$2-z$; the O(3)···O(1) distance is 2·82 Å and the angle O(3)—H(O3)···O(1) equals 164·2°. These 'dimers', 3·24 Å apart, are stacked on each other 3·47 Å along the **b** axis (Figs. 3 and 4). The H attached to N(2) is not involved in hydrogen bonding, its closest contacts being 3·48 Å to C(7a) of the hydrogen-bonded molecule and 3·10 Å to H(O3) of the molecule translated along the z axis. The closest contacts are between the five-membered portions of the bonded molecules, C(1)···C(1) = 3·257, C(1)···N(2) = 3·354 plus C(5)···C(5)($\bar{x}, \bar{y}, 1-z$) = 3·338 Å.

The authors wish to thank Dr André Boime, who supplied the original sample, Dr R. Parthasarathy for discussions, and Mrs N. Winiewicz for excellent technical assistance.

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Acta Cryst. (1978). B**34**, 2061–2063

Hexamethylenetetramine–*m*-Cresol (1:2)

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(*Received* 26 *January* 1978; *accepted* 21 *February* 1978)

Abstract. $(CH_2)_6N_4.2m\text{-}CH_3C_6H_4OH$, orthorhombic, space group *Ccc*2, a = 10·63 (1), b = 26·33 (2), c = 7·199 (5) Å; Z = 4, D_m = 1·19 (1) by flotation in hexane/CCl_4, D_x = 1·175 g cm^{-3}. The structure was determined from film data and refined to R = 0·109. The hexamethylenetetramine molecule occupies a site of symmetry 2, and is linked to two *m*-cresol molecules by N···H—O hydrogen bonds.

Introduction. Hexamethylenetetramine (HMT) forms a variety of crystalline complexes with phenol and substituted phenols (Tse, Wong & Mak, 1977; Mak, Tse, Chong & Mok, 1977). As part of our investigation on hydrogen-bonded molecular compounds of HMT, we have carried out an X-ray analysis of its 1:2 adduct with *m*-cresol to determine the nature of the molecular association.

Colourless elongated prisms of the adduct were prepared as described by Tse, Wong & Mak (1977). As the compound slowly crumbles into powder in air, specimens for diffraction study were sealed in Lindemann-glass capillaries of mean diameter 0·5 mm. Even with this precaution, the crystals deteriorated appreciably upon irradiation by X-rays, judged from the appearance of streaks and the general increase in background on the photographs. Cell dimensions were determined from high-angle reflections on all three zero-layer Weissenberg photographs calibrated with superimposed NaCl powder lines and refined by minimizing the sum of the residuals $|\sin^2\theta_m - \sin^2\theta_c|$. Seven different crystals were used to record the intensities of reflections 0*kl*–9*kl* and *hk*0–*hk*7 by the multiple-film equi-inclination Weissenberg method with Cu $K\alpha$ radiation (λ = 1·5418 Å). The intensities were estimated visually by comparison with a calibrated strip. They were corrected for Lorentz and polarization factors but not for absorption (μ = 6·29 cm^{-1}). A set of scaled structure amplitudes was derived by least squares (Hamilton, Rollett & Sparks, 1965). The data after reduction consisted of 573 independent reflections of measurable amplitude and 393 unobserved reflections.

Of the two space groups, *Ccc*2 and *Cccm*, which are consistent with the systematic absences, the latter was considered unlikely from packing considerations. The correct structure was deduced from model building in space group *Ccc*2. In view of the unfavourable data-to-parameter ratio, least-squares refinement was carried out by treating the *m*-cresol molecule as a rigid group (Doedens, 1970). The phenyl ring was taken as a regular hexagon of side 1·390 Å, the C—OH and C—CH_3 bonds were assigned lengths of 1·390 and

1·500 Å respectively, and all bond angles were fixed at 120°. Atoms of the HMT molecule were refined anisotropically, and the *m*-cresol molecule was varied as a rigid group with individual-atom isotropic temperature factors. Thus, the total number of parameters, including a scale factor, was 60, as opposed to the 118 parameters which would have been required for individual-atom anisotropic refinement. The positions of all the methylene and aromatic H atoms were generated with the program *GHMC* (Mak, Mok & Tse, 1977). Inclusion of these H atoms with $B = 6·5$ Å^2 gave a final R of 0·109 for 573 observed reflections. A calculated difference map was featureless and did not reveal the H atom involved in hydrogen-bonding.

