

Table 2. Selected interatomic distances (Å) and angles (°)

	<i>Endo</i>	<i>Exo</i>		<i>Endo</i>	<i>Exo</i>		<i>Endo</i>	<i>Exo</i>
O(1)–C(2)	1.198 (2)	1.199 (3)	C(5)–C(6)	1.539 (2)	1.527 (4)	C(7)–C(8)	1.551 (2)	1.558 (3)
C(2)–O(3)	1.341 (2)	1.339 (2)	C(6)–C(7)	1.543 (2)	1.535 (4)	C(8)–C(2)	1.499 (2)	1.499 (3)
O(3)–C(4)	1.450 (2)	1.455 (3)	C(7)–C(71)	1.455 (2)	1.452 (3)	C(8)–C(5)	1.530 (2)	1.527 (3)
C(4)–C(5)	1.506 (2)	1.496 (3)	C(71)–N(72)	1.137 (2)	1.132 (3)			
O(1)–C(2)–O(3)	120.90 (14)	121.3 (2)	C(4)–C(5)–C(8)	105.26 (11)	104.9 (2)	C(8)–C(7)–C(71)	118.03 (12)	113.9 (2)
O(1)–C(2)–C(8)	128.15 (15)	128.3 (2)	C(6)–C(5)–C(8)	89.72 (11)	90.3 (2)	C(7)–C(71)–N(72)	178.28 (16)	178.8 (2)
O(3)–C(2)–C(8)	110.94 (12)	110.5 (2)	C(5)–C(6)–C(7)	89.69 (11)	90.8 (2)	C(7)–C(8)–C(2)	114.36 (11)	113.2 (2)
C(2)–O(3)–C(4)	111.62 (11)	111.4 (2)	C(6)–C(7)–C(8)	88.78 (11)	88.8 (2)	C(7)–C(8)–C(5)	89.73 (11)	89.9 (2)
O(3)–C(4)–C(5)	106.10 (12)	107.3 (2)	C(6)–C(7)–C(71)	117.88 (13)	115.6 (2)	C(2)–C(8)–C(5)	103.86 (12)	105.1 (2)
C(4)–C(5)–C(6)	116.49 (12)	116.9 (2)						

The cyclobutane rings are also non-planar in both compounds. The puckering angle (Bucourt, 1974) for *endo* is 15.4° and for *exo* 4.6°. The increased puckering of *endo* probably arises from steric repulsion between the atoms of the lactone and the carbonitrile group.

Hydrogen bonding is not a major consideration in either structure. The shortest O...H contacts in *endo* are O(1)···H(5)( $x-\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}$ ) = 2.57 (2) and O(3)···H(6B)( $1-x, 1-y, 1-z$ ) = 2.63 (2) Å, and in *exo* O(1)···H(6B)( $\frac{1}{2}-x, \frac{1}{2}+y, z$ ) = 2.65 (3) and O(1)···H(8) = 2.69 (2) Å.

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## 2-(2-Quinoly)cyclohexanone Phenylhydrazone, C<sub>21</sub>H<sub>21</sub>N<sub>3</sub>

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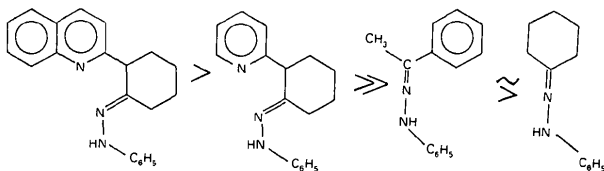
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**Abstract.**  $M_r = 315.4$ , monoclinic,  $P2_1/n$ ,  $a = 16.249$  (2),  $b = 9.058$  (1),  $c = 11.846$  (2) Å,  $\beta = 102.42$  (1)°,  $V = 1702.7$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.23$  g cm<sup>-3</sup>,  $F(000) = 672$ , Cu K $\alpha$ ,  $\lambda = 1.5418$  Å,  $\mu = 5.35$  cm<sup>-1</sup>,  $R = 0.041$  for 1698 observed reflections. The quinoline moiety is not planar and its two component rings make an angle of 1.7 (1)°. The cyclohexane ring shows partial disorder, and three of

the distances in it show significant shortening [C(5)—C(4) 1.443 (30), C(5)—C(6) 1.484 (14), C(5')—C(4) 1.433 (13) Å].

