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2-Methyl-4-nitro-1-phenylimidazole and 2-Methyl-1-(*p*-methylphenyl)-4-nitroimidazole

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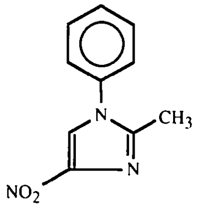
Abstract

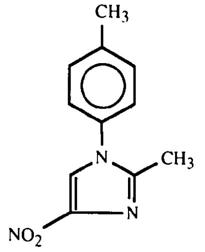
The two title compounds, $C_{10}H_9N_3O_2$, (I), and $C_{11}H_{11}N_3O_2$, (II), are monomers. No intermolecular interactions are observed. The angle between the planes of the phenyl and imidazole rings is 51.2 (6) in (I) and 53.9 (5)° in (II). The nitro groups are twisted with respect to the imidazole rings by 3.2 (5) and 3.7 (5)° in (I) and (II), respectively.

Comment

Many nitroimidazoles are used or investigated as drugs or radiosensitizing agents (Suwiński & Salwińska, 1982). 1-Arylnitroimidazoles have been synthesized recently (Salwińska & Suwiński, 1990). Examination of the Cambridge Structural Database (Allen *et al.*, 1991) indicates that this is probably the first publication of an X-ray structure of this type of compound. The structures of the title compounds, (I) and (II), are shown in Figs. 1 & 2, respectively.



(I)



(II)

None of the compounds has H atoms which are able to form hydrogen bonds. Neither the imidazole nor the phenyl rings are stacked. The aromaticity indices I_5 (Bird, 1985) and HOMA (Gdaniec, Turowska-Tyrk & Krygowski, 1989) are 67.9 and 0.823, respectively for unsubstituted imidazole [structural data for these calculations are from McMullan, Epstein, Ruble & Craven (1979)]. [Note that in the original publication (Bird, 1985), I_5 for imidazole is calculated from data corrected for libration and is 64.] The title compounds have smaller values: 61.3 and 0.565 for (I) and 64.2 and 0.664 for (II). The observed angles between the least-squares planes of the imidazole and phenyl rings are similar to those found in other 1-arylimidazoles (Ishihara *et al.*, 1992) and represent the compromise between the crystal packing and the minimum energy position, which was found by MOPAC (Quantum Chemistry Program Exchange No. 455) calculations at 90° (using default settings; rotation of the phenyl group around the N1—C4 bond with C5 and C9 related by symmetry).

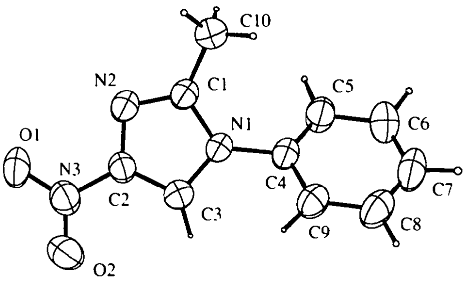


Fig. 1. The crystal structure and numbering scheme of (I). Displacement ellipsoids are drawn at the 50% probability level.

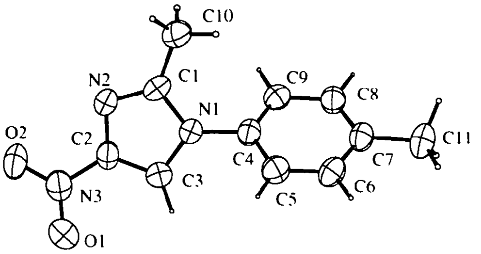


Fig. 2. The crystal structure and numbering scheme of (II). Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Colourless crystals of the title compounds (Salwińska & Suwiński, 1990) were formed by slow evaporation of water–ethanol solutions. Ther densities (D_m) were measured by flotation.

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Compound (I)

Crystal data

$C_{10}H_9N_3O_2$
$M_r = 203.20$
Monoclinic
$P2_1/n$
$a = 8.234\,(4)$ Å
$b = 7.678\,(5)$ Å
$c = 15.345\,(8)$ Å
$\beta = 99.94\,(4)°$
$V = 955.6\,(9)$ Å^3
$Z = 4$
$D_x = 1.412\,(1)$ Mg m^{-3}
$D_m = 1.44$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 18$–$27°$
$\mu = 0.851$ mm^{-1}
$T = 295\,(2)$ K
Irregularly shaped
$0.35 \times 0.22 \times 0.22$ mm
Colourless

Data collection

Kuma KM4 automated four-circle κ-axis diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2424 measured reflections
1723 independent reflections
1157 observed reflections $[I > 3\sigma(I)]$

