

### Summary

The dissociation constant of ammonia has been determined at intervals of 5° from 0 to 50° by electromotive force measurements of the extent of hydrolysis of the salt formed from ammonia

and the weak acid potassium *p*-phenolsulfonate. The results were in good agreement with those obtained previously by another electromotive force method.

WASHINGTON, D. C.

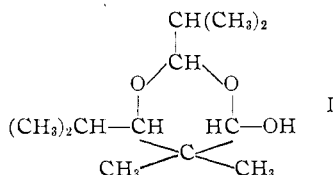
RECEIVED AUGUST 15, 1949

## NOTES

### The Trimer of Isobutyraldehyde

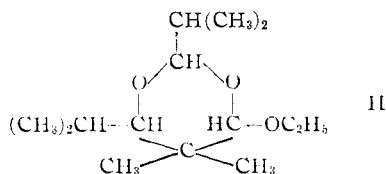
BY ELLIOT R. ALEXANDER AND ELLIOT N. MARVELL<sup>1</sup>

In an earlier communication,<sup>2</sup> Saunders, Murray and Cleveland have pointed out that Raman spectra indicate the formula I for the trimer of



isobutyraldehyde which is usually obtained by attempted aldolization. We have obtained additional chemical evidence in support of this formulation.

When the pure trimer was treated with ethyl orthoformate, a homogeneous product was obtained in good yield which did not react with metallic sodium and which did not discolor potassium permanganate or bromine in carbon tetrachloride solution. On long standing the liquid produced a weak color with a Fuchsin reagent but it gave negative tests with Fehling or Tollens solutions. Infrared analysis indicated that oxygen was present only in the form of ether linkages. These data, together with the correct analysis for carbon, hydrogen, ethoxyl groups, molecular weight and molecular refractivity, are in agreement with the formula II for our product.



### Experimental<sup>3</sup>

**Reaction of the Trimer of Isobutyraldehyde with Ethyl Orthoformate.**—By the procedure used for the reaction of hydroxypivaldehyde with ethyl orthoformate,<sup>4</sup> 48.1 g.

(1) Present address: Oregon State College, Corvallis, Oregon.

(2) Saunders, Murray and Cleveland, *THIS JOURNAL*, **65**, 1714 (1943).

(3) All melting points and boiling points are uncorrected.

(4) Alexander and Marvell, *THIS JOURNAL*, **71**, 15 (1949).

(0.222 mole) of the trimer of isobutyraldehyde<sup>1</sup> (b.p. 80° (1.0 mm.);  $n_D^{20}$  1.4487) was treated with 45.0 g. (0.31 mole) of ethyl orthoformate. After distillation through an eight inch electrically heated column packed with glass helices, the product (42.0 g., 78%) was obtained as a homogeneous colorless liquid, b.p. 61° (1.5 mm.);  $n_D^{20}$  1.4321;  $d_{20}^{20}$  0.9162. This material did not react with metallic sodium, potassium permanganate or bromine in carbon tetrachloride solution. Fehling and Tollens tests were also negative. On long standing, the liquid produced a weak color with fuchsin reagent. Infrared analysis indicated that oxygen was present only in the form of ether linkages.<sup>5</sup> These properties are those to be expected of 2,4-diisopropyl-5,5-dimethyl-6-ethoxy-1,3-dioxane (II).

*Anal.*<sup>6</sup> Calcd. for  $\text{C}_{14}\text{H}_{28}\text{O}_3$ : C, 68.82; H, 11.53; ethoxyl, 18.40; mol. wt., 244;  $M_D^{20}$ , 69.60. Found: C, 68.97; H, 11.56; ethoxyl, 18.59; mol. wt., 238;  $M_D^{20}$ , 69.23.

(5) We are indebted to Dr. Foil A. Miller and Mrs. J. L. Johnson for the determination and interpretation of these spectra.

(6) The microanalyses were performed by Miss Theta Spoor and the Howard Clark Microanalytical Laboratories, Urbana, Illinois.

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RECEIVED OCTOBER 10, 1949

### Preparation of N-Acetyl-DL-amino Acids

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It has been shown that acid hydrolysis of an alkylacetamidomalonic ester gives the amino acid as the end-product of the reaction, whereas hydrolysis with excess sodium hydroxide leads to the formation of the sodium salt of the alkylacetamidomalonic acid.<sup>1,2</sup> In the case of alkylacetamidocynoacetic ester the end-product is the amino acid with either acid or caustic hydrolysis.<sup>2,3</sup> It has now been found that hydrolysis of an alkylacetamidomalonic ester with aqueous sodium carbonate leads directly to the sodium salt of the acetyl-DL-amino acid. The same results are obtained with the alkylacetamidocynoacetic esters although the reaction time is longer. The method should prove generally useful for the preparation of N-acetyl-DL-amino acids. This procedure also affords a means of degrading alkylacetamidoma-

(1) Snyder and Smith, *THIS JOURNAL*, **66**, 350 (1944).

(2) Albertson, *ibid.*, **68**, 450 (1946).

(3) Albertson and Tullar, *ibid.*, **67**, 502 (1945).