**Introduction.** The dehydrogenation of isomerizable phenylhydrazones with many acceptors, yielding the corresponding phenylazoalkenes, proceeds through the formation of intermediate charge-transfer complexes (CTC) (Tosi, Passalacqua & Marchetti, 1971) while those not isomerizable (Marchetti & Tosi, 1972; Bruni, Cardellini & Tosi, 1981) and methylphenylhydrazones (Tosi, Bruni, Cardellini & Fava, 1983) form stable CTC's in solution. The stability of some of the above isomerizable phenylhydrazones and of the corresponding intermediate CTC's, follows the order:



However, the complexes are not isolable and yield the phenylazoalkenes through the formation of the corresponding radical ions (as evidenced by ESR spectroscopy). The quinoline ring, other substituents being equal, seems to play an important role in stabilizing the complex which decomposes at a lower rate in two radical ions. On the other hand some quinoline and isoquinoline derivatives form, with various acceptors, stable solid CTC's (Poradowska, Czuba, Lorentz & Chyla, 1976) characterized by better properties than the pyridine analogues (Tosi, Bruni, Cardellini & Mari, 1983). This behaviour seems to be due both to the possibility of an extended charge delocalization between the quinoline group and the other moiety of the donor molecule, and to structural features. Furthermore, it has been proved that the quinoline ring alone does not form CTC's with the same acceptors (Bruni *et al.*, 1981).

As the stability of the complexes involving the title compound may depend on structural features, its X-ray analysis was undertaken.

**Experimental.** Pale-yellow prismatic crystal 0.3 × 0.5 × 0.6 mm. Cell parameters from the angular values of 27 reflections automatically centred on a Siemens AED single-crystal diffractometer, Ni-filtered Cu K $\alpha$  radiation. Profile analysis following a modified version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) procedure. One check reflection every 50 measurements, max. deviation only 2.8%.  $3 < \theta < 70^\circ$ . Corrections applied for Lorentz-polarization effects and for absorption (Walker & Stuart, 1983). 3622 reflections collected, 1803 observed [ $I > 2\sigma(I)$ ], 1707 unique,  $R_{\text{int}} = 0.017$ .  $-19 \leq h \leq 19$ ,  $0 \leq k \leq 11$ ,  $0 \leq l \leq 14$ . Direct methods with *MULTAN* (Germain, Main & Woolfson, 1974). During refinement

(*SHELX76*, Sheldrick, 1976) two positions [separated by 1.01 (10) Å] compatible with a cyclohexane skeleton were identified for C(5); it was impossible to find separated positions for C(4) and C(6); furthermore, these atoms had relatively higher  $U_{22}$  thermal parameters; the occupancy values, initially both 0.5, were allowed to refine and converged to 0.30 and 0.70 for C(5) and C(5') respectively; in a difference synthesis calculated at this stage the highest peaks ( $\sim 0.25 \text{ e \AA}^{-3}$ ) around the disordered atoms were in appropriate positions for H atoms for both configurations. In the last block-matrix least-squares refinement nine reflections, with large  $\Delta F$  values, probably affected by extinction were omitted. Final  $R = 0.041$ ,  $R_w = 0.051$ ; \*  $w(\Delta F)^2$  minimized,  $w = 0.55/(\sigma^2 F + 0.0287 F^2)$ ; 122 and 227 parameters refined. For undisordered heavy atoms all  $\Delta/\sigma < 0.74$ . Max. peak in final  $\Delta F$  map  $0.14 \text{ e \AA}^{-3}$ . Scattering factors of *SHELX76*. All calculations on CDC CYBER 76 of CINECA and on GOULD SEL 32/77 computers.

**Discussion.** Atomic parameters are in Table 1, bond distances and angles in Table 2. A view of the molecule is shown in Fig. 1.

Bond distances and angles, except those of the cyclohexane ring, are comparable with those already found in similar molecular fragments. The lengths of the N—C bonds [N(1)—C(16) 1.385 (4), N(3)—C(7) 1.315 (3), N(3)—C(11) 1.356 (3) Å] are all relatively shorter than the value usually reported for N—C single bonds, while the N(2)—C(1) distance [1.278 (4) Å] is near to the value accepted for a delocalized double bond (Schenetti *et al.*, 1980; Donohue, Lavine & Rollett, 1956). The C—C bond distances of the phenyl ring average 1.383 (5) Å and the average internal C—C—C angle is  $119.8 (3)^\circ$ . Three of the distances involving the disordered C(5) atom in the ring [C(5)—C(4) 1.443 (30), C(5)—C(6) 1.484 (14), C(5')—C(4) 1.443 (13) Å] show a significant shortening with respect to the theoretical value reported by Sutton (1965) [1.537 (5) Å].

