allowing to stand for a further period of ten hours at 0°. We conclude therefore that myrtle wax contains no stearin and that the high melting-point of the older material is due to the obscure change which takes place in many fats on standing. We hope to study this change in myrtle wax further and determine how the constants, other than melting- and solidifying-points, are affected thereby.

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THE ANALYSIS OF BRONZES AND BEARING METALS.[1]

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In the laboratory with which the writers are connected, many bronzes and bearing metals are analyzed, and it was thought that the methods which are used might prove of interest to the members of this Section.

While the scheme contains much that is old, we wish to call particular attention to the volumetric method for determining lead, as the results obtained by it are as reliable as those obtained by the sulphate method.

BRONZES.

The analysis of bronzes is carried out as follows:

Weigh 1 gram of the sample (0.5 gram, if the lead is over 15 per cent.) into a No. 2 beaker, cover with a watch-glass, add 10 cc. nitric acid (sp. gr. 1.42) and warm until all is dissolved. When in solution, add 40 cc. hot water and boil five minutes, filter, wash with 2 per cent. nitric acid, burn and weigh as SnO_2. To the filtrate add 25 cc. strong ammonia and heat to boiling, then add about 5 grams ammonium persulphate and boil from five to ten minutes. Make acid with sulphuric acid, filter and wash with hot water. The lead will remain on the filter as lead peroxide. Transfer the precipitate and filter to the beaker in which precipitation was made, add water and stir well to disintegrate the filter-paper. Dilute to 600-700 cc. with cold water, add about 3 grams potassium iodide and some starch solution. When all the iodide is dissolved, add 10 cc. hydrochloric acid (1:1), stir well and titrate

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[1] Read before the Pittsburg Section of the American Chemical Society at the first monthly meeting, March 19, 1903.

with one-twentieth normal sodium thiosulphate until the solution changes from the dirty and dark yellow color to a bright lemon-yellow; or, an excess of sodium thiosulphate may be added and the excess titrated with one-twentieth normal iodine solution until the color changes from the bright yellow of the lead iodide present to the dirty and dark yellow. The number of cubic centimeters of sodium thiosulphate used multiplied by 0.5175 will give the percentage of lead. Where speed is not desirable, the lead may be determined by adding sulphuric acid to the filtrate from the oxide of tin, or the lead and copper may be deposited with the electric current.

Dilute the filtrate from the lead peroxide to 500 cc., heat to boiling and add 50 cc. of a 20 per cent. sodium thiosulphate solution,[1] boil five minutes, filter, wash with hot water, burn, and weigh as CuO.

Copper may also be determined by the following method, which is a modification of Low's method.[2] Dissolve 0.5 gram of the sample in 10 cc. nitric acid (sp. gr. 1.42). When in solution, dilute with cold water and add sodium carbonate until the solution is alkaline, make acid with acetic acid and add about 3 grams potassium iodide and some starch solution. Titrate with a sodium thiosulphate solution which has been standardized with pure copper.

Oxidize the filtrate from the copper sulphide thrown down by the thiosulphate as described above, with nitric acid and potassium chlorate and evaporate until the volume is about 300 cc. Make a basic acetate separation and determine iron or aluminum by the well-known methods. Make the filtrate from the iron or aluminum strongly alkaline with ammonia, heat to boiling and add ammonium persulphate, boil five minutes, filter, and wash with hot water, burn and weigh as Mn_3O_4.

To the filtrate from the manganese add ammonium phosphate in excess, heat to boiling and add hydrochloric acid until there is but a slight excess of ammonia, boil five minutes, filter and wash with hot water. The precipitate may be dried and weighed as $ZnNH_4PO_4$, or it may be filtered on a Gooch crucible and ignited to $Zn_2P_2O_7$. It may also be titrated with standard acid and alkali.[3]

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[1] Nissenson and Neumann: *Chem. Ztg.*, 1895. p. 1591.

[2] *Eng. and Min. J.*, 1895.

[3] Walker: This Journal, **23**, 468.

Any nickel which may be present will be found in the filtrate from the zinc and may be precipitated as sulphide and ignited to NiO. If manganese is present in small quantities, it may be determined in a separate portion by the following method: Weigh 0.2 gram of the sample into a suitable test-tube, add 10 cc. nitric acid (sp. gr. 1.20) and warm until the sample is dissolved and all nitrous fumes are driven off. Add 15 cc. silver nitrate solution (1.33 grams of the salt to 1 liter of water) and about 0.5 gram ammonium persulphate, warm until the manganese is oxidized to permanganic acid, cool, transfer to a beaker, dilute to 100 cc., and titrate with standard sodium arsenite or hydrogen peroxide until disappearance of the pink color.

DETERMINATION OF PHOSPHORUS.

