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ISSN 2414-3146

2,4-Di-*tert*-butyl-6-({[(*R*)-1-phenylethyl]amino}-methyl)phenol

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Received 26 January 2016
Accepted 2 February 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

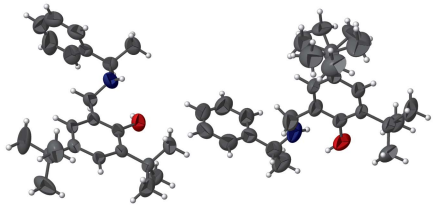
Keywords: crystal structure; phenoxy-amine; hydrogen bond.

CCDC reference: 1451497

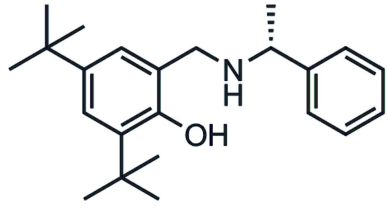
Structural data: full structural data are available from iucrdata.iucr.org

There are two molecules in the asymmetric unit of the title compound, $C_{23}H_{33}NO$, in which the dihedral angles between the aromatic rings are 72.1 (3) and 89.0 (2)°. One of the molecules features a *tert*-butyl group disordered over two sets of sites in a 0.545 (13):0.455 (13) ratio. Both molecules feature an intramolecular O—H···N hydrogen bond, which closes an *S*(6) ring. Neither of the N—H groups participates in hydrogen bonds, perhaps due to steric crowding.

3D view



Chemical scheme



Structure description

Interest in the title compound arises from the highly catalytic activity of phenoxy-imine ligated group IV metal complexes (now known FI catalysts) for olefin polymerization (Fujita *et al.*, 2014; Makio *et al.*, 2011). The structure of the title compound (phenoxy-amine) is similar to phenoxy-imine ligands of FI catalysts, but phenoxy-amines are considered to be conformationally more flexible to chelate metal ions as they are not constrained to be planar (Sreenivasulu & Vittal, 2003; Yang *et al.*, 2003). Moreover, group IV metal complexes bearing phenoxy-amine ligands have exhibited highly catalytic activities for the polymerization of olefins (Alesso *et al.*, 2011; Wan *et al.*, 2013). As a part of our studies in this area, we have prepared the title compound (Fig. 1) and determined its crystal structure.

There are two molecules in the asymmetric unit, in which the dihedral angles between the aromatic rings are 72.1 (3)° for the C1 molecule and 89.0 (2)° for the C24 molecule. Both molecules feature an intramolecular O—H···N hydrogen bond (Fig. 2, Table 1), which closes an *S*(6) ring. Neither of the N—H groups participate in hydrogen bonds, perhaps due to steric crowding.

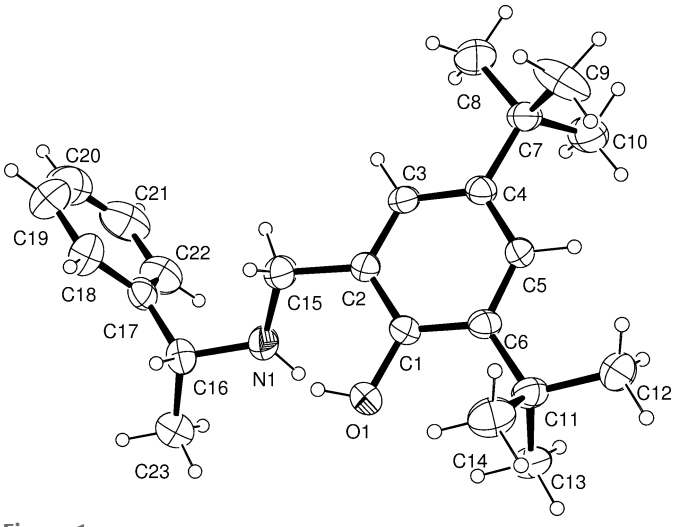


Figure 1
The molecular structure of the C1 molecule of the title compound, with displacement ellipsoids drawn at the 30% probability level.

Synthesis and crystallization

A solution of (*R*)-α-phenylethylamine (1.21 g, 10 mmol) and 3,5-di-*tert*-butylsalicylaldehyde (2.34 g, 10 mmol) in 15 ml of ethanol was stirred for 12 h at 25°C, and then the resulting precipitate was collected to give the corresponding iminophenol. A suspension of above obtained iminophenol in 20 ml of ethanol was cooled to 0°C, and $NaBH_4$ (0.57 g, 15 mmol) was added portionwise with stirring at 0°C over a period of 30 min. After the reaction mixture was stirred 12 h at 25°C, the solvents were removed under reduced pressure and the residue was dissolved in 20 ml H_2O and then extracted with CH_2Cl_2. The organic layer was washed with brine and dried over Na_2SO_4, and the solvents were removed under reduced pressure and the residue was recrystallized from ethanol solution to give the title compound (2.40 g, 70.6% yield) as white needle-like crystals. ^{1}H NMR (300 MHz, $CDCl_3$, p.p.m.): δ 10.72 (*br*, 1H), 7.36 (*m*, 2H), 7.28 (*m*, 3H), 7.20 (*s*, 1H), 6.76 (*s*, 1H), 3.85–3.69 (*m*, 3H), 1.45 (*s*, 3H), 1.43 (*s*, 9H), 1.24 (*s*, 9H). ^{13}C NMR (75 MHz, $CDCl_3$, p.p.m.): δ 155.2, 144.3, 141.2, 136.6, 129.4, 128.2, 127.2, 124.1, 123.6, 122.9, 58.1, 51.9, 35.6, 34.8, 32.4, 30.3, 24.0. Elemental analysis (calcd %) for $C_{23}H_{33}NO$: C, 81.37; H, 9.80; N, 4.13. Found: C, 81.25; H, 9.77; N, 4.12.

