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(*R*)-(+)-3,3′-(1,1′-Binaphthyl-2,2′-dioxy)-diphthalonitrile benzene solvate

Xiao-Mei Zhang[a]* and Ji-Tao Lu[b]

[a]School of Chemistry and Chemical Technology, Shandong University, Jinan 250100, People's Republic of China, and [b]School of Chemistry annd Chemical Technology, Shandong University, Jinan 250100, People's Republic of China
Correspondence e-mail: zhangxiaomei@sdu.edu.cn

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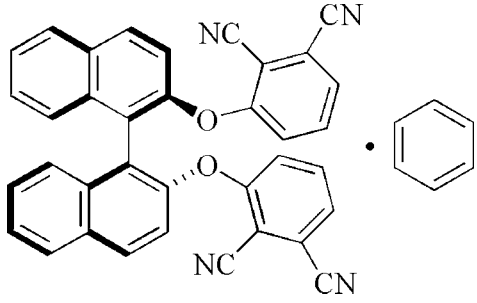
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.008 Å; R factor = 0.052; wR factor = 0.148; data-to-parameter ratio = 8.8.

White single crystals of the title compound, $C_{36}H_{18}N_4O_2\cdot C_6H_6$, were obtained by reacting (*R*)-(+)-1,1′-bi-2-naphthol with 3-nitrophthalonitrile and potassium carbonate and were recrystallized from benzene. The dihedral angle between the two naphthyl rings is 79.5(5)°. The (R)-(+)-2,2'-Bis(2,3-dicyanophenyl)-1,1'-binaphthyl units are connected together by the intermolecular \p-\p

Related literature

For related literature, see: Buchler & Ng (2000); He & Ng (2006); Jiang & Lin (2003, 2006); Kang *et al.* (2007); Kobayashi (1998); Kobayashi *et al.* (1999); Ni, Kou, Zhang *et al.* (2005); Ni, Kou, Zhao *et al.* (2005).



Experimental

Crystal data

$C_{36}H_{18}N_4O_2\cdot C_6H_6$
M_r = 616.65
Orthorhombic, $P2_12_12_1$
a = 7.87830 (10) Å
b = 12.2854 (2) Å
c = 35.3968 (5) Å
V = 3425.99 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 295 (2) K
0.28 × 0.16 × 0.12 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.979, T_{max} = 0.991
18358 measured reflections
3821 independent reflections
2647 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.149
S = 1.04
3821 reflections
433 parameters
15 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2276).

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supplementary materials

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(*R*)-(+)-3,3'-(1,1'-Binaphthyl-2,2'-dioxy)diphthalonitrile benzene solvate

X.-M. Zhang and J.-T. Lu

Comment

(*R*)-(+)-2,2'-Bis(2,3-dicyanophenyl)-1,1'-binaphthyl as well as its derivative chiral non-entrosymmetrically phthalocyanines play an important role in the molecular self-assembly and molecular recognition in chemical, physical and biological sciences, since can form metal complexes with rare earth, actinide, and some early transition metals and main-group elements. (Kobayashi *et al.*, 1999: Buchler & Ng, 2000).

To date, a variety of chiral supramolecular architectures have been obtained based on the above mentioned multifunctional ligands (Jiang & Lin, 2006). More recently, two novel interlocked chiral nanotubes formed from $Ni(acac)_2$ and C_2-symmetric 1,1-binaphthyl-6,6'-bipyridines have been reported (Jiang & Lin, 2003). They pointed out that the twisted binding sites of chiral rigid ditopic bridging ligands based on the 1,1'-binaphthyl unit will induce the formation of helical structures when linked by a linear metal-connecting point. Herein, we report the synthesis and crystal structure of a chiral organic compound (*R*)-(+)-2,2'-Bis(2,3-dicyanophenyl)-1,1'-binaphthyl benzene solvate (I).

The molecular structure of (I) is shown in Fig. 1. The cyano group C—N bond distances in this compound range from 1.138 (5) to 1.153 (6) Å and are similar to those in inorganic coordinated complexes (Ni, Kou, Zhang *et al.*, 2005; Ni, Kou, Zhao *et al.*, 2005). The C18—C19 bond distance is 1.495 (4) Å. The ten carbon atoms from two naphthyl rings in the title compound, respectively, are almost coplanar with the largest deviation value of 0.0527 (5) Å and 0.0138 (5) Å from the mean plane. The dihedral angle between the two naphthyl rings is 79.5 (5)°, which are similar to that of its derivative (*R*)-3,3'-Dibromo-2,2'-dimethoxy-5,5',6,6',7,7',8,8'-octahydro-1,1'- binaphthalene (80.2 (3)°) (He & Ng, 2006) and significantly larger than that of (*R*)-3,3'-Bis(3,4,5-trifluorophenyl)-1,1'-binaphthalene-2,2'-diol (73.5 (1)°) (Kang *et al.*, 2007) due to steric effects.

