

Structural comparisons between methylated and unmethylated nitrophenyl lophines

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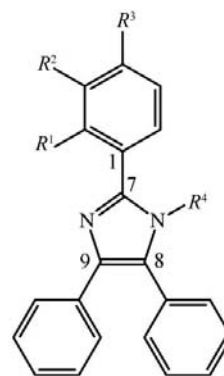
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The lophine derivative 2-(2-nitrophenyl)-4,5-diphenyl-1*H*-imidazole, $C_{21}H_{15}N_3O_2$, (I), crystallized from ethanol as a solvent-free crystal and from acetonitrile as the monosolvate, $C_{21}H_{15}N_3O_2 \cdot C_2H_3N$, (II). Crystallization of 2-(4-nitrophenyl)-4,5-diphenyl-1*H*-imidazole from methanol yielded the methanol monosolvate, $C_{21}H_{15}N_3O_2 \cdot CH_4O$, (III). Three lophine derivatives of methylated imidazole, namely, 1-methyl-2-(2-nitrophenyl)-4,5-diphenyl-1*H*-imidazole methanol solvate, $C_{22}H_{17}N_3O_2 \cdot CH_4O$, (IV), 1-methyl-2-(3-nitrophenyl)-4,5-diphenyl-1*H*-imidazole, $C_{22}H_{17}N_3O_2$, (V), and 1-methyl-2-(4-nitrophenyl)-4,5-diphenyl-1*H*-imidazole, $C_{22}H_{17}N_3O_2$, (VI), were recrystallized from methanol, acetonitrile and ethanol, respectively, but only (IV) produced a solvate. Compounds (III) and (IV) each crystallize with two independent molecules in the asymmetric unit. Five imidazole molecules in the six crystals differ in their molecular conformations by rotation of the aromatic rings with respect to the central imidazole ring. In the absence of a methyl group on the imidazole [compounds (I)–(III)], the rotation angles are not strongly affected by the position of the nitro group [44.8 (2) and 45.5 (1)° in (I) and (II), respectively, and 15.7 (2) and 31.5 (1)° in the two molecules of (III)]. However, the rotation angle is strongly affected by the presence of a methyl group on the imidazole [compounds (IV)–(VI)], and the position of the nitro group (*ortho*, *meta* or *para*) on a neighbouring benzene ring; values of the rotation angle range from 26.0 (1) [in (VI)] to 85.2 (1)° [in (IV)]. This group repulsion also affects the outer N—C—N bond angle. The packing of the molecules in (I), (II) and (III) is determined by hydrogen bonding. In (I) and (II), molecules form extended chains through N—H...N hydrogen bonds [with an N...N distance of 2.944 (5) Å in (I) and 2.920 (3) Å in (II)], while in (III) the chain is formed with a methanol solvent molecule as the mediator between two imidazole rings, with O...N distances of 2.788 (4)–2.819 (4) Å. In the absence of the imidazole N—H H-atom donor, the packing of molecules (IV)–(VI) is determined by weaker intermolecular interactions. The methanol solvent molecule in (IV) is

hydrogen bonded to imidazole [O...N = 2.823 (4) Å] but has no effect on the packing of molecules in the unit cell.

Comment

Recently, heterocyclic imidazole derivatives have attracted considerable attention because of their unique linear and nonlinear optical properties (Santos *et al.*, 2001). Lophine (2,4,5-triphenyl-1*H*-imidazole) is an attractive fluorescence and chemiluminescence compound. The chemiluminescence properties of lophine were reported for the first time by Radziszewski (1877), who showed that it emits yellow light when it reacts with oxygen in the presence of a strong base. The chemistry of lophine derivatives has a long history in relation to important physicochemical phenomena such as chemiluminescence through its oxidation and photo-, piezo- and thermochromic properties (Zimmerman *et al.*, 1961; Maeda & Hayashi, 1969, 1970). Only a few imidazole analogues or nitro derivatives of lophine are known (Inouye & Sakaino, 1985, 1986, 2000; Sakaino, Fujii *et al.*, 1990; Sakaino, Shimizu *et al.*, 1990; Kaftory *et al.*, 1998; Bu *et al.*, 2003). The spectral properties of lophine derivatives are of interest: the colour change is dependent on the solvation in the crystal and in different derivatives. We have recently synthesized various



