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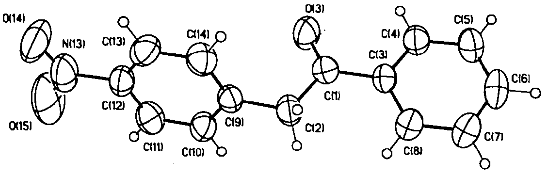

Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the molecule showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

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Structure of 2-(4-Nitrophenyl)-1-phenylethanone

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Abstract

The plane through the two central bridging C atoms and the carbonyl O atom is nearly coplanar with the phenyl ring [6.6(1)°], and forms a dihedral angle of 78.91(9)° with the plane of the six-membered ring of the 4-nitrophenyl group and a dihedral angle of 82.6(1)° with the nitro group. The carbonyl C atom in the bridging group is sp^2 hybridized; the bond angles are 117.9(2), 121.0(2) and 121.0(2)° and the C—C—C bond angle at the methylene C atom of the bridge is 114.1(2)°. The bond distances in this molecule are in the expected range indicating almost no interaction between the carbonyl group and the 4-nitrophenyl moiety.

Comment

The structure determination of the title compound was undertaken in order to obtain more structural information about deoxybenzoin derivatives and the effects of substituents in different positions on their conformations. This information is significant because this skeleton forms a part of many natural compounds and the title compound can easily be transformed into the corresponding carbanion by removal of one proton from the methylene group. These carbanions are of great interest as intermediate species in many chemical reactions. We are interested in the structural and electronic effects of the replacement of the methylene H atoms by O atoms.

Experimental

Crystal data

$C_{14}H_{11}NO_3$
$M_r = 241.25$
Monoclinic
$P2_1/c$
$a = 14.662\ (4)$ Å
$b = 10.790\ (2)$ Å
$c = 7.749\ (2)$ Å
$\beta = 104.55\ (2)°$
Cell parameters from 20 reflections
$\theta = 7.50–14.35°$
$V = 1186.6\ (5)$ Å^3
$Z = 4$
$D_x = 1.350$ Mg m^{-3}
Mo $K\alpha$
$\lambda = 0.71073$ Å
$\mu = 0.09$ mm^{-1}
$T = 293\ (1)$ K
0.60 × 0.40 × 0.24 mm
Colourless

Data collection

Nicolet *R*3*m*/*V* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1981 measured reflections
1425 independent reflections
1068 observed reflections [$F > 4.0\sigma(F)$]
$R_{\rm int} = 0.028$
$\theta_{\rm max} = 22.5°$
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 9$
6 standard reflections
frequency: 150 min
intensity variation: 0%

Refinement

Refinement on F
Final $R = 0.044$
$wR = 0.048$
$S = 1.21$
1068 reflections
164 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F) + 0.0010F^2]$
$(\Delta/\sigma)_{\rm max} = 0.027$
$\Delta\rho_{\rm max} = 0.1$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.1$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data collection: Nicolet *R*3*m*/*V* software, release 4.11. Cell refinement: Nicolet *R*3*m*/*V* software. Data reduction: Nicolet *R*3*m*/*V* software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus*, *SHELX*76 (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987).

The title compound was obtained from deoxybenzoin and HNO_3 according to Womack, Campbell & Dodds (1938). The product was purified by multifold recrystallization from anhydrous ethanol (m.p. 418–419 K). The IR spectrum of the title compound shows a band at 1687 cm^{-1} in the carbonyl stretching region while the corresponding Raman band is at 1694 cm^{-1}. Intensity data were collected with variable scan speed 3.5–15.0°

