A METHOD FOR DETERMINATION OF THE VOLATILE MATTER IN OXIDES OF LEAD.

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For calculation of glass batch compositions it is necessary to know exactly the amount of PbO in the litharge or red lead used. If special tests show that the material contains no appreciable amount of metallic impurities, the obvious method is to drive off all volatile matter (CO_2, H_2O, and O present in PbO_2) by ignition. The residue may then be figured as pure PbO. In trying to do this, however, one will meet a difficulty at the very outset. The volatile constituents are driven off only slowly and to insure their expulsion in a reasonable period of time the oxide should be carried to its melting point. If, however, the sample is heated until melting takes place the containing crucible will be badly attacked. In order to overcome this difficulty the modification of the ignition method described below was worked out.

The ignition is done in a platinum crucible of almost any size or shape, but preferably of small diameter which will give the melt a relatively small surface and thereby diminish the loss by volatilization of lead oxide. To prevent any attack of the platinum the lead oxide sample is mixed with silica (sand or ground quartz) in the proportion of about 3 weights of PbO to 1 weight of SiO_2. On account of the formation of a low melting compound and eutectics in such a mixture the melting will take place at a temperature nearly 100° C, below the melting point of pure PbO (about 880°), and the result will be a silicate melt which does not appreciably attack the platinum.

The procedure for the determination is as follows: The sample is weighed in a platinum crucible. An amount of silica[1] equal to about one-third[2] of the assumed amount of PbO in the

[1] The silica used here must not merely be dried at 100° or 200° but, previous to use in this method, it should be ignited to 1000° to completely remove volatile matter. This procedure is advisable in all cases and absolutely necessary with silica derived from carbonate-carrying sands.

[2] At the Charleroi plant we used 4 of lead oxide to 1 of silica.

sample is added to the latter and the two are carefully mixed in the crucible; finally, a thin layer of silica is spread over the top of the mixture. The crucible with full charge is weighed and placed inside an electric furnace previously heated to 800°. The temperature must be accurately measured and must not be permitted to change considerably. The heating at 800° is continued for about 20 minutes and the temperature is raised as quickly as possible to about 1000° where it is held for about 15 minutes more. Then the crucible is taken out, covered to prevent the cracking glass from flying out of the crucible, cooled and weighed. The loss in weight is the amount of volatile constituents present in the lead oxide and, in case there are no other impurities, the remainder of the original sample is pure lead oxide, PbO.

The method has been used to a considerable extent in connection with our optical glass work at the laboratories of the Pittsburgh Plate Glass Company at Charleroi, Pennsylvania, and the Spencer Lens Company at Hamburg, New York. The results checked well with a few direct analyses made on some of the samples. The average run of the figures obtained on litharge is indicated by the following examples:[1]

Average of 14 samples litharge	98.99 per cent PbO
Maximum of 14 samples litharge	99.55 per cent PbO
Minimum of 14 samples litharge	98.00 per cent PbO
Top layer of barrel which had stood open 4–6 weeks	97.27 per cent PbO
Top layer of barrel which had stood open 4–6 weeks	96.61 per cent PbO

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[1] Data furnished by R. H. Lombard.