included as fixed contributions with C—H 0·95 Å and $B = 1·3B_{eq}$ of the bonded C atom. The N—H hydrogen atoms were refined isotropically.

The function minimized was $\sum w(|F_o| - |F_c|)^2$, and weights were assigned as $w = 4F_o^2 Lp[S^2(C + R^2B) + (0·02F_o^2)^2]^{-1}$, where S = scan rate, C = total integrated peak count, R = scan time/background counting time, B = total background count, Lp = Lorentz–polarization factor, using the Enraf–Nonius *SDP* system (Frenz & Okaya, 1980); scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1) and anomalous coefficients from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). The final cycle included 334 variables and converged (largest $\Delta/\sigma = 0·01$) to $R = 0·045$, $wR = 0·039$, $S = 1·109$. An extinction coefficient refined to $g = 1·4$ (4) $\times 10^{-8}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Maximum and minimum residual electron densities were 0·58 and $-0·39$ e Å^{-3}. Table 1 shows the final positions and equivalent isotropic thermal parameters of these two molecules. Table 2 shows bond lengths and bond angles.* Fig. 1 shows the two molecules and numbering scheme, and Fig. 2 shows the unit cell. Table 3 reports the requisite parameters for evaluation of hydrogen bonding to the four possible acceptor atoms in the macrocycle.

Related literature. Complexation of alkali-metal cations by lariat ethers can be found in Gandour,

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* Tables of H-atom coordinates, anisotropic thermal parameters, torsion angles and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53554 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Selected parameters for evaluation of hydrogen bonding*

X	N2$Y^*\cdots X$ (Å)	N2Y^*—H$\cdots X$ (°)	N2Y^*—H$\cdots X$ (Å)
O1A	3·393 (7)	114 (5)	2·83 (8)
O1B	3·367 (7)	113 (6)	2·86 (9)
O2A	3·158 (8)	144 (8)	2·25 (10)
O2B	3·158 (6)	140 (7)	2·36 (8)
O3A	3·061 (7)	127 (6)	2·32 (8)
O3B	3·079 (6)	133 (7)	2·34 (8)
N1A	2·828 (8)	104 (6)	2·40 (10)
N1B	2·830 (7)	109 (6)	2·37 (9)

* If X is an atom in molecule A, then $Y = A$. If X is in molecule B, then $Y = B$.

Fronczek, Gatto, Minganti, Schultz, White, Arnold, Mazzocchi, Miller & Gokel (1986). An example of intramolecular hydrogen bonding between two tertiary nitrogens is in Shkol'nikova, Polyanchuk, Dyatlova & Polyakova (1984). The crystal structures of 10,10′-ethylenebis(1,4,7-trioxa-10-azacyclododecane) and its lithium complex are presented in Groth (1984*a*,*b*), respectively, and the potassium complex of *N*-(3,7,10-trioxaundecyl)-1,4,7-trioxa-10-azacyclododecane is presented in White, Arnold, Fronczek, Gandour & Gokel (1985).

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Two Polymorphs of 3,5-Dinitrobenzoic Acid

By PHILIPPE PRINCE, FRANK R. FRONCZEK AND RICHARD D. GANDOUR*

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA

Abstract. $C_7H_4N_2O_6$, $M_r = 212·1$, monoclinic, $P2_1/c$, $a = 10·0290$ (6), $b = 8·8711$ (7), $c = 9·514$ (2) Å, $\beta = 95·639$ (12)°, $V = 842·4$ (3) Å^3, $Z = 4$, $D_x = 1·672$ g cm^{-3}; λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 12·74$ cm^{-1}, $F(000) = 432$, $T = 297$ K, $R = 0·042$ for 1610 observations (of 1721 unique data); monoclinic, $C2/c$, $a = 21·036$ (2), $b = 8·7331$ (6), $c = 9·7659$ (8) Å, $\beta = 111·051$ (7)°, $V = 1674·3$ (5) Å^3, $Z = 8$, $D_x = 1·683$ g cm^{-3}; λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 12·82$ cm^{-1}, $F(000) = 864$, $T = 296$ K, $R = 0·043$ for