Table 1. *Atomic and rigid-group positional parameters (e.s.d.'s in parentheses)*

The internal coordinate axial system X,Y,Z for the *m*-cresol molecule is defined as follows. The origin is at the centre of the phenyl ring; the $+X$ direction is the vector from C(8) through C(5) to O(1); the $+Y$ direction is the vector from the midpoint of C(9)—C(10) to the midpoint of C(6)—C(7); and Z is the vector product $X \times Y$. For definition of the three angular parameters φ, θ and ρ, see Doedens (1970). The fractional coordinates of the group origin are denoted by x_g, y_g and z_g.

	x	y	z
Hexamethylenetetramine			
C(1)*	¼	¼	0·237
C(2)	¼	¼	−0·236 (2)
C(3)	0·1187 (10)	0·2128 (4)	0·004 (2)
C(4)	0·3434 (10)	0·1976 (4)	−0·003 (2)
N(1)	0·2306 (8)	0·2045 (3)	0·122 (2)
N(2)	0·3642 (9)	0·2420 (3)	−0·122 (2)
m-Cresol [$x_g = 0·2770$ (4), $y_g = 0·0856$ (2), $z_g = 0·639$ (2), $\varphi = -79·8$ (2)°, $\theta = 145·1$ (2)°, $\rho = -129·8$ (2)°]			
O(1)	0·1342 (5)	0·1439 (2)	0·395 (2)
C(5)	0·2056 (4)	0·1148 (2)	0·517 (2)
C(6)	0·1499 (4)	0·0935 (2)	0·673 (2)
C(7)	0·2213 (5)	0·0644 (2)	0·795 (2)
C(8)	0·3484 (5)	0·0564 (3)	0·760 (2)
C(9)	0·4041 (4)	0·0776 (3)	0·604 (2)
C(10)	0·3327 (4)	0·1068 (2)	0·482 (2)
C(11)	0·1611 (7)	0·0415 (4)	0·964 (2)

* C(1) was held stationary in the refinement.

Scattering factors for non-hydrogen atoms were taken from *International Tables for X-ray Crystallography* (1974), and that for H from Stewart, Davidson & Simpson (1965). Structure factor calculations and least-squares refinement were carried out with *NUCLS*4 (Ibers, 1969), a highly modified version of *ORFLS* (Busing, Martin & Levy, 1962) permitting the refinement of rigid groups. The function minimized was $\sum w(|F_o| - |F_c|)^2$, the weighting scheme being that of Cruickshank (1961) with $w = (2A + |F_o| + 2|F_o|^2/B)^{-1}$, where A and B were taken as 3·0 and 300·0 respectively. All computations were performed on an ICL-1904A system in the Hong Kong Universities and Polytechnic Computing Center.

The final atomic parameters are listed in Table 1.* The numbering of the atoms is shown in Fig. 1. Bond distances and angles are listed in Table 2. A *PLUTO* (Motherwell, 1975) stereoplot of the molecular packing viewed along **a** is shown in Fig. 2.

Discussion. The HMT molecule occupies a site of symmetry 2, with the crystallographic diad axis passing through C(1) and C(2). It is linked to a pair of *m*-cresol molecules by N(1)···H—O(1) hydrogen bonds (Fig. 1). The resulting winged 1:2 molecular aggregate is reminiscent of a similar one in crystalline HMT.$2(NH_2)_2CS$ (Mak, Lau, Ladd & Povey, 1978), except that in the latter the molecular components are further connected by N···H—N and S···H—N hydro-

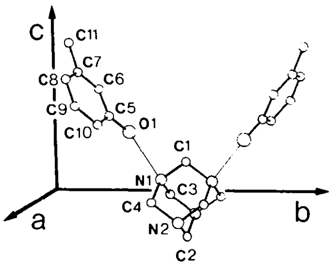

Fig. 1. Atom numbering in HMT–*m*-cresol (1:2).

