a comma after "xanthates".

Page 2, line 18. Before "Excellent" insert - These form anions and cations in solution. -

Claim 1, line 1. Change "ore" to - ores -

Claim 1, line 3. After "acid" insert - adapted to form in solution anions and cations -

30 Claim 2, line 4. After "acid" insert - adapted to form in solution anions and cations and -

Add the following claims:

10. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a sulphur derivative of carbonic acid containing an organic radical and adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

Exhibit—K-47.

11. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of a sulphur derivative of carbonic acid containing an organic radical and adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth, said agitation being so conducted as to form such a froth, and separating the froth.

R E M A R K S

The interference involving this application having been terminated by the filing of a written concession of priority by the opposing party, Sayre, the above amendment has been made to put the application in condition for immediate allowance.

During the interference the invention has gone into wide and extensive public use in the treatment of scores of thousands of tons of ore per day, and it is therefore highly desirable that the patent should be issued as soon as possible.

Claims 1 and 2 have been amended to clearly differentiate from the carbon disulphide of the prior art by describing the sulphur derivative of carbonic acid as adapted to form in solution anions and cations. Carbon disulphide does not have this property.

Two claims, 10 and 11, have been added which differentiate from the carbon disulphide of the prior art by describing the sulphur derivative as containing an organic radical. Carbon disulphide does not contain such a radical.

Respectfully,

Dated New York
August 5th, 1925

WILLIAMS & MORSE,
Attorneys for Keller.

Div. 25-S

2-181

ADDRESS ONLY
30 THE COMMISSIONER OF PATENTS
WASHINGTON, D. C.

Serial No.
670,242

UNITED STATES
DEPARTMENT OF COMMERCE

PATENT OFFICE

WASHINGTON

August nineteenth, 1925.

Cornelius H. Keller, Assor.

Sir: Your APPLICATION for a patent for an IMPROVEMENT
in

40

Froth Flotation Concentration of Ores

Exhibit—K-47.

filed Oct. 23, 1923 has been examined and ALLOWED (11 claims).

The final fee, TWENTY DOLLARS, must be paid not later than SIX MONTHS from the date of this present notice of allowance. If the final fee be not paid within that period, the patent on this application will be withheld, unless renewed within two years after the date of this notice with an additional fee of \$20, under the provisions of Section 4897, Revised Statutes.

10 The office delivers patents upon the day of their date, and on which their term begins to run. The printing, photolithographing, and engrossing of the several patent parts, preparatory to final signing and sealing, will require about four weeks, and such work will not be undertaken until after payment of the necessary fee.

20 When you send the final fee you should also send, DISTINCTLY AND PLAINLY WRITTEN, the name of the INVENTOR, TITLE OF INVENTION, AND SERIAL NUMBER AS ABOVE GIVEN, DATE OF ALLOWANCE (which is the date of this circular), DATE OF FILING, and, if assigned, the NAMES OF THE ASSIGNEES.

If you desire to have the patent issue to ASSIGNEES, an assignment containing a REQUEST to that effect, together with the FEE for recording the same, must be filed in this office on or before the date of payment of final fee.

After issue of the patent uncertified copies of the drawings and specifications may be purchased at the price of TEN CENTS EACH. The money should accompany the order. Postage stamps will not be received.

30 Final fees will NOT be received from other than the applicant, his assignee, or attorney, or a party in interest as shown by the records of the Patent Office.

Respectfully,

THOMAS E. ROBERTSON,
Commissioner of Patents.

Williams and Morse,
61 Broadway,
New York, N.Y.

MAILED
AUG. 19, 1925

40 IN REMITTING THE FINAL FEE GIVE THE SERIAL NUMBER AT THE HEAD OF THIS NOTICE.

UNCERTIFIED CHECKS WILL NOT BE ACCEPTED.

Exhibit—K-47.

WILLIAMS & MORSE
 COUNSELLORS AT LAW, PATENT ATTORNEYS
 61 BROADWAY, ADAMS BUILDING
 NEW YORK

HENRY D. WILLIAMS
 HOWARD M. MORSE

—
 EDWARD THOMAS
 10 WILLIAM C. MILES

PATENTS
 TRADEMARKS
 COPYRIGHTS
 —
 TELEPHONE
 BOWLING GREEN 6787
 —
 CABLE ADDRESS
 ALASTOR NEW YORK
 August 20th, 1925

Hon. Commissioner of Patents,
 Washington, D.C.

