distances are $OW \cdots O1$ 2·73 (2), $OW \cdots O4$ 2·96 (2) and $OW \cdots O1^i$ [(i) $\frac{1}{2} + x, \frac{1}{2} - y, -z$] 2·72 (2) Å.

References

ABRAHAM, R. J. & LOFTUS, E. P. (1980). *Proton and Carbon-13 NMR Spectroscopy*, pp. 104–107. London: Heyden.

AMICO, V., CUNSOLO, F., PIATTELLI, M. & RUBERTO, G. (1987). *Phytochemistry*, **26**, 1719–1722.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–326.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

SHELDRICK, G. M. (1976). *SHELX76. Program for crystal structure determination*. Univ. of Cambridge, England.

STEWART, R. F. (1970). *J. Chem. Phys.* **53**, 205–213.

Structure of 1,12-Dinitrododecane – an Alkane with Terminal Dipolar Substituents

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Abstract. $C_{12}H_{24}N_2O_4$, $M_r = 260·336$, monoclinic, $P2_1/n$, $a = 5·322$ (1), $b = 24·496$ (10), $c = 5·631$ (2) Å, $\beta = 100·36$ (2)°, $V = 722·2$ Å^3, $Z = 2$, $D_x = 1·197$ g cm^{-3}, $\lambda(Cu\,K\alpha) = 1·54178$ Å, $\mu = 6·54$ cm^{-1}, $F(000) = 284$, $T = 293$ K, $R = 0·041$, $wR = 0·043$ for 939 observed reflections. In the molecule, which lies on a centre of symmetry and has a fully extended *trans* configuration, the nitro group is oriented such that its plane is approximately orthogonal [78 (1)°] to that of the C_n skeleton; the molecules are arranged in the lattices such that the intermolecular $O \cdots N$ attractions are maximized whilst the $O \cdots O$ repulsions are minimized.

Introduction. This paper presents the initial structural results from a program to investigate the properties of novel materials arising from ionic and highly dipolar substituted alkanes having negligible homologous dispersion. This work is an extension of previous studies of the electrical and mechanical properties of alkanes of known crystal structures (Belmont, 1985; Belmont, Hooper & Amechi, 1985). The basic starting material in the synthesis, 12-bromododecanol, $Br(CH_2)_{12}OH$, was prepared following the route described in Bidd, Kelly, Ottley, Paynter, Simmonds & Whiting (1983), and converted to 1,12-dinitrododecane, $O_2N(CH_2)_{12}NO_2$ (Ainscow, Belmont, Henshall, Hooper & Simmonds, 1987), using modified procedures based on standard methods of functional-group conversion (Blatt, 1959; Johnson, 1954). This particular method allows very precise control of the chain length, with negligible contamination by homologues.

Experimental. Elongated rhombohedral crystals of the compound were prepared by slow evaporation of a diethyl ether solution. Single crystal $0·6 \times 0·45 \times 0·25$ mm. CAD-4 diffractometer, Ni-filtered Cu $K\alpha$ radiation, cell dimensions from setting angles for 25 automatically centred reflections, $30 < \theta < 35°$. 1227 reflections measured ($3·0 < \theta < 60°$, $h: 0 \rightarrow 14$; $k: 0 \rightarrow 5$; $l: -6 \rightarrow 6$), $\omega/2\theta$ scan, ω width $0·95° + 0·15°\tan\theta$ + background, data corrected for absorption empirically using ψ scan curves. Three standard reflections, no intensity variation. Structure solved by direct methods (*SHELXS*84, Sheldrick, 1984) and refined by full-matrix least squares (*SHELX*76, Sheldrick, 1976), minimizing $\sum w(|F_o| - |F_c|)^2$. Non-H atoms refined anisotropically, H atoms isotropically. 130 parameters, 939 observed data [$I > 1·5\sigma(I)$], weights $w = 1/[\sigma^2(F_o) + 0·00005(F_o)^2]$ with $\sigma(F_o)$ from counting statistics, $R = 0·041$, $wR = 0·043$, max. $\Delta/\sigma = 0·135$, max./min. peaks in ΔF map $+0·15$, $-0·2$ e Å^{-3}. Coefficients for atomic scattering factor calculation as in *SHELX*76. All calculations made on a DEC VAX 11/750 computer. Final atomic fractional coordinates are given in Table 1.†

