

# Monoclinic form of 1-(dicyclohexylthiophosphinoyl)-*N*-phenylthioformamide

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## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

$R$  factor = 0.040

$wR$  factor = 0.028

Data-to-parameter ratio = 15.1

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

The molecular structure of the title compound,  $\text{Cy}_2\text{P}(=\text{S})\text{C}(=\text{S})\text{N}(\text{H})\text{Ph}$  (Cy is cyclohexyl) or  $\text{C}_{19}\text{H}_{28}\text{NPS}_2$ , is virtually identical to that found in the previously reported triclinic polymorph. Thus, the conformation about the C–N bond is *Z* and the dimensions defining the central  $\text{P}(=\text{S})\text{C}(=\text{S})\text{NC}$  group in both forms are experimentally equivalent.

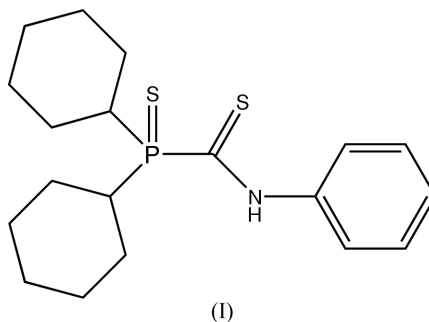
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## Comment

Compounds of the general formula  $R_2\text{P}(Y)\text{C}(=\text{S})\text{N}(\text{H})R'$  (where  $R, R' = \text{alkyl or aryl}$ , and  $Y = \text{O, S or Se}$ ) are known to adopt a variety of coordination modes in both their neutral and deprotonated forms (Siasios & Tiekink, 1996*a,b*; Horn *et al.*, 2000). Consequently, systematic structural studies have been conducted on these ligands (Cowan *et al.*, 1984; Siasios & Tiekink, 1994, 1995*a,b*). The structure of the title compound,  $\text{Cy}_2\text{P}(=\text{S})\text{C}(=\text{S})\text{N}(\text{H})\text{Ph}$ , (I), represents a second polymorph, a triclinic form having been determined previously (Siasios & Tiekink, 1994).



The key geometric parameters defining the  $\text{P}(\text{S})\text{C}(\text{S})\text{NC}$  chromophore [*e.g.*  $\text{P1}-\text{S2}$  1.9655 (8)  $\text{\AA}$ ,  $\text{C1}-\text{S1}$  1.639 (2)  $\text{\AA}$ ,  $\text{P1}-\text{C1}$  1.866 (2)  $\text{\AA}$ ,  $\text{N1}-\text{C1}$  1.334 (3)  $\text{\AA}$  and  $\text{S2}-\text{P1}-\text{C1}-\text{S1}$   $-166.1$  (1) $^\circ$ ] are equal within experimental error to those in the triclinic form (Siasios & Tiekink, 1994). The major difference between the polymorphs is found in the relative orientations of the cyclohexyl rings. Thus, whilst the  $\text{P}(\text{S})\text{C}(\text{S})\text{N}(\text{H})\text{Ph}$  portions of the molecules are essentially superimposable in the two forms, the cyclohexyl groups are not. Referring to Fig. 1, it can be seen that the methine H atom bound to C8 is directed towards the same side of the molecule as S2, and the C14 H atom is directed towards the same side as the S1 atom. Opposite orientations are observed in the triclinic form (Siasios & Tiekink, 1994).

## Experimental

Crystals of the title compound (Ojima *et al.*, 1969) were isolated by slow evaporation of a  $\text{CH}_2\text{Cl}_2$  solution at room temperature (m.p. 382–385 K).

## Crystal data

$\text{C}_{19}\text{H}_{28}\text{NPS}_2$   
 $M_r = 365.53$   
 Monoclinic,  $P2_1/c$   
 $a = 10.074$  (1) Å  
 $b = 18.139$  (2) Å  
 $c = 11.3858$  (9) Å  
 $\beta = 105.426$  (8)°  
 $V = 2005.6$  (3) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.210$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta = 11.0$ – $12.2^\circ$   
 $\mu = 0.35$  mm<sup>-1</sup>  
 $T = 293$  K  
 Prismatic, yellow  
 $0.25 \times 0.20 \times 0.15$  mm

## Data collection

Rigaku AFC-7R diffractometer  
 $\omega$ - $2\theta$  scans  
 6358 measured reflections  
 6054 independent reflections  
 3143 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$   
 $\theta_{\text{max}} = 30.1^\circ$

$h = 0 \rightarrow 14$   
 $k = 0 \rightarrow 25$   
 $l = -16 \rightarrow 15$   
 3 standard reflections  
 every 150 reflections  
 intensity decay: 0.6%

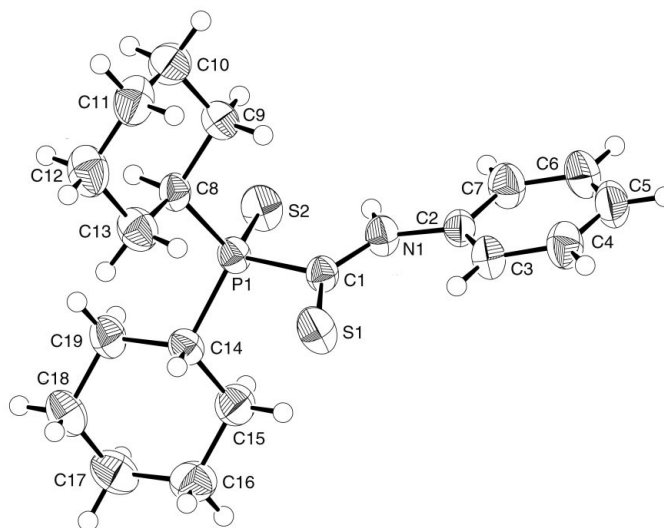
## Refinement

Refinement on  $F$   
 $R = 0.040$   
 $wR = 0.028$   
 $S = 1.69$   
 3143 reflections  
 208 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o) + 0.004|F_o|^2]$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1992); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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**Figure 1**  
 A view of  $\text{Cy}_2\text{P}(=\text{S})\text{C}(=\text{S})\text{N}(\text{H})\text{Ph}$  in the monoclinic polymorph showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level (Johnson, 1976).

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