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Biphenyl- and phenylnaphthalenyl-substituted 1*H*-imidazole-4,5-dicarbonitrile catalysts for the coupling reaction of nucleoside methyl phosphonamidites

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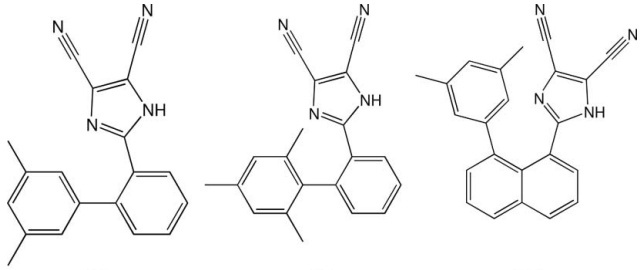
Crystal structures are reported for three substituted 1*H*-imidazole-4,5-dicarbonitrile compounds used as catalysts for the coupling reaction of nucleoside methyl phosphonamidites, namely 2-(3′,5′-dimethylbiphenyl-2-yl)-1*H*-imidazole-4,5-dicarbonitrile, $C_{19}H_{14}N_4$, (I), 2-(2′,4′,6′-trimethylbiphenyl-2-yl)-1*H*-imidazole-4,5-dicarbonitrile, $C_{20}H_{16}N_4$, (II), and 2-[8-(3,5-dimethylphenyl)naphthalen-1-yl]-1*H*-imidazole-4,5-dicarbonitrile, $C_{23}H_{16}N_4$, (III). The asymmetric unit of (I) contains two independent molecules with similar conformations. There is steric repulsion between the imidazole group and the terminal phenyl group in all three compounds, resulting in the nonplanarity of the molecules. The naphthalene group of (III) shows significant deviation from planarity. The C—N bond lengths in the imidazole rings range from 1.325 (2) to 1.377 (2) Å. The molecules are connected into zigzag chains by intermolecular N—H···$N_{imidazole}$ [for (I)] or N—H···N_{cyano} [for (II) and (III)] hydrogen bonds.

Comment

Methyl phosphonate oligonucleotides differ from conventional oligonucleotides by the replacement of a negatively charged O atom at the phosphonate linkage by a methyl group. They are more stable to degradation by cellular nucleases than are conventional oligonucleotides, and they can be applied in the antisense concept to control gene expression in mammalian cells (Uhlmann & Peyman, 1990). The P atoms in methyl phosphonate oligonucleotides are chiral, and R_P-configured molecules bind better to their target strand than do S_P-configured ones (Lebedev & Wickstrom, 1996). The phosphoramidite approach can be used for the synthesis of methyl phosphonate oligonucleotides (Jäger & Engels, 1984). The reaction is activated by the use of tetrazole but no stereocontrol occurs at the P atom. Also, the use of tetrazoles with a chiral substituent gives only weak selectivity (Schell & Engels, 1997). Therefore, substituted 1*H*-imidazole-4,5-dicarbonitriles were tested as activators for stereoselection during the synthesis of dinucleoside methyl phosphonates. Selectivities up to 84/16 (R_P/S_P) have been reported by Schell & Engels (1998). Single crystals were obtained for three different substituted 1*H*-imidazole-4,5-dicarbonitriles used for these experiments and the crystal structures of these three compounds, namely 2-(3′,5′-dimethylbiphenyl-2-yl)-1*H*-imidazole-4,5-dicarbonitrile, (I), 2-(2′,4′,6′-trimethylbiphenyl-2-yl)-1*H*-imidazole-4,5-dicarbonitrile, (II), and 2-[8-(3,5-dimethylphenyl)naphthalen-1-yl]-1*H*-imidazole-4,5-dicarbonitrile, (III), are reported here.



(I) (II) (III)

The crystal structure of (I) contains two crystallographically independent molecules, *A* and *B* (Fig. 1), which have very similar conformations. The angles between the planes of the six-membered rings of the biphenyl group are 44.91 (6)° for molecule *A* and 42.09 (6)° for molecule *B*. The angle between the plane of the imidazole group and that of the central phenyl group is 46.02 (7)° for molecule *A* and 39.57 (7)° for molecule *B*. There is significant steric repulsion between the dimethylphenyl and imidazole groups [shortest contacts: C5···C13 = 3.115 (3) Å, N5···C20 = 3.094 (3) Å, N5···C25 = 3.144 (2) Å and C20···C32 = 3.115 (3) Å]. These steric contacts are not only responsible for the nonplanar conformation of the biphenyl group, but also result in an out-of-plane distortion of the dimethylphenyl and imidazole groups with respect to the plane of the central benzene ring. Consequently, the C6—C7—C12—C13 and C25—C26—C31—C32 torsion angles are −6.6 (3) and −9.6 (3)°, respectively. The amino N atoms are planar, the sums of the three valence angles about atoms N2 and N5 being 359.4 (8) and 360.0 (9)°, respectively. The molecules are connected by N—H···N hydrogen bonds (Fig. 2 and Table 1) between the imidazole groups to form zigzag chains parallel to the *b*-axis direction. These chains are connected by weak intermolecular C—H···N contacts (Table 1) to give a three-dimensional structure.

The molecular structure of (II) is shown in Fig. 3. The angle between the planes of the six-membered rings of the biphenyl group is 71.20 (4)°, almost 30° larger than the corresponding values in (I), resulting from steric repulsions between the *ortho*-methyl groups and the imidazole group on one hand and the C10—H10*A* bond on the other [shortest contacts: C10···C18 = 3.261 (2) Å and C1···H20*C* = 2.75 Å]. The angle between the planes of the imidazole ring and the central benzene ring is 29.39 (6)°, slightly smaller than the corresponding values observed in (I), despite the short intramolecular contact distance of 2.879 (2) Å between atoms N1

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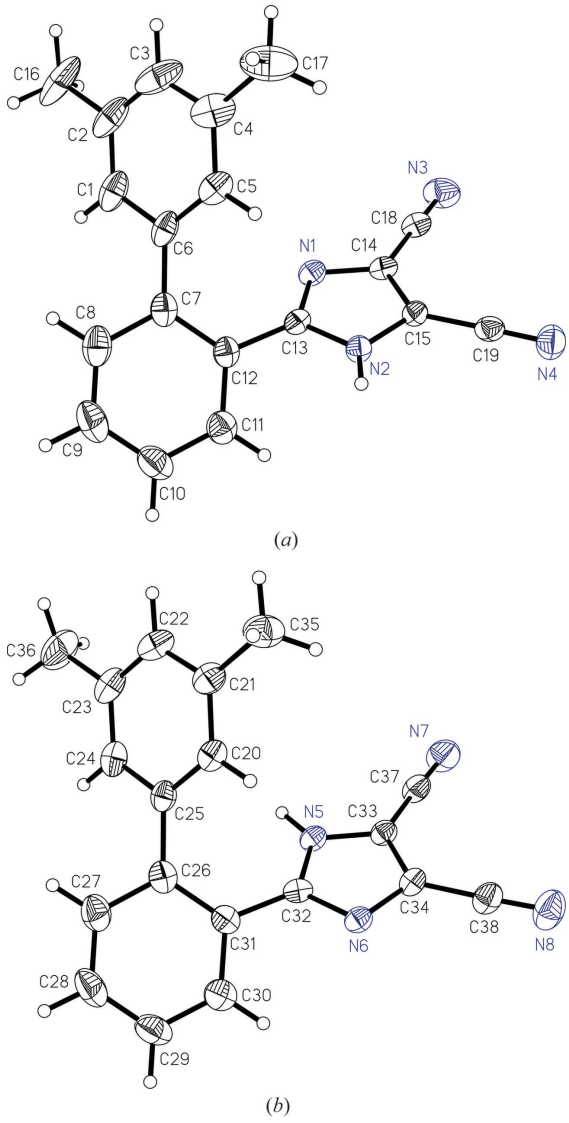

Figure 1
The structures of (*a*) molecule *A* and (*b*) molecule *B* of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

and C12. The C1—C6—C11—C12 torsion angle of 2.69 (18)° again shows a small out-of-plane distortion of the trimethylphenyl and imidazole groups from the plane of the central benzene ring, but this distortion is smaller than the corresponding distortions observed in (I). The amino N atom is planar, the sum of the three valence angles about atom N1 being 359.6 (7)°. The crystal packing of (II) is shown in Fig. 4. The molecules are connected by rather weak intermolecular hydrogen bonds between the N—H bond and a cyano N atom of a neighbouring molecule (Table 2) to form zigzag chains along the *c*-axis direction. The long H···N distance of 2.351 (15) Å and the small N—H···N angle of 127.7 (12)° show this hydrogen bond to be rather weak. The crystal packing also shows two weak intermolecular C—H···N_{cyano} contacts (Table 2). One of these results in an additional stabilization of the hydrogen-bonded chain and the other connects the chains along the *b*-axis direction. Along the *a* axis

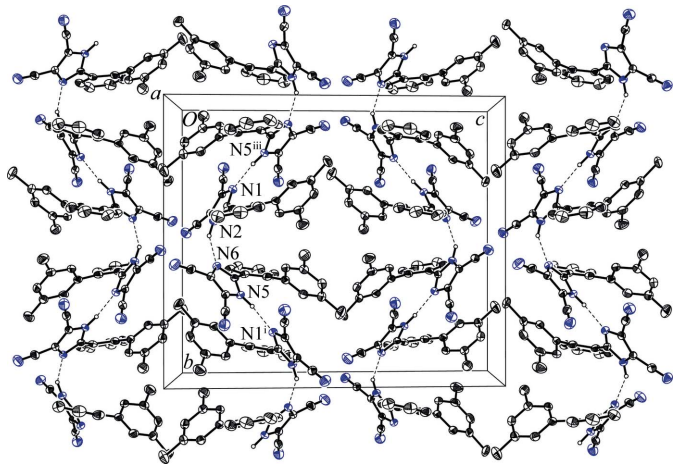

Figure 2
The crystal packing of (I), viewed down [100]. Displacement ellipsoids are drawn at the 50% probabilty level. C-bound H atoms have been omitted for clarity. Intermolecular hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.]

direction, the molecules are only connected by intermolecular C—H···$\pi_{imidazole}$ contacts (final three entries of Table 2). These contacts are very weak. Consequently, the crystal habit is a (100) plate.