The title compound (20 mg) was dissolved in dichloromethane (2 ml). The solution was allowed to evaporate slowly over several days to yield colorless blocks.

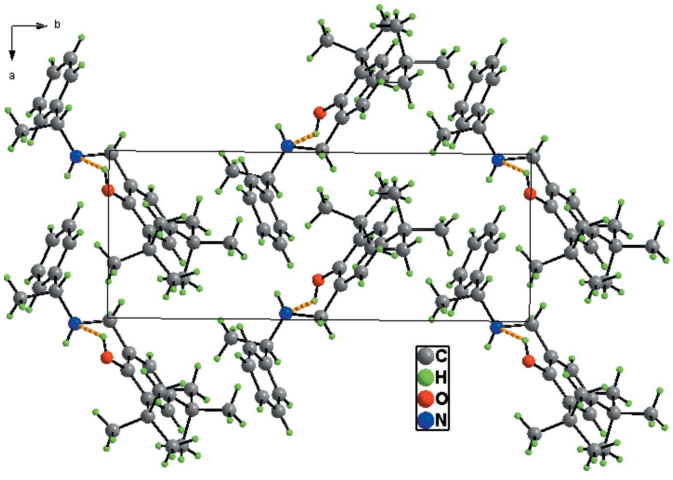


Figure 2
Part of the crystal structure, with intramolecular hydrogen bonds drawn as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.84	2.19	2.672 (5)	116
O2—H2*A*···N2	0.84	2.21	2.687 (5)	116

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. One of the *tert*-butyl groups in the C24 molecule is disordered over two sets of sites in a 0.545 (13):0.455 (13) ratio.

Acknowledgements

We are very grateful for financial support from the National Natural Science Foundation of China (No. 21272142) and the

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{33}NO$
M_r	339.50
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	200
a, *b*, *c* (Å)	6.588 (2), 16.582 (5), 19.448 (7)
β (°)	95.051 (7)
V (Å^3)	2116.3 (12)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.06
Crystal size (mm)	0.35 × 0.35 × 0.30
Data collection	
Diffractometer	Bruker SMART APEX CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22485, 10187, 5160
R_{int}	0.022
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.084, 0.212, 1.07
No. of reflections	10187
No. of parameters	487
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.18

Computer programs: *SMART* (Bruker, 2000), *SAINT* (Bruker, 2000), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *SHELXTL* (Sheldrick, 2008).

Shanxi Provincial Natural Science Foundation (No. 2012011046–14).

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full crystallographic data

IUCrData (2016). **1**, x160205 [https://doi.org/10.1107/S2414314616002054]

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Crystal data

$C_{23}H_{33}NO$
$M_r = 339.50$
Monoclinic, $P2_1$
$a = 6.588$ (2) Å
$b = 16.582$ (5) Å
$c = 19.448$ (7) Å
$\beta = 95.051$ (7)°
$V = 2116.3$ (12) Å^3
$Z = 4$
$F(000) = 744$
$D_x = 1.066$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3352 reflections
$\theta = 2.4$–$20.6°$
$\mu = 0.06$ mm^{-1}
$T = 200$ K
Block, colorless
0.35 × 0.35 × 0.30 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
22485 measured reflections
10187 independent reflections
5160 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.6°$
$h = -8 \rightarrow 6$
$k = -21 \rightarrow 22$
$l = -25 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.084$
$wR(F^2) = 0.212$
$S = 1.07$
10187 reflections
487 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.050P)^2 + 1.3036P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
C31	0.002 (2)	0.0516 (9)	0.2051 (5)	0.103 (5)	0.545 (13)
H31A	−0.0755	0.0198	0.2364	0.154*	0.545 (13)
H31B	−0.0012	0.1086	0.2182	0.154*	0.545 (13)
H31C	0.1429	0.0327	0.2080	0.154*	0.545 (13)
C32	−0.3216 (19)	0.0780 (10)	0.1401 (8)	0.117 (5)	0.545 (13)
H32A	−0.4033	0.0749	0.0956	0.175*	0.545 (13)
H32B	−0.3108	0.1344	0.1551	0.175*	0.545 (13)
H32C	−0.3873	0.0464	0.1745	0.175*	0.545 (13)
C33	−0.121 (5)	−0.0371 (11)	0.1065 (11)	0.166 (16)	0.545 (13)
H33A	−0.1441	−0.0356	0.0560	0.249*	0.545 (13)
H33B	−0.2385	−0.0617	0.1257	0.249*	0.545 (13)
H33C	0.0015	−0.0691	0.1198	0.249*	0.545 (13)
C31A	0.102 (2)	−0.0139 (8)	0.1618 (7)	0.105 (6)	0.455 (13)
H31D	0.2070	0.0205	0.1855	0.157*	0.455 (13)
H31E	0.1587	−0.0417	0.1233	0.157*	0.455 (13)
H31F	0.0568	−0.0539	0.1943	0.157*	0.455 (13)
C32A	−0.169 (3)	0.0835 (11)	0.1868 (9)	0.128 (8)	0.455 (13)
H32D	−0.2516	0.1292	0.1688	0.192*	0.455 (13)
H32E	−0.0555	0.1032	0.2182	0.192*	0.455 (13)
H32F	−0.2540	0.0471	0.2119	0.192*	0.455 (13)
C33A	−0.244 (5)	−0.0142 (18)	0.0947 (16)	0.173 (17)	0.455 (13)
H33D	−0.3062	−0.0483	0.1282	0.259*	0.455 (13)
H33E	−0.1741	−0.0482	0.0630	0.259*	0.455 (13)
H33F	−0.3503	0.0174	0.0685	0.259*	0.455 (13)
O1	0.7629 (4)	0.4986 (2)	0.42848 (15)	0.0719 (9)	
H1A	0.8874	0.4901	0.4390	0.108*	
N1	0.9640 (6)	0.4225 (2)	0.5348 (2)	0.0716 (11)	
H1	0.8473	0.4015	0.5180	0.086*	
C1	0.6895 (6)	0.5482 (2)	0.4763 (2)	0.0520 (10)	
C2	0.7895 (6)	0.5516 (2)	0.5428 (2)	0.0516 (9)	
C3	0.7135 (6)	0.6000 (2)	0.5921 (2)	0.0516 (9)	
H3	0.7803	0.6007	0.6374	0.062*	
C4	0.5437 (6)	0.6473 (2)	0.57751 (19)	0.0495 (9)	
C5	0.4506 (6)	0.6443 (2)	0.51041 (19)	0.0473 (9)	
H5	0.3335	0.6769	0.4997	0.057*	
C6	0.5179 (6)	0.5967 (2)	0.45790 (19)	0.0500 (9)	
C7	0.4572 (6)	0.7020 (3)	0.6309 (2)	0.0583 (10)	
C8	0.5766 (10)	0.6990 (5)	0.7006 (3)	0.129 (3)	
H8A	0.5738	0.6440	0.7190	0.194*	
H8B	0.5158	0.7361	0.7322	0.194*	

