

Fig. 2. Stereoscopic view [PLUTO 78 (Motherwell, 1978), modified by Drück, 1982] of the unit cell of HCB in the direction [111].

Fig. 2 shows a stereoscopic view of the packing in the unit cell. The shortest intermolecular contacts are 3.148 (3) Å between a C atom of the ring and an N atom of the neighboring molecule and 3.230 (3) Å between the N and the C atom of a cyano group. With respect to the sum of the van der Waals radii (3.47 Å; Bondi, 1964) a considerable shortening of the mentioned intermolecular contacts is observed in the crystal structure. The cavity in the center of the unit cell, which can easily accommodate a small molecule, can be clearly observed.

The planarity of the whole molecule in the crystal structure is well defined by the molecular point symmetry  $\bar{3}$  together with the calculated angles [90.1 (1), 90.1 (1) and 90.0 (1)°] between the body diagonal [111] and the lines from the molecule center to the atoms C(1), C(11) and N(1).<sup>\*</sup> A remarkable fact is, that each cyano group is pointing almost linearly towards the center of a neighboring molecule, the corresponding distance [N(1)—center] and angle [C(11)—

<sup>\*</sup> The positional deviations for the mentioned atoms from the best least-squares plane calculated including the six ring atoms are 0.002 Å.

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### 3,4-Dihydro-2-diphenylmethyl-4-diphenylmethylene-6-methyl-3-(*p*-tolyl)quinazoline, C<sub>42</sub>H<sub>34</sub>N<sub>2</sub>

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**Abstract.**  $M_r = 566.74$ , triclinic,  $P\bar{1}$ ,  $a = 12.998$  (6),  $b = 13.191$  (4),  $c = 21.864$  (3) Å,  $\alpha = 97.53$  (3),  $\beta = 90.74$  (3),  $\gamma = 118.44$  (5)°,  $V = 3255.8$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.15$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 0.072$  mm<sup>-1</sup>,  $T = 293$  K,  $R = 0.093$  for 3668 reflexions

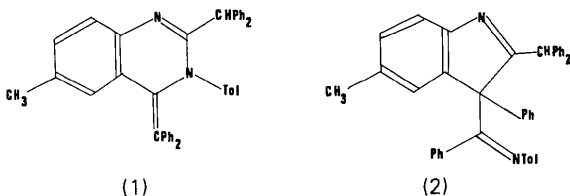
[ $F > 3\sigma(F)$ ]. The asymmetric unit consists of two molecules of the title compound related by an approximate non-crystallographic twofold axis. Bulky substituents prevent the quinazoline skeleton achieving planarity.

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**Introduction.** In the course of an investigation into the chemistry of triarylketenimines (Flowers & Halls, 1982), it was found that addition of trifluoromethanesulphonic acid readily caused the dimerization of diphenyl-*N*-tolylketenimine (Flowers & Halls, 1981). The mass, IR and NMR spectra did not lead to unambiguous determination of the structure of the dimer, and allowed, *inter alia*, both the quinazoline (1), analogous to the product of thermal dimerization of dimethyl-*N*-phenylketenimine (Barker & Rosamund, 1974), and the indolenine (2) structures; the  $^1\text{H}$  NMR chemical shifts of the aryl and methyl protons indicated considerable steric crowding, and hence the present study was undertaken.