The molecular structure of (III) is shown in Fig. 5. The naphthalene group shows considerable deviation from planarity (mean deviation from best plane = 0.041 Å). Ring atoms C2 and C10 deviate by 0.0567 (14) and 0.0788 (14) Å, respectively, in opposite directions from the best plane of the naphthalene group. The out-of-plane deviation from the naphthalene plane is even more pronounced for substituent atoms C11 and C19 [deviations of 0.221 (2) and 0.299 (2) Å, respectively] as a result of steric interactions between the dimethylphenyl and imidazole groups. The shortest contact

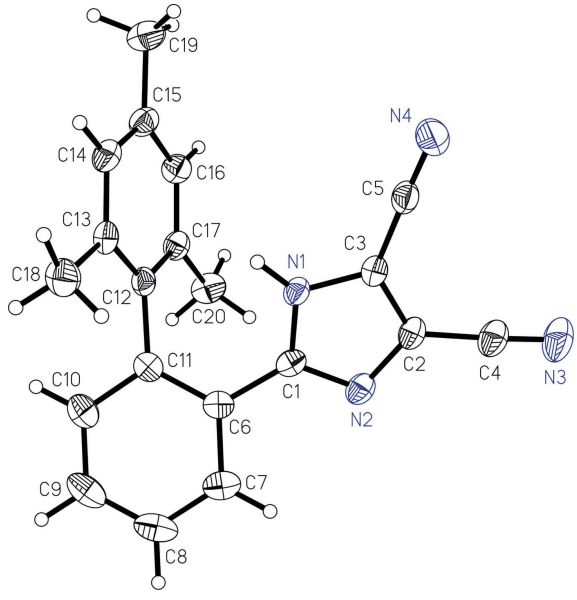

Figure 3
The molecular structure of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

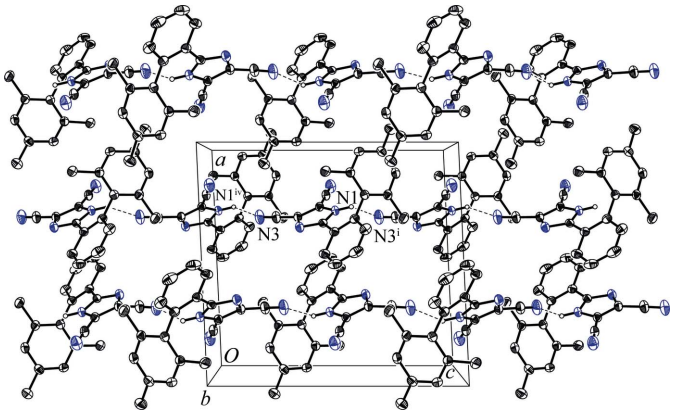

Figure 4
The crystal packing of (II), viewed down [010]. Displacement ellipsoids are drawn at the 50% probabilty level. C-bound H atoms have been omitted for clarity. Intermolecular hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (iv) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.]

distances are: C11···N2 = 2.908 (2) Å, C11···C19 = 2.988 (2) Å and C12···C19 = 2.937 (2) Å. Steric repulsion between the dimethylphenyl and imidazole groups also results in rather large values of the C1—C2—C11, C2—C1—C10 and C1—C10—C19 bond angles [124.59 (15), 126.37 (14) and 123.98 (14)°, respectively]. The dihedral angle between the naphthalene and dimethylphenyl planes is 55.20 (4)°, between the naphthalene and imidazole planes is 54.11 (6)°, and between the dimethylphenyl and imidazole planes is 22.71 (5)°. The amino N atom is again planar, the sum of the three valence angles about atom N2 being 359.9 (10)°. The crystal packing of (III) is shown in Fig. 6. The molecules are connected by intermolecular N—H···N_{cyano} hydrogen bonds (Table 3) to form zigzag chains parallel to the *c*-axis direction.

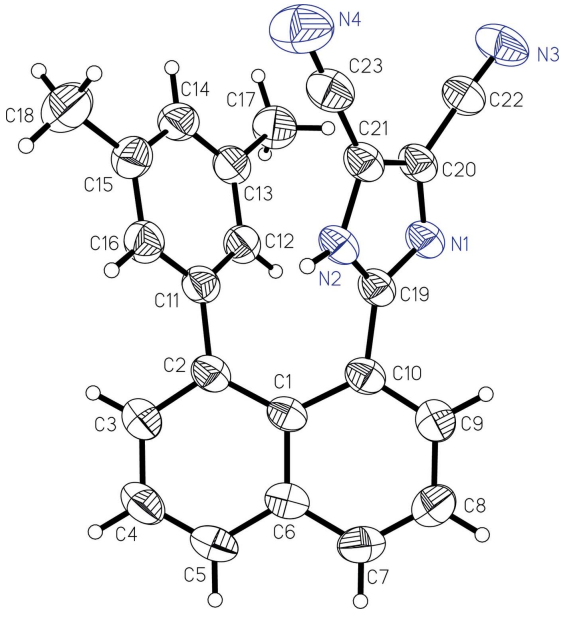

Figure 5
The molecular structure of (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

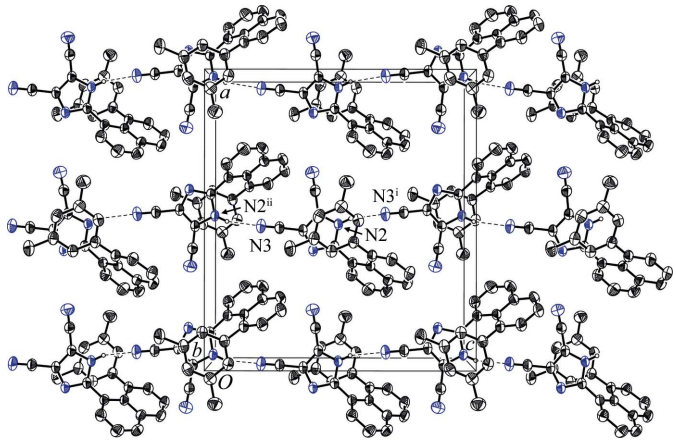

Figure 6
The crystal packing of (III), viewed down [010]. Displacement ellipsoids are drawn at the 50% probabilty level. C-bound H atoms have been omitted for clarity. Intermolecular hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $-x+1, -y, z+\frac{1}{2}$; (ii) $-x+1, -y, z-\frac{1}{2}$.]

There are no other short intermolecular contact distances. The shortest intermolecular C—H···N contact has an H···N distance of 2.74 Å.

The bond lengths and angles in the 1*H*-imidazole-4,5-dicarbonitrile groups of (I), (II) and (III) are in excellent agreement. They are also very similar to the corresponding bond lengths and angles observed in the crystal structure of 1*H*-imidazole-4,5-dicarbonitrile (Barni *et al.*, 1997). The C—N bond lengths in the imidazole rings range from 1.325 (2) to 1.377 (2) Å, thus showing a considerable degree of delocalization of the double bonds in the ring. The N—H···$N_{imidazole}$ hydrogen bond in (I) is considerably shorter than the N—H···N_{cyano} hydrogen bonds in (II) and (III).

The three title compounds have been tested as activators in the coupling reaction to produce a protected thymine dinucleoside methylphosphonate TpT (Schell & Engels, 1998). The R_P/S_P stereoselectivities were 65/35 for (I) and 77/23 for (II), while no reaction occurred for (III). Thus, the stereoselectivity of these compounds depends very much on the substituent of the imidazole group.

Experimental

The starting materials for the preparation of the title compounds were 3′,5′-dimethylbiphenyl-2-carbonitrile for (I), 2′,4′,6′-trimethylbiphenyl-2-carbonitrile for (II) and 8-(3,5-dimethylphenyl)naphthalene-1-carbonitrile for (III). The starting material (15 mmol) was reacted for 2 h with diisobutylaluminium hydride (18 mmol) in ether (20 ml), and then for 5 min with 2,3-diaminomaleonitrile (15 mmol) and concentrated H_2SO_4 (7.5 mmol) in tetrahydrofuran (40 ml). Thus, the cyano group of the starting compound was replaced by a 2-amino-3-methylideneaminobut-2-enedinitrile chain (Begland *et al.*, 1974). The products were purified by recrystallization from dichloromethane–*n*-hexane (1:1 *v*/*v*). Subsequently, they were further reacted with nicotinamide (18 mmol) and *N*-chlorosuccinimide (15 mmol) in dimethylformamide (38 ml), resulting in the cyclization of the 2-amino-3-methylideneaminobut-2-enedinitrile chain to form the desired 1*H*-imidazole-4,5-dicarbonitrile group (Moriya *et al.*,

Table 1
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N6	0.97 (2)	2.02 (2)	2.907 (2)	150.9 (17)
N5—H5B···N1^{i}	0.953 (19)	1.95 (2)	2.882 (2)	164.4 (17)
C11—H11A···N8^{ii}	0.95	2.69	3.567 (3)	154
C16—H16B···N6^{iii}	0.98	2.60	3.484 (3)	150
C16—H16C···N3^{iv}	0.98	2.65	3.497 (3)	145

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

1984). The final products were recrystallized from dichloromethane–*n*-hexane (1:1 *v*/*v*), resulting in colourless crystals of (I), (II) or (III). The yields of the three products were 74, 77 and 63%, respectively.

