

3. After the two samples just mentioned had been collected, the regulation was interrupted. The equilibrium was soon destroyed and the temperature gradually rose. Then the original temperature was re-established by regulating the addition of carbon disulphide to the boiling mixture in *B*, and two new samples were collected. They had respectively the refractive indices $39^{\circ} 41'$ and $39^{\circ} 41'$.

4. Once more the equilibrium was disturbed, and once more the original temperature re-established in *A*. While the temperature remained practically constant, again two samples of vapor were collected. They were found to have the refractive indices $39^{\circ} 40'$ and $39^{\circ} 40'$.

These few results, taken from among a considerable number of preliminary observations, are mentioned as a first proof of the reliability of the method. The conclusion that may be drawn from them is thoroughly corroborated by an extensive experience accumulated since these preliminary results were obtained.

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THE MUNROE CRUCIBLE.

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The use of platinum felt as the filtering medium in a crucible of the Gooch pattern was first suggested by Charles E. Munroe, in an article entitled "Filtration with Filters of Metallic Felt," published in the *Journal of Analytical Chemistry*, Vol. 2, July, 1888.¹

Crucibles prepared by the method suggested by Munroe have many advantages not possessed by any other type of apparatus used for filtration, and since the use of such crucibles is by no means as general as their merit would warrant, it seems desirable to again briefly describe the preparation and uses of crucibles of this type.

Preparation of the Munroe Crucible.—A concentrated solution of chlorplatinic acid is precipitated by ammonium chloride, the latter reagent being added in slight excess. The resulting precipitate of ammonium platonic chloride is washed several times with water, and is finally washed with alcohol, the excess of alcohol being poured off as soon as the ammonium chlorplatinic acid has settled. A perforated platinum crucible (one preferably in which the perforations are numerous and small) is then placed upon several layers of filter paper, and held firmly in this position while the alcohol-moist mass of ammonium chlorplatinic acid is poured into it, usually until it is filled to a height of from 0.25 to 0.5 cm. The alcohol will be rapidly absorbed by the filter paper upon which the

¹ And reprinted, *Chem. News.*, 58, p. 101, 1888.

crucible is pressed, and at the expiration of a few moments the surface of the salt in the crucible will show that all of the excess has been taken up by the filter paper, the crucible being held firmly against the layers of filter paper until this occurs. The moment at which the last of the excess of alcohol is absorbed by the filter paper is readily noted by the sudden "drying" of the surface of the ammonium chlorplatinat which takes place at that instant.

The crucible can now be removed from the filter paper, and if the foregoing operations have been properly performed, an even layer of ammonium chlorplatinat will be found to cover the bottom of the crucible, the filter paper having prevented any from having run out through the perforations. The crucible is now warmed in a water oven until the alcohol has been entirely driven out of the salt, and then, after the cap and cover have been put on, the crucible is slowly and cautiously heated until the decomposition of the ammonium chlorplatinat is effected, the heating being continued until the crucible reaches dull redness.

When the crucible cools it will be found that a layer of coherent platinum sponge covers the bottom, this layer being sometimes continuous and evenly covering the bottom of the crucible, but more often it will be interrupted by several cracks, and drawn away from the sides of the crucible. By gently pressing with the finger or with a glass rod flattened somewhat at the end, the platinum sponge can be pressed together at the places where cracks have appeared, and, if carefully done, a continuous layer of platinum sponge can thus be formed over the entire bottom of the crucible. If, however, the trial has not been successful in preparing a thoroughly continuous layer, fresh chlorplatinic acid can be used to fill up the cracks and "patch" the platinum sponge layer, or the entire layer can be removed by means of a spatula, and the trial repeated until an even coherent layer of platinum sponge completely covers the bottom of the crucible. When this has been achieved the crucible is finished.

Characteristics of the Munroe Crucible.—Prepared as described, the mat of the Munroe crucible forms a smooth, even layer, of light silver gray color, over the bottom of the crucible. To the touch the mat presents the soft and slightly elastic feeling characteristic of metallic felts.

The porosity of the mat, and at the same time its ability to retain the finest precipitates, are factors so marked as to cause surprise to one who uses the crucible for the first time. Experiments were tried by the writer to determine the relative porosity of crucible filters with asbestos, and with platinum felt, as the filtering medium, the experiments being carried out as follows: A rubber stopper was selected of such size that it would just fit into the top of the crucible, and through a perforation in this stopper the lower end of a glass tube somewhat over a meter long was placed, the upper end of this tube being so bent as to siphon from a large

vessel of water placed upon an elevated shelf. The height of water in the vessel was such that the vertical distance from the level of the water to the bottom of the crucible was just one meter. Directly under the crucible a 500 cc. graduated flask was placed, and the length of time required by the different crucible filters to allow 500 cc. of distilled water to pass, was noted.

