After refinement by least squares, H atoms were located from a difference synthesis. In the final cycles the heavier atoms were allowed to vibrate anisotropically.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1172). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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α-(2,3,5,6-Tetrachlorophenyl)-α-(3-thienyl)-acetonitrile and α-(3-Methoxyphenyl)-α-(2,3,4,5,6-pentachlorophenyl)acetonitrile

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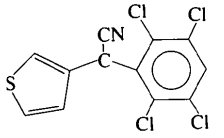
Abstract

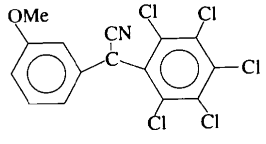
The C—Cl distances of the tetrachlorophenyl moiety in α-(2,3,5,6-tetrachlorophenyl)-α-(3-thienyl)acetonitrile, (1), $C_{12}H_5Cl_4NS$, are in the range 1.713 (3)–1.720 (3) Å, and those of the pentachlorophenyl moiety in α-(3-methoxyphenyl)-α-(2,3,4,5,6-pentachlorophenyl)acetonitrile, (2), $C_{15}H_8Cl_5NO$, are in the range 1.695 (9)–1.734 (8) Å. The thienyl ring of compound (1) is almost planar and the dihedral angle between the phenyl and thienyl rings is 78.8 (1)°, while that between the two phenyl planes of compound (2) is 88.6 (3)°.

Comment

As part of our synthetic and structural studies of the benzyne reaction, the structures of the title compounds, α-(2,3,5,6-tetrachlorophenyl)-α-(3-thienyl)acetonitrile, (1), and α-(3-methoxyphenyl)-α-(2,3,4,5,6-pentachlorophenyl)acetonitrile, (2), were determined by X-ray diffraction.



(1)



(2)

The present structure analysis reveals that compound (1) possesses an H atom in the *para* position of the tetrachlorophenyl moiety (Fig. 1). The C—Cl distances [1.713 (3)–1.720 (3) in (1) and 1.696 (9)–1.735 (8) Å in (2)] are comparable to the corresponding ranges of 1.725 (7)–1.799 (6) Å in $[Ir(C_6Cl_5)_4]^{2-}$ (Garcia, Jimenez, Oro, Lahoz, Tiripicchio & Tiripicchio, 1993), 1.673 (11)–1.738 (8) Å in perchlorotriphenylgermane (Fajari, Julia, Riera, Molins & Miravitlles, 1989) and 1.706 (11)–1.748 (11) Å in $[(PPh_3)(C_6Cl_5)BrPt(\mu\text{-}Br)Ag(PPh_3)]_2$ (Uson, Fornies, Tomas & Ara, 1991). Of the Cl atoms, Cl(5) of compound (1) and Cl(4) of compound (2) lie nearly in the plane of their respective phenyl rings, while the remaining eight Cl atoms are significantly displaced from their planes, the maximum deviation being 0.064 Å for Cl(2) in compound (1).

The orientations of the cyano groups with respect to the chlorophenyl rings are defined by the torsion

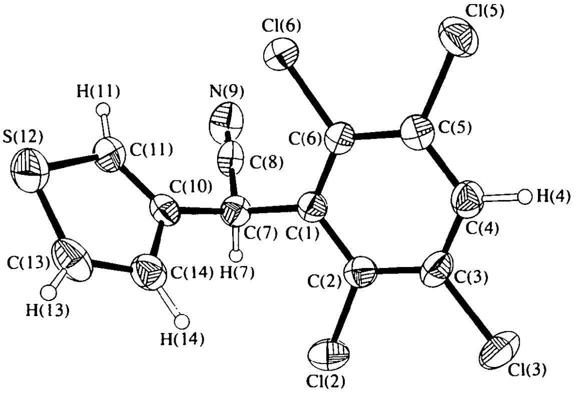


Fig. 1. The structure of compound (1) showing 50% probability displacement ellipsoids. For clarity, H atoms are drawn as small circles of arbitrary radii.