Compound (I)

Crystal data

$C_{19}H_{14}N_4$
M_r = 298.34
Monoclinic, $P2_1/n$
a = 9.771 (2) Å
b = 16.807 (2) Å
c = 19.692 (2) Å
β = 93.976 (7)°
V = 3226.1 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 134 K
0.36 × 0.30 × 0.15 mm

Data collection

Siemens SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
T_{min} = 0.867, T_{max} = 0.989
47542 measured reflections
7317 independent reflections
3808 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.093
S = 0.92
7317 reflections
428 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Compound (II)

Crystal data

$C_{20}H_{16}N_4$
M_r = 312.37
Monoclinic, $P2_1/c$
a = 12.373 (1) Å
b = 10.303 (2) Å
c = 13.279 (2) Å
β = 92.578 (7)°
V = 1691.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 133 K
0.60 × 0.40 × 0.10 mm

Data collection

Siemens SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
T_{min} = 0.784, T_{max} = 0.993
25406 measured reflections
4381 independent reflections
3014 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.04
4381 reflections
225 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N3^{i}	0.901 (14)	2.351 (15)	2.9883 (16)	127.7 (12)
C14—H14A···N4^{i}	0.95	2.62	3.5476 (17)	166
C19—H19C···N4^{ii}	0.98	2.66	3.6327 (19)	175
C16—H16A···C3^{iii}	0.95	2.96	3.8507 (17)	158
C16—H16A···C5^{iii}	0.95	3.07	3.8580 (18)	142
C19—H19A···C2^{iii}	0.98	2.91	3.7899 (19)	150

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

Compound (III)

Crystal data

$C_{23}H_{16}N_4$
M_r = 348.40
Orthorhombic, $Pca2_1$
a = 16.7042 (14) Å
b = 7.1632 (7) Å
c = 15.0528 (11) Å
V = 1801.2 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.62 mm^{-1}
T = 294 K
0.60 × 0.40 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan *MolEN* (Fair, 1990)
T_{min} = 0.776, T_{max} = 0.857
3815 measured reflections
1781 independent reflections
1753 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
3 standard reflections every 90 min
intensity decay: 2.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.070
S = 1.05
1781 reflections
251 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Friedel opposites were merged for (III) and no attempt was made to determine the direction of the *c* axis for the polar space group. C-bound H atoms were positioned geometrically and treated as riding, with planar C—H = 0.95 Å for (I) and (II), and 0.93 Å for (III), and methyl C—H = 0.98 Å for (I) and (II), and 0.96 Å for (III), with U_{iso}(H) = 1.2U_{eq}(planar C) or 1.5U_{eq}(methyl C). N-bound H atoms were located from difference Fourier syntheses and their coordinates and isotropic displacement parameters were refined.

Data collection: *SMART* (Siemens, 1995) for (I) and (II); *CAD-4 Software* (Enraf–Nonius, 1989) for (III). Cell refinement: *SMART* for (I) and (II); *CAD-4 Software* for (III). Data reduction: *SAINT* (Siemens, 1995) for (I) and (II); *MolEN* (Fair, 1990) for (III). For all three compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N3^{i}	0.81 (2)	2.26 (2)	3.001 (2)	152 (2)

Symmetry code: (i) $-x+1, -y, z+\frac{1}{2}$.

(Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: EG3118). Services for accessing these data are described at the back of the journal.

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supplementary materials

Acta Cryst. (2013). C**69**, 529-533 [doi:10.1107/S0108270113009293]

Biphenyl- and phenylnaphthalenyl-substituted 1*H*-imidazole-4,5-dicarbonitrile catalysts for the coupling reaction of nucleoside methyl phosphonamidites

Jan W. Bats, Peter Schell and Joachim W. Engels

(I) 2-(3′,5′-Dimethylbiphenyl-2-yl)-1*H*-imidazole-4,5-dicarbonitrile

Crystal data

$C_{19}H_{14}N_4$
M_r = 298.34
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 9.771 (2) Å
b = 16.807 (2) Å
c = 19.692 (2) Å
β = 93.976 (7)°
V = 3226.1 (8) Å^3
Z = 8
$F(000)$ = 1248
D_x = 1.229 Mg m^{-3}
Melting point: 425(1) K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 244 reflections
θ = 3–23°
μ = 0.08 mm^{-1}
T = 134 K
Block, colourless
0.36 × 0.30 × 0.15 mm

Data collection

Siemens SMART 1K CCD area-detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
T_{min} = 0.867, T_{max} = 0.989
47542 measured reflections
7317 independent reflections
3808 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 27.5°, θ_{min} = 2.1°
h = −12→12
k = −21→21
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.093
S = 0.92
7317 reflections
428 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0032 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.65061 (16)	0.31498 (8)	0.18696 (7)	0.0262 (4)
N2	0.66519 (16)	0.42836 (9)	0.13001 (8)	0.0240 (4)
N3	0.32788 (19)	0.22942 (10)	0.14701 (9)	0.0458 (5)
N4	0.35949 (17)	0.46877 (10)	0.03112 (9)	0.0386 (5)
N5	0.83027 (16)	0.68623 (9)	0.21618 (8)	0.0244 (4)
N6	0.78633 (15)	0.58594 (8)	0.14454 (7)	0.0246 (4)
N7	1.14192 (19)	0.77904 (11)	0.18084 (9)	0.0463 (5)
N8	1.05867 (19)	0.57761 (10)	0.03535 (9)	0.0473 (5)
C1	0.8832 (2)	0.30175 (11)	0.36994 (10)	0.0394 (6)
H1A	0.9654	0.2729	0.3648	0.047*
C2	0.8079 (3)	0.28650 (12)	0.42548 (11)	0.0461 (6)
C3	0.6906 (3)	0.33087 (13)	0.43355 (11)	0.0524 (7)
H3A	0.6398	0.3217	0.4722	0.063*
C4	0.6451 (2)	0.38853 (12)	0.38642 (11)	0.0436 (6)
C5	0.7203 (2)	0.40035 (11)	0.33000 (10)	0.0331 (5)
H5A	0.6886	0.4380	0.2965	0.040*
C6	0.8407 (2)	0.35880 (11)	0.32105 (10)	0.0306 (5)
C7	0.9277 (2)	0.37580 (10)	0.26369 (10)	0.0291 (5)
C8	1.0694 (2)	0.38304 (12)	0.27705 (12)	0.0412 (6)
H8A	1.1080	0.3738	0.3220	0.049*
C9	1.1549 (2)	0.40316 (13)	0.22702 (13)	0.0490 (6)
H9A	1.2507	0.4080	0.2380	0.059*
C10	1.1024 (2)	0.41641 (12)	0.16112 (12)	0.0448 (6)
H10A	1.1613	0.4311	0.1268	0.054*
C11	0.9627 (2)	0.40803 (11)	0.14577 (11)	0.0349 (5)
H11A	0.9257	0.4163	0.1004	0.042*
C12	0.87566 (19)	0.38757 (10)	0.19627 (10)	0.0267 (5)
C13	0.73079 (19)	0.37624 (10)	0.17362 (9)	0.0239 (4)
C14	0.52892 (19)	0.32911 (10)	0.14981 (9)	0.0248 (4)
C15	0.53520 (18)	0.39910 (10)	0.11427 (9)	0.0237 (4)
C16	0.8508 (3)	0.22143 (13)	0.47562 (11)	0.0680 (9)
H16A	0.9500	0.2245	0.4868	0.102*
H16B	0.8279	0.1694	0.4553	0.102*
H16C	0.8024	0.2282	0.5172	0.102*
C17	0.5192 (3)	0.43783 (13)	0.39721 (12)	0.0625 (8)
H17A	0.5472	0.4893	0.4170	0.094*
H17B	0.4614	0.4098	0.4282	0.094*

H17C	0.4674	0.4464	0.3534	0.094*
C18	0.4169 (2)	0.27400 (11)	0.14893 (9)	0.0301 (5)
C19	0.4373 (2)	0.43810 (11)	0.06859 (10)	0.0276 (5)
C20	0.8040 (2)	0.59259 (10)	0.34993 (10)	0.0291 (5)
H20A	0.8222	0.5587	0.3130	0.035*
C21	0.8987 (2)	0.59682 (11)	0.40554 (10)	0.0332 (5)
C22	0.8713 (2)	0.64726 (12)	0.45888 (10)	0.0389 (5)
H22A	0.9344	0.6499	0.4978	0.047*
C23	0.7535 (2)	0.69400 (12)	0.45661 (10)	0.0390 (5)
C24	0.6598 (2)	0.68791 (11)	0.40072 (10)	0.0341 (5)
H24A	0.5785	0.7191	0.3991	0.041*
C25	0.6828 (2)	0.63663 (11)	0.34664 (9)	0.0274 (5)
C26	0.5753 (2)	0.62615 (10)	0.28950 (10)	0.0291 (5)
C27	0.4381 (2)	0.62121 (12)	0.30480 (11)	0.0400 (6)
H27A	0.4155	0.6290	0.3505	0.048*
C28	0.3347 (2)	0.60536 (12)	0.25553 (12)	0.0466 (6)
H28A	0.2423	0.6027	0.2676	0.056*
C29	0.3642 (2)	0.59334 (12)	0.18868 (12)	0.0428 (6)
H29A	0.2933	0.5807	0.1549	0.051*
C30	0.4988 (2)	0.59996 (11)	0.17173 (11)	0.0342 (5)
H30A	0.5197	0.5930	0.1257	0.041*
C31	0.60429 (19)	0.61677 (10)	0.22107 (10)	0.0262 (4)
C32	0.74044 (19)	0.62799 (10)	0.19571 (9)	0.0236 (4)
C33	0.93823 (19)	0.68201 (10)	0.17594 (9)	0.0252 (4)
C34	0.9100 (2)	0.61976 (11)	0.13207 (9)	0.0260 (4)
C35	1.0268 (2)	0.54699 (12)	0.40878 (11)	0.0449 (6)
H35A	1.0887	0.5644	0.4472	0.067*
H35B	1.0726	0.5531	0.3664	0.067*
H35C	1.0027	0.4910	0.4149	0.067*
C36	0.7284 (2)	0.75154 (14)	0.51375 (11)	0.0598 (8)
H36A	0.7686	0.8034	0.5040	0.090*
H36B	0.7708	0.7308	0.5567	0.090*
H36C	0.6294	0.7576	0.5175	0.090*
C37	1.0505 (2)	0.73625 (12)	0.17970 (10)	0.0325 (5)
C38	0.9908 (2)	0.59460 (11)	0.07831 (10)	0.0329 (5)
H2A	0.700 (2)	0.4799 (12)	0.1180 (10)	0.056 (7)*
H5B	0.8208 (19)	0.7254 (11)	0.2505 (10)	0.050 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0317 (10)	0.0219 (8)	0.0251 (9)	−0.0011 (7)	0.0020 (8)	0.0026 (7)
N2	0.0248 (10)	0.0200 (8)	0.0270 (9)	−0.0018 (7)	−0.0001 (7)	0.0029 (7)
N3	0.0473 (13)	0.0432 (12)	0.0479 (12)	−0.0167 (10)	0.0091 (10)	0.0003 (9)
N4	0.0347 (11)	0.0396 (10)	0.0405 (11)	−0.0026 (9)	−0.0049 (9)	0.0061 (9)
N5	0.0305 (10)	0.0192 (8)	0.0234 (9)	−0.0011 (7)	0.0012 (7)	−0.0035 (7)
N6	0.0295 (10)	0.0209 (8)	0.0233 (9)	−0.0021 (7)	0.0009 (7)	−0.0020 (7)
N7	0.0491 (13)	0.0484 (12)	0.0416 (12)	−0.0197 (10)	0.0049 (9)	−0.0053 (9)
N8	0.0564 (14)	0.0427 (11)	0.0451 (12)	−0.0145 (10)	0.0186 (10)	−0.0117 (10)
C1	0.0572 (15)	0.0248 (11)	0.0334 (13)	−0.0024 (10)	−0.0162 (11)	−0.0026 (10)