Sir:—

Herewith we send you our check for twenty dollars in payment of the final government fee in the application for United States Letters Patent of Cornelius H. Keller, filed October 23rd, 1923. 20 Serial No. 670,242, improvements in Froth Flotation Concentration of Ores, which application was allowed on August 19th, 1925.

Please cause the patent to issue to the assignee, Minerals Separation North American Corporation, and send it to us.

Respectfully,

WILLIAMS & MORSE,
Atty. for Applicant

JEL.
 1 enc.

Patented Sept. 22, 1925

1,554.216

UNITED STATES PATENT OFFICE

CORNELIUS H. KELLER, OF SAN FRANCISCO, CALIFORNIA, ASSIGNOR
TO MINERALS SEPARATION NORTH AMERICAN CORPORATION, OF
NEW YORK, N.Y., A CORPORATION OF MARYLAND.

FROTH-FLOTATION CONCENTRATION OF ORES.

No Drawing. Application filed October 23, 1923. Serial No. 670,242.

To all whom it may concern:

Be it known that I, CORNELIUS H. KELLER, a citizen of the
10 United States, and a resident of San Francisco, county of San Fran-
cisco, State of California, have invented certain new and useful
Improvements in Froth Flotation Concentration of Ores, of which
the following is a specification.

This invention relates to the froth-flotation concentration of
ores, and is herein described as applied to the concentration of certain
ores with mineral frothing agents in the presence of certain organic
compounds containing sulphur.

It has been found that certain sulphur derivatives of carbonic
acid greatly increase the efficiency of the froth-flotation process when
20 used in connection with mineral-frothing agents. The increased
efficiency shows itself sometimes in markedly better recoveries,
sometimes in effecting the usual recoveries with greatly reduced
quantities of the usual mineral-frothing agents, and sometimes in
greatly reducing the time needed for agitation to produce the desired
recoveries.

The invention is herein disclosed in some detail as carried out
with salts of the sulphur derivatives of carbonic acid containing an
organic radical, such as an alkyl radical, and known as xanthates, as
the new substance. These form anions and cations in solution. Excel-
30 lent results were also obtained by agitating ore pulps with the complex
mixture produced when $33\frac{1}{3}\%$ of pine oil was incorporated with
an alcoholic solution of potassium hydrate, and xanthates or analo-
gous substances were produced by adding carbon disulphide to this
mixture.

The galena-bearing froth obtained with xanthates or analogous
substances used at the rate of 0.2 pounds per ton of ore had a char-
acteristic bright sheen, like a plumbago-bearing froth, and seemed
to make a more coherent froth than when other materials were used
on the same ore.

40 In general the substances referred to are not mineral-frothing
agents,—producing only a slight scum, and some evanescent frothy
bubbles, when subjected to agitation which would produce mineral-
bearing froth on an ore pulp in the presence of a mineral-frothing
agent. The substances are effective in enabling a selective flotation

Exhibit—K-47.

of lead and zinc, and cause uncombined silver, if present, to tend to go into the lead concentrate rather than with the zinc, where these are separated in separate concentrates. Usually pre-agitation is unnecessary, the brightening and other effects seeming to be practically instantaneous. The pulps may be either acid, alkaline or neutral according to circumstances. Two sticks of caustic potash weighing perhaps 15 grams were partly immersed in about 80 cc. of commercial carbon disulphide and kept for about ten days in a closed bottle containing some air in the warm region of the laboratory where were the hot plates used for drying. These eventually yielded a yellow or orange salt which was used with pine oil at the rate of approximately half a pound to a ton of ore in concentrating Hibernia ore from Timber Butte Mining Company. The test was with a neutral pulp, and the concentrates were seen to be clean with brightened lead sulphide particles.

For laboratory purposes potassium xanthate was prepared as follows:

198.4 grams of 88.5% caustic potash was dissolved in 524 grams ethyl alcohol (denatured No. 5 formula) at a temperature of 124° F., in a reflux condenser. The solution was cooled to 58° F. It contained a large excess of alcohol over the theoretical amount needed for the subsequent reactions. To this was added, while stirring, and in a cooling bath, the theoretical amount of carbon disulphide. The reaction was substantially instantaneous, producing a thick pulp of potassium xanthate. The pulp was cooled and centrifuged in a laboratory machine, yielding crystals containing about 20% moisture. The yield thus obtained was 74.7%. Another 17.5% was obtained by evaporation of the mother liquor. Both the centrifuged crystals and the residue from the mother liquor gave excellent results in flotation. It was found in cases where sulphuric acid was used that the centrifuged material yielded better results than the uncentrifuged.

