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5′-Allyl-2′-benzoyloxy-3′-methoxy-4-nitroazobenzene

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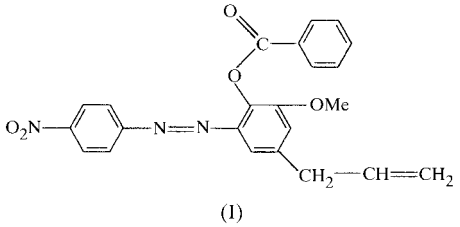
The structure of the title compound, 4-allyl-2-methoxy-6-[(4-nitrophenyl)diazenyl]phenyl benzoate, $C_{23}H_{19}N_3O_5$, displays the characteristic features of azobenzene derivatives. The azobenzene moiety of the molecule has a *trans* configuration and in this moiety, average C—N and N═N bond lengths are 1.441 (3) and 1.241 (3) Å, respectively.

Comment

The crystal and molecular structures of the title compound, (I), were investigated in order to determine the conformation, crystal packing and also to confirm the stereochemistry. In the molecule, bond lengths and angles are very similar to the structures of azo compounds studied previously (Işık *et al.*, 1997; Işık, Aygün, Kocaokutgen, Tahir *et al.*, 1998; Işık, Aygün, Kocaokutgen & Tahir, 1998).



(I)

The numbering scheme and a displacement ellipsoid plot of (I) are shown in Fig. 1. In the azo frame, the average C—N bond length is 1.441 (3) Å, indicating the single-bond character. The C—NO_2 bond length is 1.479 (4) Å, which is similar to the observed values in the literature (Zhu *et al.*, 1996; Zhang *et al.*, 1998). The N2═N3 bond length of 1.241 (3) Å is typical for a double bond. The angles C4—N2—N3 and N2—N3—C7 are 114.0 (2) and 113.7 (2)°, respectively. The torsion angles about the C—N bond [N3—N2—C4—C3 = 178.9 (2) and N3—N2—C4—C5 = −1.9 (4)°] are comparable with *trans* azo compounds. The three phenyl rings are planar within

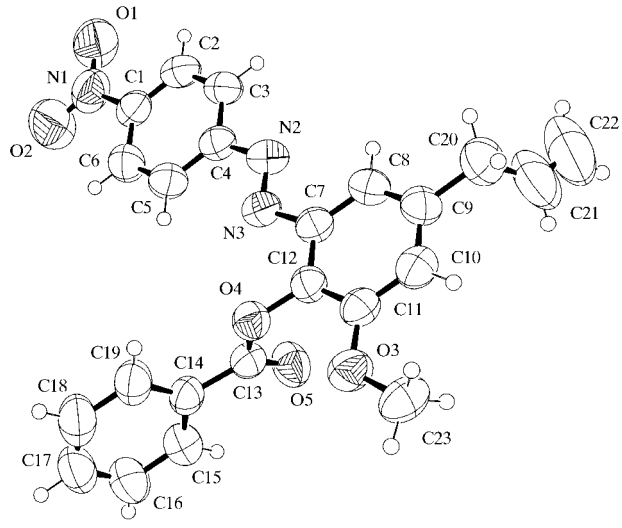


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and atom-numbering scheme.

experimental error and the largest deviation of −0.010 (2) Å is at the C18 atom of the C14–C19 ring. While the twist angle of the nitro group relative to the phenyl ring is 3.7 (1)°, the C1–C6 and C7–C12 rings in the azobenzene frame are twisted by 1.83 (2) and 1.68 (2)° out of the azo group plane, respectively. These results show that the azo group moiety is almost planar. The C7–C12 ring and the other groups adjacent to the C7–C12 ring, except the methoxy group, are not coplanar. Also, dihedral angles between the C14–C19 ring and the C1–C6, and C7–C12 rings are 97.7 (1) and 96.2 (1)°, respectively.

Displacement parameters of atoms C20, C21 and C22 of the allyl group are larger than for other atoms. This behaviour may be due to considerable freedom of movement of the group in the crystal. Also, this accounts for the apparent shortening of the C21—C22 bond length [1.245 (5) Å].

