PRACTICAL APPLICATION.

I have so far used the method and tested it only in sanitary water analysis and in the analysis of urine. To the water analyst it will be of great value. It gives the lime and sulphuric acid with almost the accuracy of the gravimetric method. It is more accurate than the soap test and is but slightly affected by the presence of magnesium salts.

For determining the sulphuric acid in urine I have found it quite satisfactory. The urine has to be diluted with nine volumes of water and then the color does not sensibly affect the determination.

I see no reason why this method may not be successfully used with all fine white precipitates. It is not suitable for precipitates that settle rapidly or gather quickly into flakes. Whether colored precipitates may be determined in this way is still to be investigated.

I desire to acknowledge obligation to Professor A. H. Buchanan for assistance in determining the equations and probable errors.

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NO. 12.]

THE SEPARATION OF TRIMETHYLAMINE FROM AMMONIA.

BY HERMANN FLECK.

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THE quantitative estimation of trimethylamine in presence of ammonia is, I believe not mentioned in the literature, although a number of publications have appeared in which the detection of trimethylamine, in presence of ammonia, by means of the different solubilities of their hydrochlorides in absolute alcohol, has been successfully carried out.

Dessaignes[1] prepared and analyzed with good results the platinum double salt of trimethylamine, by conducting the mixture of ammonia and trimethylamine vapors into hydrochloric acid, evaporating to dryness, extracting with absolute alcohol, precipitating with platinic chloride and recrystallizing the precipitate formed several times from hot water.

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[1] *Ann. Chem.* (Liebig), 81, 106.

Wicke[1] adopts the same method, using, however, alcohol-ether extract.

Winkeles,[2] in using this method, further states that while ammonium chloride is soluble to some extent in absolute alcohol, it is rendered totally insoluble by the presence of salts of such bases as trimethylamine.

Eisenberg,[3] by a similar procedure, obtained the platinum double salt in crystals of great purity and perfection.

The success in each case is undoubtedly due to the fact that large quantities of hydrochlorides were used. Winkeles,[4] for example, employed the hydrochlorides obtained from twenty-six gallons of herring brine. Further the mixtures were very rich in trimethylamine.

This method applied to a substance containing a low percentage of the latter yielded results, which clearly show that trimethylamine hydrochloride does not render ammonium chloride insoluble in absolute alcohol, and further does not serve as a good means of qualitative, much less of quantitative, separation. A portion of the mixture containing trimethylamine and ammonia was saturated with hydrochloric acid, evaporated to dryness and extracted several times with portions of several times the volume of boiling absolute alcohol. The alcoholic extract evaporated to dryness gave eighteen per cent. of supposed trimethylamine hydrochloride. To identify the latter, the residue was taken up with alcohol and platinic chloride added. The precipitate formed was redissolved in boiling water and the different fractional crystallizations, consisting of octahedra, analyzed.

	Pt found.	Required for
First crystallization......	43.6	$(NH_4Cl)_2PtCl_4$, 43.84
Last "	39.5	Corresponding to a mixture of $2\left((NH_4Cl)_2PtCl_4\right)+3\left(N(CH_3)_3.HCl\right)_2PtCl_4$, which require 39.4 per cent. Pt.

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[1] *Ann. Chem.* (Liebig), 91, 121.

[2] *Ann. Chem.* (Liebig), 93, 321.

[3] *Ber. d. chem. Ges.*, *1880*, 1669.

[4] *Loc. cit.*

Intermediate crystallizations gave intermediate, gradually decreasing results, showing that the isomorphous forms of the two salts crystallized together.

Duvillier, Buisine[1] extract the mixed *sulphates* to prepare pure trimethylamine from the technical product. The suggestion led to the use of the following method which yielded satisfactory results.

The mixed hydrochlorides are repeatedly extracted with portions of a total of five or six times the volume of boiling absolute alcohol and the solvent distilled off in a three-quarter liter distilling bulb. An excess of caustic soda is added to the residue and the gases formed on boiling driven over into a large quantity of water. Litmus is added, followed by the exact quantity of dilute sulphuric acid required to neutralize. The liquid is evaporated to dryness and extracted with one liter cold absolute alcohol, in which trimethylamine sulphate dissolves, leaving ammonium sulphate undissolved. The alcohol is distilled off, the residue transferred to a weighed dish, dried and weighed. In this manner 32,910 grams of the carefully dried mixed chlorides gave two and five-tenths grams trimethylamine sulphate, corresponding to 2.21 grams hydrochloride, or 6.71 per cent.

That the extraction was complete is evident from the total absence of the fishy odor when the extracted residues are treated with alkali. That the extracted material is pure is shown by the following analyses of the octahedral crystals of the platinum double salt prepared from the trimethylamine sulphate :

	Per cent. Pt.	Required for $[N(CH_8)_3.HCl]_2.PtCl_4$. Per cent. Pt.
I. 0.0983 gram gave	36.92	....
II. 0.3017 " "	37.12	36.93

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[1] *Ann. Chem.*, (Liebig) (5) **23**, 299.