

stance to be dried should be divided into small, thin pieces, then about six times its weight of oil put into an evaporating dish, and placed in a drying closet kept at 240° . When the oil has the temperature of the drying room, it should be weighed, and the substance to be dried, weighed and added to it. If very moist, add in successive portions. There will be a slight effervescence at first, and the whole should be kept in the drying closet for a few minutes after the effervescence has ceased.

Ordinarily the whole operation may be completed in twenty minutes. The evaporating dish containing the oil, and the substance, which is now perfectly dry, should be weighed; the loss, of course, is the moisture.

Substances like soaps, portions of which are dissolved in the oil, cannot be recovered, but those like wood pulp, none of the constituents of which are soluble in the oil, can be put in an extractor, and, after the oil is washed out, weighed again if desired.

The advantages of this process are, the quickness with which the operation may be carried out, simplicity of apparatus, ease of manipulation, and the fact that the substance to be dried is perfectly protected from any action of the air, by being immersed in a neutral liquid while heated, so that it will stand a higher temperature, without decomposition, insuring perfect dryness, than would be possible if exposed to the air.

THE VOLUMETRIC DETERMINATION OF LEAD.¹

BY J. H. WAINWRIGHT.

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HAVING been called upon some time ago to determine the percentage of metallic lead in a large number of samples of "white lead," "litharge," "red lead," etc., in a limited time, it became extremely desirable to find, if possible, some simple and rapid method, whereby twenty or thirty samples could be finished in a day. Extreme accuracy was not particularly necessary, since an error of two to three-tenths per cent. either way would not materially vitiate the results for the purpose for which they were required.

Various volumetric methods were examined, but they all with

¹ Read at the meeting of the New York Section, March 5, 1897.

one exception proved very unsatisfactory and offered no advantages whatever over the conventional gravimetric methods of precipitation and weighing the lead, either in the form of sulphate or as chromate. This one exception is the method described in Crookes' Select Methods, slightly modified, and which consists, according to Crookes, in adding an excess of sodium acetate to a neutral solution of a lead salt (nitrate carefully neutralized with either ammonia or sodium carbonate) and titrating this solution with a standardized solution of potassium bicarbonate, one cc. of which should equal 0.0207 gram metallic lead, the end of the reaction being determined by the production of the characteristic red silver chromate upon adding a drop of the solution to a drop of silver nitrate solution upon a porcelain plate. Following out this method, as described by Crookes, concordant results are difficult to obtain, but by slightly modifying some of its details, it appears to leave nothing to be desired for certain classes of work.

For the determination of metallic lead in litharge, for example, the assay is proceeded with as follows: 1.00 to 1.25 grams is dissolved in ten to fifteen cc. nitric acid (sp. gr. 1.20), the solution is neutralized with ammonia in excess and a considerable excess of acetic acid is added. It is then allowed to boil and the potassium bichromate solution is run in from a burette graduated to one-tenth cc. After the addition of bichromate solution sufficient to precipitate nearly all the lead, the solution should be again boiled until the precipitate of lead chromate, which at first is bright yellow, has become orange colored. The titration is now continued one-half cc. or so at a time, the solution being well stirred after each addition of bichromate until the reaction is almost complete, which can be observed by the sudden clearing up of the solution, the lead chromate settling promptly to the bottom of the beaker. This will, if the solution is hot, usually occur when within about one cc. of the end of the reaction, which should now be completed drop by drop, stirring and allowing to settle after each addition of bichromate and testing by adding a drop or two of the supernatant liquid to a drop of silver nitrate solution on a white porcelain plate or tile until a distinct red color is produced.

The solution of bichromate should be made of such a strength

that one cc. equals not much more nor less than 0.01 gram metallic lead and should be standardized either by means of the pure metal or by means of a pure "white lead" in which the metal has been very accurately determined gravimetrically.

The points to be particularly observed and which appear to be essential to the success of the method are :

First.—The solution of the lead salt should be as concentrated as possible before titration and decidedly acid with acetic acid.

Second.—It should be free from other metals, especially such as may exist in the lower forms of oxidation ; antimony and tin unless they have been previously thoroughly oxidized by repeated evaporation with fuming nitric acid are particularly to be avoided, since they appear to reduce the bichromate, and the results of the titration will therefore be much too high in terms of lead. Bismuth, which like lead is precipitated as a chromate, should also be avoided.

Third.—The titration should be performed in a solution kept at all times as near the boiling-point as possible.

