

Under such circumstances the neck of the inverted flask passes through a hole in the ebonite plate and into the hollow screw, while the wheel beneath raises the bottom of the flask against the ruled plate the same as when a "Petri" dish is employed.

The counting apparatus and also the "Miquel" flasks may be obtained of Eimer and Amend, 211 Third Avenue, New York City.

THE DETERMINATION OF LEAD IN ALLOYS.

BY W. E. GARRIGUES.

Received April 21, 1898.

IN the analysis of brass and bronze, the fact that we have only a single practicable method for separating lead is an unusual instance of poverty in the case of so common an element.

While the separation and determination as sulphate is admittedly excellent, there is always a more or less lengthy wait during evaporation of the filtrate from the metastannic acid, not to mention occasional losses of the assay at this stage through breakage. The latter is fortunately of rare occurrence and the former of little moment under ordinary circumstances, as no personal supervision is exacted during the operation, but it is not at all a rare occurrence that the foundryman wants his result at the earliest possible moment and it is for such occasions that the following method is especially suited. Nevertheless, the determination is fully as accurate as that with sulphuric acid and as it requires no more manipulation there is no reason why the method should not be employed at all times when the filtrate from the lead is not needed for other determinations.

The precipitation of lead as chromate, from neutral or acetic solutions, is an old and admirable process but it is inapplicable as a separation from copper, the latter being likewise thrown down. Copper chromate is known to be readily soluble in ammonia, but the writer has been unable to find any record of the behavior of ammonia toward lead chromate, the nearest approach being the statement that fixed caustic alkalies decompose it.

Experiment demonstrated that lead chromate is unaffected by

ammonia and confirmed the ready solubility of copper chromate. Hence the perfect separation.

The Analysis: To the nitric acid solution of the lead and copper, is added an excess of potassium bichromate, followed by ammonia in decided excess. After heating until the supernatant liquid clears, the precipitate of lead chromate is filtered out on a Gooch crucible (the filtering medium being a single disk of filter-paper), washed respectively with dilute ammonia, hot water, and alcohol, and dried in the water-oven ready for weighing. The results, in practice, have been found excellent.

In using the process, it must be remembered that the sulphate method is available where the chromate is not. The latter is essentially a separation from copper and zinc only. In the presence of antimony, for instance, it is entirely feasible to keep the small quantity, left in the filtrate from the tin, from coming down with lead sulphate, by the simple expedient of using a liberal amount of sulphuric acid, diluting sparingly, and filtering without undue delay. The same applies no doubt to bismuth. Both of these would obviously appear with the lead chromate. Iron would likewise interfere in the latter case.

Having now described the chromate process as being more expeditious than the sulphate, there is the somewhat paradoxical position of having to explain how the reverse may be made the case insomuch as the lead can be obtained directly, without the necessity of first removing the tin. The process is rather delicate and its indiscriminate use may be objected to on this ground with some reason. It, nevertheless, seems possible to make the separation a clean and satisfactory one. At all events the observed facts are worthy of being recorded.

The description is perhaps best given in the form of an account of an experiment:

One-half gram of soft solder, containing lead 58.83 and 58.80 per cent. by the usual sulphate and chromate methods respectively, was oxidized with nitric acid, twenty cc. of strong sulphuric acid added, and the solution evaporated to the point of dense fuming. The metastannic acid was completely dissolved.

An addition of eighty cc. cold water was made, the solution stirred for a few minutes, and the lead sulphate filtered out on a Gooch crucible. The result for lead was fifty-nine per cent.

The sulphuric acid filtrate was made alkaline with ammonia, again acid with sulphuric, but this time only until it turned methyl orange a decided red. The result was a voluminous white precipitate which was filtered out after boiling the liquid and which, on ignition over the blast, gave stannic oxide, equivalent to 41.1 per cent. tin. Total: 100.1.

If much more than eighty cc. was used for diluting the fuming acid or if the diluted liquid was allowed to stand on the water-bath for an hour or so, the tin began to precipitate slowly.

Antimony, when present in considerable quantity, sustains its reputation by coming down with both tin and lead and partly remaining in the last filtrate besides.

When it is desirable to remove lead and tin in one operation, as for instance in the determination of copper by the writer's thiocyanate method, it is essential to bear in mind the above described behavior of metastannic acid toward sulphuric acid and use the latter very sparingly. There is no difficulty in accomplishing the separation of both together in this manner. One can even go a step farther, in the determination of zinc as phosphate, by oxidizing with nitric acid, expelling the latter with as little sulphuric acid as possible, precipitating the copper as thiocyanate, and removing them all in one filtration. Three per cent. sulphuric-acid washing, to prevent re-solution of lead sulphate, has no effect on either the tin or copper precipitates.

Where small amounts of zinc alone are to be determined in bronze, this procedure is unequaled for accuracy, neatness, and despatch.

THE DUQUESNE CHEMICAL LABORATORY,
PITTSBURG, PA.

INACCURACIES IN THE DETERMINATION OF CARBON AND HYDROGEN OF COMBUSTION.

BY CHARLES F. MABERY.

Received May 16, 1898.

I WAS much interested in the results of Mr. G. Auchy which appeared in the April number of this Journal, since they are closely in line with my own observations in determining these elements in hydrocarbons, and an examination of this subject which I have now in hand. During the last five years in my