H8C	0.7180	0.7149	0.6960	0.194*
C9	0.4545 (11)	0.7894 (3)	0.6058 (3)	0.115 (2)
H9A	0.5943	0.8101	0.6082	0.172*
H9B	0.3737	0.8223	0.6351	0.172*
H9C	0.3940	0.7917	0.5579	0.172*
C10	0.2391 (7)	0.6778 (4)	0.6410 (3)	0.0892 (17)
H10A	0.1581	0.6774	0.5962	0.134*
H10B	0.1805	0.7165	0.6717	0.134*
H10C	0.2381	0.6238	0.6615	0.134*
C11	0.4145 (7)	0.5973 (3)	0.3846 (2)	0.0587 (10)
C12	0.2335 (8)	0.6556 (4)	0.3773 (3)	0.0946 (18)
H12A	0.1303	0.6382	0.4075	0.142*
H12B	0.1743	0.6559	0.3293	0.142*
H12C	0.2802	0.7100	0.3904	0.142*
C13	0.3278 (7)	0.5140 (3)	0.3651 (2)	0.0738 (13)
H13A	0.4366	0.4737	0.3711	0.111*
H13B	0.2713	0.5145	0.3167	0.111*
H13C	0.2200	0.5005	0.3948	0.111*
C14	0.5659 (8)	0.6238 (3)	0.3345 (2)	0.0840 (15)
H14A	0.6118	0.6789	0.3458	0.126*
H14B	0.5001	0.6225	0.2873	0.126*
H14C	0.6832	0.5873	0.3380	0.126*
C15	0.9848 (7)	0.5052 (3)	0.5595 (2)	0.0694 (12)
H15A	1.0971	0.5320	0.5377	0.083*
H15B	1.0196	0.5051	0.6101	0.083*
C16	1.1597 (6)	0.3768 (3)	0.5406 (2)	0.0613 (11)
H16	1.2542	0.4038	0.5103	0.074*
C17	1.2628 (6)	0.3745 (2)	0.6128 (2)	0.0583 (10)
C18	1.4481 (8)	0.4077 (4)	0.6272 (3)	0.0959 (17)
H18	1.5117	0.4331	0.5909	0.115*
C19	1.5481 (11)	0.4065 (5)	0.6913 (5)	0.133 (3)
H19	1.6758	0.4329	0.6999	0.160*
C20	1.4645 (16)	0.3675 (5)	0.7425 (4)	0.126 (3)
H20	1.5381	0.3630	0.7866	0.151*
C21	1.2768 (14)	0.3346 (4)	0.7317 (3)	0.110 (2)
H21	1.2146	0.3099	0.7686	0.132*
C22	1.1759 (9)	0.3374 (3)	0.6662 (3)	0.0870 (15)
H22	1.0451	0.3134	0.6580	0.104*
C23	1.1126 (8)	0.2931 (3)	0.5116 (3)	0.0869 (15)
H23A	1.0082	0.2677	0.5372	0.130*
H23B	1.2367	0.2603	0.5160	0.130*
H23C	1.0625	0.2974	0.4627	0.130*
O2	0.2230 (6)	0.2600 (2)	−0.05915 (18)	0.1065 (14)
H2A	0.2510	0.3033	−0.0380	0.160*
N2	0.5173 (7)	0.3004 (3)	0.0393 (2)	0.1029 (17)
H2	0.5539	0.2729	0.0037	0.124*
C24	0.1535 (7)	0.2047 (3)	−0.0145 (2)	0.0694 (12)
C25	0.2315 (7)	0.2050 (3)	0.0552 (2)	0.0788 (14)