The first crucible tested was one with asbestos mat, taken at random from a number of crucibles which had been made up for general work, and with no intention of being subjected to test. 25.6 minutes were required for 500 cc. water to pass. A Munroe crucible was next taken and 500 cc. of distilled water were found to pass in $4\frac{3}{4}$ minutes, the platinum felt mat thus being seen to have a porosity five times as great as the asbestos mat. About these same relative figures were obtained in all the experiments tried, eight crucibles with asbestos mat, and five with platinum felt mat, being tested, the average time required to pass 500 cc. of water being $5\frac{1}{4}$ minutes in the case of crucibles with the platinum felt mat, and $3\frac{1}{2}$ minutes in the case of the crucibles with asbestos mat. As the average amount of asbestos used in each crucible was 0.145 gram, and the average weight of the platinum felt mat was 0.4400 gram, it will readily be seen that weight for weight the porosity of platinum felt is more than a hundred times as great as asbestos felt.

The platinum felt retains without difficulty the finest precipitates, and many substances which cause trouble when filtered in the ordinary Gooch crucibles using asbestos are easily filtered in the Munroe crucible. Barium sulphate and calcium oxalate can be collected by the platinum felt mat without the filtrate even being clouded the first time running through.¹

Uses of the Munroe Crucible.—In the platinum felt crucible not only the body of the crucible, but the filtering medium as well, is platinum. The advantage which platinum has through its almost absolute insolubility in nearly all laboratory reagents, has made it indispensable to the chemist, and through the use of the Munroe crucible the advantage gained by the use of platinum is extended to the apparatus used in filtration. Even with the use of platinum dishes the chemist must bear in mind the slight solubility effect of certain reagents, and of course this same care must be taken to protect the platinum felt crucible from these substances. With this fact in mind, and with careful handling, the platinum felt crucible will be found to cover practically all the requirements of a crucible filter. In the ordinary crucible filter, using asbestos as a filtering medium, traces of silica, magnesium, iron and aluminum will sometimes be found to be dissolved from the asbestos and to contaminate the filtrate. While it is true that this happens but seldom and usually in such small amount that no serious harm is done, yet it is also true that after certain chemical

¹ Geo. Wash. Bull., Vol. 5, No. 4, pp. 82 (1906).

operations the decomposition of the asbestos becomes quite marked, so that very appreciable errors can be quite easily brought about, and in exact chemical work even the ordinary slight solvent action upon the asbestos is detrimental to the accuracy of the work, or requires tedious separations, and blank determinations. With the Munroe crucible the body of the crucible and the filtering medium are composed of but a single element, and consequently but a single impurity can be introduced in the process of filtration; while by the use of care in the work, and the choice of suitable reagents, the possibility of platinum being carried away becomes extremely remote. One case only requires particular mention; platinum sponge is known to be slightly soluble when acted on by hydrochloric acid in the presence of air or oxygen, and accordingly platinum felt crucibles which have just been used in the filtration of hydrochloric acid solutions, should be washed with water thoroughly before being dried.

By the choice of an appropriate solvent, the precipitate which has been collected in the Munroe crucible can be dissolved leaving the crucible ready, after drying and weighing, for the next determination, the crucible thus being used over and over, a new mat only being prepared when careless handling makes this necessary. A crucible used by the writer has now served in about two hundred analyses of the most varied nature and is still in good condition, the mat never having had to be changed in any way.

In dissolving the precipitates which have been collected in the crucible, particularly in the case of carbonates, through the effervescence of which, on contact with acid, particles of the mat are likely to be dislodged, it is desirable to place the crucible in a pipe-stem triangle over a porcelain dish,¹ so that the bottom of the crucible is immersed in a dilute solvent placed in the dish. By gradually heating the solvent, not quite to boiling, the precipitate can be quickly dissolved.

Although the platinum felt crucible is admirably adapted to nearly all the varied requirements of a crucible filter, it seems to the writer that it is in the determination of atomic weights, and similar work requiring extreme accuracy, it has its best field of application. Richards and Staehler have used the Munroe crucible with success in their work on the atomic weight of potassium,² finding in it an instrument well adapted to their needs in that work, and it is probable that the substitution of the Munroe crucible for crucibles with asbestos felt, in most atomic weight determinations, would be found to be advantageous.

The use of the Munroe crucible in the determination of reducing sugars

¹ Thesis of M. M. Brewer, Geo. Washington University, 1901, p. 28.

