very similar features. The corresponding structures of several monohomologation products that were synthesized *via* the boron-trifluoride-promoted reaction of EDA (1 equivalent) with several 1-substitued pentacyclo[5.5.0.0^{2,6}.0^{3,11}.0^{5,10}]dodecane-8,12-diones (5–8) have also been reported (Marchand, Annapurna, Reddy, Watson & Nagl, 1989). Interestingly, the regiochemistry of the ring homologation that resulted in the formation of (5–8) is the *opposite* of that observed in the case of (3a) and of (4).

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Structure of 1a,2,2a,3,3a,4,4a,5a,6,6a,7,7a,8,8a-Tetradecahydro-2,8:4,6-dimethanoanthra[2,3-*b*:6,7-*b'*]bisoxirene-3,7-diol Monohydrate

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Abstract. $C_{16}H_{20}O_4.H_2O$, $M_r = 294.34$, m.p. 422–424 K, monoclinic, Cc, $a = 16.100$ (2), $b = 7.198$ (1), $c = 12.858$ (2) Å, $\beta = 105.26$ (2)°, $V = 1437.59$ Å^3, $Z = 4$, $D_x = 1.36$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, μ(Cu $K\alpha$) = 0.735 mm^{-1}, $F(000) = 632$, $T = 297$ K, $R = 0.048$ and $wR = 0.054$ for 1294 unique reflections. The overall stereochemistry of the title compound is found to be *endo, cis, anti, cis, endo* with the orientation of the epoxy groups being *exo, exo*. All the six-membered rings assume boat conformation with varying degrees of distortion. The crystal packing is stabilized by both hydrogen bonds and van der Waals interactions.

Introduction. Polycyclic polyfunctional molecules are found to have potential use in the polymer industry. They are used as curing agents for polyurethanes containing isocyanates (NCO) as terminal groups. Urethane systems are useful in the fabrication of polymeric materials ranging from elastoplastics to rigid foams. Polyurethanes have the advantage that no by-products are formed in their production. By the combination of polyaddition followed by polycondensation, polyurethanes can be used to generate thermally stable polymers. Here we report the crystal structure of the title compound, $C_{16}H_{20}O_4.H_2O$, which belongs to the above class of polycyclic polyfunctional compounds. The synthesis of the title compound was achieved by Ramadas and Bakthavatchalam (Bakthavatchalam, 1987) and will be reported elsewhere.

Experimental. Rectangular plate-like transparent crystals, $0.35 \times 0.25 \times 0.15$ mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation, unit-cell parameters by least-squares fit of 25 reflections in the range $30 < \theta < 50°$, $\omega/2\theta$ scan technique, 1357 independent reflections in the range $2 < \theta < 70°$, *hkl* range *h*: $0 \rightarrow 19$, *k*: $0 \rightarrow 8$ and *l*: $-15 \rightarrow 14$, 1294 reflections with $I \geq 3\sigma(I)$ considered observed, two standard reflections (20$\bar{6}$ and 112) measured every hour did not show any signifi-

cant change in intensity during data collection, Lorentz and polarization corrections applied, no absorption or extinction corrections, space group Cc or $C2/c$ from systematic absences (hkl, $h + k$ odd and $h0l$, l odd), the former being confirmed by intensity statistics and subsequent satisfactory refinement of the structure. Structure solved by direct methods using multisolution technique, *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976) with anisotropic temperature factors for non-hydrogen atoms, H atoms located from difference Fourier maps and refined isotropically; the thermal parameters of five of the hydrogens were fixed at 0.05 Å^2 during refinement. Refinement converged to a final index of 0.048 and $wR = 0.054$, the weighting scheme used was $w = [\sigma^2(F_o) + 0.0005|F_o|^2]^{-1}$. $(\Delta/\sigma)_{max}$ in the final cycle of refinement for non-hydrogen atoms was 0.028 and the reflection/parameter ratio was 6.9, maximum and minimum heights in the final difference Fourier map were 0.20 and -0.23 e Å^{-3} respectively. Atomic scattering factors for non-hydrogen atoms from Cromer & Mann (1968) and for hydrogen atoms from Stewart, Davidson & Simpson (1965), anomalous-dispersion correction from Cromer & Liberman (1970).