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† Lists of structure factors, anisotropic thermal parameters and intra- and intermolecular non-bonded distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44599 (9 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* (× 10⁴) *and equivalent* (C,N,O) *or actual* (H) *isotropic U values* (× 10³) *for* $O_2N(CH_2)_{12}NO_2$

	x	y	z	$U_{eq}{}^*/U_{iso}$(Å²)
N(1)	6704 (3)	7979 (1)	−3565 (3)	45 (1)
O(1)	7930 (4)	7596 (1)	−2633 (3)	84 (1)
O(2)	7398 (3)	8251 (1)	−5115 (3)	70 (1)
C(1)	4305 (4)	8134 (1)	−2734 (4)	50 (1)
C(2)	4778 (4)	8622 (1)	−1090 (4)	45 (1)
C(3)	2416 (4)	8793 (1)	−113 (3)	44 (1)
C(4)	2915 (4)	9275 (1)	1603 (3)	43 (1)
C(5)	616 (4)	9445 (1)	2674 (3)	42 (1)
C(6)	1140 (4)	9915 (1)	4451 (3)	43 (1)
H(11)	3135 (48)	8202 (9)	−4163 (43)	74 (7)
H(12)	3806 (46)	7828 (10)	−1973 (38)	70 (7)
H(21)	5416 (43)	8917 (9)	−1926 (37)	66 (6)
H(22)	6046 (49)	8532 (9)	160 (42)	72 (7)
H(31)	1062 (43)	8882 (8)	−1396 (37)	59 (6)
H(32)	1885 (39)	8486 (9)	742 (36)	57 (6)
H(41)	4273 (41)	9184 (8)	2903 (35)	58 (6)
H(42)	3378 (39)	9580 (8)	769 (35)	56 (6)
H(51)	−771 (39)	9547 (8)	1389 (34)	52 (5)
H(52)	89 (37)	9143 (8)	3499 (33)	48 (5)
H(61)	2536 (39)	9818 (8)	5728 (35)	50 (5)
H(62)	1741 (40)	10217 (8)	3670 (34)	53 (5)

$$* U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i\mathbf{a}_j$$

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $O_2N(CH_2)_{12}NO_2$

O(1)—N(1)	1·208 (3)	O(2)—N(1)	1·208 (3)
C(1)—N(1)	1·486 (3)	C(2)—C(1)	1·505 (5)
H(11)—C(1)	0·940 (26)	H(12)—C(1)	0·925 (24)
C(3)—C(2)	1·519 (5)	H(21)—C(2)	0·958 (23)
H(22)—C(2)	0·910 (25)	C(4)—C(3)	1·518 (4)
H(31)—C(3)	0·950 (23)	H(32)—C(3)	0·962 (22)
C(5)—C(4)	1·517 (4)	H(41)—C(4)	0·958 (22)
H(42)—C(4)	0·939 (21)	C(6)—C(5)	1·518 (4)
H(51)—C(5)	0·969 (21)	H(52)—C(5)	0·942 (20)
H(61)—C(6)	0·966 (21)	H(62)—C(6)	0·945 (22)
C(6)—C(6ⁱ)	1·516 (6)		

O(2)—N(1)—O(1)	122·1 (3)	C(1)—N(1)—O(1)	119·2 (3)
C(1)—N(1)—O(2)	118·6 (3)	C(2)—C(1)—N(1)	109·7 (3)
H(11)—C(1)—N(1)	104·5 (15)	H(11)—C(1)—C(2)	113·4 (14)
H(12)—C(1)—N(1)	105·9 (16)	H(12)—C(1)—C(2)	112·8 (14)
H(12)—C(1)—H(11)	109·9 (21)	C(3)—C(2)—C(1)	112·6 (3)
H(21)—C(2)—C(1)	109·4 (14)	H(21)—C(2)—C(3)	111·1 (14)
H(22)—C(2)—C(1)	107·7 (15)	H(22)—C(2)—C(3)	109·5 (16)
H(22)—C(2)—H(21)	106·4 (21)	C(4)—C(3)—C(2)	112·6 (3)
H(31)—C(3)—C(2)	110·7 (13)	H(31)—C(3)—C(4)	109·0 (13)
H(32)—C(3)—C(2)	107·6 (14)	H(32)—C(3)—C(4)	108·8 (13)
H(32)—C(3)—H(31)	108·0 (19)	C(5)—C(4)—C(3)	113·9 (3)
H(41)—C(4)—C(3)	109·0 (13)	H(41)—C(4)—C(5)	108·2 (12)
H(42)—C(4)—C(3)	109·4 (13)	H(42)—C(4)—C(5)	106·5 (13)
H(42)—C(4)—H(41)	109·8 (19)	C(6)—C(5)—C(4)	113·9 (3)
H(51)—C(5)—C(4)	109·6 (12)	H(51)—C(5)—C(6)	108·7 (12)
H(52)—C(5)—C(4)	108·2 (13)	H(52)—C(5)—C(6)	107·8 (12)
H(52)—C(5)—H(51)	108·5 (18)	H(61)—C(6)—C(5)	109·3 (12)
H(62)—C(6)—C(5)	108·9 (12)	H(62)—C(6)—H(61)	105·0 (18)