The cyclohexane ring shows two different conformations: the ring-puckering parameters calculated following Cremer & Pople (1975) are  $Q = 0.67 (1) \text{ \AA}$ ,  $\theta = 81.6 (13)^\circ$ ,  $\phi = 57.1 (3)^\circ$  for the ring with C(5) and  $Q = 0.53 (1) \text{ \AA}$ ,  $\theta = 9.4 (9)^\circ$ ,  $\phi = 40.5 (32)^\circ$  for that with C(5'). These values correspond to a conformation which is midway between boat and twist-boat in the first case and quite near to a chair in the second. The significant degree of flattening of this second ring relative to an ideal chair, where the valency angles

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a detailed analysis of the planarity have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39559 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

would be tetrahedral and torsion angles  $60^\circ$ , is clearly illustrated by the angles at C(4) and C(5) [ $115.6(6)$ ,  $114.4(7)^\circ$ ] and by the torsion angles (see Table 2).

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and  $U_{eq}$  values

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
N(1)	4407 (1)	1339 (3)	6299 (2)	4.3
N(2)	4825 (1)	1880 (2)	5489 (2)	3.6
N(3)	5284 (1)	1878 (2)	2871 (2)	3.7
C(1)	5613 (2)	1607 (3)	5645 (2)	3.7
C(2)	6095 (2)	2192 (3)	4793 (2)	3.9
C(3)	6782 (2)	3226 (5)	5425 (3)	5.7
C(4)	7331 (3)	2454 (6)	6454 (4)	6.8
C(5)	7063 (7)	989 (34)	6693 (28)	4.4
C(5')	6889 (7)	1738 (15)	7225 (8)	5.5
C(6)	6148 (2)	774 (5)	6625 (3)	5.2
C(7)	5544 (2)	2829 (3)	3711 (2)	3.8
C(8)	5334 (2)	4343 (4)	3596 (3)	5.0
C(9)	4879 (2)	4856 (4)	2578 (3)	5.2
C(10)	4610 (2)	3873 (3)	1654 (2)	4.9
C(11)	4821 (2)	2378 (3)	1852 (2)	3.8
C(12)	4556 (2)	1354 (4)	949 (3)	4.9
C(13)	4116 (2)	1815 (5)	-88 (3)	6.2
C(14)	3911 (2)	3309 (5)	-295 (3)	6.9
C(15)	4153 (2)	4317 (4)	544 (3)	6.0
C(16)	3560 (2)	1673 (3)	6167 (2)	3.9
C(17)	3113 (2)	981 (4)	6901 (3)	5.0
C(18)	2286 (2)	1392 (6)	6867 (4)	6.6
C(19)	1899 (3)	2447 (5)	6113 (5)	7.0
C(20)	2326 (2)	3078 (4)	5366 (4)	6.2
C(21)	3155 (2)	2708 (4)	5380 (3)	4.8

Table 2. Bond distances ( $\text{\AA}$ ), bond angles ( $^\circ$ ) and selected torsion angles ( $^\circ$ )