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1583 observations (of 1725 unique data). The two polymorphs are both monoclinic, albeit with different space groups. Distances and angles are quite similar in the two polymorphs. There is no melting point difference. There is a strong intermolecular O—H···O hydrogen bond between two carboxylic groups; the donor is the acidic hydrogen on one molecule, and the acceptor is the carbonyl-type oxygen of the carboxylic group on the other molecule. There is no important difference in the strength of those interactions between the two polymorphs. The HO2···O1 distances are 1·51 (4) and 1·55 (3) Å, the O1—HO2—O2 angles are 165·5 (30) and 161·9 (30)° in the $P2_1/c$ and $C2/c$ polymorphs respectively. In both polymorphs, one nitro group is out of the plane formed by the aromatic ring by 24·0 (1) for $P2_1/c$ and 20·0 (1)° for $C2/c$. The hydroxy moiety of the carboxyl group lies on the same side of the molecule as the out-of-plane nitro group.

Experimental. The title compound, (1), was obtained in two different crystalline forms as a by-product of a reaction using 3,5-dinitrobenzoyl chloride: $P2_1/c$ polymorph recrystallized from benzene, $C2/c$ polymorph from ethanol. Data collected on Enraf–Nonius CAD-4 computer-controlled κ-axis diffractometer equipped with graphite monochromator.

O
‖
C−OH

O_2N NO_2

(1)
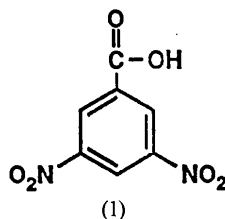
$P2_1/c$. Colorless, m.p. 485–486 K. Crystal, 0·20 × 0·40 × 0·58 mm, capillary mounted in random orientation. Cell dimensions from setting angles of 25 reflections having 25 < θ < 30°. Space group from systematic absences $h0l$ with l odd and $0k0$ with k odd. One hemisphere of data having 2 < θ < 75°, −12 ≤ h ≤ 12, 0 ≤ k ≤ 11, −11 ≤ l ≤ 11 were collected using ω–2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0·42–3·30° min^{-1}. Three reflections (400, 060, 002) were measured every 10 000 s, and exhibited only random fluctuations, thus no decay correction was applied. Background, Lorentz and polarization corrections were applied. Absorption corrections were based on ψ scans, with min. relative transmission coefficient 0·837, max. 0·998. An extinction coefficient was refined in least squares to $g = 3·27$ (9) × 10^{-7}, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. 3415 data were collected; averaging the two equivalent octants ($R_{int} = 0·013$) yielded 1721

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = (1/3)\Sigma_i\Sigma_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(Å^2)$
$P2_1/c$				
O1	0·4673 (1)	0·8262 (1)	−0·0725 (1)	4·46 (2)
O2	0·3922 (1)	0·93081 (9)	0·1190 (1)	4·52 (2)
O3	0·1883 (2)	0·1989 (1)	−0·0838 (2)	8·40 (3)
O4	0·3466 (1)	0·3026 (1)	−0·1832 (1)	4·86 (2)
O5	−0·04189 (9)	0·4811 (1)	0·2598 (1)	5·18 (2)
O6	0·0754 (1)	0·6516 (1)	0·3764 (1)	4·84 (2)
N1	0·2634 (1)	0·3023 (1)	−0·0985 (1)	4·05 (2)
N2	0·05519 (9)	0·5638 (1)	0·2780 (1)	3·37 (2)
C1	0·3193 (1)	0·6863 (1)	0·0545 (1)	2·89 (2)
C2	0·3316 (1)	0·5599 (1)	−0·0296 (1)	2·93 (2)
C3	0·2521 (1)	0·4361 (1)	−0·0087 (1)	3·02 (2)
C4	0·1601 (1)	0·4329 (1)	0·0900 (1)	3·11 (2)
C5	0·1528 (1)	0·5605 (1)	0·1719 (1)	2·86 (2)
C6	0·2301 (1)	0·6871 (1)	0·1574 (1)	2·91 (2)
C7	0·3994 (1)	0·8236 (1)	0·0309 (1)	3·17 (2)
$C2/c$				
O1	0·23089 (4)	0·0791 (1)	0·5523 (1)	4·36 (2)
O2	0·19541 (5)	0·1795 (1)	0·3265 (1)	4·36 (2)
O3	0·10164 (6)	−0·5737 (1)	0·4178 (1)	6·33 (3)
O4	0·17147 (5)	−0·4532 (1)	0·6030 (1)	4·96 (2)
O5	−0·01864 (5)	−0·2899 (2)	−0·0304 (1)	5·53 (2)
O6	0·03508 (5)	−0·1006 (1)	−0·0785 (1)	4·87 (2)
N1	0·13366 (5)	−0·4609 (1)	0·4742 (1)	3·97 (2)
N2	0·02729 (4)	−0·1965 (1)	0·0035 (1)	3·76 (2)
C1	0·15761 (4)	−0·0676 (1)	0·3551 (1)	2·95 (2)
C2	0·16413 (5)	−0·1950 (1)	0·4432 (1)	3·08 (2)
C3	0·12622 (5)	−0·3242 (1)	0·3831 (1)	3·19 (2)
C4	0·08131 (5)	−0·3298 (1)	0·2385 (1)	3·28 (2)
C5	0·07605 (5)	−0·1990 (1)	0·1563 (1)	3·10 (2)
C6	0·11354 (5)	−0·0680 (1)	0·2090 (1)	3·08 (2)
C7	0·19777 (5)	0·0730 (1)	0·4183 (1)	3·23 (2)

