

Acta Cryst. (1997). C53, 1458–1459

An Enantiomerically Pure Schiff Base Ligand

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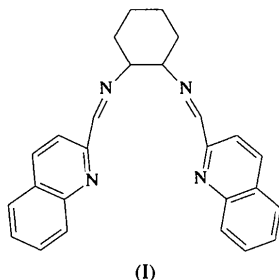
(Received 7 January 1997; accepted 22 April 1997)

Abstract

(1*R*,2*R*)-(–)-*N,N'*-Bis(quinoline-2-methylidene)-1,2-cyclohexanediamine, C₂₆H₂₄N₄, was prepared by the reaction of quinoline-2-carbaldehyde and the corresponding diamine. The crystal structure of the resulting enantiomerically pure quadridentate ligand has been determined.

Comment

Metal complexes where the central atom is coordinated to ligands with one or more N atoms have been widely studied and patented over the years as catalysts in various processes (for a review on the role of nitrogen ligands in catalytic reactions, see Togni & Venanzi, 1994). Our recent reports describe the use of nitrogen-containing quadridentate ligands, such as salen-type Schiff bases with group 4 metals (Repo *et al.*, 1997), as well as bridged-quinoline systems with later transition metals (Rieger, Abu-Surrah, Fawzi & Steimann, 1995), in the preparation of potential catalyst precursors. The title compound, (I), can also be used as a ligand in catalyst synthesis: its cationic palladium complex is active in norbornene polymerization (Abu-Surrah & Rieger, 1997).



Structurally, compound (I) (Fig. 1) resembles two recently determined Schiff base ligands with cyclohexyl bridging groups (Cannadine, Corden, Errington, Moore

& Wallbridge, 1996). The imine bond lengths, 1.263 (2) and 1.264 Å for N1—C7 and N3—C17, respectively, fall in the range of N=C double bonds.

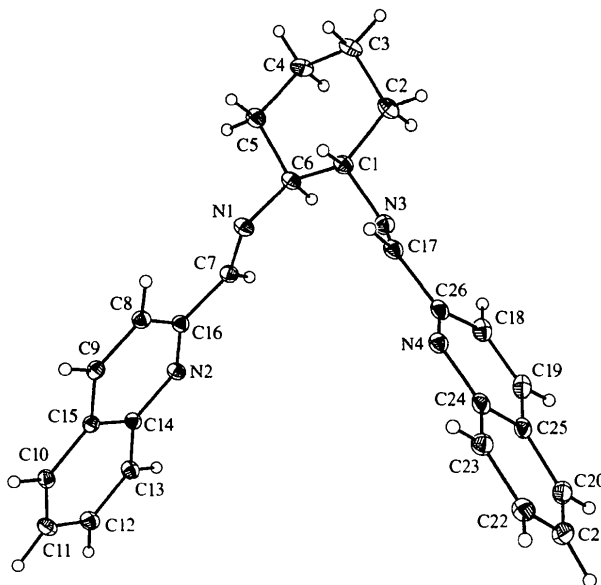


Fig. 1. View of the title compound, C₂₆H₂₄N₄. Displacement ellipsoids are drawn at the 20% probability level.

Experimental

A solution of (1*R*,2*R*)-(–)-1,2-diaminocyclohexane (0.66 g, 5.78 mmol) in ethanol (20 ml) was added to a solution of quinoline-2-carbaldehyde (2.0 g, 12.7 mmol) in ethanol (50 ml). The yellowish precipitate which started forming after 30 min was collected after 2 h of stirring and washed with ethanol (3 × 20 ml) and petroleum ether 30/50 (3 × 50 ml). Recrystallization from a mixture of dichloromethane and diethyl ether (1:1) yielded colourless needle-like crystals suitable for X-ray analysis (total yield 1.70 g, 75%).