To determine phosphorus dissolve 1 gram of the sample in 5 cc. fuming nitric acid, evaporate to expel most of the free acid, add 10 cc. concentrated hydrochloric acid and evaporate to dryness. Dissolve the residue in hydrochloric acid and water, heat to boiling and precipitate lead, tin, and copper with metallic zinc, filter and wash with hot water. To the filtrate add some iron solution free from phosphorus and 10 cc. nitric acid (sp. gr. 1.42), boil a few minutes and then precipitate with ammonia and filter to separate most of the zinc, dissolve the precipitate in hot nitric acid (sp. gr. 1.20) and precipitate the phosphorus with molybdate solution. The yellow precipitate may be weighed or titrated.

BEARING METALS.

If the sample is high in tin and low in lead, proceed as outlined for bronzes, but if the sample is high in lead and contains antimony, proceed as suggested by Mr. George Hopkins, chemist to the Carrie Furnaces of the Homestead Steel Works, he having found that the addition of an excess of pure tin will insure the complete separation of the antimony with the oxide of tin. Weigh 0.5 gram of the sample and 0.25 gram of pure tin into a tall No. 2 beaker, cover with a watch-glass, add 20 cc. nitric acid (sp. gr. 1.33) and boil down to pastiness, add 40 cc. hot water and boil a few minutes, filter and wash with 2 per cent. nitric acid, burn and weigh as $SnO_2 + Sb_2O_4$. The filtrate is made strongly alkaline with caustic potash and the lead oxidized by adding about 10 grams ammonium persulphate. The rest of the analysis is carried out as outlined for bronzes.

To determine antimony[1] weigh 1 gram of the sample and 1 gram potassium iodide into a No. 2 beaker, add 80 cc. hydrochloric acid (sp. gr. 1.10) and boil gently for one hour, filter on a weighed paper or Gooch crucible and wash with dilute hydrochloric acid and then with hot water until free from chlorides. Wash once with alcohol, dry for one hour at 100° C. and weigh. The increase in weight is metallic antimony. Calculate this to Sb_2O_4 and subtract from the weight of the mixed oxides; calculate the tin from the weight of stannic oxide found and subtract the tin which was added.

Arsenic is determined in a separate portion by any of the well-known distillation methods.

Bismuth, if present, would be found with the copper sulphide, and can be determined by dissolving the sulphides in nitric acid, and precipitating the bismuth with ammonia.

REMARKS.

It will be noted that the filtrate from the tin in bronzes is made alkaline with ammonia, while in bearing metals, caustic potash is used. If caustic potash be used for bronzes, the copper would be precipitated and the lead would not be completely oxidized, while if the filtrate of a high lead-bearing metal is made alkaline with ammonia the lead hydroxide precipitate would be of such large volume that complete oxidation would not take place.

In filtering the lead peroxide it is necessary to add some paper pulp to the filter, otherwise some of the precipitate will run through. Should manganese be present, the lead will have to be determined by the sulphate method as the oxide of manganese would be precipitated with the lead peroxide and would not dissolve in the sulphuric acid added.

While we have not made many experiments, we feel safe in saying that the manganese could be separated directly from the copper sulphide filtrate, if most of the sulphuric acid is neutralized and the solution oxidized with ammonium persulphate.

In determining lead by the sulphate method or by the electrolytic method, a large amount of time is consumed in evaporating

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[1] Andrews: This Journal, 17, 872.

to sulphuric fumes or in the long passage of the electric current, while by the titration method, if lead only is wanted, it may be determined without separating the tin, and the time required will be about thirty minutes.

If the copper is determined by the volumetric method, tin, lead, and copper results will be obtained in about one hour.

The following lead results were obtained on the same samples by the sulphate and volumetric methods.

Sample.	Sulphate method.	Volumetric method.
Bronze No. 4	2.60	2.61
" " 2	4.68	4.71
" H	5.05	4.97
" No. 1	5.39	5.33
" A	5.67	5.62
" B	7.47	7.51
" M	9.77	9.73
" S	13.72	13.62
" A.P	26.68	26.55
Babbit C	3.29	3.41
" D.S	65.85	65.93
" L	80.80	80.73
" D	89.63	89.50

We have lately had occasion to determine sulphur in bronzes and we have done this by dissolving about 10 grams of the sample in the double chloride of potassium and copper used in carbon determinations, filtering off the sulphide of copper and igniting to copper oxide. We then calculate the sulphur from the weight of oxide found; of course, in very accurate work the copper sulphide should be oxidized and the sulphur determined as barium sulphate.

Following are some analyses made by the methods outlined in this paper. All precipitates obtained were tested and in no case were impurities found.

	Babbit.	Babbit.	Bronze.	Bronze.	Bronze.	Bronze.
Tin	4.13	43.27	7.41	4.85	7.88	3.55
Lead	79.30	43.63	9.73	2.80	5.05	5.87
Copper	....	0.56	82.09	90.31	85.68	86.23
Antimony	16.30	12.60	....	....	....	....
Zinc	....	....	0.95	....	1.46	3.55
Manganese	....	....	....	1.80	....	....
Iron	....	....	....	0.21	....	0.40
Sulphur	....	....	....	....	....	0.30
	99.73	100.06	100.18	99.97	100.07	99.90

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