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### Some assay problems

James Howard Chase

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S O M E A S S A Y P R O B L E M S

-by-

James Howard Chase

A  
T H E S I S

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Approved by : D. C. Feland  
Professor of Metallurgy

14235

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The object of this thesis is to attempt to solve a few of the many problems of assaying. Each problem is handled separately from, and independently of the others.

The effect of the silicate degree on the fusibility of assay slags was the first subject studied. According to Fulton, the higher the silicate degree, the more infusible is the mixture. Also, a mixture containing a number of bases is more readily fusible than one of few bases. The purpose of this experiment, then, is to find out the fusibility and some of the physical properties of assay slags.

The first step was to find at what temperatures the various sodium silicates became sufficiently liquid to pour. Pure, finely ground  $\text{SiO}_2$  and  $\text{NaCO}_3$  were used. These two constituents were thoroughly mixed in different ratios and placed in a muffle at a temperature of about  $800^\circ\text{C}$ . The temperature was then slowly raised and each charge taken out and poured when it had become sufficiently liquid. The temperature at which the fusion of each charge started, the temperature at the time of taking out each charge, and the physical condition of the slag as poured and after cooling were noted. Table I contains the results of this series of experiments.

Table I.

grams SiO <sub>2</sub>	grams Na <sub>2</sub> CO <sub>3</sub>	silicate degree acid to base	temp. at which fus- ion starts	temp. need- ed to make charge liquid	condi- tion of slag as poured	color of slag after cooling.
25	10	8 to 1	did not become liquid up to 1140C.			
25	15	5 to 1				
25	20	4 to 1	1100C.	1120C.	viscous	green
25	25	3 to 1 tri- silicate	1030C.	1050C.	viscous	green
25	30	2 to 1 bi- silicate	990C.	1005C.	viscous	green
25	35	"	970C.	980C.	viscous	green trans- parent
25	40	"	950C.	968C.	viscous	"
25	40	---	900C.	915C.	viscous	white vitreous

The second step of the first problem was to find the effect on the melting point of a sodium silicate caused by the addition of CaO, FeO, Al<sub>2</sub>O<sub>3</sub> or PbO in known amounts .

A sodium silicate containing 10 grams SiO<sub>2</sub> and 20 grams Na<sub>2</sub>CO<sub>3</sub> was used because of its reasonably low melting point.

The CaO, PbO, FeO, and Al<sub>2</sub>O<sub>3</sub> were all pure.

The results follow in Table II.

Table II.

grams SiO <sub>2</sub>	grams Na <sub>2</sub> CO <sub>3</sub>	grams base	silicate degree	fusion began at	mixture liquid at	condition of slag as poured	appearance of slag.
10	20	10 FeO	singulo	975C	1125C	Sticky	Black-vitreous
10	20	5 FeO	sesqui	1125C	1200C	"	black-glassy
10	20	10 CaO	singulo	1200C	1275C	"	greenish white
10	20	5 CaO	sesqui	1200C	1290C	"	greenish white
10	20	10 PbO	sesqui	1000C	1137C	"	yellow
10	20	5 PbO	bi-silicate	1080C	1175C	"	greenish yellow
10	20	10 Al <sub>2</sub> O <sub>3</sub>	sub-silicate	1225C	in-fusible	--	-----
10	20	5 Al <sub>2</sub> O <sub>3</sub>	singulo	1125C	1290C	--	-----

The object of the last part of this problem was to find how much SiO<sub>2</sub> it takes to saturate a sodium silicate at any given temperature.

Five charges were prepared each containing equal parts of SiO<sub>2</sub> and Na<sub>2</sub>CO<sub>3</sub> by weight.

These were placed in muffle at a temperature of about 800°C. The temperature was then raised, and at 900°C, the first charge was removed. After each succeeding <sup>rise</sup> ~~raise~~ in temperature of 50°C, a charge was removed and poured. An assay of the resulting slags for SiO<sub>2</sub> gave the figures shown in Table III.

Table III.

grams SiO <sub>2</sub>	grams Na <sub>2</sub> CO <sub>3</sub>	silicate degree	temp.	slag sample in grams	grams SiO <sub>2</sub> in sample	% SiO <sub>2</sub>
10	10	tri-silicate	900°C.	5.82	2.98	51
10	10	"	950°C.	2.62	1.57	60
10	10	"	900°C.	3.74	2.46	65
10	10	"	1050°C.	1.52	1.02	67
10	10	"	1100°C.	1.72	1.20	70

The results on the first problem show; that the more acid the slag the more fusible it is and that a sodium silicate containing 1 part SiO<sub>2</sub> by weight to two parts of Na<sub>2</sub>CO<sub>3</sub> is preferable; that the addition of other bases lowers considerably the melting point of the silicate; that the addition of PbO and FeO makes

the sodium silicate more easily fusible even in the case where the addition of these substances makes the slag more basic; that the addition of CaO or of Al<sub>2</sub>O<sub>3</sub> makes the slag more difficult to fuse; that sodium silicates alone are viscous at assay temperatures.

