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2,2-Dimethyl-5-phenyl-4-oxa-1-aza-6,7:8,9-dibenzobicyclo[3.2.2]nonane

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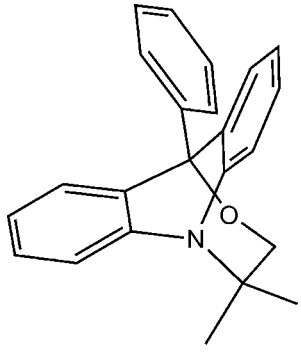
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.048; wR factor = 0.117; data-to-parameter ratio = 17.3.

In the title compound, $C_{23}H_{21}NO$, the six-membered ring of the oxaazabicyclo[3.2.2]nonane unit adopts a boat conformation. One of the seven-membered rings adopts a boat conformation while the other is in a chair conformation. The dihedral angle between the two benzene rings is 51.89 (4)°. There are no hydrogen bonds and the crystal structure is stabilized by van der Waals interactions only.

Related literature

For general background, see: Liu & Larock (2004, 2005); Pellissier & Santelli (2003); Yoshida *et al.* (2002).



Experimental

Crystal data

$C_{23}H_{21}NO$
M_r = 327.41
Monoclinic, $P2_1/c$
a = 15.707 (6) Å
b = 9.227 (4) Å
c = 12.198 (5) Å
β = 101.575 (6)°
V = 1731.7 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.20 × 0.15 × 0.11 mm

Data collection

Rigaku Mercury CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2004)
T_{min} = 0.981, T_{max} = 0.992
13049 measured reflections
3911 independent reflections
3171 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.117
S = 1.07
3911 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Data collection: *CrystalClear* (Rigaku/MSC, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2373).

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supplementary materials

Acta Cryst. (2007). E**63**, o2794 [**doi:10.1107/S1600536807021198**]

2,2-Dimethyl-5-phenyl-4-oxa-1-aza-6,7:8,9-dibenzobicyclo[3.2.2]nonane

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Comment

2-(Trimethylsilyl)phenyltriflate, an efficient precursor to benzyne, has been widely employed for syntheses of complex organic compounds (Pellissier & Santelli, 2003). Recently, Liu and Larock (2004) reported addition of H—X bonds (X = N, O and S) to arynes to construct a new carbon-heteroatom bond (Liu & Larock, 2004). There are also examples of the transition-metal-free insertion of an aryne into polar hetero atom-containing bonds such as C—N σ bond (Yoshida *et al.*, 2002) and intermolecular C—N bond reaction under very mild conditions (Liu & Larock, 2005). However, there is no example of insertion of aryne into C═N double bonds. We report here the crystal structure of the title compound, (I), which was obtained through insertion of two benzyne molecules into C═N double bonds.

As shown in Fig. 1, the oxaazabicyclo[3.2.2]nonane unit is composed of one six-membered ring and two seven-membered rings, which are connected with each other by the brigdehead atoms N1 and C7. The bond lengths and angles in (I) are normal except the slightly longer C7—C23 [1.5296 (19) Å], C7—C17 [1.5317 (19) Å] and C9—N1 [1.5209 (18) Å] bonds, which may result from the ring fusion.

The six-membered ring of the oxaazabicyclo[3.2.2]nonane unit adopts a boat conformation, with atoms N1 and C7 deviating from the C12/C17/C18/C23 plane by 0.542 (1) and 0.608 (1) Å, respectively. One of the seven-membered rings (N1,C9,C8,O1,C7,C23,C18) adopts a boat conformation, with atoms C8, C18 and C23 deviating from the plane defined by other four atoms by 0.624 (2), 1.149 (1) and 1.249 (1) Å, respectively. The other seven-membered ring (N1,C9,C8,O1,C7,C12,C17) adopts a chair conformation. The dihedral angle between the two benzene rings (C12—C17 and C18—C23) is 51.89 (4)°. The C1—C6 phenyl ring forms dihedral angles of 60.04 (5)° and 68.23 (5)°, respectively, with the C12—C17 and C18—C23 benzene rings.

No significant hydrogen bonds are observed in the crystal structure.

Experimental

2-(Trimethylsilyl)phenyl triflate (0.48 ml) and 4,4-dimethyl-2-oxazoline (0.12 ml) were added to a mixture of CsF (0.3032 g), $[RuCl_2(\eta^6\text{-}C_6H_6)]_2$ (0.02 g), PPh_3 (0.042 g) in CH_3CN (0.6 ml) and toluene (3.0 ml). The mixture was stirred under argon at 408 K for 24 h. The organic layer was filtered and concentrated in vacuo. The crude product was further purified by column chromatography on silica gel to obtain the title compound as a white solid. Colourless needle-shaped crystals of (I) suitable for X-ray analysis were grown by slow evaporation of a hexane solution at room temperature.

Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93-0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

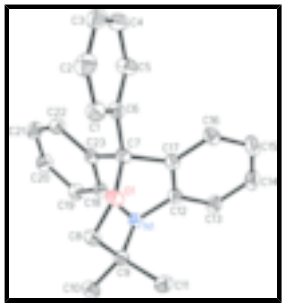


Fig. 1. The molecular structure of (I), showing 30% probability displacement ellopsoids. H atoms have been omitted for clarity.

2,2-Dimethyl-5-phenyl-4-oxa-1-aza-6,7:8,9-dibenzobicyclo[3.2.2]nonane

Crystal data

$C_{23}H_{21}NO$	F_{000} = 696
M_r = 327.41	D_x = 1.256 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3669 reflections
a = 15.707 (6) Å	θ = 2.8–27.5º
b = 9.227 (4) Å	μ = 0.08 mm^{-1}
c = 12.198 (5) Å	T = 293 (2) K
β = 101.575 (6)º	Prism, colourless
V = 1731.7 (12) Å^3	0.20 × 0.15 × 0.11 mm
Z = 4	

Data collection

Rigaku Mercury CCD area-detector diffractometer	3911 independent reflections
Radiation source: fine-focus sealed tube	3171 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.033
Detector resolution: 14.6306 pixels mm^{-1}	θ_{max} = 27.4º
T = 293(2) K	θ_{min} = 2.8º
ω scans	h = −18→20
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2004)	k = −11→11
T_{min} = 0.981, T_{max} = 0.992	l = −15→15
13049 measured reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.3898P]$ where $P = (F_o^2 + 2F_c^2)/3$

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.117$

$S = 1.07$

3911 reflections

226 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.21$ e Å^{-3}

$\Delta\rho_{min} = -0.17$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.96183 (10)	0.24037 (17)	0.28052 (13)	0.0431 (4)
H1	0.9429	0.2047	0.3428	0.052*
C2	1.04785 (10)	0.28135 (19)	0.28977 (15)	0.0519 (4)
H2	1.0861	0.2729	0.3583	0.062*
C3	1.07728 (10)	0.33424 (19)	0.19895 (16)	0.0532 (4)
H3	1.1348	0.3634	0.2061	0.064*
C4	1.02079 (11)	0.34364 (19)	0.09715 (16)	0.0532 (4)
H4	1.0406	0.3769	0.0348	0.064*
C5	0.93473 (10)	0.30390 (19)	0.08725 (13)	0.0464 (4)
H5	0.8969	0.3118	0.0183	0.056*
C6	0.90390 (9)	0.25219 (15)	0.17922 (11)	0.0347 (3)
C7	0.80819 (9)	0.21735 (15)	0.16881 (10)	0.0317 (3)
C8	0.72086 (9)	0.18203 (16)	0.31426 (11)	0.0355 (3)
H8A	0.7166	0.2861	0.3229	0.043*
H8B	0.7257	0.1387	0.3877	0.043*
C9	0.63664 (9)	0.12763 (15)	0.23859 (11)	0.0343 (3)
C10	0.56008 (10)	0.19291 (19)	0.28076 (13)	0.0473 (4)
H10A	0.5067	0.1602	0.2346	0.071*
H10B	0.5630	0.2967	0.2776	0.071*
H10C	0.5623	0.1629	0.3567	0.071*
C11	0.63202 (12)	−0.03686 (17)	0.24199 (14)	0.0484 (4)
H11A	0.5791	−0.0691	0.1944	0.073*