C26	0.1551 (7)	0.1502 (3)	0.1001 (2)	0.0677 (12)
H26	0.2130	0.1485	0.1465	0.081*
C27	0.0009 (6)	0.0988 (3)	0.0803 (2)	0.0573 (10)
C28	−0.0714 (7)	0.1005 (3)	0.0107 (2)	0.0692 (12)
H28	−0.1772	0.0640	−0.0046	0.083*
C29	0.0007 (7)	0.1515 (3)	−0.0373 (2)	0.0615 (11)
C30	−0.0932 (9)	0.0419 (3)	0.1321 (2)	0.0774 (14)
C34	−0.0804 (9)	0.1491 (3)	−0.1148 (2)	0.0850 (16)
C35	−0.2347 (12)	0.0804 (5)	−0.1276 (3)	0.156 (4)
H35A	−0.3534	0.0909	−0.1019	0.234*
H35B	−0.1713	0.0293	−0.1120	0.234*
H35C	−0.2780	0.0770	−0.1770	0.234*
C36	0.0966 (11)	0.1297 (4)	−0.1583 (3)	0.122 (2)
H36A	0.0452	0.1255	−0.2070	0.184*
H36B	0.1591	0.0784	−0.1430	0.184*
H36C	0.1986	0.1728	−0.1527	0.184*
C37	−0.1734 (10)	0.2287 (4)	−0.1375 (3)	0.111 (2)
H37A	−0.2258	0.2255	−0.1861	0.167*
H37B	−0.0695	0.2711	−0.1317	0.167*
H37C	−0.2853	0.2415	−0.1093	0.167*
C38	0.3698 (9)	0.2743 (4)	0.0855 (3)	0.108 (2)
H38A	0.2833	0.3208	0.0959	0.129*
H38B	0.4427	0.2561	0.1294	0.129*
C39	0.5943 (8)	0.3780 (3)	0.0606 (3)	0.0813 (14)
H39	0.4769	0.4161	0.0618	0.098*
C40	0.7075 (5)	0.3754 (2)	0.13118 (16)	0.0739 (13)
C41	0.8795 (6)	0.3269 (2)	0.14234 (18)	0.0938 (17)
H41	0.9194	0.2932	0.1064	0.113*
C42	0.9930 (5)	0.3279 (3)	0.2061 (2)	0.118 (2)
H42	1.1105	0.2948	0.2137	0.141*
C43	0.9346 (7)	0.3772 (3)	0.25861 (15)	0.114 (2)
H43	1.0122	0.3779	0.3022	0.136*
C44	0.7627 (8)	0.4257 (3)	0.24745 (18)	0.120 (3)
H44	0.7228	0.4594	0.2834	0.144*
C45	0.6492 (6)	0.4248 (2)	0.1837 (2)	0.109 (2)
H45	0.5317	0.4579	0.1761	0.131*
C46	0.7234 (11)	0.4047 (4)	0.0050 (3)	0.131 (3)
H46A	0.8386	0.3677	0.0030	0.197*
H46B	0.7747	0.4593	0.0152	0.197*
H46C	0.6414	0.4047	−0.0396	0.197*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C31	0.121 (11)	0.121 (11)	0.068 (6)	−0.024 (9)	0.015 (7)	0.010 (7)
C32	0.077 (9)	0.166 (14)	0.112 (10)	0.013 (8)	0.036 (7)	0.034 (10)
C33	0.36 (5)	0.057 (7)	0.091 (13)	−0.044 (15)	0.07 (2)	−0.007 (8)
C31A	0.143 (12)	0.086 (10)	0.083 (9)	−0.015 (8)	−0.002 (8)	0.042 (8)