unique data, of which 1610 had $I > 3\sigma(I)$ and were used in the refinement.

Structure solved by direct methods, using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), and successive difference Fourier syntheses. The structure was refined by weighted full-matrix least squares; non-H atoms refined anisotropically, and H atoms refined isotropically.

The function minimized was $\Sigma w(|F_o| - |F_c|)^2$ and weights were assigned as $w = 4F_o^2 Lp[S^2(C + R^2B) + (0·02F_o^2)^2]^{-1}$, where S = scan rate, C = total integrated peak count, R = scan time/background counting time, B = total background count, Lp = Lorentz–polarization factor, using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). The final cycle included 153 variables and converged (largest $\Delta/\sigma = 0·10$) to $R = 0·042$, $wR = 0·069$, $R_{all} = 0·045$, and $S = 2·627$. The max. residual density was 0·27, min. −0·26 e Å^{-3}.

$C2/c$. Data collection and reduction, structure solution and refinement were carried out as for the $P2_1/c$ polymorph. Yellow, m.p. 485–486 K. Crystal, 0·33 × 0·40 × 0·45 mm, fiber mounted in random orientation. Space group from systematic absences hkl with $h + k$ odd and $h0l$ with l odd, and successful refinement of a centrosymmetric model.

Data limits: $h + k$ even, 2 < θ < 75°, −26 ≤ h ≤ 26, 0 ≤ k ≤ 10, −12 ≤ l ≤ 12. Scan rates varied 0·57–

Table 2. *Bond distances* (Å) *and angles* (°)

			$P2_1/c$	$C2/c$
O1	C7		1·251 (2)	1·243 (1)
O2	C7		1·275 (1)	1·280 (1)
O3	N1		1·204 (2)	1·209 (1)
O4	N1		1·216 (2)	1·225 (1)
O5	N2		1·218 (1)	1·216 (2)
O6	N2		1·220 (1)	1·210 (2)
N1	C3		1·473 (1)	1·464 (2)
N2	C5		1·474 (2)	1·476 (1)
C1	C2		1·390 (1)	1·383 (2)
C1	C6		1·390 (2)	1·395 (1)
C1	C7		1·488 (1)	1·492 (1)
C2	C3		1·384 (1)	1·385 (1)
C3	C4		1·380 (2)	1·389 (1)
C4	C5		1·380 (2)	1·378 (2)
C5	C6		1·380 (1)	1·380 (1)

			$P2_1/c$	$C2/c$
O3	N1	O4	123·8 (1)	124·2 (1)
O3	N1	C3	117·5 (1)	117·9 (1)
O4	N1	C3	118·7 (1)	117·9 (1)
O5	N2	O6	124·3 (1)	124·6 (1)
O5	N2	C5	117·8 (1)	117·4 (1)
O6	N2	C5	117·9 (1)	117·97 (9)
C2	C1	C6	120·7 (1)	121·01 (9)
C2	C1	C7	119·6 (1)	119·33 (9)
C6	C1	C7	119·72 (9)	119·66 (9)
C1	C2	C3	118·2 (1)	118·61 (9)
N1	C3	C2	118·5 (1)	119·17 (9)
N1	C3	C4	118·3 (1)	118·33 (9)
C2	C3	C4	123·2 (1)	122·5 (1)
C3	C4	C5	116·4 (1)	116·51 (9)
N2	C5	C4	118·59 (9)	118·86 (9)
N2	C5	C6	118·04 (9)	117·5 (1)
C4	C5	C6	123·4 (1)	123·66 (9)
C1	C6	C5	118·2 (1)	117·7 (1)
O1	C7	O2	125·0 (1)	124·9 (1)
O1	C7	C1	118·5 (1)	119·4 (1)
O2	C7	C1	116·5 (1)	115·73 (9)

