

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.

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INACCURACIES IN THE DETERMINATION OF CARBON AND HYDROGEN OF COMBUSTION.

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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

work on the composition of the petroleum hydrocarbons, these determinations have occasioned much trouble and expense on account of the difficulty in keeping the details under control. The difficulties which I have encountered are the following :

1. An efficient and durable means for removing carbon dioxide and water from air and oxygen.
2. Maintaining a sufficient temperature to prevent gaseous hydrocarbons from escaping without complete combustion.
3. Incomplete absorption of carbon dioxide in a thirty-three and one-third per cent. solution of potassium hydroxide.
4. Escape of moisture from the potassium hydroxide bulb, over fused calcium chloride and potassium hydroxide.

I now have these details under such control that assistants and students, by following directions, may be sure of obtaining correct results.

It is not the purpose of this note to discuss these details in full since, as mentioned above, I have work in progress for the purpose of ascertaining the limits of these sources of error including the forms of potash bulbs.

That it is not safe to trust to the calcium-chloride attachment of the potash bulb even when it contains solid caustic potash, as ours always does, to retain all moisture, is shown by the following results, in which a second sulphuric-acid tube was placed after the potash bulb. These results are taken at random from notes on combustions recently made :

0.1402 gram of a California distillate, 138° - 140° , gave 0.4485 gram carbon dioxide, and 0.1897 gram water.

	Per cent.
Carbon	85.00
Hydrogen	14.60

Gain in second sulphuric-acid tube 0.0007 gram.

0.1413 gram of a California distillate gave 0.4372 gram carbon dioxide and 0.1851 gram water.

	Per cent.
Carbon	84.84
Hydrogen	14.56

Gain in second sulphuric-acid tube 0.0022 gram.

1.1420 grams of a hydrocarbon from commercial paraffin, boil-

ing-point 306° - 308° , fifty mm., gave 0.4434 gram carbon dioxide and 0.1882 gram water.

	Per cent.
Carbon	85.19
Hydrogen	14.73

Gain in second sulphuric-acid tube 0.0012 gram.

0.1384 gram of a paraffin distillate, 294° - 296° , fifty mm., gave 0.4293 gram carbon dioxide and 0.1842 gram water.

	Per cent.
Carbon	85.07
Hydrogen	14.82

Gain in second sulphuric-acid tube 0.0015 gram.

0.1443 gram of a paraffin distillate 338° - 343° , fifty mm., gave 0.4507 gram carbon dioxide and 0.1884 gram water.

	Per cent.
Carbon	85.45
Hydrogen	14.51

Gain in second sulphuric-acid tube 0.0015 gram.

0.1423 gram of a paraffin distillate 258° - 260° , fifty mm., gave 0.4441 gram carbon dioxide and 0.1423 gram water.

	Per cent.
Carbon	85.09
Hydrogen	15.08

Gain in sulphuric-acid tube 0.0016 gram.

0.1368 gram of a California distillate, boiling-point 139° - 140° , gave 0.4309 gram carbon dioxide and 0.1837 gram water.

	Per cent.
Carbon	84.96
Hydrogen	14.72

Gain in second sulphuric-acid tube 0.0009 gram.

0.1459 gram of a California distillate, boiling-point 188° - 189° , gave 0.4559 gram carbon dioxide and 0.1886 gram water.

	Per cent.
Carbon	85.70
Hydrogen	14.40

Gain in second sulphuric-acid tube 0.0016 gram.

Unless great care be exercised in the rapidity of the combustion I suspect loss of carbon dioxide, but have not yet attempted

to prove it. It will be seen that these results confirm the observations of Auchy, Drown, Blair, Dudley, and others, that the ordinary form of potash bulb will not retain all the moisture unless the operation be controlled with the greatest care. Another serious difficulty with us has been the impurities in the granular copper oxide. In blank tests it does not cease to cause an increase in weight in both sulphuric-acid tube and potash bulb until it has been heated in oxygen as long at least as it would be in six combustions. But when constant it apparently remains so.

SOME FURTHER APPLICATIONS OF HYDROGEN PEROXIDE TO QUANTITATIVE ANALYSIS.

BY PERCY H. WALKER.

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SEPARATION OF TITANIUM FROM IRON.

THE addition of a large excess of hydrogen peroxide to a solution of a ferric salt does not prevent the precipitation of ferric hydroxide on addition of ammonia in excess; but a solution of a titanium salt gives, on treatment with hydrogen peroxide, the well-known yellow solution. This solution, if treated with sufficient hydrogen peroxide and kept cold, is not precipitated at once on adding ammonia in excess.

Taking advantage of this fact titanium may be separated from iron by the following method: The cold, slightly acid solution of the mixed sulphates (in all about 150 cc.) is mixed with 100 cc. hydrogen peroxide (about two and one-half per cent. solution) and run slowly from a dropping funnel into a rather large casserole containing 100 cc. hydrogen peroxide and thirty cc. strong ammonia, stirring constantly. The precipitated ferric hydroxide is filtered at once, washed slightly with cold dilute ammonia containing a little hydrogen peroxide. The precipitate is easily dissolved from the paper with a mixture of hydrochloric acid and hydrogen peroxide, again treated with an excess of hydrogen peroxide and reprecipitated as above. This is repeated a third time. The ferric hydroxide can then be dissolved and the iron determined in the ordinary way.

The filtrates containing the titanium are boiled down, which