Discussion. A plot of the molecule showing the numbering scheme is shown in Fig. 1. The packing of the molecules in the unit cell (Motherwell, 1978) is shown in Fig. 2. Table 1 gives the fractional atomic coordinates of the non-hydrogen atoms and their equivalent isotropic thermal parameters. Table 2 shows the bond lengths and bond angles involving non-hydrogen atoms.*

The intramolecular features, namely the bond lengths and bond angles, are normal when compared with the crystal structure of 1,4,4a,5,8,8a,9a,10a-octahydro-1,4:5,8-dimethano-9,10-anthraquinone (1) (Brown, Bruce, Hudson & Mills, 1974). The $C(sp^3)$—$C(sp^3)$ bond distances in the central ring vary from 1.513 (6) to 1.586 (5) Å in the title compound with an average of 1.539 (5) Å. The C—C bonds at the ring junctions, $C(2a)$—$C(7a)$ and $C(3a)$—$C(6a)$, are the longest $[1.586$ (5) and 1.573 (4) Å]. Longer bond lengths at ring junctions are reported in (1) $[1.552$ (4) and 1.562 (4) Å]. Long bond lengths are observed for the bonds $C(2a)$—$C(2)$, $C(7a)$—$C(8)$, $C(6)$—$C(6a)$ and

$C(3a)$—$C(4)$ formed by additional reactions $[1.552$ (4) to 1.568 (7) Å].

A similar trend is observed for the corresponding bonds in (1) $[1.551$ (5) to 1.576 (5) Å]. This lengthening of bonds may be due to the influence of the methylene bridge. The bond angles in the central ring have deviations from the ideal tetrahedral angle of $109.5°$ in the range $3-6°$. The angles at the bridging methylene groups $C(9)$ and $C(10)$ $[94.1$ (4) and $95.0°]$ are in close agreement with the value 93.5 (3)°
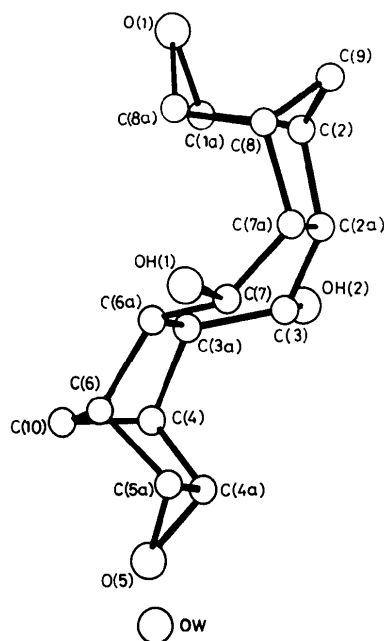

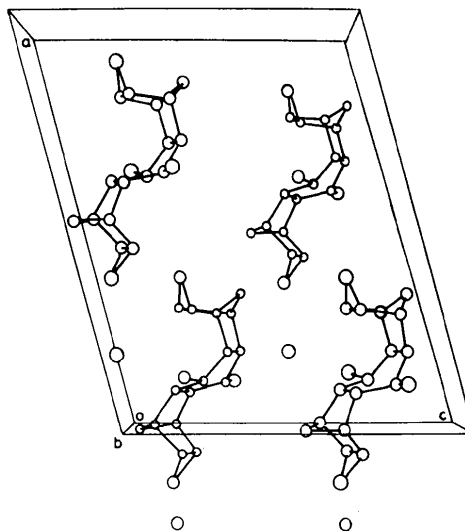Fig. 1. Plot of the molecule showing atom-numbering scheme.

Fig. 2. Packing of the molecules in the unit cell.

* Lists of calculated and observed structure factors, anisotropic thermal parameters of non-H atoms, H-atom parameters, torsion angles and equations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51942 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates for non-hydrogen atoms with e.s.d.'s in parentheses

$$U_{eq}(\text{Å}^2 \times 10^3) = (1/3)\sin^2\beta[U_{11} + U_{22}\sin^2\beta + U_{33} + 2U_{13}\cos\beta].$$