(I) and (II): $R^1 = H$, $R^3 = NO_2$, $R^2 = R^4 = H$

(III): $R^1 = NO_2$, $R^3 = H$, $R^2 = R^4 = H$

(IV): $R^1 = NO_2$, $R^2 = R^3 = H$, $R^4 = CH_3$

(V): $R^1 = R^3 = H$, $R^2 = NO_2$, $R^4 = CH_3$

(VI): $R^1 = R^2 = H$, $R^3 = NO_2$, $R^4 = CH_3$

lophine derivatives to study their spectral properties with the aim of understanding the relationship between molecular and crystal structures and colour change (Fridman, Kaftory, Eichen *et al.*, 2007; Fridman, Kaftory & Speiser, 2007; Fridman *et al.*, 2008). In order to examine the effect of the ability of the imidazole ring to form hydrogen bonds on the crystal colour, we have prepared lophine derivatives with nitrophenyl at position 7 of the imidazole (for notation see scheme), as well as compounds where the N—H hydrogen-donor ability in imidazole is blocked by replacing the H atom with a methyl group. We present here a comparison between the molecular and crystal structures of six lophine *o*-, *m*- and *p*-nitrophenyl derivatives (Figs. 1–6).

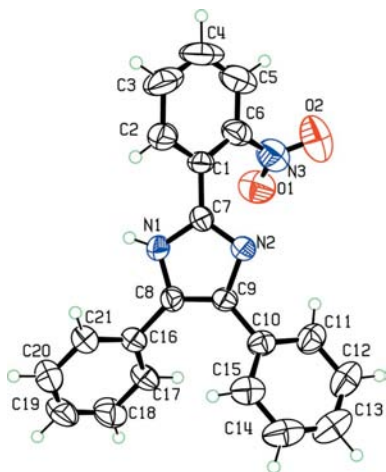


Figure 1

The molecular structure of (I), showing displacement ellipsoids at the 50% probability level and H atoms as small spheres of arbitrary radii (Farrugia, 1999).

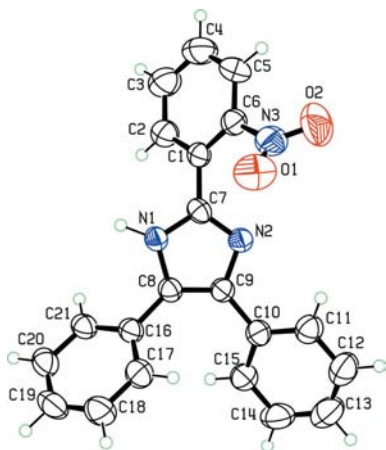
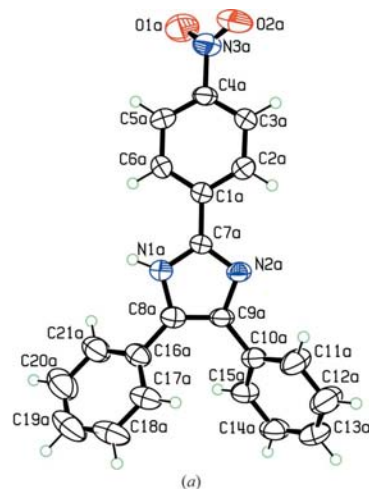


Figure 2

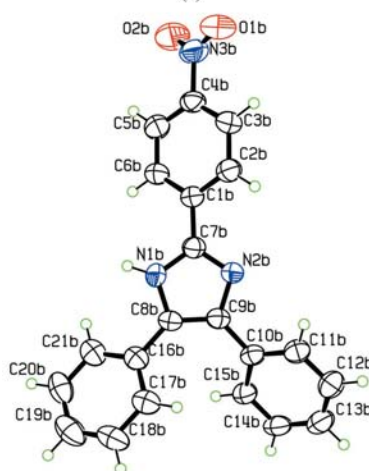
The molecular structure of (II), showing displacement ellipsoids at the 50% probability level and H atoms as small spheres of arbitrary radii (Farrugia, 1999). The acetonitrile solvent molecule is not shown.

