Fig. 2. Organisation des unités structurales en projection sur le plan *yz*.

$N(1)-H(N2)\cdots O(1) = 164 (2)°$ et $N(1)\cdots O(2) = 2,681 (4)$, $N(1)-H(N1) = 0,98 (4)$, $H(N1)\cdots O(2) = 1,72 (4)$ Å, $N(1)-H(N1)\cdots O(2) = 168 (2)°$.

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Structure of 1,2,3-Propanetriol Trinitrate (β Modification), C₃H₅N₃O₉

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Abstract. $M_r = 227.63$, orthorhombic, $Pna2_1$, $a = 8.900 (2)$, $b = 13.608 (3)$, $c = 6.762 (2)$ Å, $V = 819.0$ Å³, $Z = 4$, $D_x = 1.846$ g cm⁻³, $\lambda(Mo~K\alpha) = 0.71069$ Å, $\mu = 2.043$ cm⁻¹, $F(000) = 464$, $T = 153 (3)$ K, final $R = 0.026$ for 783 observed reflections. Bond lengths and angles are similar to those found by the microwave spectroscopic investigation of methyl nitrate in the gas phase and in some organic polynitrates in the solid state.

Introduction. The influence of nitrate groups, linked to C atoms, on the configuration of polynitrates has been studied for the stable β modification of 1,2,3-propanetriol trinitrate (PTTN) by X-ray analysis. It is also of great interest to compare the structural data with the results of a microwave-spectroscopy study of methyl nitrate (MN) in the gas phase (Cox & Waring, 1971; Dixon & Wilson, 1961).

Experimental. A suitable single crystal $0.3 \times 0.5 \times 0.4$ mm was grown from a supercooled ether solution. Syntex *P*2₁ automatic diffractometer equipped with a low-temperature device [crystal in a stream of gaseous nitrogen cooled to 153 (3) K]. Space group $Pna2_1$ unambiguously determined from systematic absences $(0kl, k+l = 2n+1; h0l, h = 2n+1)$. Unit-cell dimensions calculated by least-squares technique using 24 accurately centered reflections $(20 < \theta < 22°)$. Intensity data collected in the range $h = 0-10$, $k = 0-18$, $l = 0-8$ with graphite-monochromated Mo $K\alpha$ radiation and $\theta-2\theta$ scan; $\theta_{max} = 30°$; 863 independent reflections, 783 observed $[I \geq 2\sigma(I)]$. Three standard reflections monitored after every 97 reflections, no significant variation. Lorentz and polarization corrections applied, but no absorption correction. Structure solved by direct methods (*MULTAN*, Germain, Main & Woolfson, 1971) and refined by full-matrix least squares with anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms which were located in a difference Fourier map in the expected positions (156 parameters refined). Final $R = 0.026$; $\sum w[|F_o| - |F_c|]^2$ minimized, where $w^{-1} = \sigma^2(F_o)$; $R_w = 0.032$, $S = 2.44$. Atomic scattering factors from *International Tables for*

X-ray Crystallography (1974). In final cycles $(\Delta/\sigma)_{max} = 0.25$. $(\Delta\rho)_{max} = 10.20 |$ e Å^{-3}. All calculations performed with an Eclipse S/200 computer using *INEXTL* programs (Gerr, Yanovsky & Struchkov, 1983).

Discussion. Table 1 lists the final atomic coordinates and mean temperature factors.* The numbering scheme of the molecule is presented in Fig. 1. The bond lengths, angles and torsion angles are listed in Table 2.

Bond distances in the PTTN molecule have the usual values; the differences in the three chemically equivalent C—O—NO$_2$ groups do not exceed experimental errors, thus being comparable to appropriate parameters in MN (C—O 1·437, O—N 1·402, N—O$_{cis}$ 1·205, N—O$_{trans}$ 1·208 Å) (Cox & Waring, 1971). There is a good correlation between bond angles in PTTN and MN (C—O—C 112·72, O—N—O$_{cis}$ 118·10, O—N—O$_{trans}$ 112·38°) (Cox & Waring, 1971). The geometrical parameters in these groups in PTTN are in good agreement with those found in some organic polynitrates (Trotter, 1963; Mak & Trotter, 1964; Allen & Trotter, 1970; Halfpenny & Small, 1978; Couldwell, 1979).