	x	y	z	U_{eq}
C(1a)	0·7931 (3)	−0·1261 (7)	0·7807 (5)	72 (3)
C(2)	0·7816 (3)	−0·1289 (6)	0·8947 (5)	70 (2)
C(2a)	0·6885 (3)	−0·0600 (5)	0·8916 (4)	52 (2)
C(3)	0·6103 (3)	−0·1634 (4)	0·8263 (4)	46 (2)
C(3a)	0·5913 (3)	−0·1423 (4)	0·7048 (3)	44 (2)
C(4)	0·5008 (3)	−0·2046 (5)	0·6366 (4)	61 (2)
C(4a)	0·4354 (3)	−0·0957 (7)	0·6766 (5)	73 (3)
C(5a)	0·4402 (3)	0·0936 (7)	0·6390 (5)	77 (3)
C(6)	0·5100 (3)	0·0882 (6)	0·5800 (4)	60 (2)
C(6a)	0·5968	0·0642 (4)	0·6670	46 (2)
C(7)	0·6169 (3)	0·2017 (4)	0·7606 (4)	50 (2)
C(7a)	0·6911 (3)	0·1506 (4)	0·8564 (4)	54 (2)
C(8)	0·7859 (4)	0·1720 (6)	0·8515 (5)	68 (3)
C(8a)	0·7971 (3)	0·0676 (7)	0·7524 (4)	68 (3)
C(9)	0·8323 (4)	0·0447 (8)	0·9437 (5)	82 (3)
C(10)	0·4975 (3)	−0·1042 (6)	0·5305 (4)	64 (2)
OH(1)	0·6304 (3)	0·3774 (4)	0·7156 (3)	77 (2)
OH(2)	0·6147 (3)	−0·3579 (3)	0·8492 (3)	66 (1)
O(1)	0·8737 (3)	−0·0451 (6)	0·7725 (4)	92 (3)
O(5)	0·3658 (3)	−0·0168 (6)	0·5919 (4)	97 (3)
OW	0·1863 (3)	−0·0140 (4)	0·5478 (3)	66 (2)

Table 2. Bond lengths (Å) and bond angles (°) involving non-hydrogen atoms

C(1a)—C(2)	1·525 (9)	C(1a)—C(8a)	1·447 (7)
C(2)—C(2a)	1·568 (7)	C(2a)—C(3)	1·513 (6)
C(2a)—C(7a)	1·586 (5)	C(3)—C(3a)	1·518 (6)
C(3a)—C(4)	1·556 (6)	C(3a)—C(6a)	1·573 (4)
C(4)—C4(a)	1·508 (8)	C(4a)—C(5a)	1·455 (7)
C(5a)—C(6)	1·513 (8)	C(6)—C(6a)	1·552 (4)
C(6a)—C(7)	1·526 (5)	C(7)—C(7a)	1·518 (6)
C(7a)—C(8)	1·552 (8)	C(8)—C(8a)	1·531 (8)
C(2)—C(9)	1·534 (7)	C(8)—C(9)	1·529 (8)
C(4)—C(10)	1·533 (7)	C(6)—C(10)	1·515 (6)
O(1)—C(1a)	1·452 (7)	O(1)—C(8a)	1·442 (7)
O(5)—C(4a)	1·457 (7)	O(5)—C(5a)	1·432 (6)
C(3)—OH(2)	1·429 (4)	C(7)—OH(1)	1·431 (5)
C(8a)—C(1a)—C(2)	106·2 (4)	C(1a)—C(2)—C(2a)	109·1 (4)
C(2)—C(2a)—C(7a)	102·3 (3)	C(2a)—C(7a)—C(8)	101·9 (3)
C(7a)—C(8)—C(8a)	108·5 (4)	C(8)—C(8a)—C(1a)	104·0 (4)
C(7a)—C(2a)—C(3)	113·1 (3)	C(2a)—C(3)—C(3a)	115·7 (3)
C(3)—C(3a)—C(6a)	113·6 (3)	C(3a)—C(6a)—C(7)	112·7 (2)
C(6a)—C(7)—C(7a)	116·1 (3)	C(7)—C(7a)—C(2a)	112·9 (3)
C(6a)—C(3a)—C(4)	102·6 (3)	C(3a)—C(4)—C(4a)	107·0 (3)
C(4)—C(4a)—C(5a)	105·7 (4)	C(4a)—C(5a)—C(6)	104·7 (4)
C(5a)—C(6)—C(6a)	106·8 (3)	C(6)—C(6a)—C(3a)	102·4 (2)
C(2)—C(9)—C(8)	94·1 (4)	C(4)—C(10)—C(6)	95·0 (3)
C(1a)—C(2)—C(9)	101·5 (4)	C(2a)—C(2)—C(9)	98·9 (4)
C(8a)—C(8)—C(9)	102·2 (4)	C(7a)—C(8)—C(9)	101·2 (4)
C(3a)—C(4)—C(10)	100·0 (3)	C(4a)—C(4)—C(10)	101·3 (3)
C(5a)—C(6)—C(10)	101·5 (4)	C(6a)—C(6)—C(10)	101·3 (3)
C(2)—C(2a)—C(3)	120·6 (4)	C(3)—C(3a)—C(4)	117·1 (3)
C(6)—C(6a)—C(7)	117·4 (2)	C(7)—C(7a)—C(8)	121·0 (4)
O(1)—C(1a)—C(2)	114·3 (4)	O(1)—C(8a)—C(8)	114·2 (4)
O(5)—C(4a)—C(4)	114·6 (4)	O(5)—C(5a)—C(6)	115·0 (4)
O(1)—C(1a)—C(8a)	59·7 (3)	O(1)—C(8a)—C(1a)	60·4 (3)
C(1a)—O(1)—C(8a)	60·0 (3)	C(4a)—O(5)—C(5a)	60·5 (3)
O(5)—C(4a)—C(5a)	58·9 (3)	O(5)—C(5a)—C(4a)	60·6 (3)
C(2a)—C(3)—OH(2)	112·2 (3)	C(3a)—C(3)—OH(2)	107·2 (3)
C(6a)—C(7)—OH(1)	106·1 (3)	C(7a)—C(7)—OH(1)	111·8 (3)