H11B	0.6332	−0.0680	0.3174	0.073*
H11C	0.6808	−0.0773	0.2163	0.073*
C12	0.68217 (9)	0.08999 (14)	0.05706 (11)	0.0327 (3)
C13	0.64116 (11)	−0.00665 (16)	−0.02287 (12)	0.0411 (3)
H13	0.5811	−0.0174	−0.0360	0.049*
C14	0.69024 (12)	−0.08768 (17)	−0.08352 (13)	0.0490 (4)
H14	0.6629	−0.1526	−0.1376	0.059*
C15	0.77895 (12)	−0.07215 (18)	−0.06376 (13)	0.0499 (4)
H15	0.8115	−0.1270	−0.1044	0.060*
C16	0.82047 (10)	0.02507 (16)	0.01655 (12)	0.0411 (3)
H16	0.8806	0.0349	0.0296	0.049*
C17	0.77218 (9)	0.10735 (14)	0.07715 (11)	0.0323 (3)
C18	0.66150 (9)	0.32615 (14)	0.11765 (10)	0.0308 (3)
C19	0.60351 (10)	0.44013 (16)	0.09311 (12)	0.0389 (3)
H19	0.5440	0.4225	0.0772	0.047*
C20	0.63460 (11)	0.58057 (17)	0.09236 (13)	0.0449 (4)
H20	0.5958	0.6573	0.0751	0.054*
C21	0.72252 (11)	0.60710 (17)	0.11705 (13)	0.0452 (4)
H21	0.7430	0.7016	0.1158	0.054*
C22	0.78091 (10)	0.49345 (15)	0.14383 (12)	0.0381 (3)
H22	0.8402	0.5122	0.1622	0.046*
C23	0.75088 (9)	0.35205 (14)	0.14324 (10)	0.0307 (3)
O1	0.79830 (6)	0.15078 (11)	0.27410 (7)	0.0358 (2)
N1	0.63133 (7)	0.17812 (12)	0.11871 (9)	0.0325 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0368 (8)	0.0491 (9)	0.0415 (8)	−0.0009 (7)	0.0032 (6)	−0.0013 (7)
C2	0.0377 (9)	0.0570 (10)	0.0559 (10)	−0.0020 (7)	−0.0031 (7)	−0.0066 (8)
C3	0.0332 (8)	0.0485 (10)	0.0780 (12)	−0.0036 (7)	0.0117 (8)	−0.0092 (9)
C4	0.0452 (9)	0.0560 (10)	0.0637 (11)	−0.0010 (8)	0.0238 (8)	0.0047 (8)
C5	0.0375 (8)	0.0608 (10)	0.0419 (8)	0.0017 (7)	0.0100 (6)	0.0058 (7)
C6	0.0312 (7)	0.0361 (7)	0.0368 (7)	0.0039 (6)	0.0066 (6)	−0.0011 (6)
C7	0.0326 (7)	0.0355 (7)	0.0267 (6)	0.0028 (6)	0.0054 (5)	0.0024 (5)
C8	0.0373 (8)	0.0425 (8)	0.0279 (6)	0.0018 (6)	0.0094 (5)	0.0018 (6)
C9	0.0353 (7)	0.0369 (7)	0.0317 (7)	−0.0009 (6)	0.0089 (6)	0.0008 (6)
C10	0.0401 (8)	0.0591 (10)	0.0453 (9)	0.0001 (7)	0.0152 (7)	−0.0017 (7)
C11	0.0598 (10)	0.0403 (9)	0.0468 (9)	−0.0079 (7)	0.0150 (8)	0.0049 (7)
C12	0.0391 (7)	0.0307 (7)	0.0279 (6)	0.0021 (6)	0.0059 (5)	0.0009 (5)
C13	0.0486 (9)	0.0373 (8)	0.0353 (7)	−0.0030 (7)	0.0032 (6)	−0.0026 (6)
C14	0.0700 (12)	0.0368 (8)	0.0377 (8)	0.0021 (8)	0.0049 (7)	−0.0088 (6)
C15	0.0684 (11)	0.0413 (9)	0.0421 (9)	0.0147 (8)	0.0163 (8)	−0.0050 (7)
C16	0.0449 (8)	0.0401 (8)	0.0395 (8)	0.0095 (7)	0.0114 (6)	0.0016 (6)
C17	0.0382 (7)	0.0310 (7)	0.0276 (6)	0.0049 (6)	0.0060 (5)	0.0033 (5)
C18	0.0341 (7)	0.0312 (7)	0.0265 (6)	0.0021 (5)	0.0045 (5)	−0.0003 (5)
C19	0.0354 (7)	0.0420 (8)	0.0376 (7)	0.0075 (6)	0.0036 (6)	0.0013 (6)
C20	0.0501 (9)	0.0358 (8)	0.0476 (9)	0.0116 (7)	0.0069 (7)	0.0037 (6)

