

C6—C5—S8	120.5 (3)	C25—C24—C27	122.3 (3)
C5—C6—C7	120.5 (3)	C20—N26—C25	119.2 (3)
C5—S8—N11	110.4 (3)	C24—C27—C28	116.2 (3)
C5—S8—O10	106.6 (3)	C27—C28—C39	121.0 (3)
C5—S8—O9	106.6 (3)	C27—C28—C29	119.1 (3)
O10—S8—N11	112.8 (3)	C29—C28—C39	119.8 (3)
O9—S8—N11	105.3 (3)	C28—C29—C30	120.2 (3)
O9—S8—O10	115.0 (3)	C29—C30—C33	120.0 (3)
S8—N11—C12	118.4 (3)	C29—C30—O31	124.0 (3)
N11—C12—N18	127.2 (3)	O31—C30—C33	116.0 (3)
N11—C12—C13	120.6 (3)	C30—O31—C32	117.3 (3)
C13—C12—N18	112.2 (3)	C30—C33—C36	119.8 (3)
C12—C13—N16	116.7 (3)	C30—C33—O34	118.8 (3)
C12—C13—O14	119.5 (3)	O34—C33—C36	121.3 (3)
O14—C13—N16	124.7 (3)	C33—O34—C35	115.0 (3)
C13—O14—C15	115.8 (3)	C33—C36—C39	120.2 (3)
C13—N16—S17	106.3 (3)	C33—C36—O37	115.6 (3)
N16—S17—N18	98.4 (3)	O37—C36—C39	124.3 (3)
C12—N18—S17	107.3 (3)	C36—O37—C38	118.0 (3)
N19—C20—N26	117.7 (3)	C28—C39—C36	119.9 (3)
N19—C20—N21	119.5 (3)		

Lattice parameters were measured with Philips LAT routine. Scan speed was $0.05^\circ \text{ s}^{-1}$ with a scan width of 2.0° (intensities negligible at $\theta > 22^\circ$). Corrections for Lorentz-polarization effects were applied. The structure was solved by direct methods (MULTAN80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Coordinates of H atoms were calculated with geometrical considerations (XANADU; Roberts & Sheldrick, 1979), confirmed by a final ΔF synthesis and included in the last refinement. Refinement included anisotropic non-H atoms, isotropic H atoms, a scale factor and a secondary-extinction parameter. A locally modified version of ORFLS (Busing, Martin & Levy, 1962) and the program PARST (Nardelli, 1983) were used. Fig. 2 was drawn with ORTEPII (Johnson, 1976).

This work was partially supported by a grant from CNR to GPB.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCR (Reference: NA1061). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Molecular Co-Crystals of Carboxylic Acids. 16.† 1:1 Adduct of 3-Nitrobenzoic Acid with 3-Amino-1*H*-1,2,4-triazole

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Abstract

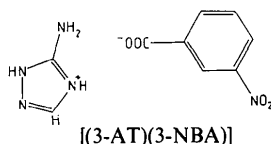
The structure of the 1:1 molecular adduct of 3-nitrobenzoic acid with the herbicide amitrole (3-amino-1*H*-1,2,4-triazole), 3-amino-1*H*,4*H*⁺-1,2,4-triazolium 3-nitrobenzoate, $\text{C}_2\text{H}_5\text{N}_4^+ \cdot \text{C}_7\text{H}_4\text{NO}_4^-$, [(3-AT)(3-NBA)], has been determined by X-ray diffraction. There are two independent but similar cyclic hydrogen-bonded hetero-dimers in the crystallographic asymmetric unit, with hydrogen bonds involving the carboxyl O atoms of the acid and both a heterocyclic N atom and an amine N atom of an amitrole [O...N 2.677–2.951 (3) Å]. The dimers also form into a ribbon-chain polymer structure.

