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Methyl 2-(cyclohexylamino)cyclopent-1-ene-1-dithiocarboxylate

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Key indicators

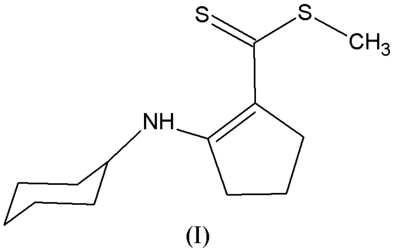
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.112
Data-to-parameter ratio = 20.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title molecule, $C_{13}H_{21}NS_2$, the Csp^2—S and C═S bond lengths are 1.770 (3) and 1.685 (3) Å, respectively. An intermolecular N—H···S hydrogen bond defines the preferred conformation of the molecule.

Comment

Over the past few decades our group has been interested in the manipulation of the reactivity of the Rh^{I} center in [Rh(BID)(CO)(PX_3)] complexes towards iodomethane oxidative addition (BID = monoanionic bidentate ligands containing different donor atoms such as O, N and S, and PX_3 = tertiary phosphine or phosphite). The utilization of the aforementioned model complexes may give insight into mechanistic aspects of homogeneous catalysis, such as the well known Monsanto process, where the oxidative addition was identified as the rate determining step in the methanol carbonylation cycle. The title compound, (I), an *S*,*N*-bidentate ligand (cacsmH), is related to the bidentate ligands methyl 2-methylamino-1-cyclopentene-1-dithiocarboxylate (macsmH) and methyl 2-imino-1-cyclopentene-dithiocarboxylate (hacsmH) used in the preparation of previously reported [Rh(BID)(CO)(PPh_3)] complexes (Steyn *et al.*, 1992, 1997). The structure of [Rh(macsm)(CO)(PPh_3)] has also been reported previously (Steyn & Roodt, 2001).



(I)

The title compound (Fig. 1) crystallizes in the monoclinic space group $P2_1/c$. All bond lengths and angles (Table 1) are within the normal ranges for this type of compound. Hydrogen bonding is observed between atoms N1 and S2 (Table 2), giving rise to the preferred conformation of the S2—C2—S1 moiety and the coplanar alignment with the 1-cyclopentene fragment. The S1—C1 bond deviates slightly from this plane (Table 1); this may be attributed to the packing effects.

Table 3 illustrates the rigidity of these types of ligands, as only slight deviations are observed in the geometry surrounding the N– and S-donor atoms bonded to the metal centre *versus* that of the free macsmH ligand. The orientations of the S—CH_3 bonds in the structures *a*–*d* (Table 3) are similar, mainly due to a packing mode favouring the less sterically hindered nature of this orientation.

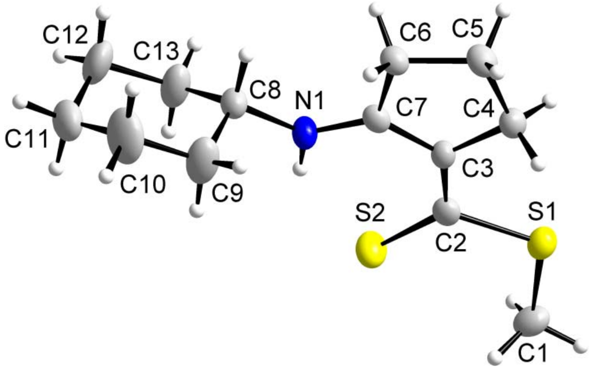


Figure 1
View of the title compound, shown with 30% probability displacement ellipsoids.

Experimental

The title compound was prepared according to the synthetic routes described previously (Bordàs *et al.*, 1972). Crystals were obtained by slow evaporation of a concentrated acetone solution.

Crystal data

$C_{13}H_{21}NS_2$
$M_r = 255.43$
Monoclinic, $P2_1/c$
$a = 19.9786$ (17) Å
$b = 5.3550$ (4) Å
$c = 13.5855$ (12) Å
$\beta = 107.507$ (2)°
$V = 1386.1$ (2) Å^3
$Z = 4$

$D_x = 1.224$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 816 reflections
$\theta = 2.3$–24.2°
$\mu = 0.36$ mm^{-1}
$T = 293$ (2) K
Plate, yellow
0.12 × 0.09 × 0.04 mm

Data collection

Bruker SMART CCD 1K diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.958$, $T_{max} = 0.986$
11770 measured reflections

3025 independent reflections
1362 reflections with $I > 2\sigma(I)$
$R_{int} = 0.099$
$\theta_{max} = 27°$
$h = -25 \rightarrow 25$
$k = -6 \rightarrow 6$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.112$
$S = 0.83$
3025 reflections
150 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C2	1.770 (3)	N1—C7	1.319 (3)
S1—C1	1.785 (3)	N1—C8	1.456 (3)
S2—C2	1.685 (3)		
C2—S1—C1	104.74 (13)	C3—C2—S1	112.41 (19)
C7—N1—C8	127.1 (2)	S2—C2—S1	120.49 (17)
C3—C2—S2	127.1 (2)	N1—C7—C3	126.3 (2)
S2—C2—C7—N1	0.9 (2)	C1—S1—C2—S2	−5.5 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S2	0.93 (3)	2.24 (3)	3.026 (3)	142 (2)

Table 3
Comparative geometrical data (Å, °) for *N*,*S*-bidentate ligands bonded to rhodium(I).

Ligand	S2—C2	S1—C1	C3—C2—S2	C3—C7—N1	S1—C2—S2	Ref.
cacsmH	1.685 (3)	1.785 (3)	127.1 (2)	126.3 (2)	120.49 (17)	*a*
cacsm	1.710 (5)	1.772 (7)	128.2 (4)	129.4 (4)	116.1 (3)	*b*
macsm	1.713 (5)	1.775 (7)	128.2 (4)	128.8 (5)	117.0 (3)	*c*
hacsm	1.707 (5)	1.776 (7)	128.2 (4)	128.8 (5)	118.1 (3)	*d*

† Notes: (*a*) This work; (*b*) Steyn & Roodt (2001); (*c*) Steyn *et al.* (1992); (*d*) Steyn *et al.* (1997).

The C-bound H atoms were placed in geometrically idealized positions (C—H = 0.97–0.98 Å) and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2$–$1.5U_{eq}(C)$. The methyl group was refined as a rigid rotor. Amine atom H1 was located in a difference Fourier map and refined isotropically.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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