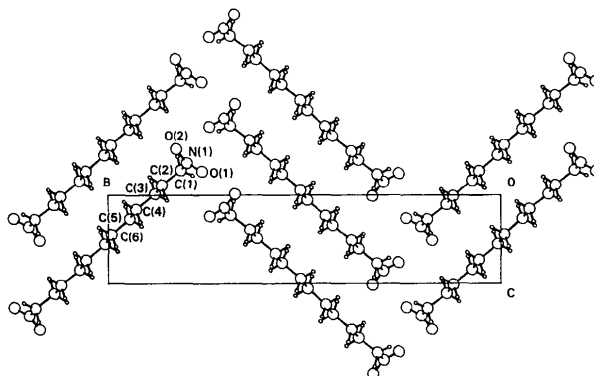Key to symmetry operation relating designated atoms to reference atoms at x, y, z: (i) −x, 2·0−y, 1·0−z.

Discussion. Table 2 gives bond lengths and angles for 1,12-dinitrododecane. Fig. 1 is a projection of the structure normal to the *yz* plane. The C—C bonded distances, excluding C(1), and the H—C—H bond angles are similar to those reported for comparable *n*-alkanes (Broadhurst, 1962; Mathesin, Norman & Pedersen, 1967; Nyburg & Luth, 1972). The nitro group is planar, as expected, with the C—N and N—O bond lengths and O—N—O and C—N—O bond angles similar to published values (Thorup, 1981). Examination of the H···H distances on vicinal C atoms* shows that the carbon skeleton of 1,12-dinitrododecane has an extended *trans* configuration and is not helically twisted, as has been observed for a non-symmetrical terminally substituted alkane chain (Hybl & Dorset, 1971). All torsion angles in the chain are 180±2°. Fig. 1 clearly shows that the molecules assume a zigzag packing arrangement. This is in marked contrast to *n*-alkanes (C_nH_{2n+2}, 6 < n < 24), where the chains have extended *trans* configurations and pack with all the molecules parallel (Broadhurst, 1962; Mathesin *et al.*, 1967; Nyburg & Luth, 1972). This difference arises from the highly polarized nitro group [*cf.* nitromethane, which has a dipole moment of 11·54 × 10^{-30} Cm (Tannenbaum, Myers & Gwinn, 1956)]. The nitro groups are arranged in sheets parallel to the *ac* planes so that the non-bonded O···N ($\delta^-\cdots\delta^+$) attraction is maximized, whilst the intermolecular O···O ($\delta^-\cdots\delta^-$) and N···N ($\delta^+\cdots\delta^+$) repulsions are minimized. Since the nitro group is of similar radius to the C_2H_4 repeat unit of the chain then the molecule as a whole appears cylindrical and it can pack conveniently in a zigzag structure.

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* See deposition footnote.

Fig. 1. Projection of the structure normal to the *yz* plane, showing atom numbering.

References

AINSCOW, T. A., BELMONT, M. R., HENSHALL, J. L., HOOPER, R. M. & SIMMONDS, D. J. (1987). *Tetrahedron*, **43**, 115–122.
BELMONT, M. R. (1985). *J. Phys. Chem. Solids*, **46**, 595–606.
BELMONT, M. R., HOOPER, R. M. & AMECHI, P. A. C. (1985). *J. Phys. Chem. Solids*, **46**, 607–617.
BIDD, I., KELLY, D. J., OTTLEY, P. M., PAYNTER, O. I., SIMMONDS, D. J. & WHITING, M. C. (1983). *J. Chem. Soc. Perkin Trans.* 1, pp. 1369–1372.

BLATT, A. H. (1959). *Organic Synthesis Collective*, Vol. 2, pp. 476–478. New York: John Wiley.

BROADHURST, M. G. (1962). *J. Res. Natl Bur. Stand. Sect. A*, **66**, 241–249.

HYBL, A. & DORSET, D. (1971). *Acta Cryst.* B27, 977–986.

JOHNSON, W. S. (1954). *Organic Synthesis*, Vol. 34, pp. 37–39. New York: John Wiley.