reported for (1). the C(sp^3)—O distances C(7)—OH(1) and C(3)—OH(2) [1·431 (5) and 1·429 (4) Å] are in conformity with the C(sp^3)—O single-bond (paraffin) distance 1·43 (1) Å (*International Tables for X-ray Crystallography*, 1974). The C(sp^3)—O distances in one epoxy ring, namely C(8a)—O(1) and C(1a)—O(1), are nearly equal [1·442 (7) and 1·452 (7) Å], while they differ slightly in the other epoxy ring, namely C(5a)—O(5) 1·432 (6) and C(4a)—O(5) 1·457 (7) Å. The bond angles in the epoxy rings are around 60°, which are normal for epoxides (Loudon, 1984).

The stereochemistry of (1) is reported to be *endo, cis, anti, cis, endo*. The same overall structure is also observed in the title compound with the orientation of the epoxy rings being *exo, exo*. The *exo* orientation of the two epoxy rings is also confirmed on the basis of chemical shift values observed for C(9) (25·94 p.p.m.) and C(10) (26·58 p.p.m.) in its ^{13}C NMR specta.

The central ring is nonplanar and occurs in a boat conformation. The atoms C(2a), C(3a), C(6a) and C(7a) are nearly planar with the maximum deviation being 0·02 Å. The deviations of the atoms C(3), C(7) and the oxygens of the hydroxyl groups OH(1), OH(2) from the above plane are −0·55, −0·54, −0·29 and −0·35 Å respectively. The endocyclic torsion angles in the central ring suggest a distortion from the ideal boat conformation with the asymmetry parameters $\Delta C_s(3) = 3\cdot84\ (5)°$ (Duax & Norton, 1975).

Both the terminal rings are forced to a strained boat conformation by the methylene bridge with the asymmetry $\Delta C_s(4)$ and $\Delta C_s(2)$ being 2·76 (5) and 3·64 (5)° respectively. The ring junctions are *cis* as

shown by the torsion angles C(4)—C(3a)—C(6a)—C(6) −1·8 (3), C(3)—C(3a)—C(6a)—C(7) −2·2 (4), C(3)—C(2a)—C(7a)—C(7) −3·1 (5) and C(2)—C(2a)—C(7a)—C(8) −3·2 (4)°.

Crystal packing is stabilized by hydrogen bonds O$W\cdots$OH(1^i) = 2·664 (6) Å [OW—H(22)$\cdots$OH(1^i) = 174 (3)°], OH(2)$\cdots$OW^{ii} = 2·674 (5) Å [OH(2)—H(20)$\cdots$OW^{ii} = 149 (3)°] and O$W\cdots$O(5) = 2·795 (7) Å [OW—H(21)$\cdots$O(5) = 174 (2)°] [symmetry codes: (i) $-0\cdot5 + x$, $-0\cdot5 + y$, z; (ii) $0\cdot5 + x$, $-0\cdot5 - y$, $0\cdot5 + z$). The presence of water molecules in the crystal lends strength to the crystal packing.