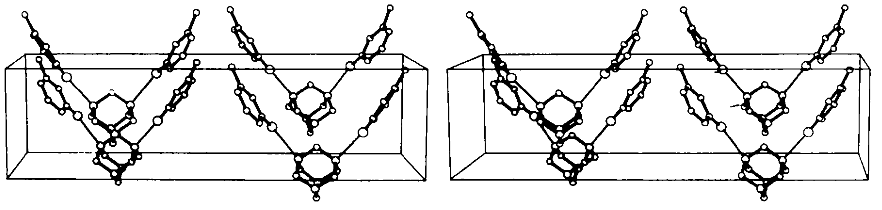

Fig. 2. A stereodrawing showing the molecular packing viewed approximately along **a** towards the origin of the unit cell at the lower left corner.

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* Lists of structure factors, anisotropic temperature factors and generated hydrogen positions, have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33423 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

The primed and unprimed atoms are related by a crystallographic diad axis.

C(1)–N(1)	1·48 (1)	C(1)–N(1)–C(3)	108·4 (7)
C(2)–N(2)	1·48 (1)	C(1)–N(1)–C(4)	108·8 (7)
C(3)–N(1)	1·47 (2)	C(3)–N(1)–C(4)	108·8 (11)
C(3)–N(2)′	1·51 (2)	C(2)–N(2)–C(3)′	108·7 (7)
C(4)–N(1)	1·51 (2)	C(2)–N(2)–C(4)	108·3 (8)
C(4)–N(2)	1·47 (2)	C(4)–N(2)–C(3)′	107·2 (12)
N(1)···O(1)	2·74 (2)	N(1)–C(1)–N(1)′	111·0 (10)
		N(2)–C(2)–N(2)′	112·7 (14)
		N(1)–C(3)–N(2)	111·3 (9)
		N(1)–C(4)–N(2)	111·8 (9)

gen bonds to give a three-dimensional network. The fact that the present adduct slowly decomposes upon exposure to air is explicable in terms of the lack of extensive hydrogen-bonding in the crystal (Fig. 2). 1:2 adducts of HMT with other substituted phenols (*e.g. p*-chlorophenol and *p*-bromophenol) are also relatively unstable; presumably in each case the basic structural unit is a 1:2 hydrogen-bonded molecular complex corresponding to the stoichiometric formula.

The measured dimensions of HMT in the present adduct (Table 2) are in good agreement with those in crystalline HMT (Becka & Cruickshank, 1963). The N(1)···H–O(1) bond of 2·74 (2) Å is normal and there are no unusual van der Waals contacts.

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Acta Cryst. (1978). B**34**, 2063–2065

Sendanolactone, a New Triterpenoid from *Melia azedarach* L. var. *japonica* Makino

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(*Received* 5 *December* 1977; *accepted* 9 *January* 1978)

Abstract. $C_{30}H_{42}O_4$, 3,6-dioxo-13α,14β,17α-lanosta-7,24-dien-21,16β-olide, orthorhombic, $P2_12_12_1$, $a = 30{\cdot}325\ (2)$, $b = 12{\cdot}121\ (1)$, $c = 7{\cdot}109\ (1)$ Å, $Z = 4$, $D_x = 1{\cdot}19$, $D_m = 1{\cdot}18$ g cm^{-3}. The structure was solved by the direct method and refined by a block-diagonal least-squares technique to $R = 0{\cdot}075$ for 1666 reflexions. Rings *A*, *C* and *D* are in the chair, boat and envelope conformations respectively.

Introduction. Crystals were obtained from a methanol solution. The systematic absences are *h*00 when *h* is odd, 0*k*0 when *k* is odd, and 00*l* when *l* is odd. Intensities were collected by the θ–2θ scan technique on a Hilger & Watts Y-290 diffractometer with Zr-filtered Mo *K*α radiation and a crystal of dimensions 0·2 × 0·2 × 0·3 mm. 1998 independent reflexions with $\theta \leq 22{\cdot}5°$ were corrected for Lorentz and polarization effects, but not for absorption. The structure was solved by the use of the program *MULTAN* (Main, Germain & Woolfson, 1970) with local modifications on a FACOM 270-30 computer. After anisotropic refinement of the non-hydrogen atoms by the block-diagonal