In (I), the (*R*)-(+)-2,2'-Bis(2,3-dicyanophenyl)-1,1'-binaphthyl units are connected together by the intermolecular π–π interactions leading to chiral helical one-dimensional supramolecular structures as shown in Fig. 2.

Experimental

(*R*)-(+)-2,2'-Bis(2,3-dicyanophenyl)-1,1'-binaphthyl was prepared according to the method reported in the literature (Kobayashi, 1998). A solid of (*R*)-(+)-2,2'-Bis(2,3-dicyanophenyl)-1,1'-binaphthyl (0.2 mmol) was added to the benzene solution (8 ml). The unsolved compounds was filtered and slowly evaporated to generate white single crystals suitable for X-ray diffraction analysis. Elemental analysis [found (calculated)] for $C_{36}H_{18}N_4O_2$: C 80.29 (80.40), H 3.37 (3.58), N 10.40% (10.62%). In the IR spectrum the cyano vibration is observed at 2238 cm^{-1}.

Refinement

The benzene solvate (C37–C42) was restrained to be planar regular hexagons, with target C⋯C distances of 1.39 (1) Å. H atoms were placed in calculated positions and were included in the refinement in the riding-model approximation, with

C—H = 0.93 Å and U_{iso}(H) = 1.2Ueq(C). The configuration of (I) was selected on the basis of the known configuration of the starting reagent, (*R*)-1,1'-binaphthalene-2,2'-diol, and Friedel pairs were merged.

Figures

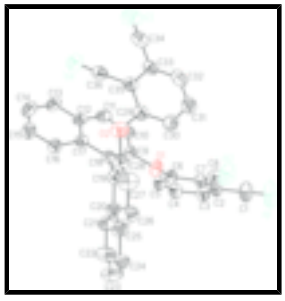


Fig. 1. A view of (I) with the unique atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. All H atoms and solvate benzene were omitted for clarity.

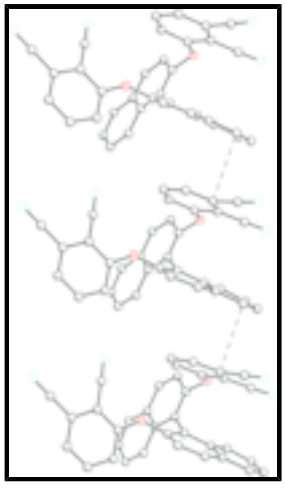


Fig. 2. The chiral helical one-dimensional supramolecular structure in (I) through inter-molecular π–π interactions.

(*R*)-(+)-3,3'-(1,1'-Binaphthyl-2,2'-dioxy)diphthalonitrile benzene solvate

Crystal data

$C_{36}H_{18}N_4O_2{\cdot}C_6H_6$	F_{000} = 1280
M_r = 616.65	D_x = 1.196 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 3821 reflections
a = 7.87830 (10) Å	θ = 2.3–26.0º
b = 12.2854 (2) Å	μ = 0.08 mm^{-1}
c = 35.3968 (5) Å	T = 295 (2) K
V = 3425.99 (9) Å^3	Block, colourless
Z = 4	0.28 × 0.16 × 0.12 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	3821 independent reflections

Radiation source: fine-focus sealed tube
Monochromator: graphite
T = 295(2) K
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.979, T_{max} = 0.991
18358 measured reflections
2647 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 26.0º
θ_{min} = 2.3º
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 15$
$l = -43 \rightarrow 43$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.149
S = 1.04
3821 reflections
433 parameters
15 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.147P)^2 + 0.3168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.1382 (3)	0.24330 (18)	0.85664 (7)	0.0668 (7)
O2	0.3103 (4)	−0.0648 (2)	0.86696 (7)	0.0737 (7)
N2	0.5117 (6)	0.3449 (3)	0.89188 (12)	0.1093 (14)
N1	0.4688 (7)	0.6613 (4)	0.87275 (19)	0.153 (2)
N3	0.4446 (6)	−0.0563 (4)	0.70672 (12)	0.1135 (14)
N4	0.2478 (5)	−0.2275 (3)	0.78731 (10)	0.0940 (12)
C1	0.3576 (7)	0.6042 (4)	0.86531 (16)	0.0984 (15)
C2	0.2210 (5)	0.5334 (3)	0.85618 (11)	0.0674 (10)