Experimental

5′-Allyl-2′-hydroxy-3′-methoxy-4-nitroazobenzene (1 g, 3.19 mmol) was dissolved in aqueous NaOH solution (1 *M*, 100 ml) by heating. After cooling the solution, benzoyl chloride was added, and the mixture was refluxed for 1 h. After cooling, a precipitate was obtained when Na_2O_3 (1 g, 9.43 mmol) was added. The precipitate was washed with pure water, filtered and dried. Diffraction-quality crystals were obtained by recrystallization from ethyl acetate (m.p. 428–434 K). ^{1}H NMR ($CDCl_3$): δ 8.5–7.0 (*m*, aromatic), 6.2–5.8 (*m*, —CH═), 5.4–5.1 (*m*, ═CH_2), 3.9 (*s*, —CH_3), 3.6–3.4 (*s*, —CH_2); ^{13}C NMR ($CDCl_3$): δ 170 (C═O), 145–120 (aromatic), 140–130 (—CH═), 116–112 (═CH_2), 54.3 (—OCH_3), 40–10 (—CH_2); IR (cm^{-1}): 3080–2800 (CH_2, CH), 1635 (allyl, C═C), 1450–1400 (N═N), 1600–1450 (C_6H_5), 1385 (OCH_3), 1740 (C═O); UV [λ (nm), ε (mol^{-1} cm^{-1})]: 271.1 (6268.1), 241.4 (9664.8), 206.2 (1229.05); analysis calculated for $C_{23}H_{19}N_3O_5$ (%): C 68.18, H 4.55, N 10.07; found (%): C 68.19, H 4.62, N 10.02.

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.213 (3)	N2—N3	1.241 (3)
O2—N1	1.209 (4)	N2—C4	1.440 (3)
N1—C1	1.479 (4)	N3—C7	1.441 (3)
C11—O3—C23	117.7 (2)	N2—N3—C7	113.7 (2)
O2—N1—O1	123.5 (3)	O5—C13—O4	122.0 (3)
N3—N2—C4	114.0 (2)		
N3—N2—C4—C3	178.9 (2)	C23—O3—C11—C10	1.3 (4)
N3—N2—C4—C5	−1.9 (4)	C12—O4—C13—O5	0.4 (4)

Crystal data

$C_{23}H_{19}N_3O_5$	D_x = 1.286 Mg m^{-3}
M_r = 417.41	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 4481 reflections
a = 7.7564 (1) Å	θ = 1.5–33°
b = 22.3441 (5) Å	μ = 0.092 mm^{-1}
c = 12.5552 (2) Å	T = 293 (2) K
β = 97.766 (1)°	Slab, light yellow
V = 2155.98 (7) Å^3	0.32 × 0.24 × 0.08 mm
Z = 4	

Data collection

Siemens SMART CCD area-detector diffractometer	1920 reflections with $I > 2\sigma(I)$
	R_{int} = 0.073
ω scans	θ_{max} = 28.28°
Absorption correction: none	$h = -10 \rightarrow 8$
15921 measured reflections	$k = -14 \rightarrow 29$
5166 independent reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0819P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.066	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.197	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
S = 0.958	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
5166 reflections	Extinction correction: *SHELXL*97 (Sheldrick, 1997)
281 parameters	Extinction coefficient: 0.0025 (11)
H atoms constrained	

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···O1	0.93	2.43	2.712 (3)	97.7
C6—H6A···O2	0.93	2.43	2.715 (3)	97.9
C15—H15A···O5	0.93	2.57	2.854 (1)	98.0
C19—H19A···O4	0.93	2.43	2.735 (1)	98.8
C16—H16A···O3^{i}	0.93	2.67	3.356 (2)	131.1
C23—H23A···O5^{ii}	0.96	3.400 (4)	2.72	128.6

Symmetry codes: (i) $-x, -y, 1-z$; (ii) $x-1, y, z$.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1219). Services for accessing these data are described at the back of the journal.

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