**Experimental.** Yellow crystal,  $0.5 \times 0.4 \times 0.4$  mm, CAD-4 diffractometer, unit-cell dimensions from 25 reflexions, 6175 reflexions measured,  $-11 \leq h \leq 11$ ,  $-13 \leq k \leq 13$ ,  $0 \leq l \leq 22$ ,  $\theta < 25^\circ$ , 3668 unique structure amplitudes with  $F > 3\sigma(F)$ , Lorentz and polarization but no absorption correction; *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) used to solve the phase problem, all non-hydrogen atoms found in Fourier map; asymmetric unit consists of two molecules (*A* and *B*) of the title compound (1), related by an approximate non-crystallographic twofold axis perpendicular to the *ac* plane; parameters of *A* and *B* refined separately by blocked full-matrix least squares based on *F* with *SHELX* (Sheldrick, 1976), final  $R = 0.093$  [ $R_w = 0.079$ ,  $w = 6.58/\sigma^2(F)$ ], isotropic thermal parameters. H atoms were constrained to chemically reasonable positions except for the lone H atom attached to the diphenylmethyl group and those attached to the central skeleton which were also refined. The hydrogen temperature factors were tied to those of the bonding atoms, except for those of the phenyl substituents, which were refined together. Fluctuations in the final difference map  $\leq \pm 0.5 \text{ e } \text{\AA}^{-3}$ , scattering factors from *International Tables for X-ray Crystallography* (1974). Computations carried out on the joint CDC 7600/ICL 1906A system of the University of Manchester Regional Computing Centre. The Cambridge Crystallographic Database was surveyed using the Crystal Structure Search and Retrieval (1980) interactive system accessed *via* the UMIST link to the DEC10 at Edinburgh.

**Discussion.** Final atomic parameters are given in Table 1.\* Fig. 1 is a labelled stereoscopic view of the heavy-atom skeleton of molecule *B* and Fig. 2 shows the molecular packing.

\* Lists of structure factors, constrained H-atom coordinates, intramolecular non-bonded distances and mean-plane calculations and a figure showing torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38360 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

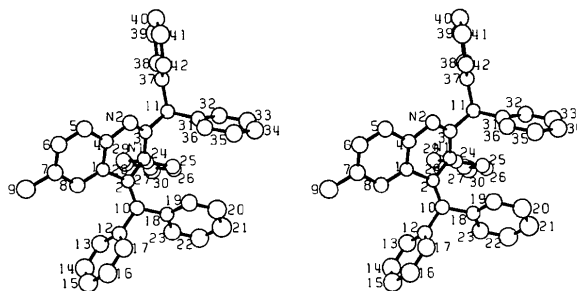


Fig. 1. Stereoscopic view of non-hydrogen atoms in molecule *B* with atoms depicted as 50% probability boundary ellipsoids.

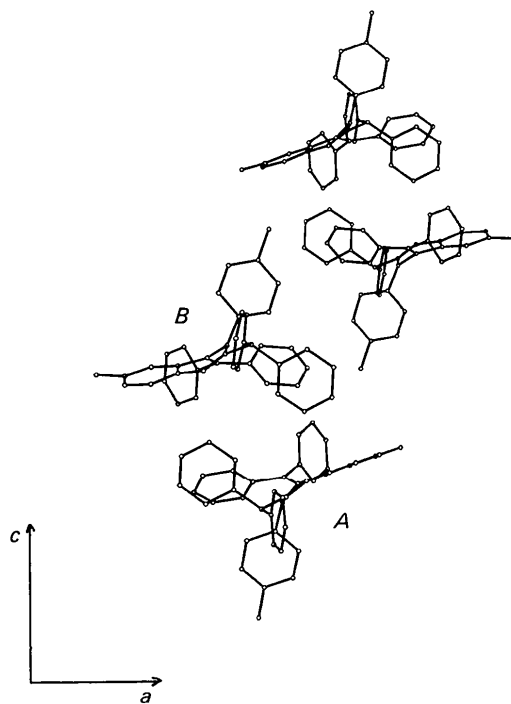


Fig. 2. View along  $b^*$  showing packing of molecule.