C3	0.0708 (5)	0.5749 (3)	0.84235 (12)	0.0767 (11)
H3	0.0586	0.6495	0.8386	0.092*
C4	−0.0598 (5)	0.5060 (3)	0.83415 (11)	0.0744 (11)
H4	−0.1625	0.5345	0.8258	0.089*
C5	−0.0423 (5)	0.3943 (3)	0.83802 (10)	0.0658 (9)
H5	−0.1318	0.3482	0.8319	0.079*
C6	0.1085 (5)	0.3523 (3)	0.85096 (9)	0.0576 (9)
C7	0.2408 (5)	0.4210 (3)	0.86122 (9)	0.0588 (9)
C8	0.3917 (6)	0.3773 (3)	0.87786 (11)	0.0729 (11)
C9	0.0561 (4)	0.1644 (3)	0.83539 (9)	0.0533 (8)
C10	0.0262 (5)	0.1796 (3)	0.79661 (9)	0.0630 (9)
H10	0.0523	0.2456	0.7852	0.076*
C11	−0.0408 (5)	0.0972 (3)	0.77607 (9)	0.0650 (10)
H11	−0.0565	0.1065	0.7502	0.078*
C12	−0.0872 (4)	−0.0024 (3)	0.79300 (9)	0.0572 (9)
C13	−0.1665 (5)	−0.0864 (4)	0.77246 (11)	0.0735 (11)
H13	−0.1823	−0.0788	0.7466	0.088*
C14	−0.2198 (5)	−0.1778 (4)	0.78994 (15)	0.0869 (13)
H14	−0.2738	−0.2320	0.7761	0.104*
C15	−0.1943 (5)	−0.1916 (3)	0.82898 (14)	0.0794 (11)
H15	−0.2339	−0.2542	0.8408	0.095*
C16	−0.1127 (5)	−0.1147 (3)	0.84941 (11)	0.0653 (9)
H16	−0.0935	−0.1261	0.8750	0.078*
C17	−0.0560 (4)	−0.0170 (3)	0.83221 (9)	0.0527 (8)
C18	0.0245 (4)	0.0673 (2)	0.85308 (8)	0.0490 (7)
C19	0.0871 (5)	0.0476 (3)	0.89238 (8)	0.0548 (8)
C20	0.0049 (5)	0.0909 (3)	0.92492 (8)	0.0609 (9)
C21	−0.1454 (6)	0.1520 (3)	0.92207 (10)	0.0766 (11)
H21	−0.1930	0.1652	0.8985	0.092*
C22	−0.2222 (7)	0.1922 (4)	0.95379 (12)	0.1017 (15)
H22	−0.3217	0.2324	0.9516	0.122*
C23	−0.1520 (9)	0.1732 (5)	0.98937 (13)	0.1127 (18)
H23	−0.2053	0.2007	1.0108	0.135*
C24	−0.0090 (8)	0.1158 (4)	0.99294 (11)	0.0977 (15)
H24	0.0367	0.1046	1.0169	0.117*
C25	0.0740 (6)	0.0717 (3)	0.96123 (9)	0.0737 (11)
C26	0.2243 (8)	0.0088 (4)	0.96445 (11)	0.0979 (15)
H26	0.2711	−0.0036	0.9882	0.117*
C27	0.3003 (6)	−0.0335 (4)	0.93352 (11)	0.0897 (13)
H27	0.3980	−0.0754	0.9360	0.108*
C28	0.2308 (5)	−0.0138 (3)	0.89762 (9)	0.0654 (9)
C29	0.3806 (5)	−0.0007 (3)	0.83978 (10)	0.0622 (9)
C30	0.4572 (5)	0.0987 (3)	0.84743 (12)	0.0740 (11)
H30	0.4622	0.1241	0.8721	0.089*
C31	0.5255 (5)	0.1595 (4)	0.81873 (14)	0.0855 (12)
H31	0.5752	0.2264	0.8241	0.103*
C32	0.5207 (5)	0.1220 (4)	0.78195 (14)	0.0827 (12)
H32	0.5661	0.1639	0.7625	0.099*
C33	0.4486 (4)	0.0224 (3)	0.77414 (10)	0.0661 (9)

