ments the ϵ-phase is produced throughout the greater part of the range, and is apparently a metastable phase at least through the approximate range 46–66% of Cd. This is consistent with the behavior of some pure metals, which are known to be electrodeposited in metastable forms.

It is important to know how the conditions of deposition affect the crystal structure of an alloy of any given composition, since hardness and other physical properties depend upon the structure.

We are at present investigating (1) the effect of current density and temperature on crystal structure, the composition of the deposit remaining constant; (2) the recrystallization temperatures of Cd–Ag alloys.

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EXPLOSION DURING THE CATALYTIC REDUCTION OF NITROANISOLE IN THE LIQUID PHASE

Sir:

Brown, Etzel and Henke [*J. Phys. Chem.*, **32**, 631–635 (1928)] quantitatively reduced a number of nitro compounds in the liquid phase at about 215°, under a hydrogen pressure of about 500 lb. per sq. in., using a nickel catalyst. The reduction could be made either without a solvent or with benzene and ethyl alcohol. Their work was done on small quantities (about 5 g.) of material, in a shaking autoclave maintained at the desired temperature by immersion in an oil-bath.

The writer attempted to apply this method to the reduction of *o*-nitroanisole. As no danger was anticipated, the work was done on a *technical* scale, using 400 g. of the nitro compound. A shaking autoclave of steel was used, of about 3000 cc. total capacity, heated electrically. The first experiment was made using benzene as a solvent, with a nickel catalyst, and gave approximately a 50% yield of amine. In the next, the solvent was omitted and the catalyst was carefully prepared to give a very active product; 250 g. of nickel nitrate was ignited at 400°, and then heated for three hours in a muffle at 560°. The oxide was reduced in the autoclave under a stream of hydrogen at a temperature of 280–300°. Four hundred grams of the nitroanisole, freshly distilled, was then added and hydrogen was passed in to a pressure of 500 lb. At a temperature of 250°, with shaking, the hydrogen was rapidly taken up, and the pressure dropped in about fifteen minutes to 250 lb. It then began to rise rapidly, and reached a total of 500 lb. in about five minutes. In thirty seconds more the pressure jumped to 1000 lb. The writer then opened the needle valve on the autoclave in the hope of relieving the pressure. Gas and liquid shot out of the valve, and an instant later the autoclave exploded with great violence.

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.

MONSANTO CHEMICAL WORKS
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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.*,