[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UPPER SCHOOL OF AGRICULTURE OF THE REPUBLIC OF COLOMBIA]

A RAPID METHOD FOR THE DETERMINATION OF ORGANIC NITROGEN

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The alkaline combustion of nitrogenous organic substances with evolution of ammonia can be utilized in a very simple manner for the quantitative determination of organically combined nitrogen. By heating the sample with solid sodium hydroxide and sodium acetate, all the nitrogen is evolved as ammonia in a very short time, the last traces of ammonia that would remain in the tube being expelled by the methane generated when the temperature is raised towards the end of the combustion.

We use a copper test-tube about 17.8 cm. long and 2.5 cm. in diameter, provided with a sound cork fitted with a delivery tube which leads the gases to a flask containing 25 cc. of 0.1 *N* sulfuric acid. The sample is heated in the copper tube with 1 g. of pure sodium hydroxide and 2 g. of crystallized sodium acetate. The heating is performed gently at first, while the water of crystallization of the sodium acetate is expelled, in order to avoid over-boiling of the fused mass; then the tube is strongly heated, and a copious evolution of methane begins, which is indicated by a rapid bubbling in the receiving flask with the discharge of dense white fumes. When the bubbling has ceased, the delivery tube is washed with distilled water into the flask, and the remaining acid is titrated with 0.1 *N* sodium hydroxide solution, using litmus as an indicator. This operation takes about 20 minutes. Blank determinations were made to test the purity of the reagents before the determinations were made for the analytical data given below in comparison with the results obtained by the Kjeldahl method. The operation takes about 30 minutes.

It is seen that for the substances rich in nitrogen the results obtained by

TABLE I

ANALYSES FOR NITROGEN

	Samples	Kjeldahl method, %	Alkaline combustion method, %
1	Wheat flour	2.52	2.59
2	Mixed fertilizer	2.10	2.15
3	Blood fertilizer	12.11	12.38
	Soils		
		0.15	0.15
		.07	.07
		.16	.15
		.16	.18
		.23	.23
		.17	.15

the alkaline combustion method are about 2% of the total nitrogen content higher than those obtained by the Kjeldahl method; this cannot be ascribed to sodium hydroxide particles carried off from the combustion tube, since this would make a great difference in the results for the samples of low nitrogen content; we must suppose, therefore, that there are some leakages in our distilling apparatus, or that one hour and a half of distillation is not enough to expel all of the ammonia from the solution.

For the determination of nitrogen in liquids such as milk or beer, we suggest that a measured volume of the liquid be evaporated to dryness in a shallow porcelain dish, lined inside with tin foil free from holes, in order to facilitate the transfer of the residue to the combustion tube.

Summary

A method for the determination of nitrogen by heating with solid sodium hydroxide and sodium acetate is described. The operation takes about thirty minutes.

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ADDITION REACTIONS OF UNSATURATED ALPHA-KETONIC ACIDS

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The action of sunlight on methyl benzalpyruvate has recently been described.[1] For further study of the effect of light on unsaturated α-ketonic esters, those of anisalpyruvic acid were chosen since it has been shown in this Laboratory[2] in a comparative study of methyl esters of benzoyl- and of anisoylacrylic acids that, whereas the former isomerizes on exposure to the light, the latter polymerizes with extreme ease. It might be expected, therefore, that the introduction of the *p*-methoxyl group into benzalpyruvic ester would increase the tendency toward polymerization already existing. Experiments have shown, however, that exposure of anisalpyruvic acid and its methyl and ethyl esters to the sunlight leads neither to polymerization nor to isomerization.

Since the polymerization of an ethylenic compound to a cyclobutane derivative is an addition reaction, whatever may be its mechanism, it seemed of interest to study the behavior of benzalpyruvic acid and of anisalpyruvic acid toward various addenda. The blocking effect of the *p*-methoxyl group in light polymerization has recently been reported also in the case of dianisalacetone by Stobbe and Färber[3] who quote, as a parallel

[1] Reimer, THIS JOURNAL, **46**, 783 (1924).

[2] Rice, *ibid.*, **45**, 222 (1923); **46**, 214 (1924).

[3] Stobbe and Färber, *Ber.*, **58**, 1548 (1925).