C34	0.4460 (5)	−0.0202 (4)	0.73636 (12)	0.0807 (12)
C35	0.3780 (4)	−0.0403 (3)	0.80310 (10)	0.0586 (9)
C36	0.3035 (5)	−0.1449 (4)	0.79493 (10)	0.0683 (10)
C37	1.108 (2)	0.6227 (16)	0.9599 (5)	0.378 (18)
H37	1.2131	0.6350	0.9710	0.454*
C38	1.0635 (19)	0.5159 (13)	0.9502 (3)	0.274 (10)
H38	1.1383	0.4580	0.9533	0.329*
C39	0.902 (2)	0.5017 (9)	0.9357 (2)	0.195 (4)
H39	0.8636	0.4330	0.9288	0.235*
C40	0.7992 (11)	0.5927 (14)	0.9319 (2)	0.182 (5)
H40	0.6924	0.5807	0.9216	0.218*
C41	0.837 (2)	0.6995 (12)	0.9418 (3)	0.305 (11)
H41	0.7594	0.7566	0.9404	0.366*
C42	1.002 (2)	0.7105 (13)	0.9537 (5)	0.356 (15)
H42	1.0442	0.7802	0.9578	0.427*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0771 (16)	0.0471 (14)	0.0762 (14)	−0.0046 (13)	−0.0215 (13)	0.0079 (11)
O2	0.0922 (19)	0.0613 (15)	0.0675 (14)	0.0211 (15)	0.0058 (14)	0.0068 (12)
N1	0.114 (4)	0.094 (3)	0.253 (7)	−0.037 (3)	−0.053 (4)	0.031 (3)
N2	0.102 (3)	0.089 (3)	0.137 (3)	0.009 (2)	−0.053 (3)	−0.006 (2)
N3	0.106 (3)	0.157 (4)	0.077 (2)	0.013 (3)	0.005 (2)	0.006 (3)
N4	0.108 (3)	0.079 (3)	0.095 (2)	−0.011 (2)	0.016 (2)	−0.014 (2)
C1	0.090 (3)	0.067 (3)	0.138 (4)	−0.015 (3)	−0.021 (3)	0.018 (3)
C2	0.062 (2)	0.053 (2)	0.088 (2)	−0.009 (2)	−0.003 (2)	0.0094 (19)
C3	0.075 (3)	0.052 (2)	0.103 (3)	0.004 (2)	−0.005 (2)	0.010 (2)
C4	0.062 (2)	0.061 (3)	0.100 (3)	0.007 (2)	−0.007 (2)	0.006 (2)
C5	0.056 (2)	0.052 (2)	0.089 (2)	−0.0064 (18)	−0.0048 (19)	0.0024 (18)
C6	0.063 (2)	0.046 (2)	0.0638 (18)	−0.0059 (18)	−0.0045 (17)	0.0084 (15)
C7	0.058 (2)	0.054 (2)	0.0645 (19)	−0.0009 (18)	−0.0020 (17)	0.0079 (16)
C8	0.068 (3)	0.061 (2)	0.089 (3)	−0.004 (2)	−0.020 (2)	−0.001 (2)
C9	0.0576 (19)	0.0480 (19)	0.0544 (16)	−0.0002 (17)	−0.0076 (15)	0.0003 (15)
C10	0.073 (2)	0.062 (2)	0.0535 (18)	0.003 (2)	0.0001 (17)	0.0139 (17)
C11	0.073 (2)	0.075 (3)	0.0467 (16)	0.020 (2)	−0.0031 (17)	0.0000 (18)
C12	0.0531 (19)	0.061 (2)	0.0572 (18)	0.0127 (17)	−0.0047 (15)	−0.0109 (17)
C13	0.067 (2)	0.071 (3)	0.082 (2)	0.016 (2)	−0.011 (2)	−0.028 (2)
C14	0.065 (3)	0.073 (3)	0.123 (4)	0.007 (2)	−0.014 (3)	−0.040 (3)
C15	0.063 (2)	0.054 (2)	0.121 (3)	−0.001 (2)	0.007 (2)	−0.011 (2)
C16	0.067 (2)	0.050 (2)	0.079 (2)	0.0003 (19)	0.0049 (19)	−0.0025 (18)
C17	0.0512 (18)	0.0486 (19)	0.0582 (17)	0.0077 (17)	0.0021 (15)	−0.0021 (15)
C18	0.0578 (19)	0.0446 (18)	0.0446 (14)	0.0023 (16)	−0.0006 (14)	0.0032 (13)
C19	0.067 (2)	0.0468 (19)	0.0503 (16)	0.0005 (17)	−0.0054 (15)	0.0030 (14)
C20	0.071 (2)	0.059 (2)	0.0529 (18)	−0.0005 (19)	0.0007 (17)	0.0012 (15)
C21	0.083 (3)	0.083 (3)	0.064 (2)	0.009 (2)	−0.001 (2)	−0.002 (2)
C22	0.110 (4)	0.114 (4)	0.081 (3)	0.030 (3)	0.022 (3)	−0.008 (3)
C23	0.149 (5)	0.119 (4)	0.070 (3)	0.014 (4)	0.024 (3)	−0.018 (3)