Comparison of the bond lengths in the imidazole rings of (I) to (IV) reveals the following ranges: N1—C7 1.350–1.375 Å, N2—C7 1.317–1.333 Å, N1—C8 1.377–1.395 Å, N2—C9 1.369–1.383 Å and C8—C9 1.374–1.377 Å. Statistical calculation of the related bond lengths in the imidazole rings in 56 lophine derivatives taken from the Cambridge Structural Database (CSD, Version 1.11 of 2009; Allen, 2002) provided corresponding average values of 1.353 (8), 1.333 (7), 1.370 (16), 1.386 (7) and 1.375 (8) Å, respectively.

The conformation of the molecules differs by rotation of the phenyl rings with respect to the central imidazole. The conformation is affected by the presence or absence of a bulky group [an H atom in (I)–(III) and a methyl group in (IV)–(VI)], and is also dependent on the position of the nitro group (*ortho*, *meta* or *para*) on the phenyl ring at position 7 of the imidazole with respect to the H atom or methyl group. An overlay of the molecules in the six crystals [only one of the two



(a)



(b)

Figure 3

The molecular structures of molecules (IIIa) and (IIIb), showing displacement ellipsoids at the 50% probability level and H atoms as small spheres of arbitrary radii (Farrugia, 1999). The methanol solvent molecule is not shown.

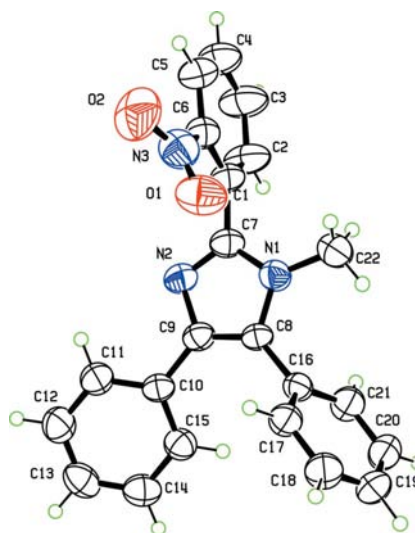
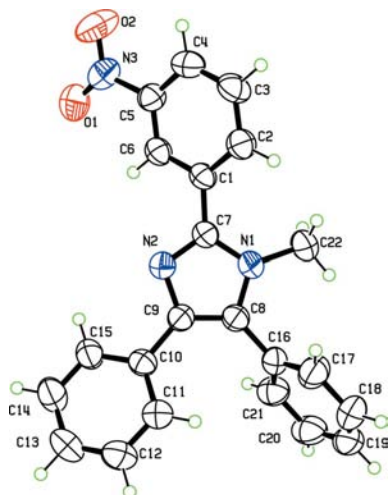