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

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

	x	y	z	U_{eq}
C(1)	0.1467 (2)	0.3577 (3)	0.1873 (3)	0.0484
C(2)	0.2151 (2)	0.4397 (3)	0.3165 (4)	0.0563
O(3)	0.1746 (1)	0.2779 (2)	0.1028 (3)	0.0743
C(3)	0.0439 (2)	0.3756 (2)	0.1710 (3)	0.0438
C(4)	−0.0195 (2)	0.2926 (2)	0.0672 (4)	0.0546
C(5)	−0.1150 (2)	0.3032 (3)	0.0531 (4)	0.0626
C(6)	−0.1477 (2)	0.3969 (3)	0.1418 (4)	0.0645
C(7)	−0.0866 (2)	0.4819 (3)	0.2413 (4)	0.0609
C(8)	0.0096 (2)	0.4709 (2)	0.2566 (4)	0.0531
C(9)	0.3169 (2)	0.4175 (2)	0.3198 (3)	0.0458
C(10)	0.3632 (2)	0.4953 (3)	0.2291 (4)	0.0553
C(11)	0.4574 (2)	0.4779 (3)	0.2354 (4)	0.0610
C(12)	0.5043 (2)	0.3824 (3)	0.3341 (4)	0.0538
C(13)	0.4607 (2)	0.3033 (3)	0.4241 (4)	0.0623
C(14)	0.3663 (2)	0.3206 (3)	0.4163 (4)	0.0599
N(13)	0.6055 (2)	0.3662 (3)	0.3463 (4)	0.0839
O(14)	0.6480 (2)	0.2864 (3)	0.4448 (4)	0.1201
O(15)	0.6415 (2)	0.4368 (3)	0.2590 (4)	0.1316

Table 2. *Geometric parameters* (Å, °)

C(1)—C(2)	1.512 (3)	C(9)—C(10)	1.377 (4)
C(1)—O(3)	1.214 (4)	C(9)—C(14)	1.380 (4)
C(1)—C(3)	1.494 (3)	C(10)—C(11)	1.383 (4)
C(2)—C(9)	1.506 (4)	C(11)—C(12)	1.363 (4)
C(3)—C(4)	1.392 (3)	C(12)—C(13)	1.359 (4)
C(3)—C(8)	1.385 (4)	C(12)—N(13)	1.473 (4)
C(4)—C(5)	1.380 (4)	C(13)—C(14)	1.382 (4)
C(5)—C(6)	1.375 (4)	N(13)—O(14)	1.214 (4)
C(6)—C(7)	1.375 (4)	N(13)—O(15)	1.223 (5)
C(7)—C(8)	1.389 (4)		
O(3)—C(1)—C(3)	121.0 (2)	C(2)—C(9)—C(10)	120.5 (2)
C(2)—C(1)—C(3)	117.9 (2)	C(10)—C(9)—C(14)	118.7 (3)
C(2)—C(1)—O(3)	121.0 (2)	C(9)—C(10)—C(11)	121.0 (3)
C(1)—C(2)—C(9)	114.1 (2)	C(10)—C(11)—C(12)	118.8 (3)
C(1)—C(3)—C(8)	122.4 (2)	C(11)—C(12)—N(13)	118.9 (3)
C(1)—C(3)—C(4)	118.7 (2)	C(11)—C(12)—C(13)	121.8 (3)
C(4)—C(3)—C(8)	119.0 (2)	C(13)—C(12)—N(13)	119.3 (3)
C(3)—C(4)—C(5)	120.6 (3)	C(12)—C(13)—C(14)	119.2 (3)
C(4)—C(5)—C(6)	119.7 (3)	C(9)—C(14)—C(13)	120.6 (3)
C(5)—C(6)—C(7)	120.7 (3)	C(12)—N(13)—O(15)	117.3 (3)
C(6)—C(7)—C(8)	119.7 (3)	C(12)—N(13)—O(14)	118.3 (3)
C(3)—C(8)—C(7)	120.3 (3)	O(14)—N(13)—O(15)	124.4 (3)
C(2)—C(9)—C(14)	120.8 (2)		

min^{-1} in θ. The lattice parameters were determined from a symmetry-constrained least-squares fit. Refinement was based on full-matrix least-squares methods with H atoms in calculated positions (C—H 0.96 Å).

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates, and a stereoscopic view of the compound have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54995 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1007]

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Structural Aspects of Reactions Based on 'Aggregative Activation': Structures of 3-Oxatricyclo[6.3.0.0^{2,7}]undec-6-en-1-ol and 6,7-Epoxy-3-oxatricyclo[6.4.0.0^{2,7}]dodecan-1-ol

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Abstract

The preparation of the title compounds is described. The former was obtained by an 'aggregative activation' reaction of dehydrodihydropyran with bases, and the latter by epoxidation of one of the so-obtained alcohols. Structure analysis shows that complexation of the oxidizing agent by the hydroxyl group directs the stereoselectivity of epoxidation. The influence of epoxidation on the conformation of the three-ring system in these molecules is discussed. The rigidity of the molecules and hydrogen bonding explain the absence of significant anisotropies in the atomic displacements, so a satisfactory description of the displacements is obtained by the rigid-body model.

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