C24	0.134 (4)	0.104 (4)	0.055 (2)	0.006 (4)	−0.003 (3)	−0.009 (2)
C25	0.100 (3)	0.070 (2)	0.0513 (19)	−0.003 (2)	−0.006 (2)	−0.0010 (17)
C26	0.128 (4)	0.110 (4)	0.055 (2)	0.014 (3)	−0.023 (2)	0.007 (2)
C27	0.100 (3)	0.100 (3)	0.070 (2)	0.032 (3)	−0.017 (2)	0.014 (2)
C28	0.084 (2)	0.057 (2)	0.0544 (18)	0.012 (2)	−0.0060 (18)	0.0039 (16)
C29	0.056 (2)	0.062 (2)	0.069 (2)	0.0143 (19)	−0.0002 (17)	0.0036 (18)
C30	0.061 (2)	0.069 (3)	0.092 (3)	0.003 (2)	−0.002 (2)	−0.011 (2)
C31	0.061 (2)	0.075 (3)	0.120 (4)	−0.001 (2)	0.008 (2)	−0.010 (3)
C32	0.061 (2)	0.078 (3)	0.110 (3)	0.005 (2)	0.018 (2)	0.025 (3)
C33	0.0502 (19)	0.070 (3)	0.078 (2)	0.010 (2)	0.0092 (18)	0.0082 (19)
C34	0.065 (2)	0.104 (3)	0.073 (3)	0.010 (3)	0.007 (2)	0.018 (2)
C35	0.0469 (18)	0.058 (2)	0.071 (2)	0.0127 (17)	0.0007 (16)	0.0024 (17)
C36	0.062 (2)	0.072 (3)	0.072 (2)	0.010 (2)	0.0113 (19)	−0.002 (2)
C37	0.31 (2)	0.63 (5)	0.191 (13)	−0.13 (3)	−0.031 (15)	−0.10 (2)
C38	0.245 (17)	0.47 (3)	0.105 (7)	0.145 (18)	0.020 (8)	0.034 (10)
C39	0.250 (13)	0.258 (13)	0.079 (5)	−0.030 (11)	0.047 (7)	0.017 (6)
C40	0.136 (6)	0.299 (14)	0.110 (5)	0.025 (9)	0.008 (4)	0.070 (8)
C41	0.36 (2)	0.41 (2)	0.141 (8)	−0.238 (19)	0.027 (10)	0.055 (11)
C42	0.33 (3)	0.52 (4)	0.218 (16)	−0.25 (3)	0.038 (17)	−0.064 (19)

Geometric parameters (Å, °)

