

cyanate. After about 40 minutes' heating, a crystalline solid began to separate, and at the close of the hour the mixture was almost solid. When cold, the mass was washed thoroughly with ether, to remove unchanged isocyanate, then with hot alcohol, and dried at 110°. Yield, nearly theoretical.

Found: N, 18.84. Calculated for $C_{16}H_{14}O_2N_4$: N, 19.04.

The purified substance forms a colorless, micro-crystalline solid, infusible at 300°, and insoluble in water, ethyl or amyl alcohol, acetone, benzene or ether.

2-Methyl-3-benzalamino-4-quinazolone, C_6H_4 $\left\{ \begin{array}{l} N = C.CH_3 \\ | \\ CO - N.N:CHC_6H_5 \end{array} \right.$. — The amino-

quinazolone was boiled for a few minutes with excess of benzaldehyde, until the water split off in the reaction was driven out. When cool, a considerable volume of alcohol was added to the mixture, and from this solution, on standing several hours, the benzal derivative slowly crystallized in stellate groups of needles. Recrystallized from alcohol, it melts at 183° (corr.).

Found: N, 16.08. Calculated for $C_{16}H_{13}ON_3$: N, 15.97.

It is insoluble in water, sparingly soluble in alcohol, and not readily soluble in mineral acids.

Acetantranil and Unsym. Methylphenylhydrazine.—Acetantranil was warmed with a slight excess of unsym. methylphenylhydrazine. Reaction occurred and heat was liberated. The product was recrystallized from alcohol, in which it is very easily soluble, although but slightly soluble in water. It melts at 106° (uncorr.) and contains 9.90 per cent. nitrogen. The expected quinazolone contains 15.85 N, and the intermediate amide 14.88. It has not been further examined.

When equimolecular amounts of 2-methyl-3-amino-4-quinazolone and *o*-benzoquinone¹ were brought together in chloroform solution, nothing definite could be isolated. With *p*-benzoquinone, an infusible, insoluble, black condensation product resulted.

m-Nitrobenzoylantranil was boiled with excess of hydrazine hydrate solution, and the white solid obtained crystallized from 95 per cent. alcohol. Colorless needles, melting with effervescence at 196-7° (corr.). Not further investigated.

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NOTES.

A Simple Fat Extraction Apparatus. — It has come to be quite generally recognized that a considerable advantage is to be gained from the use of an extraction apparatus carrying a mercury seal instead of the more usual cork or ground glass joint. A very interesting note² on the use of such seals recently appeared, and the forms of a number of flasks which have been devised for this purpose were shown. Hitherto almost all efforts at improvement seem to have been expended on this flask, the condenser and tube for holding the sample being usually of the Knorr form. Certain difficulties are encountered in the use of this condenser, and to obviate these a form of apparatus was evolved by Mr. J. W. Ames, Chemist of this Station, by combining the desirable points

¹ McPherson and Lucas, THIS JOURNAL, 31, 283 (1909).

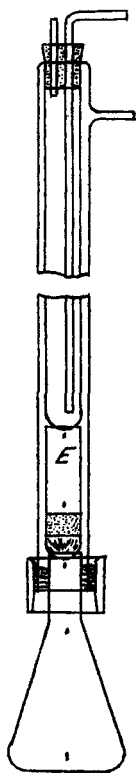
² J. Ind. Eng. Chem., 1, 314 (1909).

of several others. The arrangement is shown in the figure, and will be seen to be very simple, with no delicate or easily broken parts.

The flask used is of the form devised by Dubois,¹ but the mercury cup differs from his somewhat in the internal shape of the maple ring and in that a flat, sound cork is used instead of a rubber stopper, being more durable and satisfactory as well as inexpensive. The form of flask devised by Sy has also been used, but is not very satisfactory on account of the difficulty of cleaning out the mercury and of making adjustments of the mercury cup to fit the sample tubes.

The principle of the condenser used is the same as that of the well-known Hopkins form,² and offers a considerable advantage over all others. The cooling surface is unusually large, and condensation is so perfect that practically the only time at which the odor of ether is perceptible is when the condenser and flask are disconnected. The extraction tube used is shown at *E* in the figure, the tube used for collecting the ether differing from it only in that there is no hole in the bottom. It is dented in on two sides sufficiently to allow the ether vapors to pass upward. The filtering medium is cotton, on which are placed small circles of filter paper, the perforated plates being of aluminium. The whole apparatus may be obtained under the name of the Ames Fat Extraction Apparatus.

The use of such a form of extractor being no doubt unfamiliar to many, it may be well to add a few words of explanation as to the points most likely to cause trouble. After drying the extracted fat as usual, the flask is wiped thoroughly to remove particles of dirt, mercury, etc., after which it should stand for 30 minutes before weighing, since rubbing produces electrification on the surface, and this length of time is necessary to allow the charge to dissipate.³ Should a little mercury be accidentally jarred into the flask it will not spoil the determination, merely causing some additional labor. After drying and weighing the flask as usual from 5 to 10 cc. of ether are introduced and agitated to dissolve the fat. The ether is carefully decanted off and the washing repeated with two more portions, after which the flask is dried as before. Previous to weighing, however, the flask must be allowed to stand about 12 hours, for should it be weighed immediately it would be found to be from 20



¹ THIS JOURNAL, 30, 797 (1908).

² *Ibid.*, 20, 965 (1898).

³ In the laboratory of Prof. T. W. Richards a small amount of a salt of radium is used to dissipate such a charge. EDITOR.

to 50 mg. too heavy, while after standing this length of time it will have settled to normal condition and weight. The loss shown by this weighing gives the amount of fat extracted. The flasks are more easily cleaned by chemical than by mechanical means, thus also avoiding any danger of breakage. The bulk of the fat is removed by means of a hot strong solution of sodium hydroxide, the flask rinsed and the remaining fat removed by hot strong sulphuric acid.

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The Esterification of Certain Higher Fatty Acids on Evaporation of Their Alcoholic Solutions.—In an article by one of us¹ on the solubility of stearic acid in alcohol, attention was called to the fact that the solubility of stearic acid was apparently greater after evaporation from alcoholic solution than before. It was thought that this might be due to the presence of alkaloids in the alcohol, or to esterification. It was found, however, that the acid still showed the increased solubility when evaporated from alcohol which had been treated so as to insure the absence of alkaloids. This made esterification a probable explanation. As interaction between this acid and alcohol, on simple removal of the latter by evaporation, had not been previously noted, it seemed desirable to ascertain whether titration of the acid with a standard alkali before and after evaporation with this solvent would give confirmatory evidence.

Stearic acid was first tried with sodium hydroxide. The average of the results showed that less alkali was required for neutralization after evaporating the acid with alcohol than before. The difference, however, was small, and less than the extreme variation among the individual results in each series. The end point was very unsatisfactory, partly due to the suppression of the color of the phenolphthalein by the alcohol, and partly to the opalescence of the solution at the close of the titration.

Better results, given below, were obtained with palmitic acid and potassium hydroxide, though the end point was still rather difficult to observe with certainty. The titrations in all cases were finished in a solution containing 55 per cent. alcohol, as the results of A. Canitz² indicate that no appreciable hydrolysis of the soap was to be expected in alcohol of this strength. The palmitic acid was Kahlbaum's best grade, but, as shown by the melting point and titrations, it was not entirely pure. The alkali was made by precipitating a saturated solution of c. p. barium hydroxide with potassium sulphate, using a very slight excess of the latter. It was standardized against hydrochloric acid, which in turn was stand-

¹ THIS JOURNAL, 27, 1750.

² Ber., 26, 400.