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Structures of Some *N*-Acetylamides of Amino Acids

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Abstract. *N*-Acetylglycinamide (NAGA), $C_4H_8N_2O_2$, $M_r = 116\cdot12$, monoclinic, $P2_1/n$, $a = 5\cdot341$ (1), $b = 8\cdot041$ (2), $c = 13\cdot638$ (2) Å, $\beta = 93\cdot24$ (2)°, $V = 584\cdot8$ (3) Å^3, $Z = 4$, $D_x = 1\cdot319$ Mg m^{-3}, λ(Mo Kα) $= 0\cdot71073$ Å, $\mu = 0\cdot099$ mm^{-1}, $F(000) = 248$, room temperature, final $R = 0\cdot042$ for 1018 observed reflexions. *N*-Acetyl-L-alaninamide (L-NAAA), $C_5H_{10}N_2O_2$, $M_r = 130\cdot15$, monoclinic, $P2_1$, $a = 5\cdot942$ (1), $b = 7\cdot480$(2), $c = 8\cdot414$ (1) Å, $\beta = 106\cdot07$ (3)°, $V = 359\cdot3$ (2) Å^3, $Z = 2$, $D_x = 1\cdot203$ Mg m^{-3}, λ(Cu Kα) $= 1\cdot54178$ Å, $\mu = 0\cdot75$ mm^{-1}, $F(000) = 140$, room temperature, final $R = 0\cdot035$ for 662 observed reflexions. *N*-Acetyl-L-leucinamide (L-NALA), $C_8H_{16}N_2O_2$, $M_r = 172\cdot23$, orthorhombic, $P2_12_12_1$, $a = 5\cdot776$ (1), $b = 10\cdot205$ (4), $c = 17\cdot256$ (4) Å, $V = 1017\cdot0$ (8) Å^3, $Z = 4$, $D_x = 1\cdot125$ Mg m^{-3}, λ(Cu Kα) $= 1\cdot54178$ Å, $\mu = 0\cdot63$ mm^{-1}, $F(000) = 376$, room temperature, final $R = 0\cdot047$ for 694 observed reflexions. In the three crystals the molecular packing is ruled by the intermolecular hydrogen bonds which involve all the polar groups. However, in L-NALA the necessity to accommodate increasingly bulky alkyl side chains does not allow the maximum number of possible hydrogen bonds. Besides, the packing density and cohesiveness decrease with increasing molecular weight, from NAGA to L-NAAA to L-NALA. The crystallographic results can explain qualitatively the trend of the enthalpies of melting and sublimation recently determined.

Introduction. In this paper we present the results of X-ray studies on some *N*-acetylamides of amino

acids, of general formula: CH_3-CO-NH-CHR-CO-NH_2. These uncharged molecules have been proposed (Blackburn, Lilley & Walmsley, 1980) as models, better than amino acids, for thermodynamic and theoretical studies on polypeptides. The crystallographic study of the uncharged peptidoamides and their derivatives is part of a research program on the physico-chemical properties of these substances. In fact, accurate knowledge of the crystal structure gives an insight of the factors controlling the interactions between groups of the same or different polarity. Moreover, the geometrical parameters obtained by X-ray analysis can be used, together with spectroscopic data, dipolar moments and sublimation energies, for deriving the parameters for semiempirical potential relations and for conformational analysis. The knowledge of intermolecular interactions in the solid state can give a simple explanation for the trends of enthalpies of melting and sublimation recently determined (Ferro, Della Gatta & Barone, 1988) and will be used as support for theoretical calculations concerning the weak nonbonding interactions in solution which have recently been the subject of extensive experimental studies (Barone, Castronuovo, Elia & Giancola, 1985; Blackburn, Lilley & Millburn, 1986; Barone, Castronuovo, Del Vecchio, Elia & Giancola, 1987).

Experimental. The present compounds (NAGA, L-NAAA and L-NALA) were prepared in our laboratory using the method described by Blackburn, Lilley & Walmsley (1980). Single crystals were grown as colourless prisms by slow evaporation from