A Micro-Kjeldahl Method for Nitrogen Determination*

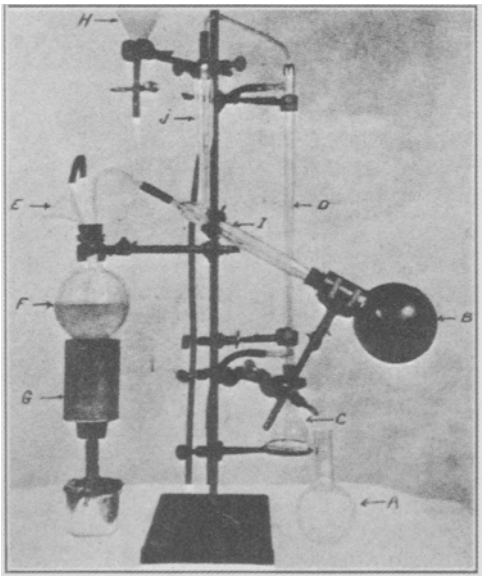
By W. F. ALLEN

THE demand for an accurate, simple, and adaptable method for the micro-determination of nitrogen to be used in connection with caffeine analysis of decaffeinated coffee resulted in the development of the apparatus shown here. It is essentially a micro-Kjeldahl distillation apparatus similar to those of Pregl's, and Parnas and Wagner's[2]. However, it was found to be easier to operate and capable of handling larger samples than the latter. The use of rubber connections was not eliminated, as was accomplished in the apparatus perfected by Kemmerer and Hallett[1], but the apparatus gave very accurate results, and was found to be easily constructed by anyone capable of doing simple glass blowing.

The entire apparatus was made of Pyrex glass. Parts *I* (23.5 cm. long) and *J* (21.0 cm. long) were made from 25 × 200 mm. test tubes. The inside condenser tubing extending over to the top of the safety trap *J* was made of 9-mm. tubing. The condenser is 60 cm. in length.

The method of procedure is as follows: The sample was digested in the 250-cc. Florence flask *A*. This size flask was found capable of handling samples requiring not over 10 cc. of concentrated sulfuric acid in the digestion mixture. With samples containing a large amount of organic matter, it was found to be very convenient to add a few drops of 30 per cent hydrogen peroxide during the last part of the digestion. The cooled digested sample was then diluted with freshly boiled distilled water, care being taken that the flask was not over one-fourth full. The flask was placed in the asbestos-lined jacket *B* and clamped into position as shown. The jacket eliminated the use of a second burner by keeping condensation of steam down to a minimum. However, very satisfactory results were obtained without the use of the jacket by applying heat to flask *A* only during the last 3 or 4 minutes of the distillation.

For samples containing up to 20 mg. of nitrogen, 15 cc. of 4 per cent boric acid[3] and 15 drops of 0.02 per cent methyl red indicator were measured into the 150-cc. evolution flask *C*, which was then placed in position



Apparatus for Micro-Kjeldahl Method

under the condenser *D* with a wide cork resting on top of the flask. Freshly boiled distilled water was next added through funnel *E* into the 500-cc. balloon flask *F* used as a steam generator. Heat was applied to flask *F* by a Bunsen burner *G*, having a shield around the top to prevent air currents from causing variation in the amount of heat applied. Immediately after starting to heat the steam generator, a slight excess of concentrated sodium hydroxide was added through funnel *H* and then washed down with a few cubic centimeters of water. This funnel was immediately closed with a pinch clamp. Distillation was continued at such rate that no bubbling of steam occurred in the receiving flask *C*. After about 125 cc. of liquid had distilled over, in about 12 minutes, the receiving flask was lowered so that the tip of the condenser was about 2 cm. above the surface, and the tip washed down with a small stream of water. Distillation was continued at an increased rate for one minute longer. The pinch clamp on funnel *H* was then released before removing the steam generator to prevent suction. A blank was always

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length of cloth, the data given above indicates that the cake must always be completely covered. Economy in press cloth would require that the lap be very small though the amount of lap will depend largely upon the kind of stripper used.

Even after the cake is properly formed it may be ruined by improper panning. The pan shover should never permit any meal to fall from the pan while he is working nor should the cake be mashed or broken as it is slid into the press.

No one can attempt to set a rule for pressing. Ram speeds, time under low pressure, maximum pressure, and drainage time are all factors which depend entirely upon the character of the seed and the market for products. We do know, however, that the press boxes should be in good condition, not bent or distorted, and the drainage channels kept open. The chokers furnished by the manufacturers of the usual change valves are ground to give good uniform action to the ram in the press and the operator need concern himself only with their proper operation.

Remember that uniformity in the press room is one of the principal factors in securing good results. Maintain a uniform operating schedule, permitting the presses to be under pressure as long as possible, speed the crew up so that the presses are charged rapidly without letting the forming of the cake suffer, and watch the hydraulic system so that every pressing receives the maximum pressure on schedule; if these things are followed religiously, good press room work will become the rule.

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Oil Chemists' Committees

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run using the same reagents and procedure as with the samples, after the apparatus had been thoroughly steamed for at least 15 minutes. Care was taken that the same amount of liquid was contained in each flask to insure the same intensity of color. The contents of the receiving flask *C* from a sample were titrated directly with dilute standard sulfuric acid from a microburet; and the end point was taken when the shade of color of the indicator exactly matched that of the blank. This proved to be a very faint pink at a pH of about 5.7. By matching the blank it was unnecessary to subtract a blank correction from the amount of acid used.

Table I—Comparative Accuracy of Micro-Kjeldahl Method

Samples Analyzed	Nitrogen in Sample	Error with Micro-Kjeldahl	Error with Micro-Kjeldahl
	Mg.	%	%
8	21.114	0.62	0.29
6	1.000		1.20
4	0.583	1.85	0.24
4	0.100		3.00

It can be readily observed that this apparatus and procedure can be used with rapidity and accuracy on samples requiring as much as 10 cc. of cencentrated sulfuric acid in the digestion mixture, as well as samples containing as little as 0.1 mg. of nitrogen. The comparative accuracy of this method with the micro-Kjeldahl method is shown in Table I. By running two sets of apparatus at the same time it was found that 20 samples could easily be analyzed in a half day.

Literature Cited

(1) Kemmerer and Hallett, IND. ENG. CHEM., *19,* 1295 (1927).
(2) Pregl. "Quantitative Organic Microanalysis," pp. 94—104, Blakiston, 1924.
(3) Scales and Harrison, J. IND. ENG. CHEM., *12,* 350 (1920).

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