C2	0.0819 (19)	0.0290 (12)	0.0248 (12)	−0.0129 (13)	−0.0139 (12)	−0.0009 (10)
C3	0.095 (2)	0.0366 (13)	0.0263 (13)	−0.0214 (14)	0.0097 (13)	−0.0066 (11)
C4	0.0660 (17)	0.0256 (12)	0.0405 (14)	−0.0087 (11)	0.0128 (12)	−0.0057 (10)
C5	0.0478 (14)	0.0218 (11)	0.0294 (12)	−0.0031 (10)	−0.0002 (10)	0.0004 (9)
C6	0.0422 (13)	0.0209 (10)	0.0269 (11)	−0.0022 (10)	−0.0108 (10)	−0.0018 (9)
C7	0.0303 (12)	0.0197 (10)	0.0362 (12)	0.0028 (9)	−0.0049 (10)	−0.0006 (9)
C8	0.0407 (15)	0.0332 (12)	0.0479 (14)	0.0065 (11)	−0.0111 (11)	0.0016 (11)
C9	0.0285 (13)	0.0482 (15)	0.0694 (18)	0.0048 (11)	−0.0040 (13)	0.0016 (13)
C10	0.0316 (14)	0.0458 (14)	0.0578 (16)	0.0026 (11)	0.0097 (12)	0.0040 (12)
C11	0.0342 (13)	0.0296 (11)	0.0406 (13)	0.0037 (10)	0.0006 (10)	0.0025 (10)
C12	0.0272 (11)	0.0194 (10)	0.0331 (12)	0.0036 (9)	−0.0017 (9)	0.0011 (9)
C13	0.0292 (12)	0.0219 (10)	0.0207 (10)	0.0023 (9)	0.0021 (9)	0.0003 (8)
C14	0.0307 (12)	0.0232 (10)	0.0210 (10)	−0.0035 (9)	0.0050 (9)	−0.0006 (8)
C15	0.0254 (11)	0.0239 (10)	0.0218 (10)	−0.0009 (9)	0.0009 (9)	0.0003 (8)
C16	0.126 (3)	0.0427 (14)	0.0311 (14)	−0.0179 (15)	−0.0274 (15)	0.0123 (11)
C17	0.084 (2)	0.0422 (15)	0.0654 (18)	−0.0048 (14)	0.0372 (16)	−0.0085 (13)
C18	0.0373 (13)	0.0285 (11)	0.0249 (11)	−0.0026 (10)	0.0049 (9)	0.0022 (9)
C19	0.0282 (12)	0.0268 (11)	0.0281 (11)	−0.0073 (9)	0.0046 (9)	0.0023 (9)
C20	0.0371 (12)	0.0234 (10)	0.0272 (11)	−0.0022 (9)	0.0051 (9)	−0.0010 (9)
C21	0.0414 (13)	0.0283 (11)	0.0297 (12)	−0.0018 (10)	0.0016 (10)	0.0026 (9)
C22	0.0527 (15)	0.0368 (12)	0.0265 (12)	−0.0021 (11)	−0.0032 (11)	0.0017 (10)
C23	0.0571 (16)	0.0340 (12)	0.0270 (12)	0.0001 (11)	0.0099 (11)	−0.0022 (10)
C24	0.0405 (14)	0.0291 (11)	0.0340 (12)	0.0039 (10)	0.0127 (10)	0.0021 (10)
C25	0.0322 (12)	0.0235 (10)	0.0275 (11)	−0.0021 (9)	0.0082 (9)	0.0018 (9)
C26	0.0314 (12)	0.0199 (10)	0.0366 (12)	0.0025 (9)	0.0065 (10)	−0.0009 (9)
C27	0.0349 (13)	0.0386 (13)	0.0478 (14)	0.0038 (11)	0.0114 (11)	−0.0053 (11)
C28	0.0261 (13)	0.0474 (15)	0.0673 (17)	0.0026 (11)	0.0103 (12)	−0.0083 (13)
C29	0.0291 (13)	0.0420 (13)	0.0560 (16)	0.0042 (11)	−0.0056 (11)	−0.0132 (12)
C30	0.0346 (13)	0.0262 (11)	0.0416 (13)	0.0042 (10)	0.0009 (10)	−0.0069 (9)
C31	0.0264 (11)	0.0181 (10)	0.0341 (11)	0.0023 (8)	0.0015 (9)	−0.0030 (9)
C32	0.0290 (12)	0.0176 (10)	0.0235 (10)	0.0000 (8)	−0.0038 (9)	0.0009 (8)
C33	0.0299 (12)	0.0231 (10)	0.0225 (10)	−0.0035 (9)	0.0017 (9)	−0.0009 (8)
C34	0.0330 (12)	0.0232 (10)	0.0221 (10)	−0.0003 (9)	0.0040 (9)	−0.0008 (8)
C35	0.0462 (15)	0.0429 (14)	0.0439 (14)	0.0080 (11)	−0.0083 (11)	−0.0007 (11)
C36	0.080 (2)	0.0621 (17)	0.0377 (15)	0.0078 (15)	0.0100 (14)	−0.0177 (13)
C37	0.0399 (13)	0.0319 (12)	0.0257 (12)	−0.0049 (10)	0.0036 (10)	−0.0042 (9)
C38	0.0400 (14)	0.0279 (11)	0.0317 (12)	−0.0095 (10)	0.0072 (10)	−0.0054 (9)

Geometric parameters (Å, º)

N1—C13	1.331 (2)	C15—C19	1.426 (3)
N1—C14	1.373 (2)	C16—H16A	0.9800
N2—C13	1.356 (2)	C16—H16B	0.9800
N2—C15	1.377 (2)	C16—H16C	0.9800
N2—H2A	0.97 (2)	C17—H17A	0.9800
N3—C18	1.147 (2)	C17—H17B	0.9800
N4—C19	1.145 (2)	C17—H17C	0.9800
N5—C32	1.357 (2)	C20—C21	1.386 (3)
N5—C33	1.365 (2)	C20—C25	1.395 (3)
N5—H5B	0.953 (19)	C20—H20A	0.9500

N6—C32	1.334 (2)	C21—C22	1.390 (3)
N6—C34	1.373 (2)	C21—C35	1.504 (3)
N7—C37	1.146 (2)	C22—C23	1.392 (3)
N8—C38	1.147 (2)	C22—H22A	0.9500
C1—C2	1.383 (3)	C23—C24	1.386 (3)
C1—C6	1.401 (2)	C23—C36	1.516 (3)
C1—H1A	0.9500	C24—C25	1.400 (2)
C2—C3	1.386 (3)	C24—H24A	0.9500
C2—C16	1.513 (3)	C25—C26	1.495 (3)
C3—C4	1.393 (3)	C26—C27	1.397 (3)
C3—H3A	0.9500	C26—C31	1.405 (2)
C4—C5	1.388 (3)	C27—C28	1.377 (3)
C4—C17	1.510 (3)	C27—H27A	0.9500
C5—C6	1.390 (3)	C28—C29	1.382 (3)
C5—H5A	0.9500	C28—H28A	0.9500
C6—C7	1.488 (3)	C29—C30	1.383 (3)
C7—C8	1.397 (3)	C29—H29A	0.9500
C7—C12	1.402 (2)	C30—C31	1.396 (2)
C8—C9	1.377 (3)	C30—H30A	0.9500
C8—H8A	0.9500	C31—C32	1.465 (2)
C9—C10	1.380 (3)	C33—C34	1.372 (2)
C9—H9A	0.9500	C33—C37	1.424 (3)
C10—C11	1.385 (3)	C34—C38	1.428 (3)
C10—H10A	0.9500	C35—H35A	0.9800
C11—C12	1.395 (3)	C35—H35B	0.9800
C11—H11A	0.9500	C35—H35C	0.9800
C12—C13	1.467 (2)	C36—H36A	0.9800
C14—C15	1.372 (2)	C36—H36B	0.9800
C14—C18	1.433 (3)	C36—H36C	0.9800