Fourth. — The bichromate solution should be neither too strong nor too weak for obvious reasons. As given in Crookes' Select Methods the solution appears to be much too strong since, unless an inconveniently large weight of the sample is taken, the experimental error is too great. On the other hand, it should be strong enough for a drop or two in excess, in a solution of about 100 to 120 cc. to react readily with the silver nitrate. The strength as given above (*i. e.* about 0.01 gram lead per cc.) is approximately correct.

Fifth.—The test solution of silver nitrate should be dilute, not over two to three per cent.

The method is particularly adapted to the assay of such substances as white lead, red lead, litharge, pig lead, etc., and for lead ores wherein the lead exists as carbonate, etc. In the case of "red lead," solution should be effected by means of nitric acid (sp. gr. 1.20), boiling, and then adding drop by drop from a pipette, a dilute solution of oxalic acid until the lead oxide formed is completely dissolved. If the "red lead" contains organic matter, as is often the case with some samples of vermilion to which a small amount of a dye has been added, the solution should be filtered before the titration. "White

lead" may be dissolved directly in dilute acetic acid and the resulting solution titrated without filtration. The method can also be used sometimes to advantage in the case of alloys containing tin and antimony, and with impure lead bullion, but the sample must be thoroughly oxidized and the solution filtered before titration, and in the latter case it is desirable before filtration to precipitate whatever silver there may be present by means of a drop or two of hydrochloric acid or solution of sodium chloride. The small amount of chloride present in the resulting lead solution does not materially interfere with the end-reaction provided, as mentioned by Crookes, the size of the drops of silver nitrate solution spread upon the porcelain plate be somewhat increased.

The following are some of the results obtained by this method:

STANDARDS.		
Lead.	Potassium carbonate	One cc. = gram lead.
Gram.	solution.	Gram.
	cc.	
0.6475	56.3	0.01150
0.6330	54.8	0.01155
0.6234	54.1	0.01152
0.6196	89.1	0.00690
0.6475	94.0	0.00696
0.6706	96.4	0.00706
0.4911	46.1	0.01065
0.5654	53.5	0.01056
0.5800	54.7	0.01060
0.5355	50.6	0.01058
0.4216	39.9	0.01056
0.4673	42.2	0.01057
0.5061	29.0	0.01742
0.5387	30.8	0.01748
0.5839	33.7	0.01733

WHITE LEAD (DRY), METALLIC LEAD DETERMINED AS ABOVE.

Sample.	Per cent.		
	1.	2.	3.
A	79.3	79.2	79.0
B	78.9	78.7	...
C	79.5	79.8	79.5
D	79.0	78.9	...

WHITE LEAD GROUND IN OIL.¹

Sample.	Per cent.		
	1.	2.	3.
E	70.2	69.7	70.1
F	72.6	72.4	...

¹ In this case the sample should be dissolved in dilute nitric acid, boiled, filtered, solution neutralized with ammonia in excess, and then acidified with acetic acid in excess.

LEAD BULLION.

Sample.	1.	2.
	Per cent.	Per cent.
A.....	97.75	97.75
B.....	97.06	96.83
C.....	95.68	95.44
D.....	95.22	95.68

E, volumetric determination = 96.27 per cent.

E, gravimetric determination = 96.36 per cent. (from PbSO_4).

ALLOY SAID TO CONTAIN LEAD EIGHTY PER CENT., ANTIMONY FIFTEEN PER CENT., AND TIN FIVE PER CENT.

Sample A (thoroughly oxidized), volumetric = 79.7 per cent., gravimetric = 79.79 per cent.

Sample B (thoroughly oxidized), volumetric = 80.6 per cent., gravimetric = 80.44 per cent.

Sample C (not completely oxidized), volumetric = 77.7-77.9 per cent., gravimetric = 75.56-75.41 per cent.

In using this method it will be found very convenient to employ the "equivalent weight" system, so that the percentage of metallic lead in the sample may be read directly upon the burette.

MANUFACTURE OF PHOSPHOR BRONZE.

BY MAX H. WICKHORST.

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PHOSPHOR bronze is bronze containing a small amount of phosphorus, varying from a few hundredths of one per cent. to over one per cent. The phosphorus is added mainly to deoxidize the metal. It may be added to the bronze in substance as phosphorus, or in the form of a high phosphorus alloy.

At the brass foundry of the Chicago, Burlington & Quincy Railroad Company, at Aurora, Ill., we use the latter method. The alloy we call "hardener" and it contains six per cent. of phosphorus with copper and tin, in the ratio of 8 to 1.

Manufacture.—Our method of making this "hardener" is as follows: Ninety pounds of copper are melted in a No. 70 crucible (which holds about 200 pounds of metal when full), under charcoal, eleven pounds of tin are added, and the whole allowed