$R_{\rm int} = 0.0344$
$\theta_{\rm max} = 82°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 9$
$l = -19 \rightarrow 19$
3 standard reflections monitored every 50 reflections intensity decay: 2.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0364$
$wR(F^2) = 0.1021$
$S = 1.188$
1549 reflections
173 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.0697P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.028$

$\Delta\rho_{\rm max} = 0.085$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.083$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin 2\theta)]^{-1/4}$
Extinction coefficient: 0.026 (11)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Compound (II)

Crystal data

$C_{11}H_{11}N_3O_2$
$M_r = 217.23$
Monoclinic
$P2_1/c$
$a = 8.259$ (2) Å
$b = 7.805$ (2) Å
$c = 16.774$ (3) Å
$\beta = 103.58$ (3)°
$V = 1051.0$ (4) Å^3
$Z = 4$
$D_x = 1.373\,(1)$ Mg m^{-3}
$D_m = 1.40$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 14$–$29°$
$\mu = 0.808$ mm^{-1}
$T = 293$ (2) K
Irregularly shaped
$0.45 \times 0.30 \times 0.30$ mm
Colourless

Data collection

Kuma KM4 automated four-circle κ-axis diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2014 measured reflections
1856 independent reflections
1044 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0237$
$\theta_{\rm max} = 82°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 10$
$l = -21 \rightarrow 20$
2 standard reflections monitored every 50 reflections intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0466$
$wR(F^2) = 0.1369$
$S = 1.266$
1783 reflections
191 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0959P)^2 + 0.0352P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.002$

$\Delta\rho_{\rm max} = 0.175$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.161$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin 2\theta)]^{-1/4}$
Extinction coefficient: 0.033 (7)
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) *for* (I)

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

	x	y	z	$U_{\rm eq}$
O1	0.1818 (2)	0.1024 (2)	0.64850 (8)	0.0839 (5)
O2	0.3670 (2)	−0.0338 (2)	0.59277 (9)	0.0825 (5)
N1	0.13878 (14)	0.1805 (2)	0.36256 (7)	0.0483 (4)
N2	0.0507 (2)	0.2358 (2)	0.48802 (8)	0.0563 (4)
N3	0.2486 (2)	0.0633 (2)	0.58621 (9)	0.0625 (4)
C1	0.0238 (2)	0.2646 (2)	0.40278 (9)	0.0516 (4)
C2	0.1849 (2)	0.1316 (2)	0.50149 (9)	0.0528 (4)
C3	0.2431 (2)	0.0961 (2)	0.42644 (9)	0.0519 (4)
C4	0.1486 (2)	0.1726 (2)	0.27056 (9)	0.0493 (4)
C5	0.0130 (2)	0.1261 (2)	0.21028 (10)	0.0601 (4)
C6	0.0265 (3)	0.1154 (3)	0.12221 (12)	0.0716 (5)
C7	0.1738 (3)	0.1478 (3)	0.09556 (12)	0.0740 (5)
C8	0.3086 (3)	0.1930 (3)	0.15628 (13)	0.0720 (5)
C9	0.2974 (2)	0.2066 (2)	0.24437 (12)	0.0597 (4)
C10	−0.1102 (2)	0.3741 (3)	0.35570 (13)	0.0658 (5)

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

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

	x	y	z	$U_{\rm eq}$
O1	0.2281 (3)	0.5272 (3)	0.59776 (12)	0.0793 (7)
O2	0.4628 (3)	0.3992 (3)	0.63850 (13)	0.0790 (6)
N1	0.2384 (2)	0.3150 (2)	0.37683 (11)	0.0493 (5)
N2	0.4369 (2)	0.2593 (3)	0.48751 (13)	0.0580 (6)
N3	0.3395 (3)	0.4348 (3)	0.58565 (13)	0.0583 (6)
C1	0.3848 (3)	0.2285 (3)	0.40852 (15)	0.0527 (6)
C2	0.3213 (3)	0.3667 (3)	0.50573 (14)	0.0513 (6)
C3	0.1979 (3)	0.4022 (3)	0.43919 (15)	0.0517 (6)
C4	0.1448 (3)	0.3201 (3)	0.29307 (14)	0.0489 (6)
C5	−0.0223 (3)	0.2822 (3)	0.2752 (2)	0.0566 (6)
C6	−0.1135 (4)	0.2940 (4)	0.1949 (2)	0.0611 (7)
C7	−0.0403 (3)	0.3417 (3)	0.1327 (2)	0.0539 (6)
C8	0.1291 (3)	0.3760 (3)	0.1525 (2)	0.0573 (7)
C9	0.2210 (3)	0.3662 (3)	0.23210 (15)	0.0542 (6)
C10	0.4672 (5)	0.1143 (5)	0.3607 (2)	0.0713 (8)
C11	−0.1412 (5)	0.3599 (5)	0.0463 (2)	0.0712 (8)