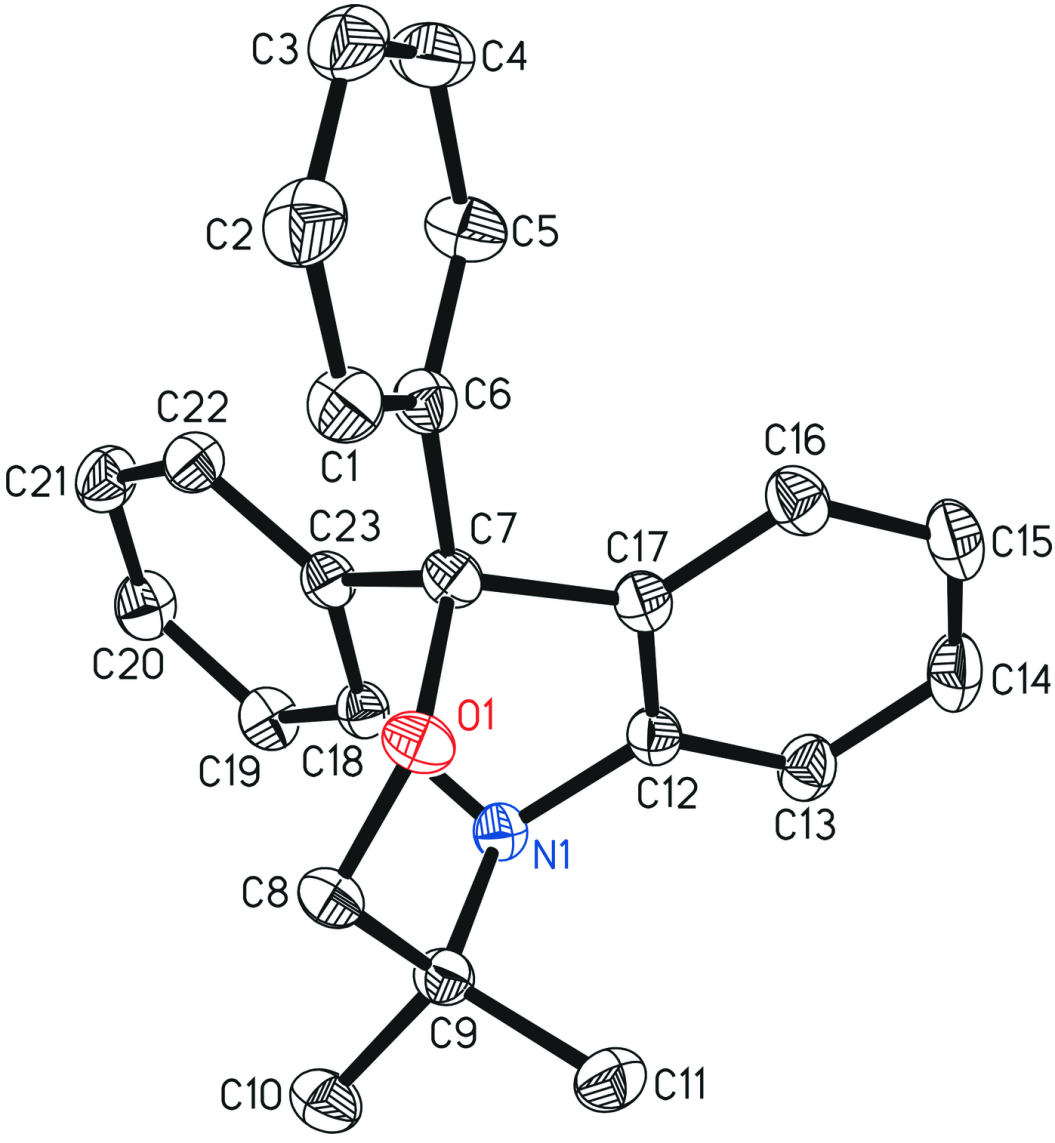
C21	0.0544 (10)	0.0315 (8)	0.0496 (9)	−0.0003 (7)	0.0106 (7)	0.0019 (6)
C22	0.0390 (8)	0.0365 (8)	0.0388 (8)	−0.0022 (6)	0.0081 (6)	−0.0002 (6)
C23	0.0328 (7)	0.0331 (7)	0.0260 (6)	0.0015 (5)	0.0050 (5)	−0.0004 (5)
O1	0.0330 (5)	0.0443 (6)	0.0305 (5)	0.0051 (4)	0.0075 (4)	0.0069 (4)
N1	0.0340 (6)	0.0326 (6)	0.0305 (6)	0.0001 (5)	0.0058 (5)	−0.0019 (5)

Geometric parameters (Å, °)