The distortions in the tetrahedral angles at C atoms can be regarded as a manifestation of the so-called 'd effect' (Slovokhotov, Timofeeva, Antipin & Struchkov, 1984), *i.e.* the R—C—O angle in the RCON moiety with R *trans* to the N atom [the torsion angle RCON (φ) is close to 180°] is less than 109·5°.
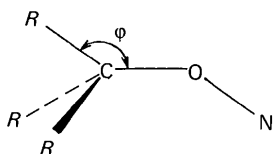
* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39681 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
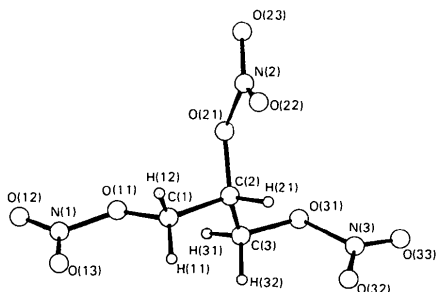

Fig. 1. A molecule of the title compound with the atomic numbering.

Table 1. *Atomic coordinates* $(\times 10^4, for$ H $\times 10^3)$ *and thermal parameters*

For non-H atoms $B_{eq} = \frac{4}{3}\sum_i\sum_j B_{ij}\mathbf{a}^*_i\mathbf{a}^*_j\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}/B (Å^2)
O(11)	992 (2)	−1096 (1)	−78	1·60 (4)
O(12)	−173 (2)	−2495 (1)	−82 (5)	2·48 (5)
O(13)	1822 (2)	−2354 (1)	−1912 (4)	2·07 (5)
O(21)	1938 (2)	464 (1)	2221 (5)	1·46 (4)
O(22)	4441 (2)	377 (1)	2633 (4)	1·98 (5)
O(23)	2939 (2)	542 (1)	5147 (4)	2·16 (5)
O(31)	697 (2)	1932 (1)	−104 (4)	1·87 (4)
O(32)	2624 (2)	2324 (1)	−2081 (5)	2·51 (5)
O(33)	1333 (3)	3448 (1)	−554 (5)	3·75 (6)
N(1)	887 (2)	−2070 (2)	−769 (5)	1·65 (5)
N(2)	3241 (3)	463 (2)	3418 (5)	1·58 (6)
N(3)	1648 (3)	2619 (2)	−1012 (6)	2·19 (5)
C(1)	2398 (3)	−621 (2)	−603 (6)	1·41 (6)
C(2)	2235 (3)	430 (2)	101 (6)	1·28 (5)
C(3)	895 (3)	939 (2)	−814 (6)	1·58 (5)
H(11)	255 (3)	−63 (2)	−187 (4)	0·2 (5)
H(12)	312 (3)	−94 (2)	4 (5)	0·7 (5)
H(2)	313 (2)	80 (2)	−19 (4)	0·5 (5)
H(31)	−5 (3)	65 (2)	−57 (4)	1·3 (5)
H(32)	97 (3)	93 (2)	−222 (5)	1·1 (5)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

O(11)—N(1)	1·408 (3)	O(32)—N(3)	1·199 (4)
O(11)—C(1)	1·453 (3)	O(33)—N(3)	1·202 (3)
O(12)—N(1)	1·200 (3)	C(1)—C(2)	1·514 (4)
O(13)—N(1)	1·199 (4)	C(1)—H(11)	0·87 (3)
O(21)—N(2)	1·414 (4)	C(1)—H(12)	0·96 (3)
O(21)—C(2)	1·458 (5)	C(2)—C(3)	1·512 (4)
O(22)—N(2)	1·198 (3)	C(2)—H(2)	0·95 (2)
O(23)—N(2)	1·204 (4)	C(3)—H(31)	0·94 (3)
O(31)—N(3)	1·402 (3)	C(3)—H(32)	0·96 (3)
O(31)—C(3)	1·445 (3)		