A pulp of Anaconda slimes which had been stored for several days assaying 2.95% copper (a part being oxidized copper mineral) was treated with cresylic acid as frothing agent and potassium xanthate, the latter used at the rate of half a pound to a ton of the slimes. With no pre-agitation, and fifteen minutes agitation in a laboratory subaeration machine these slimes yielded a concentrate containing 15.6% copper, a middling containing 0.48% copper and a tailing containing 0.082% copper. The small proportion of silver present was recovered in about the same proportions. Similar results were obtained with the use of sodium xanthate and General Naval Stores No. 5 flotation oil, a steam distilled pine oil.

A pulp of Cash Mine ore subjected first to agitation for ten minutes with a mixture of 0.3 pounds per ton of potassium xanthate with a small proportion of a 10% or a saturated solution of naphtha-

Exhibit—K-47.

lene in xylene, to yield a lead concentrate, and then subjected to agitation for ten minutes with copper sulphate 0.2 pounds per ton, Barrett No. 4 flotation oil 1.0 pound per ton, and General Naval Stores No. 5 flotation oil, 0.1 pounds per ton, yielded the results shown in the following table. Attention is called to the recovery of 95% of the lead in a concentrate containing 87% of the silver but containing only 5% of zinc, while 70% of the zinc, was recovered in the zinc concentrate.

Cash Mine ore, assays

10		% Wt.	Au. (oz.)	Ag. (oz.)	Cu.	Pb.	Zn.	Fe.
	Heads	100.0	.154	10.7	3.18	14.9	7.7	11.9
	Pb. conc.	38.0	.24	24.6	7.12	37.4	5.0	21.4
	Zn. conc.	15.4	.36	6.3	2.56	3.0	35.2	12.0
	Tails.	46.6	.015	0.9	.26	0.5	0.8	4.2

% RECOVERIES.

		Au.	Ag.	Cu.	Pb.	Zn.	Fe.
	Pb. conc.	59.4	87.1	85.2	95.3	24.7	68.4
	Zn. conc.	36.0	9.0	11.0	3.1	70.5	15.5
20	Tails.	4.6	3.9	3.8	1.6	4.8	16.4

A pulp of San Francisco Mines of Mexico ore was agitated for ten minutes with potassium xanthate 0.15 pounds per ton and the same amount of coal tar creosote to yield a lead concentrate. The remaining pulp was agitated for fifteen minutes with 0.2 pounds per ton of copper sulphate, 1.2 pounds per ton of water-gas tar, and 0.05 pounds per ton of steam distilled pine oil. The results are shown in the following table:

San Francisco Mines of Mexico

30		% ASSAYS.				% RECOVERIES.		
		% Wt.	Ag. (oz.)	Pb.	Zn.	Ag.	Pb.	Zn.
	Heads	100.0	16.5	9.4	16.1
	Pb. conc.	12.2	67.0	64.0	12.0	49.4	83.2	9.1
	Zn. conc.	28.9	23.6	3.6	43.4	41.2	11.1	78.0
	Tails.	58.9	4.5	1.5	6.0	9.4	5.7	12.9

Similar results were obtained with this ore by agitating for five minutes with 0.15 pounds each of sodium xanthate and Barrett No. 2 flotation oil per ton of ore, to yield a lead concentrate, and then agitating for ten minutes with 0.3 pounds copper sulphate, 1.3 pounds Barrett No. 4 flotation oil, and General Naval Stores No. 5 flotation oil 0.1 pounds, all per ton of ore, to yield a zinc concentrate.

During the seven and one-half hours' run on current Anaconda slimes at the rate of one hundred and eleven tons per twenty-four hours in a mineral separation standard machine from a feed containing 3.28% copper, 0.26% being acid soluble copper, there was recovered a concentrate containing 11.43% Cu. and 36.9% insoluble, —the latter being a proportion desirable for smelting. The tailings contained 0.29% copper of which 0.20% was acid soluble copper. This test was run at a temperature of 82° F., using for reagents, 7.89 pounds of kerosene acid sludge, 21.6 pounds of chamber sulphuric acid, 2.36 pounds of hard wood creosote, and 2.92 pounds of a twenty per cent solution of potassium xanthate in water all quantities being in pounds per short ton of ore.

Anaconda old gravity concentration tailings reground for flotation were treated at the rate of four hundred and eleven tons per twenty-four hours in a mineral separation standard machine provided with a porous bottom in the spitzkasten through which air was admitted. From a feed containing 0.52 copper, of which 0.12% was acid soluble copper there was recovered a concentrate containing 5.72% copper and 48% insolubles. The tailings from this contained 0.13% copper of which 0.08% was acid soluble. This test was run at a temperature of 50° F. using for reagents per ton of solids, 0.67 pounds of a 30% solution of potassium xanthate in water, 3.96 pounds of kerosene acid sludge, and 11.45 pounds of chamber sulphuric acid.

