

SHORT COMMUNICATIONS

New Method for the Study of Melamine Resin
(New Solvents for Melamine Resin)

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Melamine is slightly soluble in water, but the melamine-formaldehyde condensate is almost insoluble in water. All previous works¹⁾ on melamine resin have been carried out in aqueous media, and so the reaction mechanism of an early stage could be known because a small amount of the condensate is soluble (the solution soon becomes turbid), but as the amount of condensate increases it cannot be made clear because of the insolubility of the condensate.

For this reason, solvents other than water were tested. Melamine is soluble only with difficulty in organic solvents, but it is soluble in liquid ammonia, ethylene glycol, and the derivatives as is well known; however, ethylene glycol cannot be used as a solvent, because the iodometry (used to determine the amount of condensate) cannot be applied in the presence of a small amount of ethylene glycol. Dimethylsulfoxide (DMSO), *N*-methyl pyrrolidone, hexamethyl phosphoramide, and ϵ -caprolactam were found by the author to be new solvents for melamine, guanamine, and their hydroxymethylated products, as well as their condensates. Among these, it was found that a condensate containing a high proportion of condensation products (the latter stage) is dissolved in DMSO and that the DMSO does not affect the iodometry.* Therefore, the condensation mechanism between melamine and formaldehyde from the early to the latter stages can be studied by the use of DMSO as a solvent. The solubilities of the above substances in DMSO have been determined the results being

summarized in Table 1; hydroxymethylated products were prepared and analyzed by the usual method.²⁾

TABLE 1. SOLUBILITY IN DMSO AT 30°C

Compound	Solubility (g/100 ml)
Melamine	6
Cyclohexylmelamine	12
Benzoguanamine	51
Acetoguanamine	6
Formoguanamine	6
Phenylacetoguanamine	21
Dimethylolmelamine (M1.98F* ¹)	56
Trimethylolmelamine (M2.68F* ²)	62
Hexamethylolmelamine (M5.51F* ³)	45
Dimethylolacetoguanamine (A. G. 2.20F* ⁴)	35

*¹ formaldehyde/melamine=1.98 (mole ratio)

*² formaldehyde/melamine=2.68 (mole ratio)

*³ formaldehyde/melamine=5.51 (mole ratio)

*⁴ formaldehyde/acetoguanamine=2.20 (mole ratio)

In an aqueous solution, iodine is firmly adsorbed by the water-insoluble melamine resin (condensate) and the iodine cannot react apply iodometry with thiosulfate-solution; hence it is difficult to in this case. However, iodometry by the addition of DMSO to an aliquot solution can be used to determine the amount of condensate, because, as has been mentioned above, the condensate is easily dissolved in DMSO and DMSO does not react with the reagents used in this iodometry.

1) H. P. Wohnsiedler, *Ind. Eng. Chem.*, **44**, 2679 (1952); M. Okano and Y. Ogata, *J. Am. Chem. Soc.*, **74**, 5728 (1952); K. Koeda, *Kogyo Kagaku Zasshi (J. Chem. Soc. Japan, Ind. Chem. Sect.)*, **60**, 1567 (1957).

2) J. Kitagawa, *ibid.*, **53**, 181 (1950); N. Yoshimi, S. Yamao and M. Tanaka, *ibid.*, **66**, 279 (1963).

* Neither ethylene glycol nor DMSO affects the sulfite method (used to determine the amount of formaldehyde consumed).