Comment

In a series of studies of the formation of molecular adducts of commercially important herbicides and pesticides with suitable donor or acceptor molecules, amitrole (3-amino-1*H*-1,2,4-triazole, or 3-AT; CA Registry No. 61-82-5) has proved useful, complexing with certain nitro-substituted benzoic acids. The structures of co-crystals of 3-AT with 3,5-dinitrobenzoic acid, [(3-AT)(DNBA)(H₂O)₂] (Lynch, Smith, Byriel & Kennard, 1992), and 4-nitrobenzoic acid, [(3-AT)(4-NBA)] (Byriel, Kennard, Lynch, Smith & Thompson, 1992), are known. 3-Nitrobenzoic acid

† Part 15: Lynch, Smith, Byriel & Kennard (1994).

(3-NBA) is known to have one stable and two unstable polymorphs (Davies & Hartshorne, 1934). The structure of the stable form (Dhaneshwar, Tavole & Part, 1974) has two independent hydrogen-bonded dimer molecules in the asymmetric unit each with the nitro group in the *trans* conformation. The structure of only one of the unstable forms is known (Dhaneshwar, Kulkarni, Tavole & Part, 1975), which also has two independent dimer molecules in the asymmetric unit but with the nitro groups in the *cis* conformation. In this study we report the structure of the 1:1 molecular adduct of 3-nitrobenzoic acid with amitrole, [(3-AT)(3-NBA)]. This allows a comparison of the structural features of both homo- and hetero-associated 3-nitrobenzoic acid molecules.



The structure of the title compound comprises two independent but similar 1:1 hydrogen-bonded cyclic hetero-dimers in the asymmetric unit [graph set $R_2^2(8)$ (Etter, 1990)], involving 3-aminotriazole (molecules *A* and *C*) and 3-nitrobenzoic acid (molecules *B* and *D*). The eight-membered hydrogen-bonded ring [N(31)⋯O(10) 2.889 (3) Å, symmetry operation $\frac{1}{2} + x, \frac{1}{2} - y, z$ (*AB*), 2.951 (3) Å, symmetry operation $\frac{1}{2} - x, \frac{1}{2} + y, 1 - z$ (*CD*); N(4)⋯O(11) 2.707 (3) Å, symmetry operation $\frac{1}{2} + x, \frac{1}{2} - y, z$ (*AB*), 2.677 (3) Å, symmetry operation $\frac{1}{2} - x, \frac{1}{2} + y, 1 - z$ (*CD*)] is similar to that observed for the 3,5-dinitrobenzoic acid and 4-nitrobenzoic acid adducts with 3-AT. Similar to the [(3-AT)(DNBA)(H₂O)₂] adduct (Lynch *et al.*, 1992) the acid forms a dimer through the N atoms N(31) and N(4) of 3-AT, whereas in [(3-AT)(4-NBA)], 4-NBA forms the dimer through N(31) and N(2). The two independent pairs are involved in similar hydrogen-bonded chain networks with other symmetry generated pairs. No hydrogen bonding occurs between the *AB* and *CD* pairs. A hydrogen bond *via* N(31) completes a ribbon polymer [graph set $C(6)$] down the *a* cell direction [N(31)⋯O(11) 2.995 (3) (*AB*), 3.022 (3) Å, symmetry operation $1 - x, -y, 1 - z$ (*CD*)]. A spiral-chain polymer [graph set $C(6)$] extends down the *b* cell direction, this time linked through N(2) [N(2)⋯O(10) 2.684 (3) Å, symmetry operation $-x, -y, -z$ (*AB*), 2.684 (3) Å (*CD*)]. For the 3-NBA molecule, the dihedral angles between the benzene ring and the carboxylic group are 11.0 (2) (*B*) and 3.8 (2)° (*D*), *cf.* 4.5 and 1.6° (3-NBA, stable form, two molecules) (Dhaneshwar *et al.*, 1974), and 6.0 and 4.2° (two molecules) (Dhaneshwar *et al.*, 1975); those between the benzene ring and the nitro group are 3.7 (2) (*B*) and 11.3 (2)° (*D*), *cf.* 21.7 and 5.3°

(3-NBA, unstable form, two molecules) (Dhaneshwar *et al.*, 1974), and 2.7 and 1.3° (two molecules) (Dhaneshwar *et al.*, 1975).