Anaconda ore slimes containing 3.28% copper, of which 0.45% was acid soluble, were subjected to froth-flotation concentration at 70° F. with 7.8 pounds per ton of dry hydrated lime, 0.132 pounds per ton of steam distilled pine oil, 0.38 pounds per ton of destructively distilled pine oil (G.N.S. No. 11 flotation), and 0.69 pounds per ton of potassium xanthate in solution, yielding concentrates containing 13.76% copper and 34.7% insolubles, with a tailing of 0.317% copper of which 0.173% was acid soluble. This represented a recovery of 92.2%.

The lime was mixed with water and fed as a watery paste with the slimes to the first agitator of a series of nine standard minerals separation agitators or mixing compartments through which the pulp passed in series at the rate of 97 tons in 24 hours. The No. 11 pine oil was added at the seventh agitator, and 0.6 pounds per ton of the xanthate at the ninth. The pulp returned through fourteen spitzkastens, each equipped with a Brown aerator and circulating

Exhibit—K-47.

device. At the sixth spitzkasten the balance of the xanthate was added. At the tenth spitzkasten the steam distilled pine oil was added. The froth from the first nine spitzkastens was collected as a finished concentrate, that from the remaining five was returned to the first as a middling.

It has also been found that for the froth-flotation concentration of certain ores, such as Calumet & Hecla, Britannia, Cananea and Moctezuma, mother liquor obtained from centrifuging the above described xanthates in the process of manufacture of said xanthates
10 may be used with about the same proportions of mother liquor as of the xanthate crystals specified with results as good or better than those above given.

A pulp of 80 mesh Calumet and Hecla slimes containing 0.45% copper, mostly native, with considerable mineral occluded in the coarser particles of gangue, was agitated five minutes with 0.3 pounds potassium xanthate, then was agitated for twenty minutes with 0.4 pounds steam distilled pine oil, all per ton of ore. The concentrate thus obtained was agitated for five minutes and the froth separated to form a finished concentrate, the remainder forming a
20 middling. The results are shown in the following table:

	% Wt.	% Assays Cu.	% Re- coveries Cu.
Heads.....	100.0	0.45
Conc.	0.7	41.72	64.5
Midd.	3.9	1.44	12.3
Tails.	95.4	0.11	23.2

A Chinese graphite ore containing 49% carbon as graphite, chiefly in amorphous state, ground to 80 mesh, was agitated ten
30 minutes with 0.2 pounds potassium xanthate and 0.15 pounds pine oil, both per ton of ore, yielding a froth concentrate and a tailing. The concentrate was reagitated for five minutes to yield a finished concentrate and a middling.

The finished concentrate contained 72.8% carbon as graphite, the middling 36.8%, and the tailing 15.2%. These represented respectively 79.2%, 10.2% and 10.6% of the original graphite content of the ore.

In the absence of xanthate the same ore required a larger amount of mineral frothing agent and longer agitation to produce the same
40 results.

Having thus described certain embodiments of the invention, what is claimed is:

Exhibit—K-47.

1. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral frothing agent and a sulphur derivative of carbonic acid adapted to form in solution anions and cations and adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

2. The process of concentrating ores which consists in agitating
10 of a sulphur derivative of carbonic acid adapted to form in solution anions and cations and adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

3. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of an alkyl sulphur derivative of carbonic acid adapted to co-operate
20 with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

4. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of an ethyl-sulphur derivative of carbonic acid adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

30 5. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and an alkali-metal salt of an ethyl-sulphur derivative of carbonic acid adapted to co-operate with the mineral-frothing agent to produce by the actions of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

6. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and an alkaline xanthate adapted to co-operate with the mineral-frothing agent to
40 produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

7. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate.

Exhibit—K-47.

8. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of potassium xanthate.

9. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate and a frothing agent.

10 The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a sulphur derivative of carbonic acid containing an organic radical and adapted to co-operate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

11 The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of a sulphur derivative of carbonic acid containing an organic radical and adapted to co-operate with the mineral-frothing agent to produce
20 by the action of both a mineral-bearing froth, said agitation being so conducted as to form such a froth, and separating the froth.

In testimony whereof, I have affixed my signature to this specification.

CORNELIUS H. KELLER.