C13—N1—C14	105.12 (14)	H17A—C17—H17C	109.5
C13—N2—C15	107.45 (15)	H17B—C17—H17C	109.5
C13—N2—H2A	125.0 (12)	N3—C18—C14	178.7 (2)
C15—N2—H2A	126.9 (12)	N4—C19—C15	179.0 (2)
C32—N5—C33	107.46 (16)	C21—C20—C25	121.88 (18)
C32—N5—H5B	128.0 (12)	C21—C20—H20A	119.1
C33—N5—H5B	124.5 (12)	C25—C20—H20A	119.1
C32—N6—C34	105.18 (15)	C20—C21—C22	118.4 (2)
C2—C1—C6	121.5 (2)	C20—C21—C35	120.88 (18)
C2—C1—H1A	119.3	C22—C21—C35	120.73 (19)
C6—C1—H1A	119.3	C21—C22—C23	121.5 (2)
C1—C2—C3	118.7 (2)	C21—C22—H22A	119.3
C1—C2—C16	120.8 (2)	C23—C22—H22A	119.3
C3—C2—C16	120.5 (2)	C24—C23—C22	118.90 (19)
C2—C3—C4	121.7 (2)	C24—C23—C36	120.3 (2)
C2—C3—H3A	119.1	C22—C23—C36	120.8 (2)
C4—C3—H3A	119.1	C23—C24—C25	121.2 (2)
C5—C4—C3	118.1 (2)	C23—C24—H24A	119.4
C5—C4—C17	121.0 (2)	C25—C24—H24A	119.4

C3—C4—C17	120.9 (2)	C20—C25—C24	118.15 (19)
C4—C5—C6	122.0 (2)	C20—C25—C26	121.49 (17)
C4—C5—H5A	119.0	C24—C25—C26	120.24 (18)
C6—C5—H5A	119.0	C27—C26—C31	117.42 (19)
C5—C6—C1	118.0 (2)	C27—C26—C25	118.62 (18)
C5—C6—C7	122.20 (17)	C31—C26—C25	123.88 (18)
C1—C6—C7	119.72 (19)	C28—C27—C26	121.9 (2)
C8—C7—C12	117.13 (19)	C28—C27—H27A	119.1
C8—C7—C6	119.00 (18)	C26—C27—H27A	119.1
C12—C7—C6	123.85 (17)	C27—C28—C29	120.5 (2)
C9—C8—C7	121.9 (2)	C27—C28—H28A	119.7
C9—C8—H8A	119.0	C29—C28—H28A	119.7
C7—C8—H8A	119.0	C28—C29—C30	118.9 (2)
C8—C9—C10	120.6 (2)	C28—C29—H29A	120.6
C8—C9—H9A	119.7	C30—C29—H29A	120.6
C10—C9—H9A	119.7	C29—C30—C31	121.1 (2)
C9—C10—C11	119.0 (2)	C29—C30—H30A	119.4
C9—C10—H10A	120.5	C31—C30—H30A	119.4
C11—C10—H10A	120.5	C30—C31—C26	120.14 (18)
C10—C11—C12	120.6 (2)	C30—C31—C32	115.67 (17)
C10—C11—H11A	119.7	C26—C31—C32	124.10 (17)
C12—C11—H11A	119.7	N6—C32—N5	111.24 (17)
C11—C12—C7	120.69 (18)	N6—C32—C31	123.37 (16)
C11—C12—C13	115.99 (17)	N5—C32—C31	125.13 (17)
C7—C12—C13	123.27 (18)	N5—C33—C34	105.98 (16)
N1—C13—N2	111.46 (16)	N5—C33—C37	124.07 (17)
N1—C13—C12	127.54 (16)	C34—C33—C37	129.88 (18)
N2—C13—C12	120.83 (16)	C33—C34—N6	110.14 (16)
C15—C14—N1	110.63 (16)	C33—C34—C38	126.50 (18)
C15—C14—C18	127.52 (18)	N6—C34—C38	123.28 (16)
N1—C14—C18	121.81 (16)	C21—C35—H35A	109.5
C14—C15—N2	105.33 (16)	C21—C35—H35B	109.5
C14—C15—C19	131.59 (18)	H35A—C35—H35B	109.5
N2—C15—C19	123.06 (16)	C21—C35—H35C	109.5
C2—C16—H16A	109.5	H35A—C35—H35C	109.5
C2—C16—H16B	109.5	H35B—C35—H35C	109.5
H16A—C16—H16B	109.5	C23—C36—H36A	109.5
C2—C16—H16C	109.5	C23—C36—H36B	109.5
H16A—C16—H16C	109.5	H36A—C36—H36B	109.5
H16B—C16—H16C	109.5	C23—C36—H36C	109.5
C4—C17—H17A	109.5	H36A—C36—H36C	109.5
C4—C17—H17B	109.5	H36B—C36—H36C	109.5
H17A—C17—H17B	109.5	N7—C37—C33	177.9 (2)
C4—C17—H17C	109.5	N8—C38—C34	176.9 (2)

C6—C1—C2—C3	−2.1 (3)	C25—C20—C21—C22	−0.6 (3)
C6—C1—C2—C16	176.46 (18)	C25—C20—C21—C35	178.24 (18)
C1—C2—C3—C4	1.7 (3)	C20—C21—C22—C23	−1.3 (3)
C16—C2—C3—C4	−176.88 (19)	C35—C21—C22—C23	179.77 (19)

C2—C3—C4—C5	0.5 (3)	C21—C22—C23—C24	2.1 (3)
C2—C3—C4—C17	−178.2 (2)	C21—C22—C23—C36	−177.13 (19)
C3—C4—C5—C6	−2.5 (3)	C22—C23—C24—C25	−0.9 (3)
C17—C4—C5—C6	176.30 (19)	C36—C23—C24—C25	178.36 (19)
C4—C5—C6—C1	2.1 (3)	C21—C20—C25—C24	1.8 (3)
C4—C5—C6—C7	−174.95 (18)	C21—C20—C25—C26	−174.31 (17)
C2—C1—C6—C5	0.3 (3)	C23—C24—C25—C20	−1.0 (3)
C2—C1—C6—C7	177.39 (18)	C23—C24—C25—C26	175.13 (18)
C5—C6—C7—C8	133.6 (2)	C20—C25—C26—C27	135.43 (19)
C1—C6—C7—C8	−43.3 (3)	C24—C25—C26—C27	−40.6 (3)
C5—C6—C7—C12	−44.4 (3)	C20—C25—C26—C31	−41.1 (3)
C1—C6—C7—C12	138.66 (19)	C24—C25—C26—C31	142.82 (19)
C12—C7—C8—C9	1.9 (3)	C31—C26—C27—C28	2.0 (3)
C6—C7—C8—C9	−176.26 (19)	C25—C26—C27—C28	−174.83 (18)
C7—C8—C9—C10	−0.5 (3)	C26—C27—C28—C29	0.3 (3)
C8—C9—C10—C11	−0.9 (3)	C27—C28—C29—C30	−2.0 (3)
C9—C10—C11—C12	0.9 (3)	C28—C29—C30—C31	1.4 (3)
C10—C11—C12—C7	0.6 (3)	C29—C30—C31—C26	0.9 (3)
C10—C11—C12—C13	−176.92 (18)	C29—C30—C31—C32	−175.68 (17)
C8—C7—C12—C11	−1.9 (3)	C27—C26—C31—C30	−2.5 (3)
C6—C7—C12—C11	176.11 (17)	C25—C26—C31—C30	174.06 (17)
C8—C7—C12—C13	175.41 (17)	C27—C26—C31—C32	173.76 (17)
C6—C7—C12—C13	−6.6 (3)	C25—C26—C31—C32	−9.6 (3)
C14—N1—C13—N2	0.2 (2)	C34—N6—C32—N5	−0.5 (2)
C14—N1—C13—C12	−174.96 (18)	C34—N6—C32—C31	173.90 (16)
C15—N2—C13—N1	0.0 (2)	C33—N5—C32—N6	0.6 (2)
C15—N2—C13—C12	175.52 (16)	C33—N5—C32—C31	−173.62 (17)
C11—C12—C13—N1	130.2 (2)	C30—C31—C32—N6	−37.1 (3)
C7—C12—C13—N1	−47.2 (3)	C26—C31—C32—N6	146.47 (18)
C11—C12—C13—N2	−44.5 (2)	C30—C31—C32—N5	136.51 (18)
C7—C12—C13—N2	138.05 (18)	C26—C31—C32—N5	−39.9 (3)
C13—N1—C14—C15	−0.3 (2)	C32—N5—C33—C34	−0.5 (2)
C13—N1—C14—C18	177.64 (17)	C32—N5—C33—C37	176.68 (18)
N1—C14—C15—N2	0.3 (2)	N5—C33—C34—N6	0.2 (2)
C18—C14—C15—N2	−177.49 (18)	C37—C33—C34—N6	−176.73 (19)
N1—C14—C15—C19	179.01 (19)	N5—C33—C34—C38	177.10 (18)
C18—C14—C15—C19	1.2 (3)	C37—C33—C34—C38	0.1 (3)
C13—N2—C15—C14	−0.2 (2)	C32—N6—C34—C33	0.1 (2)
C13—N2—C15—C19	−179.03 (17)	C32—N6—C34—C38	−176.85 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···N6	0.97 (2)	2.02 (2)	2.907 (2)	150.9 (17)
N5—H5*B*···N1^{i}	0.953 (19)	1.95 (2)	2.882 (2)	164.4 (17)
C11—H11*A*···N8^{ii}	0.95	2.69	3.567 (3)	154
C16—H16*B*···N6^{iii}	0.98	2.60	3.484 (3)	150
C16—H16*C*···N3^{iv}	0.98	2.65	3.497 (3)	145

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+1/2; (ii) −*x*+2, −*y*+1, −*z*; (iii) −*x*+3/2, *y*−1/2, −*z*+1/2; (iv) *x*+1/2, −*y*+1/2, *z*+1/2.

