The Alundum Crucible as a Substitute for the Gooch Crucible.

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In order to test the efficiency of the recently introduced unglazed Alundum crucible, when used for the purposes in quantitative analytical chemistry generally assigned to the ordinary Gooch crucible, four different series of analyses were undertaken. These involved the determination of silver by precipitation as silver chloride, copper as cuprous sulpho-cyanate, aluminium as aluminium oxide from ignited aluminium hydroxide, and barium as barium sulphate. Such a selection was made in order to obtain as widely different precipitates as possible, as regards size of particles, ease of filtration and media in which produced, and at the same time be in general usage.

Both the Alundum and Gooch crucibles permit filtration, drying and weighing without disturbing the precipitate, but the porous nature of the former of course does away with the preparation of an asbestos mat. To discover whether or not such an advantage as well as others claimed for the Alundum crucible by its manufacturers, such as capability of withstanding very high temperatures, is sufficient to warrant its wide adoption for use in accurate quantitative analysis, was therefore the object in view in this study.

One crucible only was used for the determinations in one series, in order to discover what would be the effect of continuous usage and how thoroughly it might be cleaned in preparation for the next analysis. In each case the empty crucible was heated thoroughly for one hour in the drying oven at the temperature at which the precipitate was later to be dried. The apparatus for the filtration was that used with the Gooch crucible, since any more complicated or expensive apparatus would *per se* be a distinct disadvantage.

I. The determination of $AgNO_3$ as AgCl.

In these analyses 50 c. c. portions of a solution, each containing .2432 grams of pure $AgNO_3$ were used. In the first trials a solution of Kahlbaum's chemically pure NaCl was used to precipitate the AgCl from the hot solution of $AgNO_3$ while rapidly stirred. Stirring was continued for two minutes, the precipitate allowed to settle, filtered through the crucible, washed with water and then dried at 140°.

First analysis: Percent Ag in $AgNO_3$ calc. 63.50, found 65.24. No. of washings, 8.

The crucible was then washed thoroughly with pure water until all apparent traces of AgCl had been removed, dried at 140° and again weighed. Gain in weight, .0162 gr.

Second analysis: Percent Ag calc. 63.50, found 64.04. The crucible was now thoroughly washed by suction with NH_4OH to remove all AgCl and water to remove all NaCl from the pores. Weighing gave the original value for the crucible.

Pure dilute HCl was substituted for NaCl so that the error might not be due to the precipitating agent.

First analysis: Percent Ag calc. 63.50, found 63.52.

Second analysis found 63.60.

Finally a solution of purified KCl, a slightly more soluble salt than NaCl was tried. The empty crucible was in all cases washed with NH_4OH and water and it varied in weight by only one-tenth milligram.

Percent Ag calc. 63.50, found 63.98.

In all the analyses the AgCl precipitate was easily handled, the difficulty arising in the seeming impossibility of washing out of it and the pores of the crucible the NaCl and the KCl.

II. Determination of Cu in $CuSO_4.5H_2O$ as CuSCN.

This analysis seemed particularly adaptable because of the extensive use of the Gooch crucible necessitated in it. A solution of crystals of very pure $CuSO_4.5H_2O$ was made such that each 50 c. c. contained .2136 grams. Precipitation of CuSCN was affected from the hot solution in the presence of an excess of H_2SO_3 by means of $(NH_4)_2SCN$. Drying was at 140°.

Percent Cu in $CuSO_4$ calc. 25.46 found (1) 26.80 (Stirred 2 minutes and not permitted to settle.)

(2) 25.24 (Stirred 4 minutes rapidly and digested 15 minutes.)

(3) and (4) 25.39 (Stirred 4 minutes rapidly and digested 30 minutes.)

By thoroughly washing with water, drawn both ways through by suction and drying at 140°, the crucible maintained a weight varying only by

one-tenth milligram. Although the particles of CuSCN are much smaller than those of AgCl, they were retained by the pores of the crucible.

III. Determination of Al in $Al_2(SO_4)_3$ as Al_2O_3.

The effect of high temperature upon the Alundum crucible as well as its applicability for filtration of gelatinous precipitates, were discovered by this analysis. Chemically pure $Al_2(SO_4)_3.18H_2O$ was dehydrated by gentle dessication and final heating for 4½ hours at 140°. A solution was prepared such that each 50 c. c. contained .2154 grams of $Al_2(SO_4)_3$. $Al(OH)_3$ was precipitated from the hot solution in the presence of NH_4Cl by just a sufficient amount of NH_4OH. After boiling, stirring and allowing to stand, the precipitate was filtered through the crucible previously heated in the flame of a blast lamp for several minutes, and washed with water containing small amounts of NH_4Cl and NH_4OH. In seven attempts however in spite of all precautions and lengthened time of treating the precipitate, part was drawn through the crucible with the filtrate. The last three determinations were made with an entirely new solution of $Al_2(SO_4)_3$, each 50 c. c. containing .2001 grams.

EFFECT OF HEATING ON THE CRUCIBLE.

Except in one case where the change was negligible, the crucible lost in weight consistently from one analysis to the next. The first crucible was pulled apart by suction while cleaning with water following the third analysis, and the second in exactly similar fashion after the fourth analysis in which it was employed, showing that the material becomes extremely brittle after heating several times in the blast lamp.

IV. The determination of Ba in $BaCl_2$ as $BaSO_4$.

The extremely small size of the particles of $BaSO_4$ under ordinary conditions recommended a further test of the crucible. The pure $BaCl_2$ was thoroughly dried by heating three hours at 140° and a solution made such that 50 c. c. contained .2007 grams. The hot solution slightly acidified with HCl was treated with pure dilute H_2SO_4 while vigorous stirring was maintained for five minutes, after which the $BaSO_4$ was allowed to settle for five minutes.

Percent Ba in $BaCl_2$ calc. 65.96 found (1) 65.86
(2) 65.98
(3) 65.79

The crucible was washed with water under suction and maintained

practically a constant weight. A final trial of the effect upon the weight of rubbing simply with the finger was made. The crucible lost .0039 grams.

SUMMARY OF ADVANTAGES.

(1) The crucible was used to advantage in determining Cu as CuSCN, Ba as $BaSO_4$ and Ag in AgCl when HCl was used as the precipitating agent.

(2) Manipulating was found to be surprisingly simple and rapid when an analysis was once under way.

(3) Nearly a constant weight was maintained by the crucible if not heated over 140° when washed with water.

SUMMARY OF DISADVANTAGES.

(1) It was found difficult at best to wash precipitates free from precipitating agent, especially if the latter should be left as a solid residue upon evaporation of the solution.

(2) Suction was necessary, and, in the case of $AgCl,NH_4OH$, to thoroughly cleanse the crucible.

(3) It was found unadapted to filtration of such gelatinous precipitates as $Al(OH)_3$.

(4) Heating six or eight times in the blast lamp was sufficient to render the crucible so brittle as to be broken even by suction, and a consistent loss in weight was observed.

(5) In three cases the first use of crucibles led to results greatly in error.

(6) Abrasion or friction was found to have a marked effect upon the weight of the crucible.

(7) The ordinary digestion of fine precipitates to be filtered was not avoided when it was used.

Like the Gooch crucible therefore, the Alundum crucible apparently has only a limited field of usage but within that field it should be of considerable worth to the analyst.