

The autoclave, a steel shell three-sixteenths of an inch thick by about fifteen inches long and six inches in diameter, failed by splitting open on one side. Before use, it had been tested to 1500 lb. pressure. The detonation was of sufficient violence to blow out all the windows of the laboratory and shake a large concrete building.

The explosion could not have been caused by any mixture of hydrogen and oxygen in the autoclave, as the autoclave had been thoroughly swept out with hydrogen during the reduction of the catalyst. As was indicated by the initial pressure drop, reduction was taking place very satisfactorily. It seems probable that the explosion was caused by decomposition of the nitro compound itself, possibly by auto-oxidation, or possibly by rapid oxidation by the nitro body of an intermediate product of the reduction. This may have been induced by the catalyst, which was evidently quite vigorous, and it may have been aided by local high temperature attained by the rapid reduction. In the original work of Brown, Etzel and Henke, such dangers as these were eliminated by the small scale on which they worked and by the adequate cooling afforded by the oil-bath.

The purpose of this note is to call the attention of other investigators to the dangers of this type of reduction when carried out on other than a very small scale.

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#### THE RAMAN SPECTRA OF FORMALDEHYDE, TRIOXYMETHYLENE, ETHYLENE GLYCOL, AND OF SOME VISCOUS LIQUIDS

*Sir:*

The Raman spectrum of a 37% solution of formaldehyde in water consists of at least nine lines corresponding to frequencies, expressed in wave numbers of 802, 909, 1055, 1285, 1479, 2840, 2915, 3015 and 3087. Trioxymethylene gives modified lines corresponding to 497, 579, 674, 908, 1050, 1232, 1285, 1376, 1477 and 1527 wave numbers. Ethylene glycol gives lines corresponding to 341, 491, 526, 869, 1040, 1274, 1460, 2725, 2874, 2948, 3048 and 3131 wave numbers. Except for a fairly consistent shift toward the higher frequencies, of about 25 wave numbers, the stronger Raman lines from the aldehyde solution correspond both in intensity and in distribution to those from ethylene glycol. No lines are found typical of the C=O linkage (1675-1725) for formaldehyde. On the other hand, the line at 1050 typical of C-O is fairly strong. These observations would seem to indicate the formation of methylene glycol by formaldehyde in aqueous solution. This is compatible with the observations of Auerbach and Barschall [*Chem. Zentr.*, II, 1081 (1905)] and Walker [*J. Phys. Chem.*,

35, 1104 (1931)], who came to the same conclusion from cryoscopic and vapor pressure measurements. The absence of frequencies less than 800 wave numbers indicates that the concentration of higher polymers in a solution of this concentration must be small. The absence of a modified line in the 1675–1725 region for the trioxymethylene and the presence of a line corresponding to the C–O linkage may be due to the formation of a trimer in the manner suggested by Venkateswaren and Bhagavantam [*Proc. Roy. Soc. (London)*, **128**, 260 (1930)] for paraldehyde. The presence of only three frequencies below 900 wave numbers may indicate that the polymer consists of not more than three aldehyde units. Lines corresponding to C–H linkage for the trioxymethylene are very weak and as yet have not been definitely identified.

That strong continuous Raman spectra may be a property common to viscous liquids has received some substantiation from the Raman spectra of sodium silicate, orthophosphoric acid, potassium hydrogen phosphite and ethylene glycol solutions. The continuous spectra decrease on dilution and on temperature elevation. This behavior is similar to that observed with glycerin by Venkateswaren [*Indian Journal Phys.*, **3**, 105 (1928)]. This phenomenon may possibly owe its origin to intermolecular attraction, the continuous spectra arising from the probability of a large number of energy levels. The Raman spectrum of  $\alpha$ -chloronaphthalene consists of 16 lines and shows some similarities to that of chlorobenzene and naphthalene.

A detailed account of these experiments will be communicated later.

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#### THE IONIZATION CONSTANT OF ACETIC ACID

*Sir:*

We have obtained a precise value of the ionization constant of acetic acid at 25° from the results of new conductance measurements, made in this Laboratory, on acetic acid, hydrochloric acid, sodium chloride and sodium acetate at low concentrations (the lowest in each case being about 0.00003 *N*). The principles underlying the computations are essentially those which have been outlined by one of the undersigned [MacInnes, *This Journal*, **48**, 2068 (1926)]. The calculations take account of the change of the mobilities of the ions as required by the Debye–Hückel–Onsager theory and of the ion activities as predicted by the Debye–Hückel theory. The computations, which involve a short series of approximations, consist in estimating the proportion of ions at each acetic acid concentration by comparing the measured equivalent conductance with that