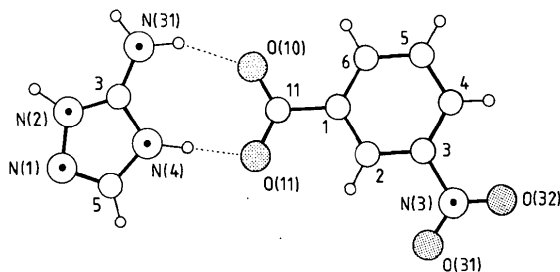


Fig. 1. The molecular conformation and atom-numbering scheme for the individual molecules of amitrole (*A* and *C*) and 3-nitrobenzoic acid (*B* and *D*) in the adduct.

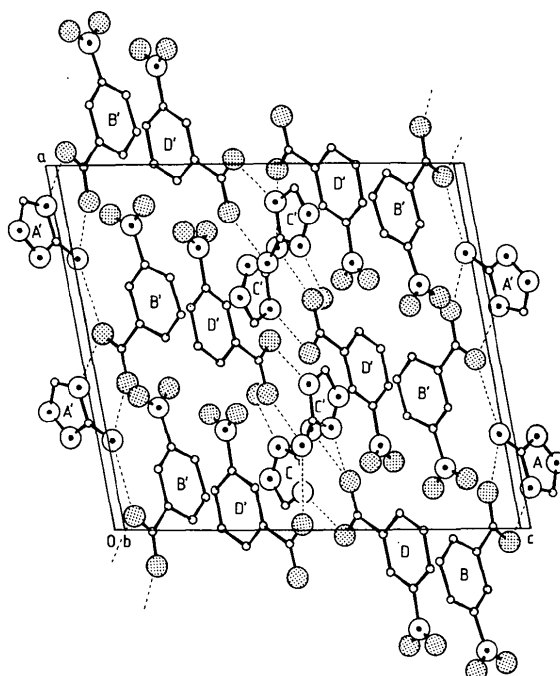


Fig. 2. Packing of the molecular adduct [(3-AT)(3-NBA)] in the unit cell.

Experimental

The title compound was prepared by refluxing equimolar amounts of 3-nitrobenzoic acid and 3-amino-1*H*-1,2,4-triazole in ethanol for 20 min at 423 K. Colourless prisms (m.p. 432–434 K) formed upon total evaporation of the solvent at room temperature.

Crystal data

$C_2H_5N_4^+ \cdot C_7H_4NO_4^-$
 $M_r = 251.2$
 Monoclinic
 $P2_1/a$
 $a = 13.316$ (5) Å
 $b = 11.192$ (1) Å
 $c = 14.935$ (5) Å

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 6-14^\circ$
 $\mu = 0.12$ mm⁻¹
 $T = 295$ K

$\beta = 100.39 (2)^\circ$
 $V = 2189 (2) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.524 \text{ Mg m}^{-3}$

Prismatic
 $0.36 \times 0.34 \times 0.10 \text{ mm}$
 Colourless

N(3D)	0.7720 (2)	0.0762 (2)	0.3091 (2)	0.0556 (9)
O(31D)	0.8168 (2)	-0.0143 (2)	0.3402 (2)	0.0826 (10)
O(32D)	0.8151 (2)	0.1593 (2)	0.2799 (2)	0.0902 (10)
C(4D)	0.6083 (2)	0.1763 (3)	0.2583 (2)	0.0493 (10)
C(5D)	0.5050 (2)	0.1845 (3)	0.2585 (2)	0.0538 (10)
C(6D)	0.4581 (2)	0.1026 (2)	0.3076 (2)	0.0441 (9)