(II) 2-(2′,4′,6′-Trimethylbiphenyl-2-yl)-1*H*-imidazole-4,5-dicarbonitrile

Crystal data

$C_{20}H_{16}N_4$
M_r = 312.37
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 12.373 (1) Å
b = 10.303 (2) Å
c = 13.279 (2) Å
β = 92.578 (7)°
V = 1691.1 (4) Å^3
Z = 4
$F(000)$ = 656
D_x = 1.227 Mg m^{-3}
Melting point: 486(1) K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 247 reflections
θ = 3–23°
μ = 0.08 mm^{-1}
T = 133 K
Plate, colourless
0.60 × 0.40 × 0.10 mm

Data collection

Siemens SMART 1K CCD area-detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
T_{min} = 0.784, T_{max} = 0.993
25406 measured reflections
4381 independent reflections
3014 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 29.0°, θ_{min} = 2.5°
h = −14→16
k = −12→14
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.104
S = 1.04
4381 reflections
225 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0160 (17)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.71987 (8)	0.43078 (10)	0.52011 (7)	0.0234 (2)

N2	0.64706 (8)	0.49068 (11)	0.37197 (7)	0.0280 (2)
N3	0.69072 (11)	0.23410 (13)	0.20203 (8)	0.0476 (3)
N4	0.83141 (11)	0.11688 (13)	0.50582 (9)	0.0430 (3)
C1	0.66536 (9)	0.52552 (12)	0.46789 (8)	0.0232 (3)
C2	0.69141 (10)	0.36986 (13)	0.36458 (8)	0.0264 (3)
C3	0.73685 (9)	0.33054 (12)	0.45601 (8)	0.0241 (3)
C4	0.68981 (11)	0.29635 (14)	0.27323 (9)	0.0341 (3)
C5	0.78988 (10)	0.21276 (14)	0.48468 (9)	0.0287 (3)
C6	0.62895 (9)	0.64746 (12)	0.51227 (8)	0.0240 (3)
C7	0.53525 (10)	0.70443 (13)	0.46939 (10)	0.0306 (3)
H7A	0.4992	0.6654	0.4124	0.037*
C8	0.49484 (11)	0.81694 (14)	0.50929 (11)	0.0373 (3)
H8A	0.4313	0.8555	0.4797	0.045*
C9	0.54702 (11)	0.87375 (14)	0.59256 (11)	0.0392 (3)
H9A	0.5179	0.9499	0.6212	0.047*
C10	0.64133 (11)	0.82023 (13)	0.63424 (10)	0.0334 (3)
H10A	0.6772	0.8612	0.6904	0.040*
C11	0.68438 (10)	0.70702 (12)	0.59482 (9)	0.0254 (3)
C12	0.78750 (9)	0.65169 (12)	0.63979 (8)	0.0237 (3)
C13	0.78784 (10)	0.59029 (12)	0.73408 (9)	0.0260 (3)
C14	0.88263 (10)	0.53157 (13)	0.77167 (9)	0.0289 (3)
H14A	0.8830	0.4901	0.8356	0.035*
C15	0.97678 (10)	0.53191 (13)	0.71823 (9)	0.0287 (3)
C16	0.97493 (10)	0.59668 (13)	0.62640 (9)	0.0284 (3)
H16A	1.0391	0.5991	0.5897	0.034*
C17	0.88270 (10)	0.65811 (12)	0.58621 (9)	0.0254 (3)
C18	0.68692 (11)	0.58439 (15)	0.79433 (10)	0.0367 (3)
H18A	0.7000	0.5280	0.8531	0.055*
H18B	0.6269	0.5495	0.7520	0.055*
H18C	0.6685	0.6719	0.8170	0.055*
C19	1.07663 (11)	0.46266 (16)	0.75969 (10)	0.0403 (4)
H19A	1.1352	0.4733	0.7131	0.060*
H19B	1.0607	0.3701	0.7674	0.060*
H19C	1.0988	0.4996	0.8254	0.060*
C20	0.88650 (11)	0.73054 (14)	0.48770 (10)	0.0350 (3)
H20A	0.9601	0.7271	0.4634	0.053*
H20B	0.8657	0.8212	0.4979	0.053*
H20C	0.8362	0.6904	0.4378	0.053*
H1A	0.7366 (12)	0.4278 (15)	0.5868 (11)	0.045 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0270 (5)	0.0252 (6)	0.0177 (5)	−0.0006 (4)	−0.0014 (4)	0.0000 (4)
N2	0.0321 (5)	0.0295 (6)	0.0221 (5)	−0.0040 (5)	−0.0020 (4)	0.0028 (4)
N3	0.0669 (9)	0.0493 (9)	0.0261 (6)	−0.0069 (7)	−0.0025 (6)	−0.0069 (6)
N4	0.0577 (8)	0.0368 (8)	0.0343 (6)	0.0118 (6)	−0.0016 (6)	−0.0040 (6)
C1	0.0204 (5)	0.0263 (7)	0.0227 (5)	−0.0040 (5)	0.0000 (4)	0.0040 (5)
C2	0.0303 (6)	0.0285 (7)	0.0205 (5)	−0.0056 (5)	0.0007 (5)	−0.0005 (5)
C3	0.0257 (6)	0.0248 (7)	0.0219 (5)	−0.0035 (5)	0.0013 (4)	−0.0019 (5)

C4	0.0441 (8)	0.0345 (8)	0.0235 (6)	−0.0069 (6)	−0.0016 (5)	0.0013 (6)
C5	0.0342 (7)	0.0303 (7)	0.0213 (6)	−0.0003 (6)	−0.0004 (5)	−0.0038 (5)
C6	0.0226 (6)	0.0236 (7)	0.0260 (6)	−0.0020 (5)	0.0026 (5)	0.0054 (5)
C7	0.0237 (6)	0.0324 (8)	0.0357 (7)	−0.0040 (6)	0.0007 (5)	0.0111 (6)
C8	0.0246 (6)	0.0329 (8)	0.0547 (8)	0.0034 (6)	0.0066 (6)	0.0159 (7)
C9	0.0363 (7)	0.0262 (8)	0.0564 (9)	0.0059 (6)	0.0159 (7)	0.0048 (7)
C10	0.0352 (7)	0.0266 (7)	0.0388 (7)	0.0008 (6)	0.0072 (6)	−0.0024 (6)
C11	0.0255 (6)	0.0234 (7)	0.0276 (6)	−0.0010 (5)	0.0045 (5)	0.0020 (5)
C12	0.0265 (6)	0.0206 (6)	0.0239 (6)	−0.0018 (5)	0.0001 (5)	−0.0047 (5)
C13	0.0298 (6)	0.0254 (7)	0.0228 (6)	−0.0042 (5)	0.0022 (5)	−0.0051 (5)
C14	0.0345 (7)	0.0299 (7)	0.0221 (6)	−0.0033 (6)	−0.0013 (5)	0.0008 (5)
C15	0.0287 (6)	0.0286 (7)	0.0282 (6)	−0.0029 (5)	−0.0047 (5)	−0.0004 (5)
C16	0.0228 (6)	0.0329 (7)	0.0297 (6)	−0.0026 (5)	0.0027 (5)	−0.0001 (5)
C17	0.0276 (6)	0.0240 (7)	0.0245 (6)	−0.0052 (5)	−0.0002 (5)	−0.0007 (5)
C18	0.0376 (7)	0.0433 (9)	0.0298 (7)	0.0013 (6)	0.0099 (6)	0.0021 (6)
C19	0.0331 (7)	0.0486 (10)	0.0386 (7)	0.0041 (7)	−0.0048 (6)	0.0059 (7)
C20	0.0313 (7)	0.0423 (9)	0.0318 (7)	−0.0029 (6)	0.0037 (5)	0.0088 (6)

Geometric parameters (Å, º)

N1—C1	1.3588 (15)	C11—C12	1.4969 (17)
N1—C3	1.3606 (15)	C12—C13	1.4027 (16)
N1—H1A	0.901 (14)	C12—C17	1.4048 (16)
N2—C1	1.3329 (15)	C13—C14	1.3921 (17)
N2—C2	1.3657 (17)	C13—C18	1.5142 (17)
N3—C4	1.1429 (17)	C14—C15	1.3911 (17)
N4—C5	1.1427 (17)	C14—H14A	0.9500
C1—C6	1.4672 (17)	C15—C16	1.3895 (17)
C2—C3	1.3758 (16)	C15—C19	1.5090 (18)
C2—C4	1.4294 (17)	C16—C17	1.3902 (17)
C3—C5	1.4232 (18)	C16—H16A	0.9500
C6—C7	1.3976 (17)	C17—C20	1.5086 (17)
C6—C11	1.4075 (17)	C18—H18A	0.9800
C7—C8	1.3778 (19)	C18—H18B	0.9800
C7—H7A	0.9500	C18—H18C	0.9800
C8—C9	1.385 (2)	C19—H19A	0.9800
C8—H8A	0.9500	C19—H19B	0.9800
C9—C10	1.3834 (19)	C19—H19C	0.9800
C9—H9A	0.9500	C20—H20A	0.9800
C10—C11	1.3940 (18)	C20—H20B	0.9800
C10—H10A	0.9500	C20—H20C	0.9800
C1—N1—C3	108.24 (10)	C17—C12—C11	119.94 (10)
C1—N1—H1A	127.8 (10)	C14—C13—C12	119.00 (11)
C3—N1—H1A	123.6 (10)	C14—C13—C18	119.64 (11)
C1—N2—C2	105.23 (10)	C12—C13—C18	121.35 (11)
N2—C1—N1	110.69 (11)	C15—C14—C13	121.83 (11)
N2—C1—C6	124.92 (11)	C15—C14—H14A	119.1
N1—C1—C6	124.37 (10)	C13—C14—H14A	119.1
N2—C2—C3	110.76 (11)	C16—C15—C14	117.89 (11)

