NN-DIMETHYLMELAMINE

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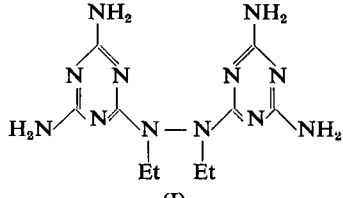
The previously unidentified product of the reaction between 2-chloro-4:6-diamino-*s*-triazine and *NNN'N'*-tetraethyldecamethylene diamine in dimethylformamide solution has been shown to be *NN*-dimethylmelamine.

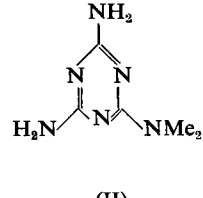
IN a previous communication[1] it was recorded that prolonged boiling under reflux of a solution of 2-chloro-4:6-diamino-*s*-triazine and *NNN'N'*-tetraethyldecamethylene diamine in dimethylformamide yielded an unidentified crystalline product containing no halogen.

The analytical figures for this material corresponded reasonably well with those required for *NN'*-diethyl-*NN'*-bis (4:6-diamino-*s*-triazin-2-yl) hydrazine (I) [Found: C, 39·2; H, 6·4; N, 54·5. $C_{10}H_{18}N_{12}$ requires C, 39·2; H, 5·9; N, 54·9 per cent] and this structure appeared to be confirmed when the same product was obtained by the prolonged boiling under reflux of a mixture of 2-chloro-4:6-diamino-*s*-triazine, *NN'*-diethylhydrazine[2], potassium carbonate and dimethylformamide. Since, however, the reaction was so unexpected, and moreover the hydrogen analysis was invariably found to be somewhat high, a further explanation was sought.

Re-examination of the analysis figures showed them to correspond even more closely with those required for *NN*-dimethylmelamine (II)



(I)



(II)

[Calc. for $C_5H_{10}N_6$: C, 39·0; H, 6·55; N, 54·55 per cent]. An authentic specimen of this material was therefore prepared by treatment of 2-chloro-4:6-diamino-*s*-triazine with dimethylamine in the presence of aqueous alkali (*cf.* general method of Kaiser and others[3]) and was found to be identical with the original product.

Prolonged refluxing of a simple solution of 2-chloro-4:6-diamino-*s*-triazine in dimethylformamide also yielded the same material, although in only approximately 5 per cent yield, whereas the best yield in the original experiments was approximately 30 per cent. It appears probable therefore that the original formation of *NN*-dimethylmelamine was due to decomposition of the solvent, dimethylformamide, particularly in the presence of bases, and reaction of the liberated dimethylamine with 2-chloro-4:6-diamino-*s*-triazine.

NN-Dimethylmelamine resists quaternisation; thus, when a methanolic solution was left with methyl iodide at room temperature for four months, the bulk of the starting material was recovered unchanged. When a 1 per cent methanolic solution of the base was heated with either methyl iodide or decamethylene di-iodide at 110–120° under pressure for 60 hours, the only product isolated was the hydriodide of the base.

Experimental

NN-*Dimethylmelamine:* To a slurry of 2-chloro-4:6-diamino-*s*-triazine (1·455 g., 1 mol.) in water (10 ml.) was added dimethylamine (2·7 g. 33 per cent w/w in ethanol, i.e. 100 per cent excess) together with one drop of phenolphthalein solution. The temperature was slowly raised until the mixture refluxed, and maintained here for 3 hours. Meanwhile the reaction mixture was kept just alkaline by adding 5 per cent sodium carbonate solution as required. After cooling, the reaction product was filtered, washed thoroughly with water, dried and recrystallised from ethanol. Rapid crystallisation gave colourless octahedral crystals, whilst slow crystallisation gave branched, fan-like needle aggregates of octahedra. The product (1·15 g., 75 per cent) had m.p. 306–307° (lit[4]. 307–308°), unaltered by admixture with the material originally described[1]. The *hydriodide* separated from ethanol as colourless glistening plates, m.p. 274–5° (decomp.) [Found: C, 21·5; H, 3·8; N, 29·9; I, 44·7. $C_5H_{11}N_6I$ requires C, 21·3; H, 3·9; N, 29·8; I, 45·0 per cent].

References

1. Austin, Lunts, Potter and Taylor, *J. Pharm. Pharmacol.*, 1959, **11**, 80.
2. Renaud and Leitch, *Canad. J. Chem.*, 1954, **32**, 545.
3. Kaiser, Thurston, Dudley, Schaefer, Hechenbleikner and Holm-Hansen, *J. Amer. chem. Soc.*, 1951, **73**, 2984.
4. U.S. Patent 2,567,847. American Cyanamid Company.