Table 1. Fractional atomic coordinates and isotropic thermal parameters for independently refined atoms (e.s.d.'s in parentheses)

Molecule A	x	y	z	U (Å <sup>2</sup> )
C(1)	0.3850 (7)	0.7695 (7)	0.3619 (3)	0.038 (2)
C(2)	0.2680 (7)	0.7292 (7)	0.3844 (3)	0.037 (2)
N(1)	0.2579 (5)	0.8240 (6)	0.4213 (3)	0.037 (2)
C(3)	0.3209 (7)	0.9359 (7)	0.4067 (3)	0.037 (2)
N(2)	0.4062 (6)	0.9675 (6)	0.3711 (3)	0.046 (2)
C(4)	0.4490 (7)	0.8873 (8)	0.3552 (3)	0.044 (2)
C(5)	0.5594 (8)	0.9311 (9)	0.3325 (4)	0.056 (3)
H(5)	0.5968 (62)	1.0112 (66)	0.3236 (29)	0.055
C(6)	0.6096 (9)	0.8593 (9)	0.3176 (4)	0.058 (3)
H(6)	0.6887 (65)	0.8908 (62)	0.2973 (30)	0.061
C(7)	0.5467 (8)	0.7416 (8)	0.3236 (4)	0.059 (3)
C(8)	0.4346 (8)	0.6950 (8)	0.3447 (4)	0.053 (3)
H(8)	0.3971 (61)	0.6166 (65)	0.3578 (30)	0.053
C(9)	0.6002 (9)	0.6619 (9)	0.3069 (5)	0.087 (3)
C(10)	0.1720 (7)	0.6235 (7)	0.3732 (3)	0.041 (2)
C(11)	0.2759 (7)	1.0201 (8)	0.4294 (4)	0.046 (2)
H(11)	0.2644 (55)	1.0111 (55)	0.4689 (30)	0.043
C(12)	0.1761 (7)	0.5142 (8)	0.3473 (4)	0.047 (2)
C(13)	0.2015 (8)	0.4532 (9)	0.3866 (4)	0.069 (3)
C(14)	0.2041 (9)	0.3481 (10)	0.3613 (5)	0.085 (3)
C(15)	0.1802 (9)	0.3134 (10)	0.2996 (5)	0.090 (3)
C(16)	0.1557 (9)	0.3701 (10)	0.2604 (5)	0.088 (3)
C(17)	0.1518 (8)	0.4734 (9)	0.2844 (4)	0.072 (3)
C(18)	0.0510 (7)	0.6063 (7)	0.3850 (4)	0.045 (2)
C(19)	0.0110 (8)	0.6703 (8)	0.3583 (4)	0.055 (3)
C(20)	-0.1007 (9)	0.6594 (9)	0.3695 (4)	0.072 (3)
C(21)	-0.1674 (9)	0.5827 (9)	0.4081 (4)	0.079 (3)
C(22)	-0.1311 (9)	0.5140 (9)	0.4328 (4)	0.073 (3)
C(23)	-0.0200 (7)	0.5244 (8)	0.4224 (4)	0.055 (3)
C(24)	0.2287 (7)	0.8081 (7)	0.4845 (4)	0.042 (2)
C(25)	0.1239 (8)	0.7983 (8)	0.5053 (4)	0.053 (2)
C(26)	0.1019 (8)	0.7843 (8)	0.5665 (4)	0.061 (3)
C(27)	0.1811 (8)	0.7789 (8)	0.6059 (4)	0.061 (3)
C(28)	0.2851 (8)	0.7892 (8)	0.5851 (4)	0.063 (3)
C(29)	0.3114 (8)	0.8073 (8)	0.5256 (4)	0.054 (3)
C(30)	0.1561 (9)	0.7629 (10)	0.6729 (4)	0.093 (4)
C(31)	0.1659 (7)	0.9934 (7)	0.3903 (4)	0.045 (2)
C(32)	0.0768 (8)	1.0087 (8)	0.4194 (4)	0.061 (3)
C(33)	-0.0222 (9)	0.9883 (9)	0.3811 (5)	0.080 (3)
C(34)	-0.0337 (9)	0.9558 (9)	0.3198 (5)	0.084 (3)
C(35)	0.0537 (8)	0.9402 (9)	0.2905 (4)	0.073 (3)
C(36)	0.1535 (8)	0.9599 (8)	0.3272 (4)	0.057 (3)
C(37)	0.3682 (7)	1.1494 (7)	0.4372 (4)	0.046 (2)
C(38)	0.4084 (8)	1.2120 (9)	0.4961 (4)	0.067 (3)
C(39)	0.4893 (10)	1.3354 (10)	0.5039 (5)	0.088 (3)
C(40)	0.5244 (9)	1.3860 (10)	0.4537 (5)	0.080 (3)
C(41)	0.4882 (9)	1.3296 (10)	0.3954 (5)	0.085 (3)
C(42)	0.4038 (8)	1.2060 (9)	0.3873 (4)	0.063 (3)