Data collection

Enraf-Nonius CAD-4
 diffractometer

$2\theta/\omega$ scans

Absorption correction:
 empirical

$T_{\min} = 0.100$, $T_{\max} =$
 0.947

4248 measured reflections

3838 independent reflections

2319 observed reflections

$[I > 2.5\sigma(I_o)]$

$R_{\text{int}} = 0.0148$

$\theta_{\max} = 25^\circ$

$h = 0 \rightarrow 7$

$k = -10 \rightarrow 10$

$l = -10 \rightarrow 10$

3 standard reflections

monitored every 250

reflections

intensity variation: -2.6%

Refinement

Refinement on F

$R = 0.036$

$wR = 0.040$

$S = 1.05$

2319 reflections

397 parameters

All H-atom parameters

refined

$w = 1/[\sigma^2(F_o)$
 $+ 0.00139(F_o)^2]$

$(\Delta/\sigma)_{\max} = 0.01$

$\Delta\rho_{\max} = 0.14 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	x	y	z	U_{eq}
N(1A)	0.3235 (2)	0.0259 (2)	-0.1078 (2)	0.0479 (8)
N(2A)	0.2515 (2)	0.0838 (2)	-0.0679 (1)	0.0393 (7)
C(3A)	0.2914 (2)	0.1760 (2)	-0.0185 (2)	0.0335 (7)
N(31A)	0.2445 (2)	0.2503 (2)	0.0294 (2)	0.0483 (8)
N(4A)	0.3902 (1)	0.1813 (2)	-0.0269 (1)	0.0371 (7)
C(5A)	0.4047 (2)	0.0884 (3)	-0.0821 (2)	0.0469 (9)
C(1B)	0.0652 (2)	-0.0081 (2)	0.1447 (2)	0.0305 (7)
C(11B)	-0.0036 (2)	0.0847 (2)	0.0920 (2)	0.0313 (2)
O(10B)	-0.0979 (1)	0.0749 (2)	0.0889 (1)	0.0416 (6)
O(11B)	0.0374 (1)	0.1667 (1)	0.0535 (1)	0.0396 (5)
C(2B)	0.1670 (2)	-0.0134 (2)	0.1372 (2)	0.0340 (8)
C(3B)	0.2275 (2)	-0.1015 (2)	0.1820 (2)	0.0359 (8)
N(3B)	0.3343 (2)	-0.1073 (2)	0.1687 (2)	0.0495 (8)
O(31B)	0.3652 (2)	-0.0297 (2)	0.1232 (2)	0.0684 (8)
O(32B)	0.3874 (2)	-0.1892 (2)	0.2047 (2)	0.0792 (9)
C(4B)	0.1920 (2)	-0.1855 (2)	0.2361 (2)	0.0445 (9)
C(5B)	0.0911 (2)	-0.1783 (2)	0.2458 (2)	0.0470 (9)
C(6B)	0.0278 (2)	-0.0909 (2)	0.1998 (2)	0.0406 (8)
N(1C)	0.1370 (2)	0.0283 (2)	0.4035 (2)	0.0618 (9)
N(2C)	0.2246 (2)	0.0904 (2)	0.4382 (2)	0.0460 (8)
C(3C)	0.2042 (2)	0.1878 (2)	0.4813 (2)	0.0378 (8)
N(31C)	0.2706 (2)	0.2673 (2)	0.5224 (2)	0.0529 (9)
N(4C)	0.1024 (2)	0.1920 (2)	0.4744 (1)	0.0440 (7)
C(5C)	0.0658 (2)	0.0931 (3)	0.4265 (2)	0.0580 (11)
C(1D)	0.5133 (2)	0.0102 (2)	0.3553 (2)	0.0330 (7)
C(11D)	0.4628 (2)	-0.0795 (2)	0.4083 (2)	0.0340 (7)
O(10D)	0.3704 (1)	-0.0644 (1)	0.4117 (1)	0.0443 (6)
O(11D)	0.5161 (1)	-0.1643 (2)	0.4460 (1)	0.0443 (6)
C(2D)	0.6163 (2)	0.0009 (2)	0.3532 (2)	0.0351 (8)
C(3D)	0.6615 (2)	0.0845 (2)	0.3062 (2)	0.0382 (8)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Table 2. Selected geometric parameters (\AA , $^\circ$)