² THIS JOURNAL, 29, 623 (1907).

has been described by Pelet,¹ and by Zerban and Naquin.² Gooch and Beyer, in their work on the use of filtering crucibles in electrolytic analysis, have recently³ pointed out advantages which can be gained in certain cases through the use of platinum felt as the filtering medium.

Phelps has used the Munroe crucible in his work on the analysis of barium sulphate, and has found that results difficult or impossible to reach by the use of crucibles containing asbestos mats can be easily obtained by the use of the platinum felt crucible.

General Notes.—In his original paper⁴ Munroe refers to rubbing the platinum sponge with a glass rod, to smooth out the mat and remove cracks, and this procedure compresses the mat and burnishes its surface to a considerable degree. By gently rubbing all parts of the mat with some hard object until the surface becomes considerably burnished, a very smooth and polished surface is obtained, which for some purposes becomes of considerable advantage, although it should be noted that the porosity of the felt is greatly diminished.

In preparing platinum felt crucibles, advantages can be gained by placing a circular piece of fine platinum wire gauze of suitable size in the bottom of the crucible, before pouring in the ammonium platinic chloride, the mat being tougher and less easily injured, and also being less liable to crack and curl, when it has the support of the skeleton formed by the platinum gauze.

Precipitates often cling very tenaciously to the surface of the platinum felt, and if removed mechanically will often take particles of the felt with them. In cases where it is necessary to mechanically remove precipitates, the method suggested by Richards and Staehler,⁵ of placing a perforated disk of platinum of suitable size on the top of the layer of platinum sponge, will be found to be of advantage.

If two or three drops of chlorplatinic acid are placed upon the platinum felt mat in a crucible, the porosity of the felt will cause the solution to distribute itself through the mat, and if the crucible is then ignited the chlorplatinic acid will be decomposed with the separation of platinum. It is probable that the platinum thus separated within the body of the mat cements the particles of platinum sponge together to a certain degree, and also cements the sponge to the body of the crucible (and to the platinum gauze, if such has been used), so that when so treated the felt mat is somewhat toughened.

Several names have been used by previous writers in referring to cru-

¹ *Bull. assoc. chim. sucr. dist.*, **24**, 1392.

² *THIS JOURNAL*, **30**, 1456 (1908).

³ *Am. J. Sci.*, **25**, 249 (1908).

⁴ *J. Anal. Chem.*, **2**, 241 (1888).

⁵ *THIS JOURNAL*, **29**, 623 (1907).

cibles with platinum felt as the filtering medium. Munroe in his original publication refers to his device simply as "Filters of Metallic Felt," and other writers have used the name "Gooch-Munroe"¹ and "Munroe-Neubauer"² crucible. The writer sees no necessity for this hyphenation, since Munroe has undoubted priority in the use of platinum felt as a filtering medium in a filtering crucible, in all the three elements which are involved in the conception and presentation to science of an idea; *viz.*, invention, experimentation, and published description, and accordingly the only name which seems suitable is that used as the title of this article.

THE ACTION OF COKE ON FERRIC CHLORIDE AND AURIC CHLORIDE IN SOLUTION.

BY ALFRED TINGLE.

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W. O. de Coninck³ states that when solutions of auric, platinumic or ferric chloride are filtered through animal charcoal or powdered coke the metal is retained by the filter. No quantitative data are given in the paper cited.

This behavior of perchlorides with coke came to be of interest to the present writer, and so led to a short series of experiments under definite working conditions, which may be of sufficient interest to be recorded as showing some of the limitations which should qualify de Coninck's statements. The action of animal charcoal was outside the scope of the present work.

The coke used was powdered to such size that it passed through a sieve of 20 meshes to the inch, but not through one of 40 meshes. Two series of experiments were made, one being on coke which was merely thus pulverized and another on "washed coke." To prepare the latter, previously pulverized coke was boiled with pure concentrated hydrochloric acid, washed with distilled water till the washings gave only a faint precipitate with silver nitrate solution, and dried by heating in a dish on the hot plate.

Each coke filter was made by placing a plug of glass wool just above the top of a Jones "reductor" and filling in the space above to a height of 20 cm. with coke. The approximate weight of this amount of coke was 40 grams. It may be remarked that this method of procedure differs slightly from that of de Coninck, who states that he used (in his experiments with animal charcoal) a funnel 8 to 10 cm. in diameter, three-quarters filled with the filtering material, but gives no details of his experiments with coke. The solutions used were auric chloride, ferric

¹ THIS JOURNAL, 29, 633 (1907).

² *Ibid.*, 30, 1456 (1908).

³ *Compt. rend.*, 130, 1551-1552