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endo-2-Norborneol 2,4-Dinitrobenzoate

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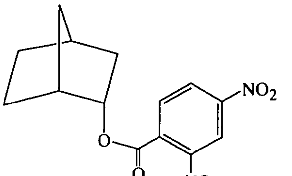
Abstract

The structure of the title compound, $C_{14}H_{14}N_2O_6$ (*endo*-bicyclo[2.2.1]heptan-2-yl 2,4-dinitrobenzoate), was determined at 130 K to obtain an accurate measurement of the C—O bond length for comparison purposes. Steric interactions between the carboxyl function and the 2-nitro group of the ester result in these two groups twisting out of the plane of the aromatic ring. The C(alkyl)—O bond length is 1.465 (2) Å.

Comment

As part of our studies on the factors affecting C—O bond lengths (White & Robertson, 1992), we determined the structure of the title compound, (1), to obtain an accurate measurement of the C—O bond length for comparison purposes.



(1)

The X-ray data were measured at low temperature to remove the unwanted effects of thermal motion. The ester function exists in the *cisoid* conformation as defined by the torsion angle O2—C8—O1—C2 of −3.2 (2)°. Both the carboxyl and 2-nitro groups are rotated out of the plane of the phenyl ring; torsion angle O2—C8—C9—C14 is −34.7, O3—N1—C10—C9 is −51.6 (2) and O4—N1—C10—C11 is −53.7 (2)°. This acts to minimize steric interactions between these two substituents. The 4-nitro group is twisted slightly away from the plane of the phenyl ring; O5—N2—C12—C11 is 13.6 (2)°. The C(alkyl)—O bond length was determined to be 1.465 (2) Å.

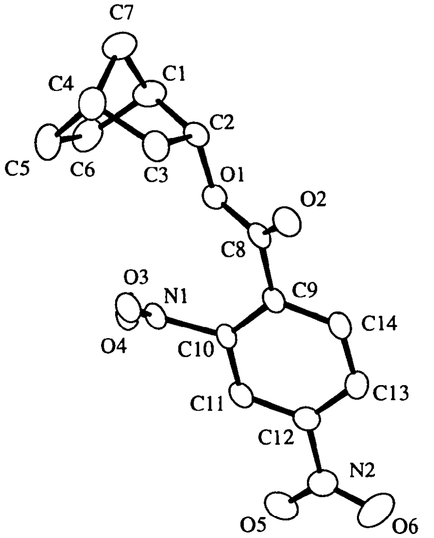


Fig. 1. *ORTEPII* (Johnson, 1976) drawing of (1). Displacement ellipsoids are shown at the 50% probability level.

Experimental

Crystals of (1) were obtained by slow evaporation from pentane.

Crystal data

$C_{14}H_{14}N_2O_6$
$M_r = 306.27$
Orthorhombic
Pbca
$a = 9.0574 (9)$ Å
$b = 11.658 (4)$ Å
$c = 25.697 (4)$ Å
$V = 2713.3 (11)$ Å^3
$Z = 8$
$D_x = 1.500$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13$–$17°$
$\mu = 0.119$ mm^{-1}
$T = 130 (1)$ K
Block
0.57 × 0.50 × 0.27 mm
Colourless

Data collection

CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4693 measured reflections
3946 independent reflections
3015 observed reflections [$I > 2\sigma(I)$]