In the second experiment I attempted to determine the amount of Ag held in the filtrate from a KCN solution after the Ag had been precipitated by each of three different methods. Four cyanide solutions were used containing at the start free KCN as follows:  
No. 1, 0.05 %; No.2, 0.15 % ; No.3, 0.50 %; No.4, 0.50 %.  
To these solutions was added AgNO<sub>3</sub> solution until No.1 contained 0.86 Oz. Ag, No.2, 28.5 oz. Ag, No.3, 1.6oz. Ag, No.4, 42.7 oz. Ag per ton of solution. The addition of the silver, of course, greatly reduced the amount of free cyanide present by forming  $\text{K Ag (Cn)}_2$ .

The methods of precipitating the Ag from the solution, I will designate as A, B and C.

- (A) Take 1 A.T. of solution  
Add CuSO<sub>4</sub> as long as precipitate forms.  
Filter.  
Transfer filtrate to lead boat.  
Evaporate  
Cupel the boat and residue.



(B) Take 1 A.T. of solution  
 Add  $\text{CuSO}_4$  until precipitate just starts  
 to form.

Add small amount of  $\text{H}_2\text{SO}_4$

Filter, etc., as in (A).

(C) Take 1 A.T. of solution

Add  $\text{CuSO}_4$  as in (A)

Add excess of  $\text{H}_2\text{SO}_4$

Filter, etc., as in preceding cases.

All three methods were tried on each of the four solutions and the results are shown in Table IV.

Table IV.

% KCN in solution before adding $\text{AgNO}_3$	assay of solution oz. Ag per ton	method used	Ag in filtrate oz. per ton.
0.05	0.86	A	0.00
		B	0.00
		C	0.08
0.15	28.50	A	2.20
		B	1.40
		C	2.00
0.50	1.60	A	0.08
		B	0.00
		C	6.12
0.50	42.70	A	3.80
		B	0.00
		C	6.30

From the results obtained, it would appear that the method designated as "B" gave the best results as in each case the loss of Ag due to an imperfect precipitation was less in this method than in either of the others.

In the case where a large excess of acid is used it may be that a considerable amount of free cyanogen is regenerated which may redissolve a part of the silver. In the case of the solution low in free cyanide, but high in silver, none of the schemes mentioned gave good precipitation.

The third problem investigated dealt with the nail assay and in this I attempted to find the amount of Ag held up by a possible formation of matte in fusing a sulphide ore. For this purpose a pure pyrite was ground thru 10 mesh. A solution of  $\text{AgNO}_3$  was added sufficient to give the ore a content of <sup>about</sup> 50 oz. Ag per ton and, after drying, the ore was ground thru 100 mesh. Four different charges were used consisting of the following mixtures:

- No. I - 15 grams soda
- 80 grams  $\text{PbO}$
- 24 grams  $\text{KNO}_3$
- $\frac{1}{2}$  A.T. ore
- 10 grams borax glass

No.2 - 15 grams soda  
- 40 grams PbO  
- 24 grams  $\text{KNO}_3$   
-  $\frac{1}{2}$  A.T. ore  
- 10 grams borax glass

NO.3 - 25 grams soda  
- 20 grams PbO  
- 3 grams argol  
- 10 grams borax glass  
-  $\frac{1}{2}$  A.T. ore

No.4 - 25 grams soda  
- 20 grams PbO  
- 3 grams argol  
-  $\frac{1}{2}$  A.T. ore  
- 10 grams borax glass  
- 5 nails

No matte was formed in the nail method. (No 4.)

The assay of the other mattes for Ag is shown in the following table.

charge used	weight of matte	Ag in matte	Ag in matte per ton ore used	assay raw ore	oz per ton recovery	ore matte
# 1	4.1 grams	0.04mgms	0.08 oz.	57.20 oz	57.10	0.08
	6.2 "	0.10 "	0.20 "		56.80	0.20
	3.4 "	0.04 "	0.08 "		57.06	0.08
	4.0 "	0.06 "	0.12 "		57.02	0.12
# 2	7.6 grams	0.22mgm.	0.44 oz	57.20 oz	56.72	0.44
	8.8 "	0.18 "	0.36 "		56.74	0.36
	7.2 "	0.22 "	0.44 "		56.50	0.44
	6.6 "	0.22 "	0.44 "		56.46	0.44
# 3	11.0 gms.	0.42mgm.	0.84 oz	57.20 oz	56.20	0.84
	12.2 "	0.48 "	0.96 "		56.08	0.96
	16.3 "	0.58 "	1.16 "		53.20	1.16
	12.6 "	0.52 "	1.04 "		54.40	1.04
# 4	no matte formed					

The high litharge or niter assay should, of course, show no matte provided the amount of oxidizing material is enough, and sufficient time is allowed for the reducing material to be burned. It is of interest to note that altho the amount of matte formed when the nails are omitted at the same time that the charge lacks oxidizing material is large yet it does not carry a large percentage of the values, a result to be expected, of course, when it is remembered that there is thrown down at the same time

at least 20 grams of lead. When the behavior of the pyrite, on being heated when oxidizing conditions are lacking, is considered, the question arises, "Why is it that the presence of metallic iron is necessary to enable the soda to dissolve the iron matte formed." It certainly is true that pyrite heated to the full temperature of the assay furnace must give a matte, provided that a matte forms at all, which is approximately  $\text{FeS}$  and it seems that this must also be the matte formed when the metallic iron decomposes the pyrite. There is here a field for considerable research and reasoning.

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