

through which the cooling water overflows and passes to the drain (Fig. 1, *f*). Immediately underneath the cocks (ordinary brass gas cocks) is a trough which catches the water leakage (if any) from the cocks.

"Low heat" of the electric heater is, in the Raleigh climate, adequate and best.

The glass cylinders are $5\frac{1}{4}$ in. high, $1\frac{9}{16}$ in. in diameter (inside) and weigh 40 to 50 g. But instead of receiving the ether extract in these larger cylinders it may be received in smaller cylinders, set inside the larger ones. These smaller cylinders will have a height of $1\frac{7}{8}$ in., and diameter (outside) of $1\frac{7}{16}$ in., and, if of glass, weigh 14 to 18 g., if of metal (aluminum) 5 to 8 g. In this arrangement the large cylinders have no projections for the support of the crucibles. The supporting is done by projections in the smaller cylinders or by a small triangle (aluminum wire) set inside. If the bottom of the larger cylinder is covered with sand or, better, copper filings (No. 80), the heat conducting contact between outer and inner cylinder is improved. Mercury would make a perfect contact, but the avoidance of mercury, corks, and ground glass joints is a virtue—and no small one—of this type of extractor. In this arrangement (of an inner receiver), the "medium heat" of the heater has been found best.

Cylinders or jars of appreciably smaller size ($1\frac{7}{16}$ in. instead of $1\frac{9}{16}$ in. inside diameter) have proven efficient. There is space for twenty-two of these smaller jars on the $4\frac{1}{2} \times 24$ in. heaters, eleven on each side.

Vertical sections of the parts (except the water-distributing system), with dimensions and brief description, made originally over a year ago for Mr. Glenn H. Pickard, Chicago, are given in Fig. 3.

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A COLORIMETRIC DETERMINATION OF LEAD DIOXIDE IN LITHARGE

By WALLER V. MORGAN
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The object of this note is to present a simple method for the determination, in a semi-quantitative way, of lead dioxide in the presence of litharge. The method has been used to advantage in connection with rubber work where it is important to know the amount of oxidizing agent present. The method with slight alterations is applicable to the determination of oxidizing agents in general where they occur as an impurity with non-oxidizing agents.

METHOD

A definite amount of litharge, *e. g.*, 5 g., is boiled for a minute with a solution containing 2 g. of aniline hydrochloride dissolved in 10 cc. of water and 5 cc. of concentrated hydrochloric acid. The solution is then cooled to separate any lead chloride which has formed, then filtered to remove the lead chloride and any litharge which has not been dissolved. It is not necessary to convert all or part of the litharge into lead chloride as the action depends upon the oxidizing power of the dioxide only. The filtrate is compared

with standards made by adding definite amounts of lead dioxide to the above-mentioned solution. A colorimeter may be employed to estimate the amount of aniline purple formed, or the solutions may be compared in suitable receptacles.

The lead dioxide oxidizes the aniline to aniline purple. The intensity of the color is proportional to the lead dioxide content.

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A METHOD FOR BRINGING ELEMENTARY SULFUR INTO SOLUTION FOR ANALYSIS

By A. P. BJERREGAARD
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This method was devised to analyze some samples of flowers of sulfur intended to be used for making sheep dips. The work was done in June 1909 at the laboratory of the New Mexico Agricultural Experiment Station.

On ignition these sulfurs all left a small amount of coke, which slowly oxidized away on continuing the ignition. Practically no incombustible ash was left. The presence of this carbonaceous matter made the combustion method of analysis unavailable for determining the percentage of actual sulfur present.

An attempt was made to extract the sulfur with carbon bisulfide, but it was found that most of the samples left a very large residue of insoluble sulfur, making this method also unavailable. Some of the samples containing large proportions of insoluble sulfur were then boiled for a few minutes with alcohol, the alcohol poured off, and carbon bisulfide added. There appeared to be no change in the proportion of insoluble sulfur. Heating the samples to fusion and then to 125° C. in the air bath did not render the insoluble sulfur soluble; moreover, small amounts of sulfur were volatilized in this procedure, thus tending to vitiate the results even if the sulfur had become soluble. A higher temperature would of course aggravate this error.

Boiling with a mixture of nitric acid and bromine dissolved part of the sulfur, but about half of it fused to small globules which dissolved with extreme slowness. Boiling with a mixture of bromine and water resulted in the evolution of gas of a strong sulfurous odor, nor did this procedure dissolve all the sulfur.

Finally advantage was taken of the solubility of sulfur in dry liquid bromine, and it was dissolved in that substance and concentrated nitric acid added. On slightly warming this solution a vigorous action ensued, copious bromine and nitrous fumes were given off, and the sulfur dissolved completely in a few minutes.

In practice about 0.1 g. of sulfur was dissolved in 1 cc. bromine, and 10 cc. nitric acid added. After removing the excess of bromine and the nitrous fumes by heat, 100 cc. water and a few cubic centimeters of hydrochloric acid were added, and the solution boiled again to expel the rest of the nitric acid, after which the sulfuric acid was precipitated in the hot solution by barium chloride in the usual manner. The results were entirely satisfactory.

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