Figure 4

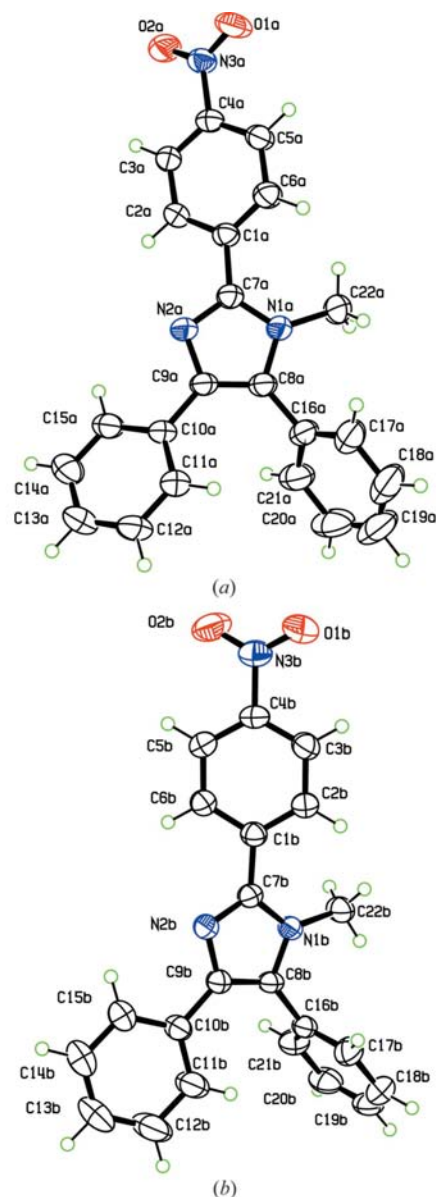
The molecular structure of (IV), showing displacement ellipsoids at the 50% probability level and H atoms as small spheres of arbitrary radii (Farrugia, 1999). The methanol solvent molecule is not shown.

**Figure 5**

The molecular structure of (V), showing displacement ellipsoids at the 50% probability level and H atoms as small spheres of arbitrary radii (Farrugia, 1999).

independent molecules of (III) and (VI) is shown] is shown in Figs. 7(a) and 7(b), and the rotation angles of the phenyl rings are given in Table 1. The main difference between the conformations of molecules (I) and (III) is the rotation of the phenyl ring at position 7. The rotation angles in (I) and (II) are 44.8 (7) and 45.5 (4)°, respectively, compared with 15.7 (5) and 31.5 (6)° in molecules (IIIa) and (IIIb), respectively. This difference can be explained by the larger repulsion of the nitro group in the *ortho* position compared with that in the *para* position. In the presence of a methyl group, the rotation angle decreases in the following sequence: *o*-nitro in (IV) [85.2 (1)°] > *m*-nitro in (V) [36.0 (1)°] > *p*-nitro in (VI) [26.0 (1)° in (VIa) and 30.8 (1)° in (VIb)]. It should be noted that the conformations of the two crystallographically independent molecules in (VI) are different with respect to the rotation of the phenyl rings in positions 8 and 9 (see notation in the scheme). The effect of the methyl group is also seen in the angles C1–C7–N1 and C1–C7–N2, where the angle is larger on the side with the methyl group: 124.7 (3) and 123.2 (3)° in (IV), 126.1 (2) and 122.6 (2)° in (V), 126.9 (2) and 122.2 (2)° in (VIa), and 125.8 (2) and 123.2 (2)° in (VIb), respectively. In the absence of the methyl group [compounds (I)–(III)] the opposite relation between values of the equivalent bond angles is observed: 123.2 (3) and 126.1 (3)° in (I), 122.9 (2) and 125.2 (2)° (*o*-nitro group) in (II), 124.4 (3) and 125.2 (3)° in (IIIa), and 124.1 (3) and 125.3 (3)° (*p*-nitro group) in (IIIb).

The molecular packing is similar in compounds (I)–(III) as they all form folded chains comprising molecules bonded to each other by hydrogen bonds. However, while in the crystals of (I) and (II) the molecules are connected to each other through N–H···N hydrogen bonds (Figs. 8 and 9, respectively), in (III) a mediator molecule, *viz.* methanol, forms hydrogen bonds between each pair of molecules of (III). The geometry of the hydrogen bonds is given in Table 2. A similar chain formation by hydrogen bonds has been observed in five other lophine derivatives (Seethalakshmi *et al.*, 2006; Inouye

**Figure 6**

The molecular structures of molecules (VIa) and (VIb), showing displacement ellipsoids at the 50% probability level and H atoms as small spheres of arbitrary radii (Farrugia, 1999).