$3\cdot30°$ min^{-1}. Three standard reflections (400, 020, 004), no decay correction was applied. Min. relative transmission coefficient 0·924, max. 0·992. Extinction coefficient $g = 1\cdot45$ (6) $\times$ 10^{-5}. 3499 data were collected, 1725 unique data (R_{int} $-$ 0·017) and 1583 data observed with $I > 3\sigma(I)$. Weighting scheme as for $P2_1/c$ polymorph; 153 variables, largest $\Delta/\sigma = 0\cdot05$, $R = 0\cdot043$, $wR = 0\cdot069$, $R_{all} = 0\cdot046$, and $S = 3\cdot319$. The max. residual density was $0\cdot26$ e Å^{-3}, min. $-0\cdot24$ e Å^{-3}. Table 1 presents the final coordinates* and equivalent isotropic thermal parameters, and Table 2 presents bond distances and angles. Fig. 1 illustrates the molecules and the numbering scheme, and Fig. 2 shows the unit cells.

Related literature. Colapietro, Domenicano, Marciante & Portalone (1983) report the space group of 3,5-dinitrobenzoic acid as $C2/c$. The Cambridge Structural Database (Allen, Kennard & Taylor, 1983) contains 47 molecules having the 3,5-dinitrobenzoate fragment. However only one structure was found containing the 3,5-dinitrobenzoic

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53555 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

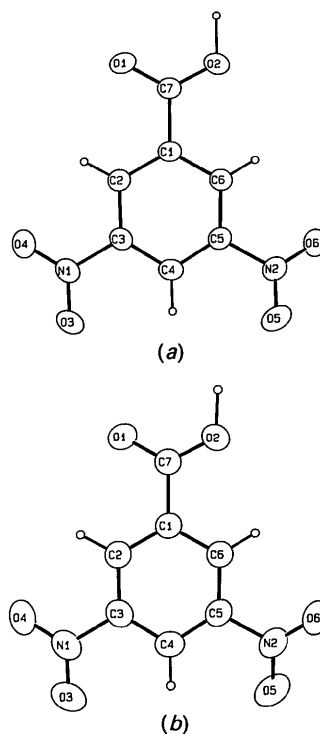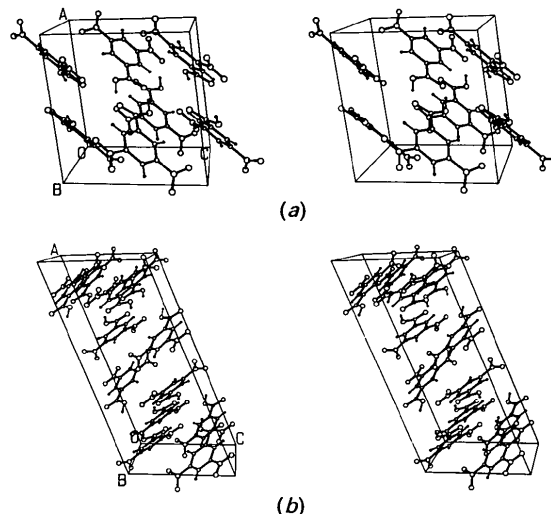Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level for (a) the $P2_1/c$ polymorph, and (b) the $C2/c$ polymorph. H atoms are drawn as circles of arbitrary radius.

Fig. 2. Stereoviews of the unit cells for (a) the $P2_1/c$ polymorph, and (b) the $C2/c$ polymorph.

acid residue (Fritchie & Trus, 1968), and coordinates are not given.