2-401

Div. 25

TITLE REPORT

No. 1,554,216

Name C. H. Keller

30 The title appears from the assignment records to be vested in:

Minerals Separation North American Corporation,
61 Broadway, New York, N.Y.

Examined up to and including 6/15/27
This certificate dated 6/29/27

WILLIS B. MAGRUDER
Chief of Assignment Division.

No further assignments appear to have been received for record including

Exhibit—K-47.

213

Div. 25

Room 314

Paper No. 12

TFM/CLB

Address only
 "The Commissioner of Patents,
 Washington, D.C.,"
 and not any official by name

All communications respecting this
 application should give the serial
 number, date of filing, and name of
 the applicant

DEPARTMENT OF COMMERCE

UNITED STATES PATENT OFFICE

WASHINGTON

10

*Please find below a communication from the EXAMINER in
 charge of this application*

THOMAS E. ROBERTSON
Commissioner of Patents

Applicant: Cornelius H. Keller
 Ser. No. 670,242 Pat. 1554216
 Filed Oct. 23, 1923

Williams & Morse,
 61 Broadway,
 New York, N.Y.

For Froth Flotation Concentra-
 tion of Ores.
 Pat. granted Sept. 22/25.

20

The case, above referred to, is forwarded to the Examiner of Interferences because it is adjudged to interfere with others, hereafter specified. The question of priority will be determined in conformity with the Rules. The interference will be identified as No. 55642. On or before Sep. 5, 1927 the statement demanded by rule 110 must be sealed up and filed with the subject of invention, and name of party filing it, indorsed on the envelope. The subject-matter involved in the interference is

1. The process of concentrating ores which consists in agitating
 30 a suitable pulp of an ore with a mineral-frothing agent and a sulphur derivative of carbonic acid adapted to form in solution anions and cations and adapted to cooperate with the mineral-frothing agent to produce by the action of both a mineral bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

2. The process of concentrating ores which consists in agitating
 a suitable pulp of an ore with a mineral-frothing agent and a salt of sulphur derivative of carbonic acid adapted to form in solution
 anions and cations and adapted to cooperate with the mineral-
 40 frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said

agitation being so conducted as to form such a froth, and separating the froth.

3. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of an alkyl sulphur derivative of carbonic acid adapted to cooperate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

10 4. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral frothing agent and a salt of an ethyl-sulphur derivative of carbonic acid adapted to cooperate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

20 5. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and an alkali-metal salt of an ethyl-sulphur derivative of carbonic adapted to cooperate with the mineral-frothing agent to produce by the actions of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

6. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and an alkaline xanthate adapted to cooperate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth containing a large proportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

30 7. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate.

8. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of potassium xanthate.

9. The improvement in the concentration of minerals by flotation which comprises subjecting the mineral in the form of a non-acid pulp to a flotation operation in the presence of a xanthate and a frothing agent.

40 10. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a sulphur derivative of carbonic acid containing an organic radical and adapted to cooperate with the mineral-frothing agent to produce by the action of both a mineral bearing-froth containing a large porportion of a mineral of the ore, said agitation being so conducted as to form such a froth, and separating the froth.

Exhibit—K-47.

11. The process of concentrating ores which consists in agitating a suitable pulp of an ore with a mineral-frothing agent and a salt of a sulphur derivative of carbonic acid containing an organic radical and adapted to cooperate with the mineral-frothing agent to produce by the action of both a mineral-bearing froth, said agitation being so conducted as to form such a froth, and separating the froth.

The interference involves your patent above identified and

An application of Rhetherford B. Martin of Colonia, New Jersey, for Concentrating Process.

10 The relation of the counts of the interference to the claims of the respective parties is as follows:

Counts	Martin	Keller
1	1	1
2	2	2
3	3	3
4	4	4
5	5	5
6	6	6
7	7	7
20 8	8	8
9	9	9
10	10	10
11	11	11

Counts compared.

T.F.M.

J. H. LIGHTFOOT,
Examiner.

I N T E R F E R E N C E

Interference No. 55642

Paper No. 13

30 *Name,* Cornelius H. Keller

Serial No. 670,242, Pat. 1,554,216

Title, Froth Flotation Concentration of Ores.

Filed, Oct. 23, 1923

Interference with Rhetherford B. Martin

DECISION OF

Law Examiner, Interference dissolved

Dated, May 26, 1930

Ex'r of Interferences,

Dated,

Board, Law Ex. Affirmed

Dated, Apr. 27, 1931

Commissioner,

Dated,

40

REMARKS

This should be placed in each application or patent involved in interference in addition to the interference letters by Primary Examiner.