

volatilized to any extent, but passes mainly into the lead button, and to a lesser extent into the slag, since the conditions, which favour accurate estimation of the lead, also promote the more or less complete reduction of the antimony.

The fusions were made in a muffle, in a reducing atmosphere. Charges thus:—

Ore—10 grams,	} Flux composed thus:—
Flux—30 grams,	
Salt cover and 2 iron nails.	
	Bicarb. soda—16 parts, Carb. potash—
	16 parts, Borax—8 parts, Flour—
	5 parts.

DURATION OF FUSIONS $1\frac{1}{4}$ HOURS.

Per cent. Antimony in Ore.	Per cent. Lead in Ore wet analysis.	Per cent. Lead in Ore fire assay.	Condition of button resulting from fire assay.
0.63	54.2	53.6	No evidence of brittleness.
0.90	55.7	54.7	Slightly brittle.
1.35	55.3	54.9	{ Buttons crack under the hammer.
1.80	54.8	54.8	
2.70	50.3	51.0	
3.60	47.7	48.5	

Since then it is impossible to guard against this latter source of error, it would seem that attention should centre upon the loss of lead from the various causes mentioned, and upon the conditions affecting that loss.

First as regards loss of lead:—

Loss by volatilization.—While lead and its compounds are sensibly volatile at comparatively low temperatures when exposed to the air, yet it must be borne in mind that, in process of assay, the reduced lead, and that still undergoing reduction, are covered with slag and so protected, while the temperature during the greater part of the operation is low, hence it would seem improbable that a material amount of lead would be volatilized provided the proper conditions prevailed.

Loss in the Slag.—Whatever properties the flux used may possess, a certain chemical loss of lead is bound to occur in the slag, since complete reduction, under the most favourable conditions, cannot be effected. The analysis of slags from a number of very carefully conducted assays showed a mean percentage of 0.62 per cent. lead. Further investigation showed that this chemical loss largely increases with the temperature, and that in cases of improper firing, it may amount to almost double this last figure.

The following assays will illustrate the loss in the slags as compared with that by volatilization. The assays were made in precisely the same manner, as regards amount and proportion of the charges of ore and flux, and duration of fusion as those above quoted:—

Per cent. Lead in Ore wet analysis.	Per cent. Lead in Ore fire assay.	Difference between wet and fire assays in units of Lead.	Per cent. Lead in slag wet analysis.	Weight of slag from fire assay (approximate)	Units of Lead accounted for in slag.
50.9	49.2	1.7	0.62	28 grams.	1.7
52.2	50.7	1.5	0.53	28 "	1.5
52.7	51.1	1.6	0.47	29 "	1.4
62.6	61.0	1.6	0.53	28 "	1.5

It would seem from these analysis that the greater part of the missing lead can be accounted for in the slag, taking the difference between the wet and fire determinations as representing the ultimate loss. The loss by volatilization in these instances must therefore of necessity have been not appreciable.

Loss from Spirling.—This mechanical loss of lead caused by too high initial temperature is probably the greatest source of error—a conclusion justified by the evidence afforded both by observation and experiment.

The following are typical of the loss which may be occasioned by this cause:—

Charges of ore and flux same as before:

Per cent. Lead in Ore. Time of fusion $1\frac{1}{4}$ hours.	Per cent. Lead in Ore. Time of fusion $\frac{1}{2}$ hour.	Per cent. Lead in Slag by wet analysis.
53.6	51.0	} Slags average 0.74 per cent. Pb.
54.5	52.9	
51.0	50.1	
49.4	47.1	
61.2	59.3	
53.0	51.0	

Second as regards the conditions which affect the loss of lead.

The loss of lead from the various causes considered, may be said to depend essentially upon, and to be governed by the following factors:—

- (1). The temperature and duration of the fusion.
- (2). The atmosphere, neutral reducing or oxidizing in which the fusion is conducted.
- (3). The nature and properties of the flux employed.

Temperature.—The great secret is to start the assays at a low temperature. For the first 45 or 50 minutes, it should not exceed that of incipient fusion of the charge—dull redness (say about 550° C.), and should then be gradually raised till at pouring it about equals that of a gold assay. The total time of the operation should be from 1—1½ hours—certainly not less than 1 hour—carbonate ores not requiring so long for their reduction as sulphides.

While a low initial temperature is a *sine qua non* of success, as guarding against loss of lead by spiriting and a minor loss by volatilization, yet on the other hand the temperature during the earlier stages must not be too low, otherwise the necessary reactions will not have taken place, and the right conditions will not have been arrived at when the temperature is raised, and the results will be vitiated.

The atmosphere in which the operation is conducted should be reducing. In the case of a muffle furnace, this may be attained by placing a few pieces of charcoal in the front part of the muffle, and a more even temperature throughout the muffle is also thereby engendered. This measure ensures conditions which combat volatilization of lead. Protected by a layer of slag and surrounded by an envelope of reducing gas, loss from this cause is minimized. Arsenic, when present in any considerable quantity would seem to promote this loss.

The Flux.—The essential features of a good lead flux may be stated thus:—

- (1). It must be readily fusible to protect the lead from volatilization.
- (2). It must contain excess of carbon.
- (3). The amount of borax present must not be excessive, otherwise lead will tend to pass into the slag chemically combined as a borate.

- (4). It must act as a desulphurizer.

The following well known lead flux composed of:—

Bicarb. soda.....	16 parts.
Carb. potash.....	16 "
Borax glass.....	8 "
Flour.....	5 "

When used in the proportion of 3 of flux to 1 of ore, with a salt cover and a couple of iron nails in the charge, will be found to give satisfactory results in the case of oxidized and sulphide ore alike.

Upon an average, in the case of ores of medium and high grade, free from interfering elements, the wet assay exceeds the dry by about 1.7 per cent., though in some instances the dry assay can undoubtedly be made to more nearly approach the wet.