needed only to remove the last traces. Unless very large volumes of air are to be run through, a copper column 4 or 5 in. high (if punchings are used) is quite sufficient. Where turnings are used, a much higher column is necessary to present the same surface. The bottle should hold a couple of liters to make frequent filling unnecessary.

The reagent recommended is made by diluting one part of commercial ammonia with one part of water and saturating the mixture with ammonium chloride. This mixture is a quantitative absorbent of oxygen until so heavy a precipitate forms that the apparatus is clogged.

If the tube D is not a part of the apparatus there is a stagnation of reagent in the tower and that becomes clogged long before the apparatus is exhausted. The tube D furnishes a slow circulation which effectually prevents stagnation in the tower. The circulation so obtained is not nearly as rapid as in Van Brunt's apparatus, but is amply sufficient to keep the tower in operation.

That this reagent gives very pure nitrogen is shown by the fact that

Ferguson¹ used this apparatus in his electrotitrimetric determination of iron; and after reaching the end-point, if the nitrogen so prepared was allowed to flow for some minutes, no change in the end-point could be detected. Mr. R. K. MacAlpine² of the chemistry laboratory of the University of Michigan used this apparatus to prepare nitrogen for use in atomic weight work. He found that the nitrogen so prepared, very slowly reduced a dilute chromate solution. The action was exceedingly slow and took some time for reduction enough to cause a visible color change in a solution of chromate so dilute that the yellow color was just apparent.

¹ THIS JOURNAL, 9 (1917), 941.

² Personal communication.

Using 8 mm. glass tubing for connections and a tower containing about 5 in. of copper punchings, the author has been able to put a stream of 95 per cent commercial nitrogen through this apparatus faster than ordinary Drechsel gas wash bottles could remove the ammonia, and yet the resulting gas gave no test for oxygen with colorless cuprous chloride solution. The introduction, following the generator, of a wash bottle containing a little cuprous chloride solution with some metallic copper is very useful in indicating a leak or other failure of the apparatus.

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FAT EXTRACTION APPARATUS

By J. M. PICKEL

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The fat extractor pictured here (Figs. 1 and 2) was designed by the writer and has now entered on the third year of its use in his laboratory. It is compact and economical—more so than any other that has come under his notice. Twenty fat extractions are made simultaneously on one electric heater $4\frac{1}{2} \times 24$ in., ten on each side of the heater, as against a total of seven by the leading extractor of this general style now on the market. About 15 cc. of ether, one-third

FIG. 1

to one-half of which is recovered for future use, are required for each extraction. The ether is distilled off from the extract (recovered) by merely giving the condenser a slight turn on its axis; there is no interruption of the distillation and no time or ether lost in taking the apparatus apart, removing the substance extracted, putting in its place a tube or receiver for the ether, putting the apparatus together again, and starting up the distillation again.

FIG. 2

APPARATUS

CONDENSER, metal (Fig. 2, *a*; Fig. 3, A). Note the eccentric position of the ether drip-point, and the smaller diameter of the lower half-inch of the condenser.

CYLINDER, glass (Fig. 2, *b*; Fig. 3, B). A cylinder is used rather than a flask for the reason that it takes up less room on the heater.

ETHER RECOVERY CUP,¹ metal (Fig. 2, *c*; *c'* side view; *c''* bottom view; *c'''* top view; and Fig. 3, C). Note the small funnel (top view, *c'''*) which, by means of its bent stem passing through and soldered to the center of the cup (bottom view, *c''*), conveys the ether to the substance being extracted. A small triangle is soldered to the bottom of the cup. This supports the cup on the crucible below. During the extraction the drip-point of the condenser is over the funnel. A slight turn of the condenser brings the point to one side; the ether then drops into the cup and is thus distilled off from the extract.

CRUCIBLE, alundum RA 360, No. 5204 cut down to a depth of $\frac{7}{8}$ in. (Fig. 2, *d*; Fig. 3, D). This will accommodate 2 (and often as much as 4 or 5) g. of ordinary feeding stuffs. A small disc of filter paper or, better, of perforated metal is laid on the stuff to prevent the constant dropping of the ether from drilling a canal through the stuff.

¹ THIS JOURNAL, 7 (1915), 236, the writer described an ether recovery tube adapted for use with the Knorr fat extractor.

FIG. 3—VERTICAL SECTIONS OF THE PICKEL FAT EXTRACTOR

- A—Condenser, metal (copper or brass); *a*—eccentric drip point.
 B—Jar, glass, weight 30 to 40 g.; *b*—three support points for crucible.
 C—Ether recovery cup with funnel (both metal, but glass possible).
 D—Crucible (alundum) RA 360, No. 5204 cut down to a depth of $\frac{7}{8}$ in. (Norton Co., Worcester, Mass.).
 E—Receiver for fat, glass or metal (preferably aluminum). Use optional, but if used, it sits in B, in which case B is without the support points *b*; and in which case, also, it is E, not B, that receives the fat and is weighed.
 F—Shows position of crucible whose top should be about $\frac{1}{8}$ in. below top of E. F is supported by suitable indentations in E or else on a little aluminum wire tripod to be weighed along with E. A little sand or copper filings in the bottom of B improves the heat-conducting contact between B and E; or mercury may be used, but it is otherwise a nuisance best avoided. It would improve E if its bottom diameter were $1\frac{1}{2}$ in., its top diameter remaining $1\frac{3}{8}$ in., but in this, reference must be had to the inside diameter of B.

WATER SYSTEM (Fig. 1, *e*). The small pipes which convey the cooling water from the cocks down into the condensers end at the point where the larger tubes join the condenser, thus making it possible to raise and lower the condensers for the admission and removal of the cylinders. Momentum carries the water down to the bottom of the condensers; the cooling effect is thoroughly adequate. Practically all the ether condenses on the lower quarter-inch of the condenser,

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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