& Sakaino, 1986; Thuer *et al.*, 2004; Liu *et al.*, 2005; Thiruvalluvar *et al.*, 2007). While the methanol solvent molecule in (III) has a specific role in the packing arrangement as described above, the role of the acetonitrile molecules in solvate (II) is to occupy lattice voids. In the absence of the imidazole N–H H-atom donor, the packing of molecules in (IV)–(VI) is determined by much weaker intermolecular interactions, such as weak C–H···O hydrogen bonds between the O atoms of the nitro group and aromatic H atoms (Table 2). The methanol solvent molecule in (IV), although hydrogen bonded to the imidazole [O33···N2 = 2.823 (4) Å], does not play any significant role in the unit-cell packing. Differential scanning calorimetry (DSC) thermographs (see Figs. s1 and s2 in the supplementary material) show that the

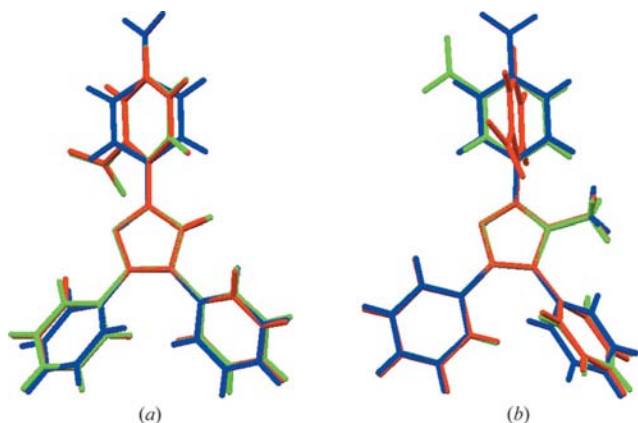


Figure 7

Overlay of molecules (a) of (I)–(III) (red, green and blue, respectively, in the electronic version of the paper) and (b) of (IV)–(VI) (red, green and blue, respectively, in the electronic version of the paper). Only one molecule [(IIIa) and (VIa)] is shown for compounds (III) and (VI).

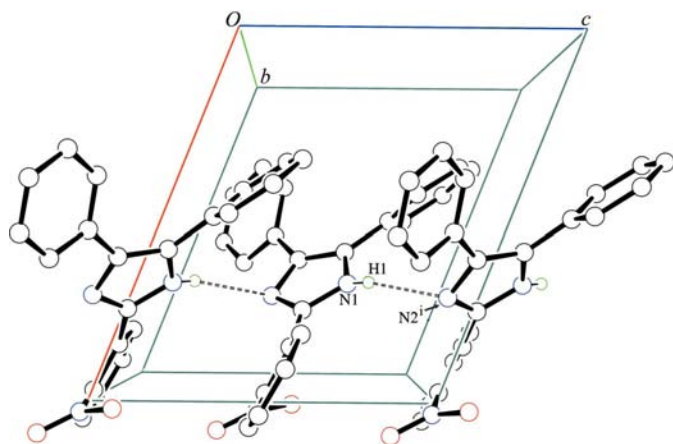


Figure 8

Hydrogen-bonding in (I). [Symmetry code: (i) $x, -y, \frac{1}{2} + z$.]

solvent molecules are removed at 394 and 377 K for (II) and (III), respectively. Upon removal of acetonitrile from (II) there is no change in the crystal colour, whereas removal of the methanol from (III) changes the crystal colour from light yellow to red. It is therefore suggested that the change of colour is a result of the change in the hydrogen bonding. This assumption needs further investigation.

Experimental

Benzil (1 mmol), the appropriate benzaldehyde (1 mmol) and ammonium acetate (1.2 g) were dissolved in boiling glacial acetic acid (16 ml) and refluxed for 5–24 h. On completion of the reaction, the reaction mixture was poured into ice water, washed with NaHCO_3 and then washed several times with ethyl acetate. The combined extracts were dried over MgSO_4 . The purification was carried out by flash column chromatography. Compound (I) crystallized solvent-free from ethanol and from acetonitrile as monosolvate (II);