C32A	0.14 (2)	0.146 (15)	0.105 (13)	0.009 (14)	0.063 (15)	0.012 (11)
C33A	0.22 (3)	0.19 (3)	0.116 (15)	−0.13 (3)	0.016 (17)	0.058 (17)
O1	0.0629 (19)	0.082 (2)	0.0702 (19)	0.0199 (17)	0.0000 (15)	−0.0235 (17)
N1	0.056 (2)	0.062 (2)	0.094 (3)	0.0100 (18)	−0.010 (2)	−0.016 (2)
C1	0.052 (2)	0.047 (2)	0.056 (2)	0.0031 (19)	0.0048 (19)	−0.0104 (19)
C2	0.050 (2)	0.047 (2)	0.057 (2)	0.0044 (18)	−0.0043 (18)	−0.0071 (18)
C3	0.055 (2)	0.048 (2)	0.051 (2)	−0.0015 (19)	−0.0064 (18)	−0.0095 (18)
C4	0.049 (2)	0.046 (2)	0.054 (2)	−0.0036 (18)	0.0065 (18)	−0.0066 (18)
C5	0.045 (2)	0.045 (2)	0.052 (2)	0.0019 (17)	0.0016 (17)	−0.0030 (17)
C6	0.058 (2)	0.040 (2)	0.051 (2)	−0.0046 (19)	0.0025 (18)	−0.0033 (19)
C7	0.062 (3)	0.059 (3)	0.055 (2)	−0.005 (2)	0.0062 (19)	−0.012 (2)
C8	0.113 (5)	0.191 (8)	0.078 (4)	0.040 (5)	−0.020 (3)	−0.067 (4)
C9	0.168 (6)	0.060 (3)	0.127 (5)	0.001 (4)	0.074 (5)	−0.021 (3)
C10	0.073 (3)	0.110 (4)	0.088 (4)	−0.008 (3)	0.025 (3)	−0.030 (3)
C11	0.071 (3)	0.060 (3)	0.044 (2)	0.000 (2)	−0.0016 (19)	−0.002 (2)
C12	0.098 (4)	0.108 (4)	0.072 (3)	0.037 (4)	−0.026 (3)	−0.013 (3)
C13	0.080 (3)	0.078 (3)	0.062 (3)	−0.013 (3)	−0.003 (2)	−0.015 (2)
C14	0.106 (4)	0.090 (4)	0.056 (3)	−0.010 (3)	0.004 (3)	0.008 (2)
C15	0.070 (3)	0.057 (2)	0.077 (3)	0.012 (2)	−0.015 (2)	−0.007 (2)
C16	0.053 (3)	0.060 (3)	0.071 (3)	0.007 (2)	0.008 (2)	0.003 (2)
C17	0.055 (3)	0.045 (2)	0.075 (3)	0.009 (2)	0.004 (2)	−0.004 (2)
C18	0.063 (3)	0.112 (4)	0.111 (5)	−0.004 (3)	−0.004 (3)	−0.010 (4)
C19	0.097 (5)	0.163 (8)	0.131 (6)	0.014 (5)	−0.039 (5)	−0.050 (6)
C20	0.162 (8)	0.114 (6)	0.094 (5)	0.053 (6)	−0.033 (5)	−0.015 (4)
C21	0.174 (8)	0.077 (4)	0.083 (4)	0.020 (5)	0.032 (5)	0.007 (3)
C22	0.108 (4)	0.063 (3)	0.092 (4)	−0.003 (3)	0.021 (3)	0.003 (3)
C23	0.087 (4)	0.084 (3)	0.089 (3)	0.007 (3)	0.009 (3)	−0.018 (3)
O2	0.126 (3)	0.111 (3)	0.077 (2)	−0.051 (3)	−0.019 (2)	0.037 (2)
N2	0.105 (4)	0.138 (4)	0.063 (2)	−0.064 (3)	−0.006 (2)	0.012 (3)
C24	0.079 (3)	0.071 (3)	0.057 (3)	−0.020 (3)	−0.005 (2)	0.014 (2)
C25	0.078 (3)	0.091 (4)	0.063 (3)	−0.028 (3)	−0.016 (2)	0.007 (3)
C26	0.077 (3)	0.076 (3)	0.047 (2)	−0.012 (3)	−0.009 (2)	0.003 (2)
C27	0.057 (3)	0.053 (2)	0.061 (3)	0.004 (2)	0.002 (2)	−0.001 (2)
C28	0.072 (3)	0.071 (3)	0.062 (3)	−0.019 (2)	−0.012 (2)	0.000 (2)
C29	0.067 (3)	0.065 (3)	0.050 (2)	−0.009 (2)	−0.012 (2)	0.001 (2)
C30	0.089 (4)	0.082 (3)	0.061 (3)	−0.024 (3)	0.006 (3)	0.004 (3)
C34	0.108 (4)	0.082 (3)	0.059 (3)	−0.029 (3)	−0.026 (3)	0.008 (3)
C35	0.207 (8)	0.164 (7)	0.084 (4)	−0.113 (7)	−0.064 (5)	0.012 (4)
C36	0.190 (7)	0.117 (5)	0.060 (3)	0.028 (5)	0.008 (4)	−0.001 (3)
C37	0.125 (5)	0.117 (5)	0.084 (4)	0.015 (4)	−0.028 (4)	0.022 (4)
C38	0.107 (5)	0.131 (5)	0.083 (4)	−0.042 (4)	−0.007 (3)	−0.006 (4)
C39	0.068 (3)	0.090 (4)	0.085 (3)	−0.005 (3)	−0.001 (3)	0.005 (3)
C40	0.081 (3)	0.067 (3)	0.074 (3)	0.006 (3)	0.010 (3)	0.001 (3)
C41	0.084 (4)	0.102 (4)	0.091 (4)	0.017 (3)	−0.021 (3)	−0.016 (3)
C42	0.111 (5)	0.133 (6)	0.102 (5)	0.019 (4)	−0.024 (4)	0.007 (4)
C43	0.159 (6)	0.101 (5)	0.076 (4)	−0.062 (5)	−0.015 (4)	0.015 (4)
C44	0.214 (9)	0.069 (4)	0.078 (4)	−0.014 (5)	0.021 (5)	−0.006 (3)
C45	0.145 (6)	0.089 (4)	0.093 (4)	0.040 (4)	0.005 (4)	−0.003 (3)

C46	0.151 (6)	0.152 (6)	0.088 (4)	−0.039 (5)	−0.005 (4)	0.030 (4)

Geometric parameters (Å, º)

