

measured is equal to the partial specific volume ( $dV/dg$ ) provided these quantities are independent of concentration. This appears to be true, at least in the case of zein. Less precision was obtained with gliadin than with zein, due in part to the greater fragility of the gliadin pellets and possibly in part to the fact that pressing into pellets caused a partial denaturation and insolubilization of gliadin<sup>6</sup> (of the order of a few tenths of one per cent.).

TABLE I  
SUMMARY OF RESULTS

Preparation <sup>a</sup>	Initial concn.	Final concn.	$\Delta V / \Delta g^b$
Standard Zein (commercial zein not further purified)	0.00	0.41	0.770
	.00	.71	.766
	.00	1.15	.773
	1.15	2.21	.772
	2.21	3.20	.773
			Av. = 0.771 ± 0.002
Washed Standard Zein (commercial zein repeatedly washed by suspension in H <sub>2</sub> O)	0.00	0.83	0.773
	.83	1.59	.771
	1.59	2.49	.771
Fractionated Standard Zein (main component of commercial sample after removal of the most and least soluble material)	0.00	0.50	0.778
	.00	.81	.776
	.81	1.81	.776
	1.81	2.81	.776
Fractionated Standard Zein in 52% ethanol ( $\bar{V}_w = 0.959$ )	0.00	0.67	0.778
	.67	1.54	.773
Laboratory Zein (prepared by low temperature extraction of white corn)	0.00	0.80	0.775
	.80	1.72	.777
Gliadin (Connecticut Agricultural Experiment Station preparation)	0.00	0.63	0.723
	.63	1.31	.723
	1.31	2.03	.733
	2.33	3.11	.718
			Av. = 0.724 ± 0.004

<sup>a</sup> The measurements on zein, except in the two cases indicated, were in 73% aqueous alcohol ( $\bar{V}_w = 0.927$ ). In the case of gliadin the solvent was 62% alcohol ( $\bar{V}_w = 0.944$ ). <sup>b</sup> The precision of the measurements is indicated in terms of mean deviations.

The differences between the various zein preparations are slight and probably due in large part to inorganic impurities which would be expected to cause a decrease in specific volume by electrostriction. The higher value, 0.776, obtained on the more purified preparations is probably closest to the true value for zein. To the authors' knowledge this is the highest value yet reported for any unconjugated protein (most values range from 0.73 to 0.75) and reflects the unusually high proportion of non-polar amino acids in zein. The results also indicate the partial specific volume of zein to be independent of alcohol concentration over the range 52–73%.

The large difference in partial specific volume between zein and gliadin is not unexpected on the basis of their amino acid compositions. Thus using the corrected molal volumes of the amino

(6) This denaturing effect of pressing into pellets was also observed in an experiment with serum albumin.

acid residues as given by Cohn and Edsall<sup>7</sup> together with the amino acid compositions collected by them, one calculates the values 0.75<sub>2</sub> and 0.70<sub>6</sub> for zein and gliadin, respectively, both of which are lower by 3% than the values found experimentally. This might be interpreted as indicating deficiencies in the present amino acid analyses of these proteins.

The dilatometric technique is most applicable to systems involving organic solvents (a large temperature coefficient of expansion and good drainage qualities are essential). Experiments involving water as solvent have proved quite unsatisfactory.

(7) E. J. Cohn and J. T. Edsall, "Proteins, Amino Acids and Peptides," Chaps. 15 and 16, Reinhold Publishing Corporation, New York, N. Y., 1943.

DEPARTMENT OF PHYSICAL CHEMISTRY  
HARVARD MEDICAL SCHOOL

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### Preparation of Chloroacetaldehyde Hydrate

BY LEWIS F. HATCH AND HAROLD E. ALEXANDER<sup>1</sup>

Recently a method has been described<sup>2</sup> for the preparation of alkoxyacetaldehydes by the oxidation of the  $\alpha$ -alkyl ethers of glycerol using periodic acid as the oxidizing agent. This method for the preparation of substituted acetaldehydes has been extended to the preparation of chloroacetaldehyde hydrate by the oxidation of glycerol  $\alpha$ -monochlorohydrin with periodic acid.

#### Experimental

One-half mole of Eastman Kodak Co., "practical" grade glycerol  $\alpha$ -monochlorohydrin was oxidized by 0.50 mole of periodic acid under the same conditions as used for the oxidation of the  $\alpha$ -alkyl ethers of glycerol.<sup>2</sup> Eighteen ml. (25.2 g.) of material boiling at 84° (742 mm.) was obtained. Chloroacetaldehyde and its hydrate are both reported<sup>3</sup> to boil at 85–85.5°.

Refluxing two 3-g. samples of the product for three hours with 20% alcoholic potassium hydroxide gave 35.6% saponifiable chlorine. The theoretical chlorine for  $\text{CH}_2\text{-ClCHO}\cdot\text{H}_2\text{O}$  is 36.8%. This would indicate a purity of 97% and a yield of 50%. This yield can be increased to about 60% by redistilling the material boiling between 84–98° obtained after removal of the material boiling at 84°.

The semicarbazone melted at 133–134° (literature 134°<sup>4</sup>) and decomposed in hot aqueous solutions.

- (1) At present on active duty with the U. S. N. R.
- (2) Hatch and Nesbitt, *THIS JOURNAL*, **67**, 39 (1945).
- (3) Natterer, *Monatsh.*, **3**, 442 (1882).
- (4) Kling, *Compt. rend.*, **148**, 568 (1909).

DEPARTMENT OF CHEMISTRY  
THE UNIVERSITY OF TEXAS  
AUSTIN, TEXAS

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### Derivatives of Ipral, Neonol, Nostal and Sandopal: an Optical Crystallographic Study

BY MARTIN E. HULTQUIST, CHARLES F. POE AND NORMAN F. WITT

In previous studies, the optical properties of ten substituted benzyl esters of barbital and