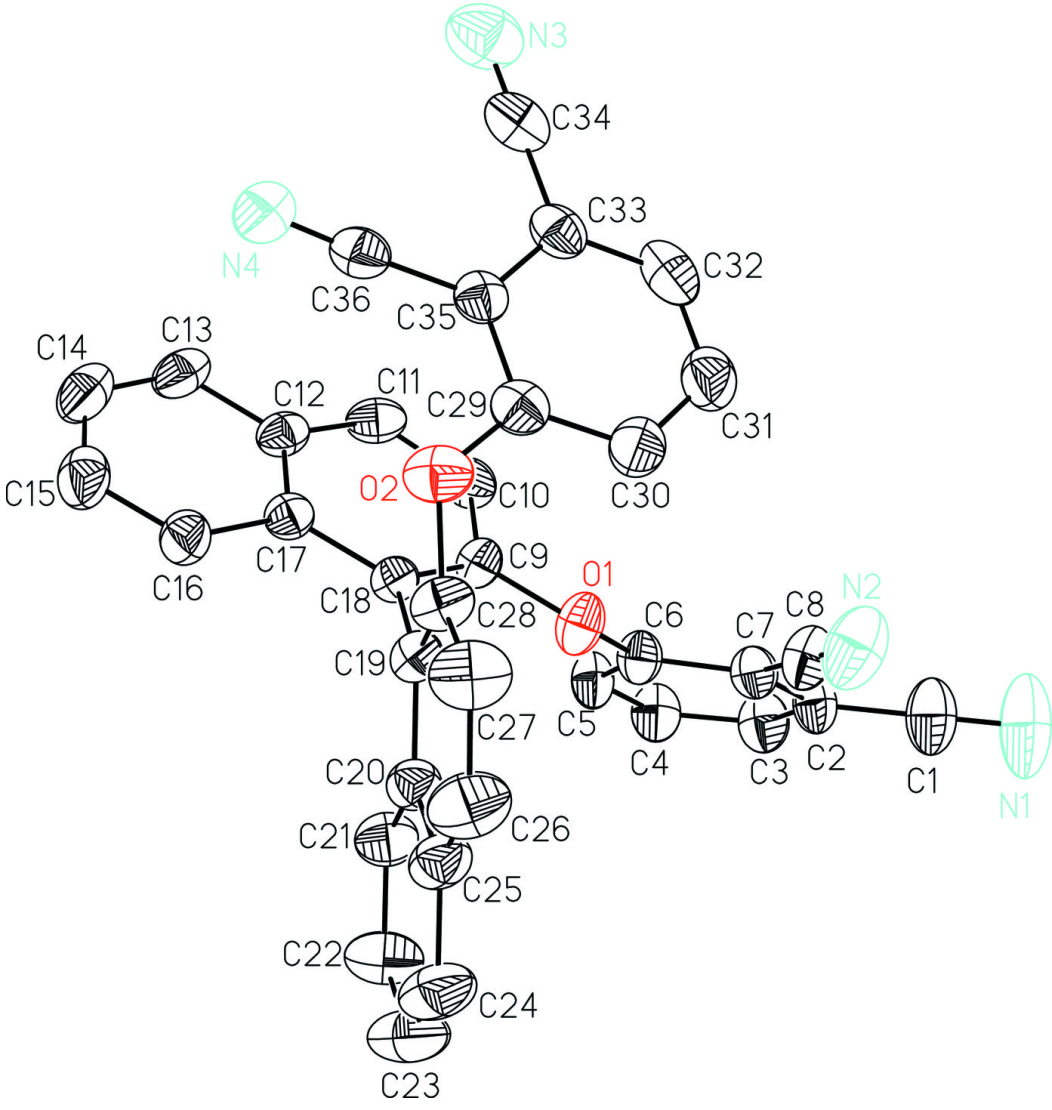
O1—C6	1.374 (4)	C20—C21	1.406 (6)
O1—C9	1.387 (4)	C20—C25	1.415 (5)
O2—C29	1.361 (4)	C21—C22	1.367 (6)
O2—C28	1.401 (4)	C21—H21	0.9300
N1—C1	1.153 (6)	C22—C23	1.395 (7)
N2—C8	1.140 (5)	C22—H22	0.9300
N3—C34	1.139 (5)	C23—C24	1.336 (7)
N4—C36	1.138 (5)	C23—H23	0.9300
C1—C2	1.421 (6)	C24—C25	1.407 (6)
C2—C3	1.379 (5)	C24—H24	0.9300
C2—C7	1.402 (5)	C25—C26	1.419 (7)
C3—C4	1.364 (6)	C26—C27	1.352 (6)
C3—H3	0.9300	C26—H26	0.9300
C4—C5	1.386 (5)	C27—C28	1.404 (5)
C4—H4	0.9300	C27—H27	0.9300
C5—C6	1.374 (5)	C29—C35	1.386 (5)
C5—H5	0.9300	C29—C30	1.389 (5)
C6—C7	1.390 (5)	C30—C31	1.371 (6)
C7—C8	1.431 (6)	C30—H30	0.9300
C9—C18	1.370 (4)	C31—C32	1.381 (6)
C9—C10	1.405 (4)	C31—H31	0.9300
C10—C11	1.353 (5)	C32—C33	1.378 (6)
C10—H10	0.9300	C32—H32	0.9300
C11—C12	1.411 (5)	C33—C35	1.398 (5)
C11—H11	0.9300	C33—C34	1.436 (6)
C12—C13	1.409 (5)	C35—C36	1.442 (6)
C12—C17	1.421 (4)	C37—C42	1.382 (10)