C31—C30	1.508 (12)	C15—H15B	0.9900
C31—H31A	0.9800	C16—C17	1.506 (6)
C31—H31B	0.9800	C16—C23	1.520 (6)
C31—H31C	0.9800	C16—H16	1.0000
C32—C30	1.640 (13)	C17—C18	1.346 (7)
C32—H32A	0.9800	C17—C22	1.374 (6)
C32—H32B	0.9800	C18—C19	1.358 (8)
C32—H32C	0.9800	C18—H18	0.9500
C33—C30	1.408 (19)	C19—C20	1.346 (10)
C33—H33A	0.9800	C19—H19	0.9500
C33—H33B	0.9800	C20—C21	1.351 (9)
C33—H33C	0.9800	C20—H20	0.9500
C31A—C30	1.649 (14)	C21—C22	1.385 (8)
C31A—H31D	0.9800	C21—H21	0.9500
C31A—H31E	0.9800	C22—H22	0.9500
C31A—H31F	0.9800	C23—H23A	0.9800
C32A—C30	1.397 (15)	C23—H23B	0.9800
C32A—H32D	0.9800	C23—H23C	0.9800
C32A—H32E	0.9800	O2—C24	1.370 (5)
C32A—H32F	0.9800	O2—H2A	0.8400
C33A—C30	1.50 (3)	N2—C39	1.431 (7)
C33A—H33D	0.9800	N2—C38	1.446 (7)
C33A—H33E	0.9800	N2—H2	0.8800
C33A—H33F	0.9800	C24—C29	1.381 (6)
O1—C1	1.361 (4)	C24—C25	1.407 (6)
O1—H1A	0.8400	C25—C26	1.385 (6)
N1—C15	1.456 (5)	C25—C38	1.550 (7)
N1—C16	1.491 (5)	C26—C27	1.355 (6)
N1—H1	0.8800	C26—H26	0.9500
C1—C2	1.399 (5)	C27—C28	1.396 (6)
C1—C6	1.408 (5)	C27—C30	1.548 (6)
C2—C3	1.379 (5)	C28—C29	1.376 (6)
C2—C15	1.510 (5)	C28—H28	0.9500
C3—C4	1.376 (5)	C29—C34	1.554 (6)
C3—H3	0.9500	C34—C37	1.506 (8)
C4—C5	1.393 (5)	C34—C35	1.532 (7)
C4—C7	1.525 (5)	C34—C36	1.533 (8)
C5—C6	1.394 (5)	C35—H35A	0.9800
C5—H5	0.9500	C35—H35B	0.9800
C6—C11	1.525 (5)	C35—H35C	0.9800
C7—C8	1.507 (7)	C36—H36A	0.9800
C7—C10	1.521 (6)	C36—H36B	0.9800
C7—C9	1.529 (7)	C36—H36C	0.9800
C8—H8A	0.9800	C37—H37A	0.9800

C8—H8B	0.9800	C37—H37B	0.9800
C8—H8C	0.9800	C37—H37C	0.9800
C9—H9A	0.9800	C38—H38A	0.9900
C9—H9B	0.9800	C38—H38B	0.9900
C9—H9C	0.9800	C39—C46	1.500 (7)
C10—H10A	0.9800	C39—C40	1.505 (5)
C10—H10B	0.9800	C39—H39	1.0000
C10—H10C	0.9800	C40—C41	1.3900
C11—C14	1.520 (6)	C40—C45	1.3900
C11—C13	1.529 (6)	C41—C42	1.3900
C11—C12	1.532 (7)	C41—H41	0.9500
C12—H12A	0.9800	C42—C43	1.3900
C12—H12B	0.9800	C42—H42	0.9500
C12—H12C	0.9800	C43—C44	1.3900
C13—H13A	0.9800	C43—H43	0.9500
C13—H13B	0.9800	C44—C45	1.3900
C13—H13C	0.9800	C44—H44	0.9500
C14—H14A	0.9800	C45—H45	0.9500
C14—H14B	0.9800	C46—H46A	0.9800
C14—H14C	0.9800	C46—H46B	0.9800
C15—H15A	0.9900	C46—H46C	0.9800

C30—C31—H31A	109.5	C19—C20—C21	120.5 (7)
C30—C31—H31B	109.5	C19—C20—H20	119.8
C30—C31—H31C	109.5	C21—C20—H20	119.8
C30—C32—H32A	109.5	C20—C21—C22	119.3 (7)
C30—C32—H32B	109.5	C20—C21—H21	120.4
C30—C32—H32C	109.5	C22—C21—H21	120.4
C30—C33—H33A	109.5	C17—C22—C21	120.8 (6)
C30—C33—H33B	109.5	C17—C22—H22	119.6
C30—C33—H33C	109.5	C21—C22—H22	119.6
C30—C31A—H31D	109.5	C16—C23—H23A	109.5
C30—C31A—H31E	109.5	C16—C23—H23B	109.5
H31D—C31A—H31E	109.5	H23A—C23—H23B	109.5
C30—C31A—H31F	109.5	C16—C23—H23C	109.5
H31D—C31A—H31F	109.5	H23A—C23—H23C	109.5
H31E—C31A—H31F	109.5	H23B—C23—H23C	109.5
C30—C32A—H32D	109.5	C24—O2—H2A	109.5
C30—C32A—H32E	109.5	C39—N2—C38	109.3 (5)
H32D—C32A—H32E	109.5	C39—N2—H2	125.4
C30—C32A—H32F	109.5	C38—N2—H2	125.4
H32D—C32A—H32F	109.5	O2—C24—C29	119.9 (4)
H32E—C32A—H32F	109.5	O2—C24—C25	119.6 (4)
C30—C33A—H33D	109.5	C29—C24—C25	120.5 (4)
C30—C33A—H33E	109.5	C26—C25—C24	118.8 (4)
H33D—C33A—H33E	109.5	C26—C25—C38	118.8 (4)
C30—C33A—H33F	109.5	C24—C25—C38	121.4 (5)
H33D—C33A—H33F	109.5	C27—C26—C25	122.6 (4)