N(1)-N(2)	1.379 (3)	C(4)-C(5)	1.443 (30)	C(12)-C(13)	1.348 (5)
N(1)-C(16)	1.385 (4)	C(4)-C(5')	1.433 (13)	C(13)-C(14)	1.403 (6)
N(2)-C(1)	1.278 (4)	C(5)-C(6)	1.484 (14)	C(14)-C(15)	1.345 (5)
N(3)-C(7)	1.315 (3)	C(5')-C(6)	1.532 (1)	C(16)-C(17)	1.396 (5)
N(3)-C(11)	1.356 (3)	C(7)-C(8)	1.413 (5)	C(16)-C(21)	1.385 (4)
C(1)-C(2)	1.501 (4)	C(8)-C(9)	1.354 (5)	C(17)-C(18)	1.387 (5)
C(1)-C(6)	1.496 (4)	C(9)-C(10)	1.406 (4)	C(18)-C(19)	1.366 (7)
C(2)-C(3)	1.525 (5)	C(10)-C(11)	1.404 (4)	C(19)-C(20)	1.362 (7)
C(2)-C(7)	1.511 (3)	C(10)-C(15)	1.422 (4)	C(20)-C(21)	1.385 (5)
C(3)-C(4)	1.518 (6)	C(11)-C(12)	1.411 (4)		
N(2)-N(1)-C(16)	118.0 (2)	C(8)-C(9)-C(10)	119.7 (3)		
N(1)-C(2)-C(1)	117.3 (2)	C(9)-C(10)-C(15)	126.3 (3)		
C(7)-N(3)-C(11)	118.8 (2)	C(11)-C(10)-C(15)	119.2 (3)		
N(2)-C(1)-C(6)	127.4 (2)	N(3)-C(11)-C(10)	122.7 (2)		
N(2)-C(1)-C(2)	119.1 (2)	C(10)-C(11)-C(12)	118.9 (3)		
C(2)-C(1)-C(6)	113.6 (3)	N(3)-C(11)-C(12)	118.4 (3)		
C(1)-C(2)-C(7)	113.9 (3)	C(11)-C(12)-C(13)	120.2 (3)		
C(1)-C(2)-C(3)	108.6 (2)	C(12)-C(13)-C(14)	121.2 (3)		
C(3)-C(2)-C(7)	114.8 (3)	C(13)-C(14)-C(15)	120.4 (4)		
C(2)-C(3)-C(4)	110.7 (3)	C(10)-C(15)-C(14)	120.2 (3)		
C(3)-C(4)-C(5)	115.6 (6)	N(1)-C(16)-C(21)	122.7 (3)		
C(3)-C(4)-C(5')	115.5 (8)	N(1)-C(16)-C(17)	118.0 (3)		
C(4)-C(5)-C(6)	116.8 (20)	C(17)-C(16)-C(21)	119.2 (3)		
C(4)-C(5)-C(6')	114.4 (7)	C(16)-C(17)-C(18)	119.5 (3)		
C(1)-C(6)-C(5)	112.6 (9)	C(17)-C(18)-C(19)	120.9 (4)		
C(1)-C(6)-C(5')	109.9 (6)	C(18)-C(19)-C(20)	119.4 (4)		
N(3)-C(7)-C(2)	115.3 (2)	C(19)-C(20)-C(21)	121.4 (4)		
C(2)-C(7)-C(8)	122.6 (2)	C(16)-C(21)-C(20)	119.5 (3)		
N(3)-C(7)-C(8)	122.1 (2)				
C(7)-C(8)-C(9)	119.7 (3)				
C(1)-C(2)-C(3)-C(4)	54.5 (4)	C(4)-C(5)-C(6)-C(1)	39.1 (21)		
C(2)-C(3)-C(4)-C(5')	-50.9 (7)	C(4)-C(5)-C(6)-C(1)	-47.7 (9)		
C(2)-C(3)-C(4)-C(5)	-5.2 (13)	C(5)-C(6)-C(1)-C(2)	54.8 (6)		
C(3)-C(4)-C(5)-C(6)	47.4 (10)	C(2)-C(1)-C(6)-C(5)	13.0 (12)		
C(3)-C(4)-C(5)-C(6')	-43.3 (22)	C(6)-C(1)-C(2)-C(3)	-59.5 (3)		

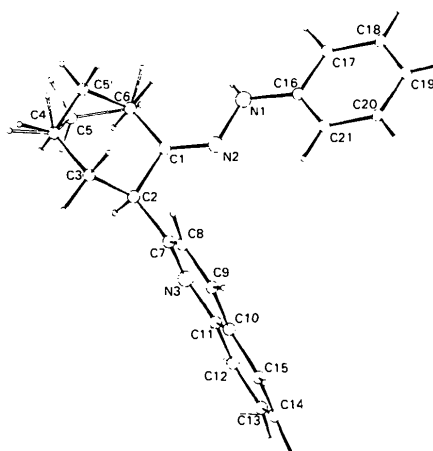


Fig. 1. Projection of the molecule with the atomic numbering scheme.

The quinoline group is not planar, the dihedral angle formed by the mean pyridine ring and the benzene planes being  $1.7(1)^\circ$ . This slight bend around the C(10)-C(11) bond has always been observed in this type of compound (Skrzypczak-Jankun & Kałuski, 1977; Skrzypczak-Jankun, Hoser, Grezesiak, Kałuski & Rozwadowska, 1979). The plane of the quinolyl group and the plane of the cyclohexane rings subtend angles of  $62.4(1)$  and  $62.2(1)^\circ$  while these rings are inclined at  $41.7(1)$  and  $42.6(1)^\circ$  with respect to the aromatic ring.