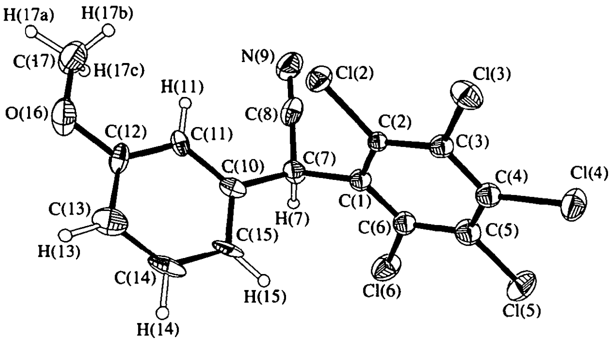


Fig. 2. The structure of compound (2) showing 50% probability displacement ellipsoids. For clarity, H atoms are drawn as small circles of arbitrary radii.

angles C(8)—C(7)—C(1)—C(2) [−123.3 (3) in (1) and −113 (1)° in (2)], and with respect to the thienyl ring in (1) and the methoxy-substituted phenyl ring in (2) by the torsion angles C(11)—C(10)—C(7)—C(8) [−12.0 (4) in (1) and −1 (1)° in (2)]. The C(8)—N(9) distances [1.136 (4) in (1) and 1.129 (11) Å in (2)] are typical of C≡N triple-bond distances (Rabinovich & Shakked, 1978). The thienyl ring in (1) is almost planar with an r.m.s. deviation of 0.003 Å. The S—C bond lengths of the thienyl ring [1.688 (3) and 1.682 (3) Å] are slightly shorter than those of 1.70 (1)–1.72 (1) Å found in a thenoyl-substituted cyclopropane structure (Saba, 1994).

Experimental

α-(2,3,5,6-Tetrachlorophenyl)-α-(3-thienyl)acetonitrile was prepared by the reaction of pentachlorophenyllithium and α-lithioarylacetonitrile, while α-(3-methoxyphenyl)-α-(2,3,4,5,6-pentachlorophenyl)acetonitrile was synthesized by the reaction of perchlorobenzene and α-lithioarylacetonitrile (Refat, Waggenspack, Dutt, Zhang, Faddo & Biehl, 1995). Colorless crystals of both compounds were recrystallized from ethanol solution.

Compound (1)

Crystal data

$C_{12}H_5Cl_4NS$
$M_r = 337.03$
Monoclinic
$P2_1/n$
$a = 9.288$ (2) Å
$b = 10.809$ (2) Å
$c = 13.379$ (2) Å
$\beta = 100.91$ (1)°
$V = 1318.9$ (4) Å^3
$Z = 4$
$D_x = 1.697$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 1.033$ mm^{-1}
$T = 293$ (2) K
Plate
0.35 × 0.15 × 0.10 mm
Colorless

Data collection

R3m/V diffractometer
$\omega/2\theta$ scans
Absorption correction:
  ψ scan
  $T_{\min} = 0.795$, $T_{\max} = 0.936$
2794 measured reflections
2619 independent reflections
2062 observed reflections
  [$I > 2\sigma(I)$]

$R_{\rm int} = 0.013$
$\theta_{\max} = 26.06°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 13$
$l = -16 \rightarrow 16$
3 standard reflections
  monitored every 150 reflections
  intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.043$
$wR(F^2) = 0.131$
$S = 1.036$
2448 reflections
164 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 0.9350P]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.720$ e Å^{-3}
$\Delta\rho_{\min} = -0.384$ e Å^{-3}
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* (1)