C13—C14	1.348 (6)	C37—C38	1.400 (9)
C13—H13	0.9300	C37—H37	0.9300
C14—C15	1.407 (6)	C38—C39	1.381 (9)
C14—H14	0.9300	C38—H38	0.9300
C15—C16	1.352 (5)	C39—C40	1.388 (8)
C15—H15	0.9300	C39—H39	0.9300
C16—C17	1.419 (5)	C40—C41	1.390 (9)
C16—H16	0.9300	C40—H40	0.9300
C17—C18	1.421 (4)	C41—C42	1.375 (9)
C18—C19	1.495 (4)	C41—H41	0.9300
C19—C28	1.373 (5)	C42—H42	0.9300
C19—C20	1.425 (5)		
C6—O1—C9	121.5 (3)	C20—C21—H21	119.8
C29—O2—C28	118.1 (3)	C21—C22—C23	120.4 (5)
N1—C1—C2	179.7 (6)	C21—C22—H22	119.8
C3—C2—C7	120.4 (4)	C23—C22—H22	119.8
C3—C2—C1	120.3 (4)	C24—C23—C22	120.5 (4)
C7—C2—C1	119.3 (4)	C24—C23—H23	119.7
C4—C3—C2	119.6 (4)	C22—C23—H23	119.7
C4—C3—H3	120.2	C23—C24—C25	121.3 (4)
C2—C3—H3	120.2	C23—C24—H24	119.4
C3—C4—C5	121.2 (4)	C25—C24—H24	119.4
C3—C4—H4	119.4	C24—C25—C20	118.8 (4)
C5—C4—H4	119.4	C24—C25—C26	122.2 (4)
C6—C5—C4	119.4 (4)	C20—C25—C26	119.0 (4)
C6—C5—H5	120.3	C27—C26—C25	120.9 (4)
C4—C5—H5	120.3	C27—C26—H26	119.5
C5—C6—O1	124.2 (3)	C25—C26—H26	119.5
C5—C6—C7	120.5 (3)	C26—C27—C28	119.6 (4)
O1—C6—C7	115.2 (3)	C26—C27—H27	120.2
C6—C7—C2	118.8 (3)	C28—C27—H27	120.2
C6—C7—C8	120.2 (3)	C19—C28—O2	120.6 (3)
C2—C7—C8	121.0 (3)	C19—C28—C27	122.6 (3)
N2—C8—C7	178.0 (5)	O2—C28—C27	116.7 (3)
C18—C9—O1	116.4 (3)	O2—C29—C35	116.9 (3)
C18—C9—C10	122.1 (3)	O2—C29—C30	123.2 (3)
O1—C9—C10	121.0 (3)	C35—C29—C30	119.8 (3)
C11—C10—C9	119.4 (3)	C31—C30—C29	120.4 (4)
C11—C10—H10	120.3	C31—C30—H30	119.8
C9—C10—H10	120.3	C29—C30—H30	119.8
C10—C11—C12	121.4 (3)	C30—C31—C32	120.4 (4)
C10—C11—H11	119.3	C30—C31—H31	119.8
C12—C11—H11	119.3	C32—C31—H31	119.8
C13—C12—C11	122.1 (3)	C33—C32—C31	119.8 (4)
C13—C12—C17	119.2 (4)	C33—C32—H32	120.1
C11—C12—C17	118.6 (3)	C31—C32—H32	120.1
C14—C13—C12	120.7 (4)	C32—C33—C35	120.4 (4)
C14—C13—H13	119.6	C32—C33—C34	121.1 (4)
C12—C13—H13	119.6	C35—C33—C34	118.5 (4)

C13—C14—C15	120.5 (4)	N3—C34—C33	178.5 (5)
C13—C14—H14	119.8	C29—C35—C33	119.2 (4)
C15—C14—H14	119.8	C29—C35—C36	120.4 (3)
C16—C15—C14	120.6 (4)	C33—C35—C36	120.4 (3)
C16—C15—H15	119.7	N4—C36—C35	177.6 (4)
C14—C15—H15	119.7	C42—C37—C38	122.8 (12)
C15—C16—C17	120.8 (4)	C42—C37—H37	118.6
C15—C16—H16	119.6	C38—C37—H37	118.6
C17—C16—H16	119.6	C39—C38—C37	116.0 (10)
C16—C17—C12	118.1 (3)	C39—C38—H38	122.0
C16—C17—C18	122.3 (3)	C37—C38—H38	122.0
C12—C17—C18	119.6 (3)	C38—C39—C40	118.2 (9)
C9—C18—C17	118.5 (3)	C38—C39—H39	120.9
C9—C18—C19	120.4 (3)	C40—C39—H39	120.9
C17—C18—C19	120.9 (3)	C39—C40—C41	127.8 (9)
C28—C19—C20	118.1 (3)	C39—C40—H40	116.1
C28—C19—C18	119.1 (3)	C41—C40—H40	116.1
C20—C19—C18	122.8 (3)	C42—C41—C40	111.8 (10)
C21—C20—C25	118.6 (3)	C42—C41—H41	124.1
C21—C20—C19	121.6 (3)	C40—C41—H41	124.1
C25—C20—C19	119.8 (4)	C41—C42—C37	122.9 (12)
C22—C21—C20	120.4 (4)	C41—C42—H42	118.5
C22—C21—H21	119.8	C37—C42—H42	118.5
N1—C1—C2—C3	−175 (100)	C18—C19—C20—C21	2.5 (5)
N1—C1—C2—C7	5(99)	C28—C19—C20—C25	1.2 (5)
C7—C2—C3—C4	1.1 (6)	C18—C19—C20—C25	−178.1 (3)
C1—C2—C3—C4	−179.2 (4)	C25—C20—C21—C22	0.3 (6)
C2—C3—C4—C5	−2.6 (6)	C19—C20—C21—C22	179.7 (4)
C3—C4—C5—C6	1.1 (6)	C20—C21—C22—C23	0.1 (7)
C4—C5—C6—O1	178.5 (3)	C21—C22—C23—C24	0.1 (8)
C4—C5—C6—C7	2.0 (6)	C22—C23—C24—C25	−0.8 (8)
C9—O1—C6—C5	29.3 (5)	C23—C24—C25—C20	1.1 (7)
C9—O1—C6—C7	−154.0 (3)	C23—C24—C25—C26	−178.8 (5)
C5—C6—C7—C2	−3.5 (5)	C21—C20—C25—C24	−0.9 (6)
O1—C6—C7—C2	179.7 (3)	C19—C20—C25—C24	179.7 (4)
C5—C6—C7—C8	174.3 (3)	C21—C20—C25—C26	179.1 (4)
O1—C6—C7—C8	−2.5 (5)	C19—C20—C25—C26	−0.3 (6)
C3—C2—C7—C6	1.9 (6)	C24—C25—C26—C27	179.3 (5)
C1—C2—C7—C6	−177.8 (4)	C20—C25—C26—C27	−0.7 (7)
C3—C2—C7—C8	−175.8 (4)	C25—C26—C27—C28	0.8 (7)
C1—C2—C7—C8	4.4 (6)	C20—C19—C28—O2	175.5 (3)
C6—C7—C8—N2	−135 (14)	C18—C19—C28—O2	−5.1 (5)
C2—C7—C8—N2	43 (14)	C20—C19—C28—C27	−1.1 (6)
C6—O1—C9—C18	−148.6 (3)	C18—C19—C28—C27	178.2 (4)
C6—O1—C9—C10	38.8 (5)	C29—O2—C28—C19	65.2 (5)
C18—C9—C10—C11	2.1 (5)	C29—O2—C28—C27	−117.9 (4)
O1—C9—C10—C11	174.3 (3)	C26—C27—C28—C19	0.2 (7)
C9—C10—C11—C12	2.4 (6)	C26—C27—C28—O2	−176.6 (4)
C10—C11—C12—C13	176.1 (3)	C28—O2—C29—C35	−147.4 (3)