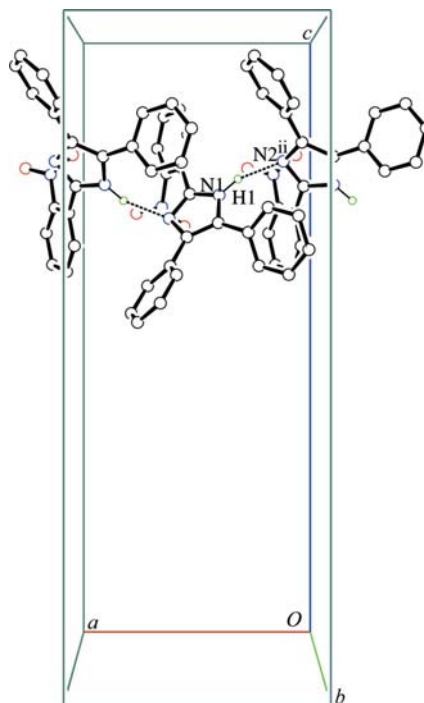


Figure 9

Hydrogen-bonding in (II). [Symmetry code: (ii) $-\frac{1}{2} + x, y, \frac{3}{2} - z$.]

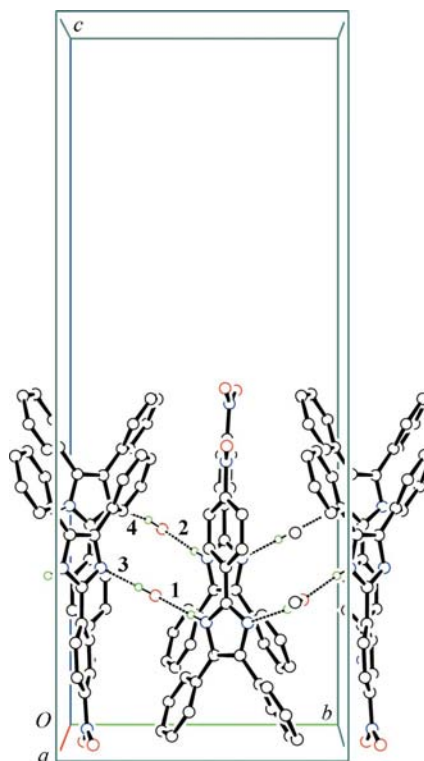


Figure 10

Hydrogen-bonding in (III). The bold numbers 1–4 refer to the hydrogen bonds as they are listed for (III) in Table 2.

compounds (III) and (IV) both crystallized from methanol as monosolvates; compound (V) crystallized from acetonitrile and compound (VI) from ethanol.

Compound (I)

Crystal data

$C_{21}H_{15}N_3O_2$ $V = 1752.6$ (7) Å³
 $M_r = 341.36$ $Z = 4$
 Monoclinic, Cc Mo $K\alpha$ radiation
 $a = 10.521$ (2) Å $\mu = 0.09$ mm⁻¹
 $b = 19.769$ (4) Å $T = 293$ K
 $c = 9.056$ (2) Å $0.59 \times 0.37 \times 0.08$ mm
 $\beta = 111.49$ (3)°

Data collection

Nonius KappaCCD diffractometer 943 reflections with $I > 2\sigma(I)$
 6192 measured reflections $R_{int} = 0.083$
 1533 independent reflections

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$ 2 restraints
 $wR(F^2) = 0.090$ H-atom parameters constrained
 $S = 0.88$ $\Delta\rho_{max} = 0.12$ e Å⁻³
 1533 reflections $\Delta\rho_{min} = -0.13$ e Å⁻³
 236 parameters

Compound (II)

Crystal data

$C_{21}H_{15}N_3O_2 \cdot C_2H_5N$ $V = 4075.2$ (15) Å³
 $M_r = 382.41$ $Z = 8$
 Orthorhombic, $Pbca$ Mo $K\alpha$ radiation
 $a = 9.111$ (2) Å $\mu = 0.08$ mm⁻¹
 $b = 18.830$ (4) Å $T = 293$ K
 $c = 23.754$ (5) Å $0.50 \times 0.09 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer 1506 reflections with $I > 2\sigma(I)$
 6703 measured reflections $R_{int} = 0.080$
 3584 independent reflections