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

	x	y	z	$U_{\rm eq}$
Cl(2)	0.49429 (10)	0.35142 (8)	0.21653 (7)	0.0604 (3)
Cl(3)	0.72619 (9)	0.54436 (10)	0.31357 (9)	0.0705 (3)
Cl(5)	0.34530 (10)	0.75150 (8)	0.51244 (6)	0.0569 (2)
Cl(6)	0.10363 (8)	0.56620 (7)	0.41431 (6)	0.0486 (2)
C(1)	0.3052 (3)	0.4685 (2)	0.3157 (2)	0.0363 (6)
C(2)	0.4474 (3)	0.4627 (3)	0.2958 (2)	0.0415 (6)
C(3)	0.5526 (3)	0.5471 (3)	0.3410 (2)	0.0457 (7)
C(4)	0.5205 (3)	0.6351 (3)	0.4068 (2)	0.0453 (7)
C(5)	0.3826 (3)	0.6401 (3)	0.4292 (2)	0.0406 (6)
C(6)	0.2750 (3)	0.5573 (2)	0.3838 (2)	0.0367 (6)
C(7)	0.1878 (3)	0.3779 (3)	0.2637 (2)	0.0402 (6)
C(8)	0.0592 (3)	0.4414 (3)	0.2065 (2)	0.0445 (7)
N(9)	−0.0400 (3)	0.4880 (3)	0.1587 (2)	0.0646 (8)
C(10)	0.1500 (3)	0.2804 (3)	0.3359 (2)	0.0394 (6)
C(11)	0.0174 (4)	0.2648 (3)	0.3627 (3)	0.0521 (8)
S(12)	0.01935 (12)	0.14780 (8)	0.44648 (8)	0.0683 (3)
C(13)	0.1964 (4)	0.1131 (3)	0.4483 (2)	0.0544 (8)
C(14)	0.2540 (4)	0.1945 (3)	0.3834 (2)	0.0500 (7)

Table 2. *Selected geometric parameters* (Å, °) *for* (1)

Cl(2)—C(2)	1.713 (3)	C(5)—C(6)	1.392 (4)
Cl(3)—C(3)	1.720 (3)	C(7)—C(8)	1.462 (4)
Cl(5)—C(5)	1.719 (3)	C(7)—C(10)	1.515 (4)
Cl(6)—C(6)	1.719 (3)	C(8)—N(9)	1.136 (4)
C(1)—C(6)	1.389 (4)	C(10)—C(11)	1.357 (4)
C(1)—C(2)	1.398 (4)	C(10)—C(14)	1.403 (4)
C(1)—C(7)	1.530 (4)	C(11)—S(12)	1.688 (3)
C(2)—C(3)	1.389 (4)	S(12)—C(13)	1.682 (4)
C(3)—C(4)	1.366 (4)	C(13)—C(14)	1.411 (5)
C(4)—C(5)	1.371 (4)		
C(6)—C(1)—C(2)	118.1 (3)	C(1)—C(6)—Cl(6)	120.3 (2)
C(6)—C(1)—C(7)	121.7 (2)	C(5)—C(6)—Cl(6)	118.8 (2)
C(2)—C(1)—C(7)	120.2 (3)	C(8)—C(7)—C(10)	113.3 (2)
C(3)—C(2)—C(1)	120.0 (3)	C(8)—C(7)—C(1)	112.2 (2)
C(3)—C(2)—Cl(2)	119.4 (2)	C(10)—C(7)—C(1)	112.7 (2)
C(1)—C(2)—Cl(2)	120.6 (2)	N(9)—C(8)—C(7)	177.2 (4)
C(4)—C(3)—C(2)	121.1 (3)	C(11)—C(10)—C(14)	112.4 (3)
C(4)—C(3)—Cl(3)	118.5 (2)	C(11)—C(10)—C(7)	125.9 (3)
C(2)—C(3)—Cl(3)	120.4 (3)	C(14)—C(10)—C(7)	121.7 (3)
C(3)—C(4)—C(5)	119.7 (3)	C(10)—C(11)—S(12)	111.8 (3)
C(4)—C(5)—C(6)	120.2 (3)	C(13)—S(12)—C(11)	93.7 (2)
C(4)—C(5)—Cl(5)	118.7 (2)	C(14)—C(13)—S(12)	109.6 (2)
C(6)—C(5)—Cl(5)	121.1 (2)	C(10)—C(14)—C(13)	112.5 (3)
C(1)—C(6)—C(5)	120.9 (3)		

Compound (2)

Crystal data

$C_{15}H_8Cl_5NO$
$M_r = 395.47$
Triclinic
$P\bar{1}$
$a = 7.681$ (2) Å
$b = 8.780$ (2) Å
$c = 12.761$ (3) Å
$\alpha = 100.24$ (2)°
$\beta = 99.65$ (2)°
$\gamma = 108.39$ (2)°
$V = 780.2$ (3) Å^3
$Z = 2$
$D_x = 1.683$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 8$–14°
$\mu = 0.928$ mm^{-1}
$T = 293$ (2) K
Needle
0.35 × 0.10 × 0.05 mm
Colorless