N(2A)—N(1A)	1.379 (3)	N(2C)—N(1C)	1.376 (3)
C(5A)—N(1A)	1.287 (3)	C(5C)—N(1C)	1.289 (3)
C(3A)—N(2A)	1.323 (3)	C(3C)—N(2C)	1.319 (3)
N(31A)—C(3A)	1.325 (3)	N(31C)—C(3C)	1.324 (3)
N(4A)—C(3A)	1.345 (3)	N(4C)—C(3C)	1.342 (3)
C(5A)—N(4A)	1.362 (3)	C(5C)—N(4C)	1.360 (3)
C(11B)—C(1B)	1.509 (3)	C(11D)—C(1D)	1.510 (3)
C(2B)—C(1B)	1.382 (3)	C(2D)—C(1D)	1.382 (3)
C(6B)—C(1B)	1.390 (3)	C(6D)—C(1D)	1.389 (3)
O(10B)—C(11B)	1.253 (2)	O(10D)—C(11D)	1.252 (2)
O(11B)—C(11B)	1.259 (2)	O(11D)—C(11D)	1.256 (3)
C(3B)—C(2B)	1.369 (3)	C(3D)—C(2D)	1.372 (3)
N(3B)—C(3B)	1.472 (3)	N(3D)—C(3D)	1.467 (3)
C(4B)—C(3B)	1.378 (3)	C(4D)—C(3D)	1.373 (4)
O(31B)—N(3B)	1.219 (3)	O(31D)—N(3D)	1.224 (3)
O(32B)—N(3B)	1.222 (3)	O(32D)—N(3D)	1.214 (3)
C(5B)—C(4B)	1.379 (4)	C(5D)—C(4D)	1.379 (4)
C(6B)—C(5B)	1.389 (4)	C(6D)—C(5D)	1.390 (4)
C(5A)—N(1A)—N(2A)	103.2 (2)	C(5C)—N(1C)—N(2C)	103.3 (2)
C(3A)—N(2A)—N(1A)	111.5 (2)	C(3C)—N(2C)—N(1C)	111.5 (2)
N(31A)—C(3A)—N(2A)	127.6 (2)	N(31C)—C(3C)—N(2C)	127.0 (2)
N(4A)—C(3A)—N(2A)	106.5 (2)	N(4C)—C(3C)—N(2C)	106.6 (2)
N(4A)—C(3A)—N(31A)	125.9 (2)	N(4C)—C(3C)—N(31C)	126.4 (2)
C(5A)—N(4A)—C(3A)	105.7 (2)	C(5C)—N(4C)—C(3C)	105.9 (2)
N(4A)—C(5A)—N(1A)	113.1 (2)	N(4C)—C(5C)—N(1C)	112.8 (2)
C(2B)—C(1B)—C(11B)	120.0 (2)	C(2D)—C(1D)—C(11D)	120.0 (2)
C(6B)—C(1B)—C(11B)	121.3 (2)	C(6D)—C(1D)—C(11D)	121.3 (2)
C(1B)—C(11B)—O(10B)	118.8 (2)	C(1D)—C(11D)—O(10D)	118.7 (2)
O(10B)—C(11B)—C(1B)	118.0 (2)	O(10D)—C(11D)—C(1D)	117.7 (2)
O(11B)—C(11B)—C(1B)	117.9 (2)	O(11D)—C(11D)—C(1D)	117.9 (2)
O(11B)—C(11B)—O(10B)	124.1 (2)	O(11D)—C(11D)—O(10D)	124.3 (2)
C(3B)—C(2B)—C(1B)	119.4 (2)	C(3D)—C(2D)—C(1D)	119.3 (2)
N(3B)—C(3B)—C(2B)	117.6 (2)	N(3D)—C(3D)—C(2D)	118.2 (2)
C(4B)—C(3B)—C(2B)	123.0 (2)	C(4D)—C(3D)—C(2D)	123.0 (2)
C(4B)—C(3B)—N(3B)	119.4 (2)	C(4D)—C(3D)—N(3D)	118.9 (2)
O(31B)—N(3B)—C(3B)	118.4 (2)	O(31D)—N(3D)—C(3D)	118.8 (2)
O(32B)—N(3B)—C(3B)	118.0 (2)	O(32D)—N(3D)—C(3D)	118.5 (3)
O(32B)—N(3B)—O(31B)	123.6 (2)	O(32D)—N(3D)—O(31D)	122.7 (2)
C(5B)—C(4B)—C(3B)	117.7 (2)	C(5D)—C(4D)—C(3D)	118.0 (2)
C(6B)—C(5B)—C(4B)	120.3 (2)	C(6D)—C(5D)—C(4D)	120.0 (3)
C(5B)—C(6B)—C(1B)	120.8 (2)	C(5D)—C(6D)—C(1D)	121.0 (2)

