

$C_{21}H_{48}O_7$ : C, 69.89; H, 9.08;  $CH_3CO$ , 16.12. Found by Haslewood for hepatol diacetate: C, 71.2; H, 9.2. Found in this work for hepatol diacetate: C, 70.1, 70.2; H, 9.1, 9.2. Found for digitogenin diacetate:  $CH_3CO$ , 16.25.

In view of the evidence presented above it is suggested that the hepatols are decomposition products of digitonin. No further work has been done to determine the nature of the second hepatol, which has the properties of a more contaminated digitogenin. There also seems to be room for some doubt whether 7-hydroxycholesterol is a natural constituent of liver, or rather an autoxidation product of cholesterol.

I wish to thank Dr. E. Fernholz for his interest and advice during this investigation.

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### Preparation of "Siliconyl Alcohol"

BY EDMUND L. NIEDZIELSKI

In the course of an investigation it was found necessary to have larger amounts of pure "siliconyl alcohol" at our disposal. The literature reports only one vague and cumbersome description<sup>1</sup> for its preparation which does not contain either the analysis of the substance in question or data on yields. In order to justify the continued inclusion of this compound in the literature, the following procedure for its successful preparation may be suggested.

As starting material tetraethylsilane<sup>2</sup> was used which was transformed into triethyl-chloroethyl-monosilane according to Friedel and Crafts. Ten grams of the chloro compound was refluxed for three hours with 5 g. of fused potassium acetate and 1.5 g. of acetic acid. The reaction product was poured into excess of water, separated, dried and submitted to fractionation, yielding 3.2 g. of a fraction boiling between 208 and 214° which consisted of triethyl-acetoxyethyl-monosilane (yield 28%). Three and two-tenths grams of triethyl-acetoxyethyl-monosilane was refluxed with 10 cc. of a 22% solution of alcoholic potassium hydroxide. After three hours the product was transferred into excess of water and the isolation procedure given above for the acetate was applied. One and two-tenths grams of siliconyl alcohol (b. p. 190° (uncor.)) was obtained amounting to a yield of 48%. Calcd. for  $C_8H_{20}OSi$ : C, 60.00; H, 12.50. Found: C, 58.26; H, 11.72.

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- (1) C. Friedel and J. M. Crafts, *Ann.*, **138**, 19 (1866).  
(2) S. Sugden and H. Wilkins, *J. Chem. Soc.*, 128 (1931).

### Effect of Aging Cottonseed Meal on the Solubility of the Proteins

BY H. S. OLCOTT AND T. D. FONTAINE

A sample of ethyl ether-extracted cottonseed meal<sup>1</sup> was stored in a closed glass container at room temperature (75–92°F.). At three to five week intervals separate portions were extracted with water and 0.5 *N* sodium chloride solution according to the method previously described<sup>2</sup> for determining relative protein solubilities. Over a period of fifteen months, during which the meal, originally light yellow, acquired a brownish cast, there were no significant changes in solubility. The average percentage of the total nitrogen soluble in water was  $26.5 \pm 0.3$ ; that soluble in 0.5 *N* sodium chloride solution was  $79.1 \pm 0.4$ . These findings are in marked contrast to the decrease in solubility described for the proteins of soy bean meal during storage.<sup>3,4</sup>

(1) The meal used contained 9.8%  $H_2O$ , 9.15% N (dry basis), and 1.9% residual oil (by chloroform extraction).

(2) H. S. Olcott and T. D. Fontaine, *THIS JOURNAL*, **61**, 2037 (1939); **62**, 1334 (1940).

(3) D. B. Jones and C. E. F. Gersdorff, *ibid.*, **60**, 723 (1938).

(4) A. K. Smith and S. J. Circle, *Ind. Eng. Chem.*, **30**, 1414 (1938).

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### Note on the Heats of Dilution of Amino Acids

BY JULIAN M. STURTEVANT

Doehlemann and Lange<sup>1</sup> have pointed out that appreciable heat effects arising from disturbance of the water equilibrium may be encountered at very low concentrations in the dilution of various types of electrolytes. It is important to note that analogous effects should be encountered with amphoteric electrolytes, particularly amino acids.

An aliphatic amino acid at its isoelectric point in aqueous solution is almost entirely in the form of zwitter ions,  $Z^{\pm}$ . In the case of glycine, for example, only about 0.04% is present as positive and negative amino acid ions.<sup>2</sup> A solution of a pure amino acid in pure water is in general not isoelectric, though it becomes very nearly so at sufficiently high concentrations. At infinite dilution the solution is necessarily at *pH* 7, so that in the dilution of a concentrated solution,

(1) Doehlemann and Lange, *Z. physik. Chem.*, **170**, 391 (1934).

(2) Edsall and Blanchard [*THIS JOURNAL*, **55**, 2337 (1933)] have shown that an entirely negligible fraction of the amino acid is in the form  $NH_2RCOOH$ .