There are no significant intermolecular contacts shorter than those of van der Waals.

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*Acta Cryst.* (1984). C40, 1955–1956

## Structure of 8-Dimethylsulphido-7,9-dicarba-*nido*-undecaborane(11),\* C<sub>4</sub>H<sub>17</sub>B<sub>9</sub>S

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**Abstract.**  $M_r = 194.6$ ,  $P2_1/c$ ,  $a = 10.043$  (4),  $b = 8.936$  (4),  $c = 12.946$  (6) Å,  $\beta = 94.48$  (3)°,  $V = 1158.3$  (9) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.116$  Mg m<sup>-3</sup>,  $Cu K\alpha$ ,  $\lambda = 1.54178$  Å,  $\mu = 1.94$  mm<sup>-1</sup>,  $F(000) = 408$ , room temperature,  $R = 0.071$  for 1368 observed independent reflections. The S(CH<sub>3</sub>)<sub>2</sub> group, which has normal bond lengths and angles, is bonded to the B atom located between two C atoms in an open face of the C<sub>2</sub>B<sub>9</sub> *nido*-carbaborane cage. Centroids of the cages have twelve nearest neighbours and simulate a distorted cubic close packing.

**Introduction.** X-ray investigation of the title compound, which forms part of our continuing studies on structures of the thio derivatives of the carbaboranes, has been undertaken in order to clarify the position of the S(CH<sub>3</sub>)<sub>2</sub> group on the 7,9-C<sub>2</sub>B<sub>9</sub> *nido* skeleton. Crystals were prepared in the Institute of Inorganic Chemistry, Czechoslovak Academy of Sciences (Plešek, Janoušek & Heřmánek, 1978).

**Experimental.** Transparent colourless crystal 0.35 × 0.30 × 0.40 mm, Syntex  $P2_1$  diffractometer,  $Cu K\alpha$  radiation, graphite monochromator. Cell parameters and standard deviations by least squares from 23 high-order reflections. 1574 independent reflections measured by  $\omega/2\theta$  scans to  $2\theta = 115^\circ$  (for  $h 0 \rightarrow 10$ ,  $k 0 \rightarrow 9$ ,  $l -14 \rightarrow +14$ ). 1368 reflections with  $I > 1.96\sigma(I)$  regarded as observed [ $\sigma(I)$  from counting statistics]. Intensities of three standards measured every 25 reflections showed insignificant changes. Data scaled according to standard reflections and corrected for Lorentz and polarization effects, but not for absorption

or extinction. Structure solved by direct methods with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974), all non-H atoms located on *E* map. C cage atoms distinguished from B atoms by their lower temperature factors and shorter bond distances. Refinement with local version of full-matrix least-squares program *ORFLS* (Busing, Martin & Levy, 1962), anisotropic temperature factors for non-H atoms. H atoms from difference Fourier synthesis, refined with individual isotropic temperature factors. Function minimized  $\sum w(|F_o| - |F_c|)^2$  with  $w^{-1} = \sigma^2(F_o) + (0.015F_o)^2$ . In final cycle  $R = 0.071$ ,  $R_w = 0.088$ ,  $av. \Delta/\sigma = 0.07$ ,  $max. = 0.47$  for H atom in methyl group. Max. and min. heights in final  $\Delta\rho$  map +0.36 and -0.60 e Å<sup>-3</sup>. Scattering factors from *International Tables for X-ray Crystallography* (1974) except for H (Stewart, Davidson & Simpson, 1965). Calculations performed on IBM 370/135 and Siemens 7536 computers.

**Discussion.** The positional parameters are in Table 1, † bond lengths and selected angles in Table 2. The molecular structure is shown in Fig. 1. The orientation of the S(CH<sub>3</sub>)<sub>2</sub> group [bonded to B(8) in an open face of the *nido*-carbaborane cage] is defined by the torsion angles given in Table 2. The bond distances B–S = 1.886 (4), average S–C = 1.790 (6) Å and B–S–C and C–S–C angles are in good agreement with those found for other S(CH<sub>3</sub>)<sub>2</sub> groups in related borane

† Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and a unit-cell packing diagram have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39584 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

\* 8-Dimethylsulphonio-7,9-dicarba-*nido*-undecaborate(1-).