MATHESIN, H., NORMAN, N. & PEDERSEN, B. F. (1967). *Acta Chem. Scand.* **21**, 127–135.

NYBURG, S. C. & LUTH, H. (1972). *Acta Cryst.* B28, 2992–2995.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1984). *SHELXS84*. Program for the automatic solution of crystal structures. Univ. of Göttingen, Federal Republic of Germany.

TANNENBAUM, E., MYERS, R. J. & GWINN, W. D. (1956). *J. Chem. Phys.* **25**, 42–47.

THORUP, N. (1981). *Acta Cryst.* B37, 1442–1444.

Structure of 5'-Acetylthymidine

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Abstract. $C_{12}H_{16}N_2O_6$, $M_r = 284 \cdot 3$, orthorhombic, $P2_12_12_1$, $a = 9 \cdot 262$ (1), $b = 30 \cdot 658$ (2), $c = 4 \cdot 788$ (1) Å, $V = 1359 \cdot 5$ (2) Å^3, $Z = 4$, $D_x = 1 \cdot 389$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1 \cdot 54178$ Å, $\mu = 0 \cdot 97$ mm^{-1}, $F(000) = 600$, room temperature, $R = 0 \cdot 034$ for 1490 observed reflections. The molecule has a C(2')-*endo* furanose ring associated with the base in the *anti* conformation. Atomic coordinates are presented for a standard thymine residue, which was derived from the bond distances and angles averaged over 21 structures retrieved from the Cambridge Crystallographic Database.

Introduction. Raman spectroscopic examination of the crystals of nucleosides and nucleotides whose structures are known from X-ray analysis has revealed several correlations between structure and spectrum, which have, in turn, been used for diagnosing the conformation of nucleic acids (Nishimura, Tsuboi, Sato & Aoki, 1986; Katahira, Nishimura, Tsuboi, Sato, Mitsui & Iitaka, 1986). The present study was undertaken as part of such investigations.

Experimental. Crystals grown from aqueous solution by slow evaporation, colorless plate, D_m not measured. Crystal dimensions: $0 \cdot 10 \times 0 \cdot 15 \times 0 \cdot 30$ mm, Rigaku AFC-5 diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Lattice parameters from 24 reflections ($56 < 2\theta < 81°$). $\omega/2\theta$ scan, $2\theta \le 140°$, $0 \le h \le 11$, $0 \le k \le 38$, $0 \le l \le 5$, three standard reflections: no variation. 1606 independent reflections measured, 1490 with $I \ge 2\sigma(I)$ considered observed. No absorption correction. Structure solved by direct methods, H atoms located from a difference Fourier map and their positions and isotropic thermal parameters refined. Full-matrix least-squares refinement,

anisotropic thermal parameters for non-H atoms, isotropic type I extinction correction (Becker & Coppens, 1974) with $g = 0 \cdot 24$ (1) $\times 10^{-4}$, $\sum w(\Delta F)^2$ minimized, $w = 1$, $R = 0 \cdot 034$, $wR = 0 \cdot 033$, final $|\text{shift}/\sigma|_{\text{max}} < 0 \cdot 1$, $-0 \cdot 16 \le \Delta\rho \le 0 \cdot 14$ e Å^{-3}. The final atomic parameters are given in Table 1.* Bond distances and angles are listed in Table 2. All crystallographic calculations were performed on a VAX11/780 computer using the program system *XTAL*2·2 (Hall & Stewart, 1987) with the scattering factors as included in the program.

Discussion. A perspective view of the molecule is shown in Fig. 1. The conformation of the nucleoside moiety is quite normal. The pseudorotation coordinates (Sato, 1983) of the furanose ring are $\Pi = 37 \cdot 8$ (3)° and $\Phi = 257 \cdot 4$ (4)° and, therefore, the ring is puckered in a C(2')-*endo* form. The conformations about the C(1')—N(1) and the C(4')—C(5') bonds are *anti* and *trans–gauche*, the torsion angles O(4')—C(1')—N(1)—C(6), C(2')—C(1')—N(1)—C(6) and C(3')—C(4')—C(5')—O(5') being 48·5 (3), 103·4 (3) −174·3 (2)°, respectively.

To see whether the molecular dimensions of the base are normal or not, crystal structures containing the unsubstituted thymine residue were retrieved from the Cambridge Crystallographic Database (Allen *et al.*, 1979) and the bond distances and angles were averaged over 21 accurate structures* using the program *GEOM*

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* Lists of H-atom parameters, anisotropic thermal parameters, structure factors and reference codes for the crystal structures used in the analysis of the thymine residue have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44610 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.