$R_{\text{int}} = 0.0170$
$\theta_{\max} = 29.96°$
$h = -1 \rightarrow 12$
$k = -1 \rightarrow 16$
$l = -1 \rightarrow 36$
3 standard reflections frequency: 160 min intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0437$
$wR(F^2) = 0.1163$
$S = 1.132$
3946 reflections
256 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 1.0647P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.420$ e Å^{-3}
$\Delta\rho_{\min} = -0.247$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0012 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
C1	0.2260 (2)	0.2506 (2)	0.41977 (7)	0.0338 (4)
C2	0.0978 (2)	0.24206 (13)	0.38140 (6)	0.0262 (3)
C3	−0.0301 (2)	0.30396 (14)	0.40934 (7)	0.0298 (3)
C4	0.0351 (2)	0.33044 (15)	0.46322 (7)	0.0334 (3)
C5	0.1414 (2)	0.4317 (2)	0.45661 (8)	0.0390 (4)
C6	0.2716 (2)	0.3773 (2)	0.42670 (7)	0.0365 (4)
C7	0.1412 (2)	0.2295 (2)	0.47087 (7)	0.0396 (4)
C8	0.0385 (2)	0.28715 (11)	0.29394 (6)	0.0234 (3)
C9	0.08702 (14)	0.34711 (11)	0.24513 (6)	0.0219 (3)
C10	0.16768 (15)	0.44910 (11)	0.24419 (5)	0.0210 (3)
C11	0.21216 (15)	0.50139 (11)	0.19867 (6)	0.0234 (3)
C12	0.1692 (2)	0.45034 (12)	0.15267 (6)	0.0246 (3)
C13	0.0901 (2)	0.34868 (13)	0.15106 (6)	0.0275 (3)
C14	0.0500 (2)	0.29782 (12)	0.19738 (6)	0.0260 (3)
N1	0.20135 (14)	0.51181 (10)	0.29229 (5)	0.0254 (3)
N2	0.20700 (15)	0.50911 (12)	0.10373 (5)	0.0304 (3)
O1	0.13831 (11)	0.29572 (8)	0.33186 (4)	0.0242 (2)
O2	−0.07677 (12)	0.23594 (10)	0.29665 (5)	0.0308 (3)
O3	0.09855 (13)	0.53387 (9)	0.32155 (5)	0.0325 (3)
O4	0.32971 (14)	0.54062 (12)	0.29909 (5)	0.0406 (3)
O5	0.29734 (14)	0.58721 (10)	0.10565 (5)	0.0383 (3)
O6	0.1451 (2)	0.47725 (14)	0.06423 (5)	0.0495 (4)

Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.526 (2)	C9—C14	1.395 (2)
C1—C7	1.541 (2)	C9—C10	1.396 (2)
C1—C6	1.544 (3)	C10—C11	1.379 (2)
C2—O1	1.465 (2)	C10—N1	1.468 (2)
C2—C3	1.542 (2)	C11—C12	1.379 (2)
C3—C4	1.536 (2)	C12—C13	1.385 (2)
C4—C7	1.533 (3)	C12—N2	1.472 (2)
C4—C5	1.533 (3)	C13—C14	1.378 (2)
C5—C6	1.544 (3)	N1—O4	1.223 (2)
C8—O2	1.204 (2)	N1—O3	1.224 (2)
C8—O1	1.333 (2)	N2—O6	1.218 (2)
C8—C9	1.502 (2)	N2—O5	1.225 (2)
C2—C1—C7	99.26 (13)	C10—C9—C8	124.35 (13)
C2—C1—C6	109.91 (13)	C11—C10—C9	122.96 (13)
C7—C1—C6	100.83 (15)	C11—C10—N1	115.68 (11)
O1—C2—C1	110.06 (12)	C9—C10—N1	121.23 (13)
O1—C2—C3	113.13 (12)	C10—C11—C12	116.97 (13)
C1—C2—C3	103.90 (12)	C11—C12—C13	122.72 (14)
C4—C3—C2	103.00 (13)	C11—C12—N2	117.73 (13)
C7—C4—C5	102.20 (15)	C13—C12—N2	119.53 (14)
C7—C4—C3	101.67 (13)	C14—C13—C12	118.57 (14)
C5—C4—C3	107.23 (14)	C13—C14—C9	121.26 (13)
C4—C5—C6	102.64 (15)	O4—N1—O3	125.30 (13)
C1—C6—C5	104.24 (14)	O4—N1—C10	117.04 (12)
C4—C7—C1	94.61 (13)	O3—N1—C10	117.63 (12)
O2—C8—O1	125.63 (14)	O6—N2—O5	124.60 (15)
O2—C8—C9	122.20 (14)	O6—N2—C12	117.56 (14)
O1—C8—C9	112.14 (12)	O5—N2—C12	117.84 (13)
C14—C9—C10	117.46 (13)	C8—O1—C2	115.68 (11)
C14—C9—C8	118.19 (12)		

Data were collected at 130 K using an Oxford Cryostream low-temperature cooling device. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). Refinement was performed by *SHELXL*93 (Sheldrick, 1993) using full-matrix least squares, with anisotropic displacement parameters for all non-H atoms. H atoms were refined without constraint. The figure was generated using *ORTEP*II (Johnson, 1976) and tables prepared using *SHELXL*93. All calculations were carried out on a VAX 4000VLC computer system.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1023). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-D-Glucopyranosyl-*S*-phenylsulfenamide: Structure and Evaluation as a Novel β-Glucosidase Inhibitor

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Abstract

The structure of 2,3,4,6-tetra-*O*-acetyl-*N*-β-D-glucopyranosyl-*S*-phenylsulfenamide, $C_{20}H_{25}NO_9S$, at low temperature is reported. The glucopyranosyl residue adopts

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