H33E—C33A—H33F	109.5	C27—C26—H26	118.7
C1—O1—H1A	109.5	C25—C26—H26	118.7
C15—N1—C16	113.5 (3)	C26—C27—C28	116.5 (4)
C15—N1—H1	123.3	C26—C27—C30	122.1 (4)
C16—N1—H1	123.3	C28—C27—C30	121.4 (4)
O1—C1—C2	119.3 (4)	C29—C28—C27	124.2 (4)
O1—C1—C6	119.8 (3)	C29—C28—H28	117.9
C2—C1—C6	120.9 (3)	C27—C28—H28	117.9
C3—C2—C1	119.8 (4)	C28—C29—C24	117.3 (4)
C3—C2—C15	120.1 (4)	C28—C29—C34	122.1 (4)
C1—C2—C15	120.1 (4)	C24—C29—C34	120.5 (4)
C4—C3—C2	121.8 (4)	C32A—C30—C33	133.2 (13)
C4—C3—H3	119.1	C32A—C30—C33A	114.1 (15)
C2—C3—H3	119.1	C33—C30—C33A	36.2 (18)
C3—C4—C5	117.1 (3)	C32A—C30—C31	51.5 (9)
C3—C4—C7	123.0 (4)	C33—C30—C31	117.5 (12)
C5—C4—C7	120.0 (4)	C33A—C30—C31	136.3 (11)
C4—C5—C6	124.4 (4)	C32A—C30—C27	112.7 (8)
C4—C5—H5	117.8	C33—C30—C27	112.5 (10)
C6—C5—H5	117.8	C33A—C30—C27	110.4 (10)
C5—C6—C1	115.9 (3)	C31—C30—C27	113.0 (6)
C5—C6—C11	122.4 (4)	C32A—C30—C32	49.7 (9)
C1—C6—C11	121.6 (3)	C33—C30—C32	106.5 (14)
C8—C7—C10	107.4 (4)	C33A—C30—C32	72.5 (14)
C8—C7—C4	113.4 (4)	C31—C30—C32	100.3 (9)
C10—C7—C4	110.2 (3)	C27—C30—C32	105.3 (6)
C8—C7—C9	108.0 (5)	C32A—C30—C31A	109.3 (11)
C10—C7—C9	107.9 (4)	C33—C30—C31A	70.9 (13)
C4—C7—C9	109.9 (4)	C33A—C30—C31A	106.5 (14)
C7—C8—H8A	109.5	C31—C30—C31A	58.9 (8)
C7—C8—H8B	109.5	C27—C30—C31A	103.0 (5)
H8A—C8—H8B	109.5	C32—C30—C31A	150.0 (7)
C7—C8—H8C	109.5	C37—C34—C35	111.0 (5)
H8A—C8—H8C	109.5	C37—C34—C36	109.4 (5)
H8B—C8—H8C	109.5	C35—C34—C36	106.3 (6)
C7—C9—H9A	109.5	C37—C34—C29	110.9 (5)
C7—C9—H9B	109.5	C35—C34—C29	110.1 (4)
H9A—C9—H9B	109.5	C36—C34—C29	109.1 (4)
C7—C9—H9C	109.5	C34—C35—H35A	109.5
H9A—C9—H9C	109.5	C34—C35—H35B	109.5
H9B—C9—H9C	109.5	H35A—C35—H35B	109.5
C7—C10—H10A	109.5	C34—C35—H35C	109.5
C7—C10—H10B	109.5	H35A—C35—H35C	109.5
H10A—C10—H10B	109.5	H35B—C35—H35C	109.5
C7—C10—H10C	109.5	C34—C36—H36A	109.5
H10A—C10—H10C	109.5	C34—C36—H36B	109.5
H10B—C10—H10C	109.5	H36A—C36—H36B	109.5
C14—C11—C6	109.7 (4)	C34—C36—H36C	109.5

C14—C11—C13	110.7 (4)	H36A—C36—H36C	109.5
C6—C11—C13	110.7 (4)	H36B—C36—H36C	109.5
C14—C11—C12	107.8 (4)	C34—C37—H37A	109.5
C6—C11—C12	111.8 (4)	C34—C37—H37B	109.5
C13—C11—C12	106.0 (4)	H37A—C37—H37B	109.5
C11—C12—H12A	109.5	C34—C37—H37C	109.5
C11—C12—H12B	109.5	H37A—C37—H37C	109.5
H12A—C12—H12B	109.5	H37B—C37—H37C	109.5
C11—C12—H12C	109.5	N2—C38—C25	113.0 (5)
H12A—C12—H12C	109.5	N2—C38—H38A	109.0
H12B—C12—H12C	109.5	C25—C38—H38A	109.0
C11—C13—H13A	109.5	N2—C38—H38B	109.0
C11—C13—H13B	109.5	C25—C38—H38B	109.0
H13A—C13—H13B	109.5	H38A—C38—H38B	107.8
C11—C13—H13C	109.5	N2—C39—C46	105.5 (5)
H13A—C13—H13C	109.5	N2—C39—C40	111.9 (4)
H13B—C13—H13C	109.5	C46—C39—C40	113.5 (4)
C11—C14—H14A	109.5	N2—C39—H39	108.6
C11—C14—H14B	109.5	C46—C39—H39	108.6
H14A—C14—H14B	109.5	C40—C39—H39	108.6
C11—C14—H14C	109.5	C41—C40—C45	120.0
H14A—C14—H14C	109.5	C41—C40—C39	119.4 (3)
H14B—C14—H14C	109.5	C45—C40—C39	120.5 (3)
N1—C15—C2	110.8 (3)	C40—C41—C42	120.0
N1—C15—H15A	109.5	C40—C41—H41	120.0
C2—C15—H15A	109.5	C42—C41—H41	120.0
N1—C15—H15B	109.5	C41—C42—C43	120.0
C2—C15—H15B	109.5	C41—C42—H42	120.0
H15A—C15—H15B	108.1	C43—C42—H42	120.0
N1—C16—C17	113.5 (3)	C44—C43—C42	120.0
N1—C16—C23	106.8 (4)	C44—C43—H43	120.0
C17—C16—C23	112.6 (4)	C42—C43—H43	120.0
N1—C16—H16	107.9	C45—C44—C43	120.0
C17—C16—H16	107.9	C45—C44—H44	120.0
C23—C16—H16	107.9	C43—C44—H44	120.0
C18—C17—C22	117.2 (5)	C44—C45—C40	120.0
C18—C17—C16	120.6 (5)	C44—C45—H45	120.0
C22—C17—C16	122.1 (5)	C40—C45—H45	120.0
C17—C18—C19	122.8 (7)	C39—C46—H46A	109.5
C17—C18—H18	118.6	C39—C46—H46B	109.5
C19—C18—H18	118.6	H46A—C46—H46B	109.5
C20—C19—C18	119.3 (7)	C39—C46—H46C	109.5
C20—C19—H19	120.3	H46A—C46—H46C	109.5
C18—C19—H19	120.3	H46B—C46—H46C	109.5