The data were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refined by full-matrix least squares (*SHELX76*; Sheldrick, 1976) with anisotropic displacement parameters for all non-H atoms. Molecular graphics were prepared using *PLUTO* (Motherwell & Clegg, 1978).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HL1044). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(3-Benzyloxy-4-nitrophenyl)oxirane, an Intermediate in the Synthesis of Formoterol

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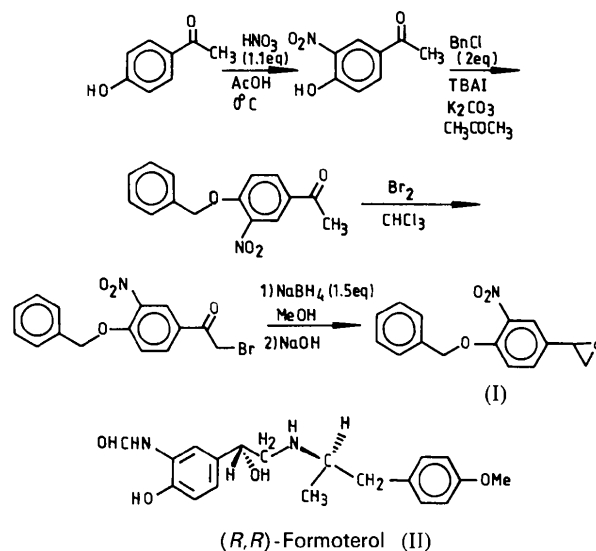
Abstract

In the title compound, C₁₅H₁₃NO₄, the two planar nitrophenyl and benzyloxy groups are inclined at 152.3 (2)°. The torsion angle about the central C—O bond is 177.5 (5)°, giving an extended C—C—O—C chain. The nitro group is twisted out of the plane of the phenyl ring by 28.4 (3)° to diminish steric hindrance; O atoms of the nitro and benzyloxy groups are separated by only 2.625 (7) Å. The dihedral angles between the epoxy ring and the two aromatic rings are 98.3 (3) and 102.6 (3)°. There is a possible C—H⋯O intermolecular interaction.

Comment

The title compound, (I), is one of the intermediates in the synthesis of the anti-asthmatic agent formoterol, (II), a β-adrenoreceptor-stimulating

catecholamine analogue with selective bronchodilator activity (Murase, Mase, Ida, Takahashi & Murakami, 1977; Trofast, Osterberg, Kallstrom & Waldeck, 1991). It was prepared in four stages as shown in the scheme below.



The structure determination of (I) was undertaken to gain insight into the reaction pathways. In all essential details the geometry of the molecule is normal. The dihedral angles between the plane defined by C(6)—O(4)—C(9)—C(10) and the two aromatic rings present in the structure are 2.7 (3) and 149.6 (4)°. The nitro group is twisted 28.4 (3)° out of the plane of the phenyl ring and this gives a short O(4)⋯O(3) intramolecular contact of 2.625 (7) Å; the sum of van der Waals radii for O is 2.80 Å (Pauling, 1960). The O(4)⋯O(3)—N(1) angle is 86.1 (7)°.

One C—H⋯O intermolecular contact [C(9)—H⋯O(1)(y - 3/4, -x + 3/4, -z + 3/4) 3.437 (8) Å] is geometrically appropriate for a C—H⋯O hydrogen bond (Berkovitch-Yellin & Leiserowitz, 1984). Other contacts are all of van der Waals type.

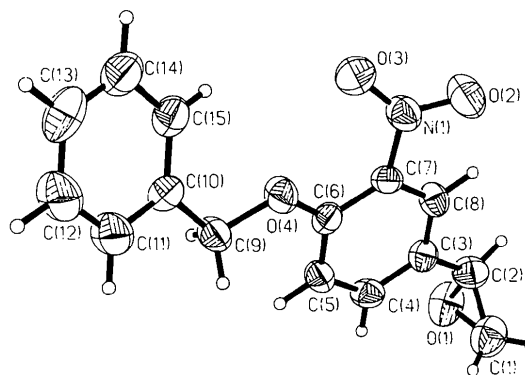


Fig. 1. A perspective view of the molecule with atom labelling.