C1—C6	1.384 (2)	C11—H11A	0.96
C1—C2	1.386 (2)	C11—H11B	0.96
C1—H1	0.93	C11—H11C	0.96
C2—C3	1.373 (3)	C12—C13	1.381 (2)
C2—H2	0.93	C12—C17	1.395 (2)
C3—C4	1.376 (3)	C12—N1	1.4510 (17)
C3—H3	0.93	C13—C14	1.389 (2)
C4—C5	1.382 (2)	C13—H13	0.93
C4—H4	0.93	C14—C15	1.373 (2)
C5—C6	1.392 (2)	C14—H14	0.93
C5—H5	0.93	C15—C16	1.390 (2)
C6—C7	1.518 (2)	C15—H15	0.93
C7—O1	1.4596 (16)	C16—C17	1.3868 (19)
C7—C23	1.5296 (19)	C16—H16	0.93
C7—C17	1.5317 (19)	C18—C19	1.384 (2)
C8—O1	1.4289 (16)	C18—C23	1.3963 (19)
C8—C9	1.537 (2)	C18—N1	1.4467 (18)
C8—H8A	0.97	C19—C20	1.385 (2)
C8—H8B	0.97	C19—H19	0.93
C9—C11	1.520 (2)	C20—C21	1.375 (2)
C9—N1	1.5209 (18)	C20—H20	0.93
C9—C10	1.524 (2)	C21—C22	1.388 (2)
C10—H10A	0.96	C21—H21	0.93
C10—H10B	0.96	C22—C23	1.387 (2)
C10—H10C	0.96	C22—H22	0.93
C6—C1—C2	120.45 (15)	H11A—C11—H11B	109.5
C6—C1—H1	119.8	C9—C11—H11C	109.5
C2—C1—H1	119.8	H11A—C11—H11C	109.5
C3—C2—C1	120.80 (16)	H11B—C11—H11C	109.5
C3—C2—H2	119.6	C13—C12—C17	120.72 (13)
C1—C2—H2	119.6	C13—C12—N1	120.02 (13)
C2—C3—C4	119.33 (15)	C17—C12—N1	119.24 (12)
C2—C3—H3	120.3	C12—C13—C14	119.55 (15)
C4—C3—H3	120.3	C12—C13—H13	120.2
C3—C4—C5	120.28 (16)	C14—C13—H13	120.2
C3—C4—H4	119.9	C15—C14—C13	120.16 (15)
C5—C4—H4	119.9	C15—C14—H14	119.9
C4—C5—C6	120.84 (15)	C13—C14—H14	119.9
C4—C5—H5	119.6	C14—C15—C16	120.47 (14)
C6—C5—H5	119.6	C14—C15—H15	119.8
C1—C6—C5	118.27 (14)	C16—C15—H15	119.8

C1—C6—C7	121.39 (13)	C17—C16—C15	119.95 (15)
C5—C6—C7	120.27 (12)	C17—C16—H16	120.0
O1—C7—C6	107.10 (10)	C15—C16—H16	120.0
O1—C7—C23	111.05 (10)	C16—C17—C12	119.14 (13)
C6—C7—C23	112.18 (11)	C16—C17—C7	126.19 (13)
O1—C7—C17	105.93 (11)	C12—C17—C7	114.66 (11)
C6—C7—C17	114.58 (11)	C19—C18—C23	120.45 (13)
C23—C7—C17	105.87 (10)	C19—C18—N1	121.12 (13)
O1—C8—C9	114.67 (11)	C23—C18—N1	118.43 (11)
O1—C8—H8A	108.6	C18—C19—C20	119.65 (14)
C9—C8—H8A	108.6	C18—C19—H19	120.2
O1—C8—H8B	108.6	C20—C19—H19	120.2
C9—C8—H8B	108.6	C21—C20—C19	120.32 (14)
H8A—C8—H8B	107.6	C21—C20—H20	119.8
C11—C9—N1	109.77 (11)	C19—C20—H20	119.8
C11—C9—C10	109.89 (12)	C20—C21—C22	120.27 (14)
N1—C9—C10	107.99 (11)	C20—C21—H21	119.9
C11—C9—C8	110.46 (12)	C22—C21—H21	119.9
N1—C9—C8	110.59 (11)	C23—C22—C21	120.09 (14)
C10—C9—C8	108.09 (12)	C23—C22—H22	120.0
C9—C10—H10A	109.5	C21—C22—H22	120.0
C9—C10—H10B	109.5	C22—C23—C18	119.20 (12)
H10A—C10—H10B	109.5	C22—C23—C7	125.29 (13)
C9—C10—H10C	109.5	C18—C23—C7	115.51 (12)
H10A—C10—H10C	109.5	C8—O1—C7	117.70 (10)
H10B—C10—H10C	109.5	C18—N1—C12	108.01 (11)
C9—C11—H11A	109.5	C18—N1—C9	110.05 (10)
C9—C11—H11B	109.5	C12—N1—C9	113.39 (10)

Fig. 1


C3
C4
C2
C5
C6
C22
C1
C16
C21
C23
C7
C17
C15
C20
C14
O1
C19
C18
C12
C13
N1
C8
C9
C10
C11