N(1)—O(11)—C(1)	113·3 (2)	C(2)—C(1)—H(11)	110 (2)
N(2)—O(21)—C(2)	114·5 (2)	C(2)—C(1)—H(12)	111 (2)
N(3)—O(31)—C(3)	113·9 (2)	H(11)—C(1)—H(12)	109 (2)
O(11)—N(1)—O(12)	112·1 (2)	O(21)—C(2)—C(1)	110·9 (2)
O(11)—N(1)—O(31)	118·1 (2)	O(21)—C(2)—C(3)	104·2 (2)
O(12)—N(1)—O(13)	129·8 (3)	O(21)—C(2)—H(2)	110 (1)
O(21)—N(2)—O(22)	118·5 (3)	C(1)—C(2)—C(3)	112·3 (2)
O(21)—N(2)—O(23)	111·9 (3)	C(1)—C(2)—H(2)	110 (2)
O(22)—N(2)—O(23)	129·6 (3)	C(3)—C(2)—H(2)	109 (2)
O(31)—N(3)—O(32)	118·5 (3)	O(31)—C(3)—C(2)	112·9 (2)
O(31)—N(3)—O(33)	111·8 (3)	O(31)—C(3)—H(31)	103 (2)
O(32)—N(3)—O(33)	129·7 (3)	O(31)—C(3)—H(32)	110 (2)
O(11)—C(1)—C(2)	105·2 (2)	C(2)—C(3)—H(31)	116 (2)
O(11)—C(1)—H(11)	111 (2)	C(2)—C(3)—H(32)	110 (2)
O(11)—C(1)—H(12)	111 (2)	H(31)—C(3)—H(32)	104 (3)

O(12)—N(1)—O(11)—C(1)	169·8 (4)	O(22)—N(2)—O(21)—C(2)	−4·3 (3)
O(13)—N(1)—O(11)—C(1)	−10·2 (3)	O(23)—N(2)—O(21)—C(2)	176·4 (4)
N(1)—O(11)—C(1)—C(2)	175·1 (4)	N(2)—O(21)—C(2)—C(3)	−152·2 (4)
N(1)—O(11)—C(1)—H(11)	56 (3)	N(2)—O(21)—C(2)—H(2)	−35 (3)
N(1)—O(11)—C(1)—H(12)	−65 (3)	O(21)—C(2)—C(3)—O(31)	58·0 (3)
O(11)—C(1)—C(2)—C(3)	−57·9 (3)	O(21)—C(2)—C(3)—H(31)	−60 (3)
O(11)—C(1)—C(2)—O(21)	58·2 (3)	O(21)—C(2)—C(3)—H(32)	−178 (3)
O(11)—C(1)—C(2)—H(2)	180 (3)	H(2)—C(2)—C(3)—O(31)	−59 (3)
H(11)—C(1)—C(2)—C(3)	62 (3)	H(2)—C(2)—C(3)—H(31)	−177 (4)
H(11)—C(1)—C(2)—O(21)	178 (3)	H(2)—C(2)—C(3)—H(32)	65 (4)
H(11)—C(1)—C(2)—H(2)	−60 (4)	C(2)—C(3)—O(31)—N(3)	79·8 (4)
H(12)—C(1)—C(2)—C(3)	−178 (3)	H(31)—C(3)—O(31)—N(3)	−154 (3)
H(12)—C(1)—C(2)—O(21)	−62 (3)	H(32)—C(3)—O(31)—N(3)	−44 (3)
H(12)—C(1)—C(2)—H(2)	60 (4)	C(3)—O(31)—N(3)—O(32)	−8·4 (3)
C(1)—C(2)—O(21)—N(2)	86·8 (4)	C(3)—O(31)—N(3)—O(33)	171·9 (5)
C(1)—C(2)—C(3)—O(31)	178·0 (4)		