Data collection

$R3m/V$ diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan
$T_{\min} = 0.880$, $T_{\max} = 0.988$
1841 measured reflections
1679 independent reflections
882 observed reflections [$I > 2\sigma(I)$]

$R_{\text{int}} = 0.042$
$\theta_{\max} = 21°$
$h = 0 \rightarrow 7$
$k = -8 \rightarrow 8$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.056$
$wR(F^2) = 0.105$
$S = 1.016$
1392 reflections
200 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0156P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.302$ e Å^{-3}
$\Delta\rho_{\min} = -0.312$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

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

	x	y	z	U_{eq}
Cl(2)	0.1317 (4)	0.4144 (3)	0.8622 (2)	0.0468 (8)
Cl(3)	0.3504 (4)	0.5960 (3)	1.0992 (2)	0.0480 (8)
Cl(4)	0.4258 (4)	0.9697 (3)	1.1845 (2)	0.0451 (8)
Cl(5)	0.2778 (4)	1.1601 (3)	1.0301 (2)	0.0397 (7)
Cl(6)	0.0383 (3)	0.9755 (3)	0.7965 (2)	0.0360 (7)
C(1)	0.0950 (12)	0.7027 (11)	0.8381 (7)	0.024 (2)
C(2)	0.1657 (12)	0.6221 (10)	0.9072 (7)	0.029 (2)
C(3)	0.2661 (12)	0.7029 (12)	1.0144 (7)	0.031 (2)
C(4)	0.3004 (12)	0.8681 (11)	1.0526 (7)	0.027 (3)
C(5)	0.2306 (12)	0.9545 (10)	0.9842 (7)	0.025 (2)
C(6)	0.1300 (11)	0.8685 (11)	0.8790 (6)	0.020 (2)
C(7)	−0.0191 (12)	0.6101 (11)	0.7207 (7)	0.030 (3)
C(8)	−0.2153 (16)	0.6005 (11)	0.7078 (7)	0.034 (3)
N(9)	−0.3673 (13)	0.5905 (10)	0.6984 (6)	0.050 (3)
C(10)	0.0690 (13)	0.6778 (10)	0.6321 (7)	0.028 (2)
C(11)	−0.0335 (13)	0.7204 (10)	0.5490 (7)	0.028 (2)
C(12)	0.0452 (14)	0.7738 (11)	0.4680 (7)	0.032 (2)
C(13)	0.2318 (15)	0.7911 (11)	0.4681 (9)	0.048 (3)
C(14)	0.3324 (14)	0.7487 (11)	0.5520 (9)	0.045 (3)
C(15)	0.2538 (13)	0.6930 (10)	0.6321 (8)	0.037 (3)
O(16)	−0.0459 (10)	0.8193 (8)	0.3840 (5)	0.055 (2)
C(17)	−0.2381 (15)	0.7969 (12)	0.3789 (7)	0.059 (3)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