Crystal data

C₂₆H₂₄N₄
M_r = 392.49
Orthorhombic
P2₁2₁2₁
a = 5.932 (1) Å
b = 15.120 (5) Å
c = 23.564 (6) Å
V = 2114 (1) Å³
Z = 4
D_x = 1.233 Mg m⁻³
D_m not measured

Mo Kα radiation

λ = 0.71073 Å
Cell parameters from 30 reflections
θ = 15–32°
μ = 0.074 mm⁻¹
T = 173 (2) K
Needle
0.55 × 0.20 × 0.20 mm
Colourless

Data collection

Siemens P4 diffractometer
ω scans

R_{int} = 0.041
θ_{max} = 25.05°

Absorption correction: $h = -7 \rightarrow 7$
 ψ scan (Sheldrick, 1993) $k = -17 \rightarrow 10$
 $T_{\min} = 0.899$, $T_{\max} = 0.985$ $l = -28 \rightarrow 20$
 14322 measured reflections 2 standard reflections
 3711 independent reflections every 58 reflections
 2890 reflections with intensity decay: 6%
 $I > 2\sigma(I)$

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\max} < 0.001$
 $R[F^2 > 2\sigma(F^2)] = 0.032$ $\Delta\rho_{\max} = 0.132 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.072$ $\Delta\rho_{\min} = -0.116 \text{ e } \text{\AA}^{-3}$
 $S = 1.373$ Extinction correction:
 3711 reflections *SHELXL93*
 272 parameters Extinction coefficient:
 H atoms riding 0.0115 (12)
 $w = \exp[5.00(\sin\theta/\lambda)^2] /$ Scattering factors from
 $[\sigma^2(F_o^2) + (0.0349P)^2]$ *International Tables for*
 where $P = (0.333F_o^2 + 0.667F_c^2)$ *Crystallography* (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

N1—C7	1.263 (2)	C1—C6	1.529 (2)
N1—C6	1.461 (2)	C7—C16	1.473 (2)
N3—C17	1.264 (2)	C17—C26	1.482 (3)
N3—C1	1.460 (2)		
C7—N1—C6	117.5 (2)	N1—C6—C1	108.86 (15)
C17—N3—C1	119.2 (2)	N1—C7—C16	122.2 (2)
N3—C1—C6	109.30 (14)	N3—C17—C26	121.0 (2)

The intensity data were corrected for Lorentz and polarization effects and for absorption. A decay correction was also applied. All non-H atoms were refined anisotropically.

Data collection: *P3/P4-PC Diffractometer Program* (Siemens, 1991a). Cell refinement: *P3/P4-PC Diffractometer Program*. Data reduction: *XDISK* (Siemens, 1991b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL93*.

The work of ASA-S was supported financially by the Deutscher Akademischer Austausch-Dienst (DAAD) through the award of a PhD scholarship. TVL and TR were supported by the Academy of Finland and the DAAD exchange program.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1231). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1459–1462

2,4,6,8-Tetraazabicyclo[3.3.1]nonane-3,7-dione and 2,4,6,8-Tetraacetyl-2,4,6,8-tetraazabicyclo[3.3.1]nonane-3,7-dione

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(Received 31 January 1997; accepted 29 April 1997)

Abstract

The title compounds, 2,4,6,8-tetraazabicyclo[3.3.1]nonane-3,7-dione, (I) (C₅H₈N₄O₂), and 2,4,6,8-tetraacetyl-2,4,6,8-tetraazabicyclo[3.3.1]nonane-3,7-dione, (II) (C₁₃H₁₆N₄O₆), display similar conformations, each with the cyclic urea moieties and bridgehead atoms forming two mean planes having a dihedral angle of approximately 110°. The CO of each acetyl group is *anti* to the CO of the cyclic urea to which it is attached.

Comment

The stereochemistry of bicyclo[3.3.1]nonane and hetero analogues has been studied by a variety of physical and theoretical methods (Zefirov & Palyulin, 1991; Eliel & Wilen, 1994) because their structures are related to those of natural products (alkaloids) and adamantanoid compounds (Eres'ko *et al.*, 1979; Peters, 1979; Vogtle, 1992). While exploring predictive structure–property relationships of new molecules (Piacenza, Legsai, Blaise & Gallo, 1996), it was found that no structural data were