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Structure of Ethyl 1-Cyano-1,2,3,4-tetrahydro-2-isoquinolinecarboxylate, $C_{13}H_{14}N_2O_2$

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Abstract. $M_r = 230 \cdot 27$, triclinic, $P\bar{1}$, $a = 8 \cdot 868$ (1), $b = 10 \cdot 119$ (1), $c = 8 \cdot 2858$ (9) Å, $\alpha = 83 \cdot 624$ (8), $\beta = 119 \cdot 147$ (8), $\gamma = 111 \cdot 051$ (8)°, $V = 604 \cdot 2$ (1) Å³, $Z = 2$, $D_m = 1 \cdot 28$, $D_x = 1 \cdot 27$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1 \cdot 54178$ Å, $\mu = 0 \cdot 623$ mm⁻¹, $F(000) = 244$, room temperature, $R = 0 \cdot 053$ for 1361 observed reflexions. The hydrogenated heterocyclic ring has the conformation of a slightly deformed half-chair. The C(3)–N(2)–C(13)–O(14) torsion angle [$-172 \cdot 4$ (4)°] characterizes the amide bond as *anti*. The NCOO group is exactly planar and makes an angle of $31 \cdot 2$ (4)° with the aromatic ring, minimizing steric interactions between the carbonyl group and neighboring H atoms.

Introduction. The present work is the second part of the investigation of a series of Reissert compounds. The structure investigation of the title compound has been undertaken to determine the effect of substituents on the molecular conformation and to facilitate interpretation of ¹³C NMR data (Brózda, 1982).

Experimental. Elongated prismatic crystals from ethanol, D_m by flotation, crystal $0 \cdot 3 \times 0 \cdot 3 \times 0 \cdot 2$ mm, Syntex $P2_1$ diffractometer, cell parameters from least-squares treatment of setting angles of 15 reflections, profiles measured for 1717 reflexions with $2\theta \le 115°$, range of *hkl*: *h* $-9 \rightarrow 7$, *k* $-10 \rightarrow 11$, *l* $0 \rightarrow 9$, profile analysis according to Lehmann & Larsen (1974), no significant intensity variation for two standard reflexions, no absorption correction, 1366 observed reflexions with $I \ge 1 \cdot 96\sigma(I)$; structure solved by direct methods using *SHELX*76 (Sheldrick, 1976), full-matrix least-squares refinement on F, $w^{-1} = \sigma^2(F)$, five

extinction-affected reflexions excluded from final refinement, H atoms located in a ΔF map and included as fixed isotropic contributions to F_c, anisotropic thermal parameters for non-H atoms, $R = 0 \cdot 053$, $R_w = 0 \cdot 064$, $(\Delta/\sigma)_{max} = 0 \cdot 05$, max. and min. height in final ΔF map $0 \cdot 17$ and $-0 \cdot 19$ e Å⁻³; computer programs: *SHELX*76 (Sheldrick, 1976) and local programs (Jaskólski, 1982), molecular illustrations drawn using *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates and bond lengths and angles are given in Table 1 and 2 respectively.† The labeling sequence is shown in the formula below.
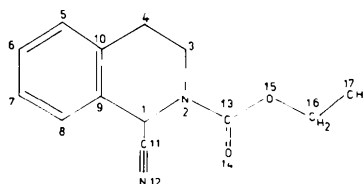
The ethoxycarbonyl substituent is joined to the tetrahydroisoquinoline ring by a urethane bond. The cyano group is bonded to the C(1) asymmetric center. The N(2)–C(3) bond distance [1·463 (5) Å] agrees with the corresponding value characteristic of piperidine amides [1·464 (5) Å; Jaskólski, 1979], while

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† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39636 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.