Cl(2)—C(2)	1.732 (9)	C(5)—C(6)	1.376 (10)
Cl(3)—C(3)	1.726 (9)	C(7)—C(8)	1.462 (13)
Cl(4)—C(4)	1.715 (8)	C(7)—C(10)	1.527 (11)
Cl(5)—C(5)	1.695 (9)	C(8)—N(9)	1.127 (11)
Cl(6)—C(6)	1.734 (8)	C(10)—C(15)	1.383 (11)
C(1)—C(2)	1.369 (11)	C(10)—C(11)	1.389 (11)
C(1)—C(6)	1.379 (10)	C(11)—C(12)	1.363 (11)
C(1)—C(7)	1.533 (11)	C(12)—C(13)	1.393 (13)
C(2)—C(3)	1.389 (11)	C(13)—C(14)	1.388 (12)
C(3)—C(4)	1.369 (11)	C(14)—C(15)	1.360 (12)
C(4)—C(5)	1.400 (11)		
C(2)—C(1)—C(6)	117.2 (8)	C(5)—C(6)—C(1)	123.4 (8)
C(2)—C(1)—C(7)	120.8 (8)	C(5)—C(6)—Cl(6)	117.4 (6)
C(6)—C(1)—C(7)	122.1 (8)	C(1)—C(6)—Cl(6)	119.2 (6)
C(1)—C(2)—C(3)	121.6 (8)	C(8)—C(7)—C(10)	112.4 (8)
C(1)—C(2)—Cl(2)	120.4 (7)	C(8)—C(7)—C(1)	110.3 (7)
C(3)—C(2)—Cl(2)	118.0 (7)	C(10)—C(7)—C(1)	114.1 (7)
C(4)—C(3)—C(2)	119.9 (8)	N(9)—C(8)—C(7)	178.8 (11)
C(4)—C(3)—Cl(3)	120.0 (7)	C(15)—C(10)—C(11)	118.8 (9)
C(2)—C(3)—Cl(3)	120.1 (7)	C(15)—C(10)—C(7)	119.5 (9)
C(3)—C(4)—C(5)	120.0 (8)	C(11)—C(10)—C(7)	121.7 (8)
C(3)—C(4)—Cl(4)	120.4 (7)	C(12)—C(11)—C(10)	120.8 (9)
C(5)—C(4)—Cl(4)	119.6 (7)	C(11)—C(12)—C(13)	120.7 (9)
C(6)—C(5)—C(4)	117.9 (8)	C(14)—C(13)—C(12)	117.7 (10)
C(6)—C(5)—Cl(5)	122.0 (7)	C(15)—C(14)—C(13)	121.8 (10)
C(4)—C(5)—Cl(5)	120.1 (7)	C(14)—C(15)—C(10)	120.2 (9)

Data were corrected for both structures for Lorentz, polarization and absorption effects. Refinement was carried out for all reflections, except for 164 in structure (1) and 280 in structure (2) with negative F^2, and for seven reflections in each structure with largest weighted $|F_o^2-F_c^2|$. Weighted wR and S factors were based on F^2, while conventional R factors were based on F, with F set to zero for negative F^2. Full-matrix least-squares refinement was performed for both structures using *SHELXL*93 (Sheldrick, 1993). All non-H atoms were refined anisotropically. H atoms in both structures were treated using a riding model and their isotropic displacement parameters were refined as common free variables.

For both compounds, data collection: *R3m/V* diffractometer software; cell refinement: *R3m/V* diffractometer software; data reduction: *R3m/V* diffractometer software; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990*a*); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1176). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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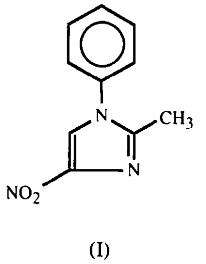
Abstract

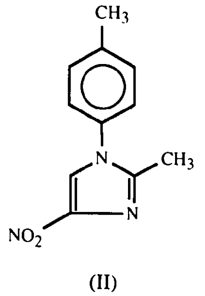
The two title compounds, $C_{10}H_9N_3O_2$, (I), and $C_{11}H_{11}N_3O_2$, (II), are monomers. No intermolecular interactions are observed. The angle between the planes of the phenyl and imidazole rings is 51.2 (6) in (I) and 53.9 (5)° in (II). The nitro groups are twisted with respect to the imidazole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

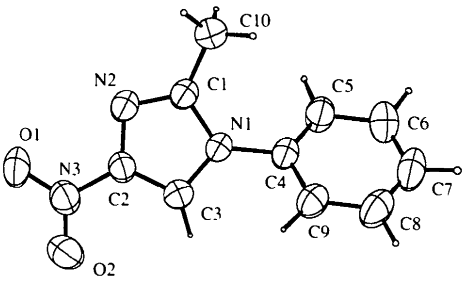


Fig. 1. The crystal structure and numbering scheme of (I). Displacement ellipsoids are drawn at the 50% probability level.

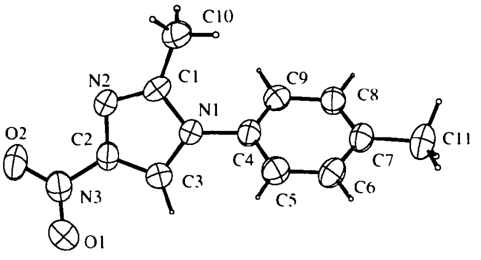


Fig. 2. The crystal structure and numbering scheme of (II). Displacement ellipsoids are drawn at the 50% probability level.

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.