C10—C11—C12—C17	−2.6 (5)	C28—O2—C29—C30	34.6 (5)
C11—C12—C13—C14	−175.5 (4)	O2—C29—C30—C31	−179.9 (3)
C17—C12—C13—C14	3.1 (5)	C35—C29—C30—C31	2.1 (5)
C12—C13—C14—C15	−1.2 (6)	C29—C30—C31—C32	−0.8 (6)
C13—C14—C15—C16	−1.5 (6)	C30—C31—C32—C33	−0.7 (6)
C14—C15—C16—C17	2.1 (6)	C31—C32—C33—C35	1.0 (6)
C15—C16—C17—C12	−0.1 (5)	C31—C32—C33—C34	−178.3 (4)
C15—C16—C17—C18	177.6 (3)	C32—C33—C34—N3	141 (18)
C13—C12—C17—C16	−2.5 (5)	C35—C33—C34—N3	−38 (19)
C11—C12—C17—C16	176.2 (3)	O2—C29—C35—C33	−180.0 (3)
C13—C12—C17—C18	179.8 (3)	C30—C29—C35—C33	−1.8 (5)
C11—C12—C17—C18	−1.5 (5)	O2—C29—C35—C36	0.0 (5)
O1—C9—C18—C17	−178.6 (3)	C30—C29—C35—C36	178.1 (3)
C10—C9—C18—C17	−6.1 (5)	C32—C33—C35—C29	0.3 (5)
O1—C9—C18—C19	−3.8 (5)	C34—C33—C35—C29	179.5 (3)
C10—C9—C18—C19	168.7 (3)	C32—C33—C35—C36	−179.6 (3)
C16—C17—C18—C9	−171.9 (3)	C34—C33—C35—C36	−0.4 (5)
C12—C17—C18—C9	5.7 (5)	C29—C35—C36—N4	−161 (11)
C16—C17—C18—C19	13.3 (5)	C33—C35—C36—N4	19 (12)
C12—C17—C18—C19	−169.0 (3)	C42—C37—C38—C39	−3(2)
C9—C18—C19—C28	−101.1 (4)	C37—C38—C39—C40	0.3 (10)
C17—C18—C19—C28	73.6 (4)	C38—C39—C40—C41	−1.5 (13)
C9—C18—C19—C20	78.2 (4)	C39—C40—C41—C42	5.3 (17)
C17—C18—C19—C20	−107.1 (4)	C40—C41—C42—C37	−8(3)
C28—C19—C20—C21	−178.2 (4)	C38—C37—C42—C41	8(3)

Fig. 1


N3
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C30
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C21
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C22
C24
C23

Fig. 2