N2—C2—C4	123.62 (11)	C16—C15—C19	121.88 (12)
C3—C2—C4	125.62 (12)	C14—C15—C19	120.22 (12)
N1—C3—C2	105.08 (11)	C15—C16—C17	122.44 (11)
N1—C3—C5	124.20 (10)	C15—C16—H16A	118.8
C2—C3—C5	130.72 (11)	C17—C16—H16A	118.8
N3—C4—C2	177.46 (16)	C16—C17—C12	118.47 (11)
N4—C5—C3	178.45 (14)	C16—C17—C20	120.06 (11)
C7—C6—C11	120.03 (12)	C12—C17—C20	121.47 (11)
C7—C6—C1	117.39 (11)	C13—C18—H18A	109.5
C11—C6—C1	122.58 (11)	C13—C18—H18B	109.5
C8—C7—C6	120.35 (13)	H18A—C18—H18B	109.5
C8—C7—H7A	119.8	C13—C18—H18C	109.5
C6—C7—H7A	119.8	H18A—C18—H18C	109.5
C7—C8—C9	119.90 (13)	H18B—C18—H18C	109.5
C7—C8—H8A	120.1	C15—C19—H19A	109.5
C9—C8—H8A	120.1	C15—C19—H19B	109.5
C10—C9—C8	120.36 (13)	H19A—C19—H19B	109.5
C10—C9—H9A	119.8	C15—C19—H19C	109.5
C8—C9—H9A	119.8	H19A—C19—H19C	109.5
C9—C10—C11	120.87 (13)	H19B—C19—H19C	109.5
C9—C10—H10A	119.6	C17—C20—H20A	109.5
C11—C10—H10A	119.6	C17—C20—H20B	109.5
C10—C11—C6	118.44 (11)	H20A—C20—H20B	109.5
C10—C11—C12	120.14 (11)	C17—C20—H20C	109.5
C6—C11—C12	121.42 (11)	H20A—C20—H20C	109.5
C13—C12—C17	120.29 (11)	H20B—C20—H20C	109.5
C13—C12—C11	119.74 (10)		

C2—N2—C1—N1	0.25 (13)	C1—C6—C11—C10	−177.61 (11)
C2—N2—C1—C6	−177.98 (11)	C7—C6—C11—C12	−177.38 (11)
C3—N1—C1—N2	−0.45 (13)	C1—C6—C11—C12	2.69 (18)
C3—N1—C1—C6	177.79 (10)	C10—C11—C12—C13	72.88 (16)
C1—N2—C2—C3	0.04 (14)	C6—C11—C12—C13	−107.42 (13)
C1—N2—C2—C4	−179.89 (12)	C10—C11—C12—C17	−108.95 (14)
C1—N1—C3—C2	0.45 (13)	C6—C11—C12—C17	70.75 (16)
C1—N1—C3—C5	−178.74 (11)	C17—C12—C13—C14	−2.52 (18)
N2—C2—C3—N1	−0.31 (13)	C11—C12—C13—C14	175.64 (11)
C4—C2—C3—N1	179.63 (12)	C17—C12—C13—C18	178.75 (12)
N2—C2—C3—C5	178.80 (12)	C11—C12—C13—C18	−3.09 (18)
C4—C2—C3—C5	−1.3 (2)	C12—C13—C14—C15	−0.14 (19)
N2—C1—C6—C7	28.19 (17)	C18—C13—C14—C15	178.61 (12)
N1—C1—C6—C7	−149.80 (12)	C13—C14—C15—C16	1.96 (19)
N2—C1—C6—C11	−151.88 (12)	C13—C14—C15—C19	−177.59 (12)
N1—C1—C6—C11	30.13 (17)	C14—C15—C16—C17	−1.2 (2)
C11—C6—C7—C8	−1.86 (18)	C19—C15—C16—C17	178.37 (12)
C1—C6—C7—C8	178.07 (11)	C15—C16—C17—C12	−1.41 (19)
C6—C7—C8—C9	−0.24 (19)	C15—C16—C17—C20	178.07 (12)
C7—C8—C9—C10	1.9 (2)	C13—C12—C17—C16	3.27 (18)
C8—C9—C10—C11	−1.4 (2)	C11—C12—C17—C16	−174.89 (11)

C9—C10—C11—C6	−0.73 (19)	C13—C12—C17—C20	−176.21 (12)
C9—C10—C11—C12	178.97 (12)	C11—C12—C17—C20	5.63 (18)
C7—C6—C11—C10	2.32 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N3^{i}	0.901 (14)	2.351 (15)	2.9883 (16)	127.7 (12)
C14—H14*A*···N4^{i}	0.95	2.62	3.5476 (17)	166
C19—H19*C*···N4^{ii}	0.98	2.66	3.6327 (19)	175
C16—H16*A*···C3^{iii}	0.95	2.96	3.8507 (17)	158
C16—H16*A*···C5^{iii}	0.95	3.07	3.8580 (18)	142
C19—H19*A*···C2^{iii}	0.98	2.91	3.7899 (19)	150

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) −*x*+2, *y*+1/2, −*z*+3/2; (iii) −*x*+2, −*y*+1, −*z*+1.

(III) 2-[8-(3,5-Dimethylphenyl)naphthalen-1-yl]-1*H*-imidazole-4,5-dicarbonitrile

Crystal data

$C_{23}H_{16}N_4$
M_r = 348.40
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
a = 16.7042 (14) Å
b = 7.1632 (7) Å
c = 15.0528 (11) Å
V = 1801.2 (3) Å^3
Z = 4
$F(000)$ = 728
D_x = 1.285 Mg m^{-3}
Melting point: 514(1) K
Cu $K\alpha$ radiation, λ = 1.54180 Å
Cell parameters from 25 reflections
θ = 46–62°
μ = 0.62 mm^{-1}
T = 294 K
Block, colourless
0.60 × 0.40 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: ψ scan *MolEN* (Fair, 1990)
T_{min} = 0.776, T_{max} = 0.857
3815 measured reflections
1781 independent reflections
1753 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 69.9°, θ_{min} = 5.3°
h = −20→20
k = 0→8
l = −18→18
3 standard reflections every 90 min
intensity decay: 2.0%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.070
S = 1.05
1781 reflections
251 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 0.15P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0137 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.39034 (9)	0.03608 (19)	0.39285 (9)	0.0420 (3)
N2	0.48444 (8)	0.0795 (2)	0.49401 (9)	0.0394 (3)
N3	0.47805 (12)	0.0167 (3)	0.18351 (11)	0.0665 (5)
N4	0.66997 (11)	0.1656 (3)	0.38986 (14)	0.0759 (6)
C1	0.33666 (9)	0.1751 (2)	0.62080 (10)	0.0393 (3)
C2	0.37449 (10)	0.3547 (2)	0.62529 (11)	0.0432 (4)
C3	0.36311 (12)	0.4641 (3)	0.70001 (13)	0.0534 (4)
H3A	0.3887	0.5791	0.7032	0.064*
C4	0.31469 (13)	0.4082 (3)	0.77064 (14)	0.0593 (5)
H4A	0.3093	0.4842	0.8204	0.071*
C5	0.27531 (11)	0.2422 (3)	0.76655 (13)	0.0550 (5)
H5A	0.2421	0.2062	0.8130	0.066*
C6	0.28460 (10)	0.1244 (3)	0.69229 (12)	0.0456 (4)
C7	0.24059 (12)	−0.0446 (3)	0.68892 (15)	0.0551 (5)
H7A	0.2076	−0.0769	0.7362	0.066*
C8	0.24593 (14)	−0.1599 (3)	0.61781 (15)	0.0605 (5)
H8A	0.2144	−0.2667	0.6148	0.073*
C9	0.29923 (11)	−0.1173 (3)	0.54884 (13)	0.0516 (4)
H9A	0.3033	−0.1982	0.5007	0.062*
C10	0.34525 (10)	0.0400 (2)	0.55056 (11)	0.0409 (3)
C11	0.42305 (11)	0.4389 (2)	0.55251 (12)	0.0426 (4)
C12	0.39158 (11)	0.4619 (2)	0.46758 (12)	0.0438 (4)
H12A	0.3397	0.4219	0.4557	0.053*
C13	0.43646 (12)	0.5437 (2)	0.40042 (12)	0.0462 (4)
C14	0.51353 (12)	0.6051 (2)	0.41944 (13)	0.0501 (4)
H14A	0.5440	0.6588	0.3745	0.060*
C15	0.54637 (11)	0.5882 (3)	0.50413 (14)	0.0503 (4)
C16	0.49942 (13)	0.5053 (3)	0.56997 (13)	0.0486 (4)
H16A	0.5200	0.4943	0.6272	0.058*
C17	0.40170 (15)	0.5662 (3)	0.30870 (15)	0.0640 (6)
H17A	0.3921	0.4454	0.2832	0.096*
H17B	0.4387	0.6340	0.2720	0.096*
H17C	0.3522	0.6337	0.3124	0.096*
C18	0.62974 (14)	0.6562 (4)	0.52483 (18)	0.0712 (6)
H18A	0.6681	0.5668	0.5037	0.107*
H18B	0.6355	0.6711	0.5879	0.107*
H18C	0.6386	0.7740	0.4960	0.107*