Table 3. *Selected geometric parameters* (Å, °) *for* (I) *and* (II)

	(I)	(II)		(I)	(II)
O1—N3	1.220 (2)	1.223 (3)	C2—C3	1.349 (2)	1.352 (4)
O2—N3	1.218 (2)	1.215 (3)	C4—C5	1.368 (2)	1.374 (4)
N1—C3	1.352 (2)	1.354 (3)	C4—C9	1.379 (2)	1.369 (4)
N1—C1	1.377 (2)	1.378 (3)	C5—C6	1.377 (2)	1.383 (4)
N1—C4	1.429 (2)	1.436 (3)	C6—C7	1.367 (3)	1.374 (4)
N2—C1	1.307 (2)	1.315 (3)	C7—C8	1.365 (3)	1.386 (4)
N2—C2	1.351 (2)	1.359 (3)	C7—C11	-	1.500 (4)
N3—C2	1.416 (2)	1.417 (3)	C8—C9	1.375 (3)	1.374 (4)
C1—C10	1.474 (3)	1.469 (4)			

	(I)	(II)
C3—N1—C1	107.42 (12)	107.6 (2)
C3—N1—C4	123.96 (12)	124.6 (2)
C1—N1—C4	128.56 (12)	127.8 (2)
C1—N2—C2	104.24 (12)	105.0 (2)
O2—N3—O1	123.30 (15)	123.2 (2)
O2—N3—C2	117.85 (14)	119.3 (2)
O1—N3—C2	118.9 (2)	117.5 (2)
N2—C1—N1	111.02 (14)	110.4 (2)
N2—C1—C10	124.76 (14)	125.2 (2)
N1—C1—C10	124.22 (14)	124.3 (2)
C3—C2—N2	112.98 (13)	112.0 (2)
C3—C2—N3	125.4 (2)	126.6 (2)
N2—C2—N3	121.58 (13)	121.4 (2)
C2—C3—N1	104.33 (13)	105.0 (2)
C5—C4—C9	120.98 (15)	120.5 (2)
C5—C4—N1	120.03 (13)	119.3 (2)
C9—C4—N1	118.96 (14)	120.2 (2)
C4—C5—C6	119.0 (2)	119.2 (2)
C7—C6—C5	120.6 (2)	121.4 (2)
C8—C7—C6	120.0 (2)	118.0 (2)
C6—C7—C11	-	121.0 (3)
C8—C7—C11	-	120.9 (3)
C7—C8—C9	120.4 (2)	121.2 (2)
C8—C9—C4	119.1 (2)	119.6 (2)

Unit-cell parameters and space groups were found from oscillation and Weissenberg photographs and confirmed by preliminary diffractometer data collection. All non-H atom positions were found using direct methods and refined. H-atom positions were calculated from geometry and also refined (not using any restrains).

For both compounds, data collection: *Kuma KM*4 *Software* (1989); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976).

The author wishes to thank Professor J. Suwiński for samples of compounds.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1197). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methyl 6-[(4-Bromobenzoyloxy)(5,5,8,8-tetramethyl-5,6,7,8-tetrahydro-2-naphthyl)methyl]-2-naphthalenecarboxylate

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Abstract

The structure of the title compound, $C_{34}H_{33}BrO_4$, determined using direct methods and refined including the anomalous scattering from the Br atoms, shows that the absolute configuration at the C14 atom is *R*. High displacement parameters of several C atoms indicate conformational disorder of the saturated ring in the tetramethyltetrahydronaphthyl group. The packing of the molecules is stabilized by van der Waal's forces.

Comment

Retinoic acid is known to exert its biological effects through gene regulation mediated by at least two classes of nuclear receptors: retinoic acid receptors (RAR α,β,γ) and retinoid X receptors (RXR α,β,γ) (Lehmann *et al.*, 1992). In attempts to identify biological functions of each receptor, recent efforts have focused on finding receptor-specific ligands for these receptors (Bernard *et al.*, 1992; Shroot, 1991; Lehmann, Dawson, Hobbs, Husmann & Pfahl, 1991). Graupner *et al.* (1991) have reported that the racemate of the 2-naphthoate derivative (1) is an RAR β,γ-selective agonist. We have synthesized both enantiomers of (1) and tested their biological activity (Reczek *et al.*, 1994).

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