O1—C1—C2—C3	−178.4 (4)	C29—C24—C25—C38	−167.4 (5)
C6—C1—C2—C3	3.2 (6)	C24—C25—C26—C27	−3.4 (8)
O1—C1—C2—C15	4.3 (6)	C38—C25—C26—C27	165.6 (5)

C6—C1—C2—C15	−174.1 (4)	C25—C26—C27—C28	3.4 (7)
C1—C2—C3—C4	−1.8 (6)	C25—C26—C27—C30	−175.6 (5)
C15—C2—C3—C4	175.6 (4)	C26—C27—C28—C29	−1.5 (7)
C2—C3—C4—C5	0.1 (6)	C30—C27—C28—C29	177.6 (5)
C2—C3—C4—C7	−179.5 (4)	C27—C28—C29—C24	−0.5 (7)
C3—C4—C5—C6	0.2 (6)	C27—C28—C29—C34	178.0 (5)
C7—C4—C5—C6	179.8 (4)	O2—C24—C29—C28	−176.3 (5)
C4—C5—C6—C1	1.2 (6)	C25—C24—C29—C28	0.6 (7)
C4—C5—C6—C11	−178.1 (4)	O2—C24—C29—C34	5.3 (8)
O1—C1—C6—C5	178.7 (3)	C25—C24—C29—C34	−177.9 (5)
C2—C1—C6—C5	−2.9 (5)	C26—C27—C30—C32A	57.3 (13)
O1—C1—C6—C11	−1.9 (6)	C28—C27—C30—C32A	−121.7 (12)
C2—C1—C6—C11	176.5 (4)	C26—C27—C30—C33	−134.9 (14)
C3—C4—C7—C8	0.3 (6)	C28—C27—C30—C33	46.2 (15)
C5—C4—C7—C8	−179.4 (5)	C26—C27—C30—C33A	−173.8 (16)
C3—C4—C7—C10	−120.2 (5)	C28—C27—C30—C33A	7.2 (17)
C5—C4—C7—C10	60.2 (5)	C26—C27—C30—C31	1.1 (10)
C3—C4—C7—C9	121.2 (5)	C28—C27—C30—C31	−177.9 (8)
C5—C4—C7—C9	−58.4 (5)	C26—C27—C30—C32	109.6 (8)
C5—C6—C11—C14	119.7 (4)	C28—C27—C30—C32	−69.4 (9)
C1—C6—C11—C14	−59.6 (5)	C26—C27—C30—C31A	−60.4 (8)
C5—C6—C11—C13	−117.8 (4)	C28—C27—C30—C31A	120.6 (7)
C1—C6—C11—C13	62.9 (5)	C28—C29—C34—C37	119.8 (6)
C5—C6—C11—C12	0.1 (6)	C24—C29—C34—C37	−61.8 (7)
C1—C6—C11—C12	−179.2 (4)	C28—C29—C34—C35	−3.4 (8)
C16—N1—C15—C2	171.7 (3)	C24—C29—C34—C35	175.0 (6)
C3—C2—C15—N1	134.9 (4)	C28—C29—C34—C36	−119.7 (5)
C1—C2—C15—N1	−47.7 (6)	C24—C29—C34—C36	58.7 (7)
C15—N1—C16—C17	56.4 (5)	C39—N2—C38—C25	162.6 (5)
C15—N1—C16—C23	−178.9 (4)	C26—C25—C38—N2	150.3 (5)
N1—C16—C17—C18	−117.7 (5)	C24—C25—C38—N2	−41.1 (8)
C23—C16—C17—C18	120.8 (5)	C38—N2—C39—C46	−172.7 (5)
N1—C16—C17—C22	63.8 (5)	C38—N2—C39—C40	63.4 (6)
C23—C16—C17—C22	−57.7 (6)	N2—C39—C40—C41	60.8 (5)
C22—C17—C18—C19	−0.8 (8)	C46—C39—C40—C41	−58.4 (6)
C16—C17—C18—C19	−179.3 (6)	N2—C39—C40—C45	−123.3 (4)
C17—C18—C19—C20	3.1 (11)	C46—C39—C40—C45	117.5 (5)
C18—C19—C20—C21	−4.6 (12)	C45—C40—C41—C42	0.0
C19—C20—C21—C22	3.7 (11)	C39—C40—C41—C42	175.9 (4)
C18—C17—C22—C21	−0.1 (7)	C40—C41—C42—C43	0.0
C16—C17—C22—C21	178.4 (5)	C41—C42—C43—C44	0.0
C20—C21—C22—C17	−1.3 (9)	C42—C43—C44—C45	0.0
O2—C24—C25—C26	178.1 (5)	C43—C44—C45—C40	0.0
C29—C24—C25—C26	1.2 (8)	C41—C40—C45—C44	0.0
O2—C24—C25—C38	9.4 (8)	C39—C40—C45—C44	−175.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.84	2.19	2.672 (5)	116
O2—H2*A*···N2	0.84	2.21	2.687 (5)	116