C19	0.40546 (10)	0.0538 (2)	0.47883 (11)	0.0384 (3)
C20	0.46288 (11)	0.0564 (2)	0.35248 (10)	0.0414 (4)
C21	0.52226 (11)	0.0854 (2)	0.41370 (11)	0.0404 (3)
C22	0.47209 (12)	0.0383 (3)	0.25815 (12)	0.0486 (4)
C23	0.60471 (11)	0.1274 (3)	0.40170 (12)	0.0501 (4)
H2A	0.5051 (13)	0.085 (3)	0.5428 (16)	0.047 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0488 (7)	0.0474 (7)	0.0300 (7)	0.0024 (6)	−0.0010 (6)	−0.0004 (6)
N2	0.0462 (7)	0.0457 (7)	0.0262 (7)	0.0063 (6)	−0.0019 (6)	−0.0015 (5)
N3	0.0735 (12)	0.0953 (13)	0.0307 (8)	0.0048 (10)	0.0045 (8)	−0.0014 (9)
N4	0.0537 (9)	0.1049 (15)	0.0692 (12)	−0.0031 (10)	0.0037 (9)	0.0089 (12)
C1	0.0414 (7)	0.0462 (8)	0.0303 (7)	0.0045 (6)	0.0005 (6)	0.0025 (7)
C2	0.0496 (8)	0.0474 (9)	0.0326 (8)	0.0027 (7)	0.0007 (7)	0.0002 (7)
C3	0.0627 (11)	0.0539 (10)	0.0437 (10)	−0.0017 (8)	0.0045 (8)	−0.0079 (8)
C4	0.0665 (12)	0.0699 (12)	0.0414 (10)	0.0048 (9)	0.0095 (9)	−0.0134 (10)
C5	0.0570 (10)	0.0697 (12)	0.0383 (9)	0.0047 (8)	0.0131 (8)	−0.0004 (9)
C6	0.0445 (8)	0.0542 (9)	0.0380 (9)	0.0050 (7)	0.0046 (7)	0.0044 (8)
C7	0.0539 (10)	0.0593 (10)	0.0522 (11)	−0.0027 (8)	0.0127 (9)	0.0087 (9)
C8	0.0622 (10)	0.0530 (10)	0.0664 (13)	−0.0115 (9)	0.0110 (10)	0.0020 (10)
C9	0.0590 (10)	0.0486 (9)	0.0473 (10)	−0.0011 (8)	0.0024 (8)	−0.0045 (8)
C10	0.0444 (8)	0.0455 (8)	0.0327 (8)	0.0047 (6)	0.0000 (7)	0.0026 (6)
C11	0.0524 (9)	0.0378 (8)	0.0377 (8)	0.0022 (6)	0.0030 (7)	0.0011 (6)
C12	0.0521 (9)	0.0383 (8)	0.0410 (9)	0.0020 (7)	−0.0002 (7)	0.0011 (7)
C13	0.0624 (10)	0.0369 (8)	0.0393 (9)	0.0035 (7)	0.0033 (8)	0.0024 (7)
C14	0.0628 (11)	0.0396 (8)	0.0478 (10)	−0.0004 (7)	0.0108 (8)	0.0040 (8)
C15	0.0549 (10)	0.0416 (8)	0.0543 (10)	−0.0027 (7)	0.0031 (8)	0.0015 (8)
C16	0.0568 (9)	0.0479 (8)	0.0411 (9)	−0.0023 (7)	−0.0043 (8)	0.0010 (8)
C17	0.0856 (15)	0.0629 (12)	0.0436 (10)	−0.0065 (11)	−0.0042 (10)	0.0110 (9)
C18	0.0625 (12)	0.0749 (15)	0.0762 (16)	−0.0162 (11)	−0.0018 (11)	0.0082 (12)
C19	0.0454 (8)	0.0400 (8)	0.0299 (8)	0.0047 (6)	−0.0017 (6)	−0.0004 (6)
C20	0.0533 (9)	0.0430 (8)	0.0279 (8)	0.0043 (7)	−0.0006 (7)	0.0009 (6)
C21	0.0486 (9)	0.0413 (8)	0.0314 (8)	0.0067 (7)	0.0005 (6)	0.0008 (6)
C22	0.0557 (9)	0.0576 (10)	0.0326 (9)	0.0020 (8)	0.0004 (8)	0.0007 (7)
C23	0.0517 (10)	0.0600 (10)	0.0386 (9)	0.0051 (8)	0.0003 (7)	0.0011 (8)

Geometric parameters (Å, º)

N1—C19	1.325 (2)	C9—C10	1.365 (3)
N1—C20	1.363 (2)	C9—H9A	0.9300
N2—C19	1.352 (2)	C10—C19	1.479 (2)
N2—C21	1.365 (2)	C11—C16	1.387 (3)
N2—H2A	0.81 (2)	C11—C12	1.392 (2)
N3—C22	1.138 (2)	C12—C13	1.388 (2)
N4—C23	1.138 (3)	C12—H12A	0.9300
C1—C6	1.430 (2)	C13—C14	1.390 (3)
C1—C2	1.435 (2)	C13—C17	1.506 (3)
C1—C10	1.440 (2)	C14—C15	1.393 (3)

C2—C3	1.384 (3)	C14—H14A	0.9300
C2—C11	1.491 (2)	C15—C16	1.397 (3)
C3—C4	1.394 (3)	C15—C18	1.508 (3)
C3—H3A	0.9300	C16—H16A	0.9300
C4—C5	1.361 (3)	C17—H17A	0.9600
C4—H4A	0.9300	C17—H17B	0.9600
C5—C6	1.409 (3)	C17—H17C	0.9600
C5—H5A	0.9300	C18—H18A	0.9600
C6—C7	1.417 (3)	C18—H18B	0.9600
C7—C8	1.355 (3)	C18—H18C	0.9600
C7—H7A	0.9300	C20—C21	1.370 (2)
C8—C9	1.401 (3)	C20—C22	1.434 (2)
C8—H8A	0.9300	C21—C23	1.421 (3)

C19—N1—C20	104.82 (14)	C13—C12—H12A	119.5
C19—N2—C21	107.84 (14)	C11—C12—H12A	119.5
C19—N2—H2A	125.1 (15)	C12—C13—C14	118.92 (17)
C21—N2—H2A	127.0 (15)	C12—C13—C17	120.30 (17)
C6—C1—C2	117.37 (14)	C14—C13—C17	120.77 (17)
C6—C1—C10	116.25 (15)	C13—C14—C15	121.71 (17)
C2—C1—C10	126.37 (14)	C13—C14—H14A	119.1
C3—C2—C1	119.04 (16)	C15—C14—H14A	119.1
C3—C2—C11	116.30 (16)	C14—C15—C16	117.73 (17)
C1—C2—C11	124.59 (15)	C14—C15—C18	121.65 (19)
C2—C3—C4	122.47 (18)	C16—C15—C18	120.62 (19)
C2—C3—H3A	118.8	C11—C16—C15	121.86 (18)
C4—C3—H3A	118.8	C11—C16—H16A	119.1
C5—C4—C3	119.78 (18)	C15—C16—H16A	119.1
C5—C4—H4A	120.1	C13—C17—H17A	109.5
C3—C4—H4A	120.1	C13—C17—H17B	109.5
C4—C5—C6	120.41 (18)	H17A—C17—H17B	109.5
C4—C5—H5A	119.8	C13—C17—H17C	109.5
C6—C5—H5A	119.8	H17A—C17—H17C	109.5
C5—C6—C7	118.87 (17)	H17B—C17—H17C	109.5
C5—C6—C1	120.78 (16)	C15—C18—H18A	109.5
C7—C6—C1	120.35 (16)	C15—C18—H18B	109.5
C8—C7—C6	120.99 (18)	H18A—C18—H18B	109.5
C8—C7—H7A	119.5	C15—C18—H18C	109.5
C6—C7—H7A	119.5	H18A—C18—H18C	109.5
C7—C8—C9	119.63 (18)	H18B—C18—H18C	109.5
C7—C8—H8A	120.2	N1—C19—N2	111.36 (15)
C9—C8—H8A	120.2	N1—C19—C10	125.26 (15)
C10—C9—C8	121.58 (18)	N2—C19—C10	123.33 (14)
C10—C9—H9A	119.2	N1—C20—C21	111.10 (14)
C8—C9—H9A	119.2	N1—C20—C22	121.80 (16)
C9—C10—C1	120.84 (16)	C21—C20—C22	127.02 (17)
C9—C10—C19	115.11 (15)	N2—C21—C20	104.83 (15)
C1—C10—C19	123.98 (14)	N2—C21—C23	124.60 (16)
C16—C11—C12	118.75 (16)	C20—C21—C23	130.42 (16)

C16—C11—C2	120.00 (16)	N3—C22—C20	177.1 (2)
C12—C11—C2	121.15 (16)	N4—C23—C21	177.5 (2)
C13—C12—C11	120.98 (17)		
C6—C1—C2—C3	4.0 (2)	C16—C11—C12—C13	2.3 (2)
C10—C1—C2—C3	−176.64 (17)	C2—C11—C12—C13	178.74 (15)
C6—C1—C2—C11	−172.89 (16)	C11—C12—C13—C14	−0.9 (2)
C10—C1—C2—C11	6.5 (3)	C11—C12—C13—C17	179.54 (17)
C1—C2—C3—C4	−1.5 (3)	C12—C13—C14—C15	−0.6 (2)
C11—C2—C3—C4	175.69 (18)	C17—C13—C14—C15	178.99 (17)
C2—C3—C4—C5	−1.4 (3)	C13—C14—C15—C16	0.6 (3)
C3—C4—C5—C6	1.5 (3)	C13—C14—C15—C18	−179.54 (19)
C4—C5—C6—C7	−177.90 (19)	C12—C11—C16—C15	−2.3 (3)
C4—C5—C6—C1	1.2 (3)	C2—C11—C16—C15	−178.80 (17)
C2—C1—C6—C5	−3.9 (2)	C14—C15—C16—C11	0.9 (3)
C10—C1—C6—C5	176.62 (16)	C18—C15—C16—C11	−178.97 (19)
C2—C1—C6—C7	175.19 (16)	C20—N1—C19—N2	−1.6 (2)
C10—C1—C6—C7	−4.2 (2)	C20—N1—C19—C10	−179.23 (14)
C5—C6—C7—C8	178.2 (2)	C21—N2—C19—N1	2.2 (2)
C1—C6—C7—C8	−0.9 (3)	C21—N2—C19—C10	179.93 (14)
C6—C7—C8—C9	3.8 (3)	C9—C10—C19—N1	52.5 (2)
C7—C8—C9—C10	−1.2 (3)	C1—C10—C19—N1	−130.50 (18)
C8—C9—C10—C1	−4.3 (3)	C9—C10—C19—N2	−124.88 (18)
C8—C9—C10—C19	172.79 (18)	C1—C10—C19—N2	52.1 (2)
C6—C1—C10—C9	6.8 (2)	C19—N1—C20—C21	0.35 (19)
C2—C1—C10—C9	−172.56 (17)	C19—N1—C20—C22	177.34 (16)
C6—C1—C10—C19	−170.01 (15)	C19—N2—C21—C20	−1.85 (17)
C2—C1—C10—C19	10.6 (3)	C19—N2—C21—C23	174.04 (16)
C3—C2—C11—C16	55.2 (2)	N1—C20—C21—N2	0.94 (18)
C1—C2—C11—C16	−127.87 (19)	C22—C20—C21—N2	−175.85 (18)
C3—C2—C11—C12	−121.27 (19)	N1—C20—C21—C23	−174.61 (17)
C1—C2—C11—C12	55.7 (2)	C22—C20—C21—C23	8.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···N3^{i}	0.81 (2)	2.26 (2)	3.001 (2)	152 (2)

Symmetry code: (i) −*x*+1, −*y*, *z*+1/2.