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$ 263 parameters
 $wR(F^2) = 0.106$ H-atom parameters constrained
 $S = 0.81$ $\Delta\rho_{max} = 0.15$ e Å⁻³
 3584 reflections $\Delta\rho_{min} = -0.16$ e Å⁻³

Compound (III)

Crystal data

$C_{21}H_{15}N_3O_2 \cdot CH_4O$ $V = 3884.8$ (12) Å³
 $M_r = 373.40$ $Z = 8$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 11.728$ (2) Å $\mu = 0.09$ mm⁻¹
 $b = 11.365$ (2) Å $T = 293$ K
 $c = 29.241$ (6) Å $0.20 \times 0.20 \times 0.05$ mm
 $\beta = 94.62$ (2)°

Data collection

Nonius KappaCCD diffractometer 2718 reflections with $I > 2\sigma(I)$
 22154 measured reflections $R_{int} = 0.105$
 6747 independent reflections

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.067$ 505 parameters
 $wR(F^2) = 0.156$ H-atom parameters constrained
 $S = 0.88$ $\Delta\rho_{max} = 0.17$ e Å⁻³
 6747 reflections $\Delta\rho_{min} = -0.18$ e Å⁻³

Compound (IV)

Crystal data

$C_{22}H_{17}N_3O_2 \cdot CH_4O$ $V = 2017.8$ (7) Å³
 $M_r = 387.43$ $Z = 4$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 5.8130$ (10) Å $\mu = 0.09$ mm⁻¹
 $b = 15.003$ (3) Å $T = 293$ K
 $c = 23.243$ (5) Å $0.09 \times 0.02 \times 0.02$ mm
 $\beta = 95.480$ (3)°

Data collection

Nonius KappaCCD diffractometer 1604 reflections with $I > 2\sigma(I)$
 11012 measured reflections $R_{int} = 0.084$
 3292 independent reflections

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$ 263 parameters
 $wR(F^2) = 0.150$ H-atom parameters constrained
 $S = 0.91$ $\Delta\rho_{max} = 0.15$ e Å⁻³
 3292 reflections $\Delta\rho_{min} = -0.24$ e Å⁻³

Compound (V)

Crystal data

$C_{22}H_{17}N_3O_2$ $V = 1802.6$ (6) Å³
 $M_r = 355.39$ $Z = 4$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 16.242$ (3) Å $\mu = 0.09$ mm⁻¹
 $b = 9.583$ (2) Å $T = 293$ K
 $c = 11.615$ (2) Å $0.08 \times 0.03 \times 0.01$ mm
 $\beta = 94.36$ (2)°

Data collection

Nonius KappaCCD diffractometer 1975 reflections with $I > 2\sigma(I)$
 10123 measured reflections $R_{int} = 0.055$
 3139 independent reflections

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$ 244 parameters
 $wR(F^2) = 0.154$ H-atom parameters constrained
 $S = 1.09$ $\Delta\rho_{max} = 0.26$ e Å⁻³
 3139 reflections $\Delta\rho_{min} = -0.21$ e Å⁻³

Compound (VI)

Crystal data

$C_{22}H_{17}N_3O_2$ $\gamma = 109.63$ (3)°
 $M_r = 355.39$ $V = 1788.2$ (10) Å³
 Triclinic, $P\bar{1}$ $Z = 4$
 $a = 11.855$ (2) Å Mo $K\alpha$ radiation
 $b = 12.384$ (2) Å $\mu = 0.09$ mm⁻¹
 $c = 14.791$ (3) Å $T = 293$ K
 $\alpha = 111.95$ (3)° $0.50 \times 0.05 \times 0.05$ mm
 $\beta = 99.56$ (3)°

Data collection

Nonius KappaCCD diffractometer 4081 reflections with $I > 2\sigma(I)$
 14497 measured reflections $R_{int} = 0.091$
 6250 independent reflections