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Structure of Nucleoside Analogues 3'-Fluoro-2',3'-dideoxyuridine, 3'-Fluoro-2',3'-dideoxy-5-bromouridine and 3'-Azido-2',3'-dideoxy-5-bromouridine*

By D. H. Everaert, O. M. Peeters, N. M. Blaton and C. J. De Ranter†

Laboratorium voor Analytische Chemie en Medicinale Fysicochemie,
Instituut voor Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4,
B-3000 Leuven, Belgium

and A. Van Aerschot and P. Herdewijn

Laboratorium voor Farmaceutische Chemie, Rega Institute for Medical Research,
Katholieke Universiteit Leuven, B-3000 Leuven, Belgium

Abstract. (I) 1-(3-Fluoro-2,3-dideoxy-β-D-*erythro*-pentofuranosyl)uracil, $C_9H_{11}FN_2O_4$, $M_r = 230.20$, monoclinic, $P2_1$, $a = 9.046(5)$, $b = 6.994(4)$, $c = 15.826(8)$ Å, $\beta = 101.19(4)°$, $V = 982.3(9)$ Å^3, $Z = 4$, $D_m = 1.56$, $D_x = 1.557$ Mg m^{-3}, λ(Mo $K\alpha$ radiation) $= 0.71073$ Å, $\mu = 0.127$ mm^{-1}, $F(000) = 480$, $T = 290$ K, final $R = 0.029$ for 1495 unique observed reflections. The asymmetric unit contains two molecules (A and B). For molecule A: the N-glycosidic torsion angle χ has a value of $-121.1(2)°$ in the *anti* range; the sugar pucker is ^2E with $P = 165(1)°$ and $\psi_m = 35(1)°$ and the C4'—C5' conformation is $+sc$ (synclinal) with $\gamma = 51.3(3)°$. For molecule B: the N-glycosidic torsion angle χ has a value of $-150.5(2)°$ in the *anti* range; the sugar pucker is ^2_3T with $P = 176(1)°$ and $\psi_m = 26(1)°$ and the C4'—C5' conformation is $+sc$ with $\gamma = 47.4(3)°$. (II) 1-(3-Fluoro-2,3-dideoxy-β-D-*erythro*-pentofuranosyl)-5-bromouracil, $C_9H_{10}BrFN_2O_4$, $M_r = 309.09$, monoclinic, $P2_1$, $a = 5.701(4)$, $b = 18.192(9)$, $c = 10.538(5)$ Å, $\beta = 95.67(5)°$, $V = 1088(1)$ Å^3, $Z = 4$, $D_m = 1.89$, $D_x = 1.888$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0.71073$ Å, $\mu = 3.759$ mm^{-1}, $F(000) = 616$, $T = 290$ K, final $R = 0.034$ for 1597 unique observed reflections. The asymmetric unit contains two molecules (A and B). For molecule A: the N-glycosidic torsion angle χ has a value of $-168.8(6)°$ in the *anti* range; the sugar pucker is ^2_3T with $P = 179(1)°$ and $\psi_m = 31(1)°$ and the C4'—C5' conformation is $+sc$ with $\gamma = 53.9(9)°$. For molecule B: the N-glycosidic torsion angle χ has a value of $-131.8(7)°$ in the *anti* range; the sugar pucker is ^2E with $P = 161(1)°$ and $\psi_m = 35(1)°$ and the C4'—C5' conformation is $+sc$ with $\gamma = 51.2(9)°$. (III) 1-(3-Azido-2,3-dideoxy-β-D-*erythro*-pentofuranosyl)-5-bromouracil, $C_9H_{10}BrN_5O_4$, $M_r = 332.11$, monoclinic, $P2_1$, $a = 5.858(3)$, $b = 11.813(6)$, $c = 17.757(9)$ Å, $\beta = 92.70(4)°$, $V = 1227(1)$ Å^3, $Z = 4$, $D_m = 1.80$, $D_x = 1.797$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0.71073$ Å, $\mu = 3.332$ mm^{-1}, $F(000) = 664$, $T = 290$ K, final $R = 0.027$ for 1713 unique observed reflections. The asymmetric unit contains two molecules (A and B). For molecule A: the N-glycosidic torsion angle χ has a value of $-168.4(4)°$ in the *anti* range; the sugar pucker is $_3T^4$ with $P = 203(1)°$ and $\psi_m = 37(1)°$ and the C4'—C5' conformation is $+sc$ with $\gamma = 172.1(5)°$. For molecule B: the N-glycosidic torsion angle χ has a value of $-129.8(5)°$ in the *anti* range; the sugar pucker is $^2T_3$ with $P = 173(1)°$ and $\psi_m = 33(1)°$ and the C4'—C5' conformation is ap (antiperiplanar) with $\gamma = 50.5(7)°$. All the conformational parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–