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$ 489 parameters
 $wR(F^2) = 0.130$ H-atom parameters constrained
 $S = 0.89$ $\Delta\rho_{max} = 0.16$ e Å⁻³
 6248 reflections $\Delta\rho_{min} = -0.19$ e Å⁻³

Table 1

Comparison of phenyl ring (Ph) rotation ($^{\circ}$) with respect to the imidazole ring.

| | Ph rotation at position 2 | Ph rotation at position 4 | Ph rotation at position 5 |
|--------|---------------------------|---------------------------|---------------------------|
| (I) | 44.8 (2) | 45.0 (2) | 34.3 (2) |
| (II) | 45.5 (1) | 41.0 (2) | 33.1 (1) |
| (IIIa) | 15.7 (2) | 37.4 (2) | 46.5 (2) |
| (IIIb) | 31.5 (1) | 30.9 (2) | 35.3 (2) |
| (IV) | 85.2 (1) | 69.5 (1) | 24.3 (2) |
| (V) | 30.5 (1) | 65.5 (1) | 24.2 (1) |
| (VIa) | 26.0 (1) | 54.5 (1) | 25.8 (2) |
| (VIb) | 30.8 (1) | 81.1 (1) | 12.5 (2) |

Table 2

Strong and weak hydrogen-bond geometry.

| Compound | $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------|-----------------------------|-------|-------------|-------------|---------------|
| (I) | $N1-H1\cdots N2^i$ | 0.86 | 2.10 | 2.942 (5) | 165 |
| (II) | $N1-H1\cdots N2^{ii}$ | 0.99 | 1.97 | 2.920 (3) | 161 |
| (III) | $N1a-H1a\cdots O3a$ | 0.86 | 1.96 | 2.819 (4) | 175 |
| | $N1b-H1b\cdots O3b^{iii}$ | 0.86 | 1.95 | 2.788 (4) | 166 |
| | $O3a-H3Oa\cdots N2b^{iii}$ | 0.82 | 1.98 | 2.791 (4) | 171 |
| | $O3b-H3Ob\cdots N2a$ | 0.82 | 1.98 | 2.792 (4) | 169 |
| (IV) | $C18-H18\cdots O2^{iv}$ | 0.96 | 2.56 | 3.426 (4) | 150 |
| (V) | $C4-H4\cdots O1^v$ | 0.96 | 2.48 | 3.148 (3) | 127 |
| (VI) | $C12a-H12a\cdots O1a^{vi}$ | 0.96 | 2.66 | 3.544 (3) | 154 |
| | $C14b-H14b\cdots O2b^{vii}$ | 0.96 | 2.63 | 3.258 (3) | 124 |
| | $C18b-H18b\cdots O1a^{vi}$ | 0.96 | 2.69 | 3.241 (3) | 118 |
| | $C19b-H19b\cdots O1b^{vi}$ | 0.96 | 2.58 | 3.431 (3) | 148 |

Symmetry codes: (i) $x, -y, \frac{1}{2} + z$; (ii) $\frac{1}{2} - x, y, \frac{3}{2} - z$; (iii) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (iv) $1 - x, 1 - y, 1 - z$; (v) $x, \frac{3}{2} - y, \frac{1}{2} + z$; (vi) $x, -1 + y, -1 + z$; (vii) $x, y, -1 + z$.

H atoms were clearly found in difference Fourier maps. The amine H atom was refined in the earlier stages of the refinement and was fixed in the later stages. All other H atoms were refined at idealized positions riding on the parent C and N atoms, with $C-H = 0.96 \text{ \AA}$, $N-H = 0.86 \text{ \AA}$ and $O-H = 0.82 \text{ \AA}$, and with $U_{iso}(H) = 1.2U_{eq}(C, N \text{ or } O)$.

For all compounds, data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO HKL-2000* (Otwinowski & Minor, 1997); data reduction: *DENZO HKL-2000*; program(s) used to solve structure:

SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3195). Additional figures are also available. Services for accessing these data are described at the back of the journal.

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