## Molecule B

C(1)	-0.1018 (7)	0.8529 (7)	0.1328 (3)	0.035 (2)
C(2)	-0.0262 (7)	0.7989 (7)	0.1167 (3)	0.033 (2)
N(1)	0.0780 (5)	0.8762 (6)	0.0899 (3)	0.036 (2)
C(3)	0.1288 (7)	0.9965 (7)	0.1066 (3)	0.036 (2)
N(2)	0.0790 (6)	1.0472 (6)	0.1402 (3)	0.044 (2)
C(4)	-0.0422 (7)	0.9742 (7)	0.1472 (3)	0.038 (2)
C(5)	-0.1060 (8)	1.0324 (8)	0.1693 (4)	0.050 (3)
H(5)	-0.0565 (60)	1.1125 (65)	0.1842 (28)	0.050
C(6)	-0.2255 (8)	0.9692 (8)	0.1724 (4)	0.053 (2)
H(6)	-0.2706 (57)	1.0257 (59)	0.1936 (28)	0.052
C(7)	-0.2866 (8)	0.8492 (8)	0.1553 (4)	0.052 (2)
C(8)	-0.2246 (8)	0.7898 (8)	0.1361 (4)	0.047 (2)
H(8)	-0.2619 (58)	0.7053 (64)	0.1323 (27)	0.046
C(9)	-0.4207 (8)	0.7788 (8)	0.1533 (4)	0.073 (3)
C(10)	-0.0398 (7)	0.6961 (7)	0.1279 (3)	0.040 (2)
C(11)	0.2512 (7)	1.0694 (8)	0.0861 (4)	0.040 (2)
H(11)	0.2428 (53)	1.0323 (57)	0.0451 (29)	0.038
C(12)	-0.1544 (7)	0.5981 (7)	0.1439 (4)	0.045 (2)
C(13)	-0.2407 (8)	0.5228 (8)	0.0963 (4)	0.064 (3)
C(14)	-0.3452 (9)	0.4305 (9)	0.1122 (5)	0.072 (3)
C(15)	-0.3585 (9)	0.4168 (9)	0.1712 (4)	0.072 (3)
C(16)	-0.2763 (9)	0.4842 (9)	0.2191 (5)	0.074 (3)
C(17)	-0.1673 (8)	0.5809 (8)	0.2039 (4)	0.065 (3)
C(18)	0.0637 (7)	0.6732 (7)	0.1293 (3)	0.041 (2)
C(19)	0.1599 (8)	0.7461 (8)	0.1703 (4)	0.056 (3)
C(20)	0.2574 (9)	0.7250 (9)	0.1749 (4)	0.073 (3)
C(21)	0.2481 (9)	0.6283 (10)	0.1378 (4)	0.081 (3)
C(22)	0.1558 (9)	0.5552 (10)	0.0970 (4)	0.076 (3)
C(23)	0.0574 (8)	0.5755 (8)	0.0923 (4)	0.059 (3)
C(24)	0.0942 (7)	0.8280 (7)	0.0292 (3)	0.041 (2)

Table 1 (cont.)

	x	y	z	U(Å <sup>2</sup> )
C(25)	0.1889 (7)	0.8071 (7)	0.0196 (4)	0.046 (2)
C(26)	0.1977 (8)	0.7617 (8)	-0.0404 (4)	0.050 (2)
C(27)	0.1164 (8)	0.7372 (8)	-0.0888 (4)	0.054 (3)
C(28)	0.0228 (8)	0.7585 (8)	-0.0775 (4)	0.059 (3)
C(29)	0.0119 (7)	0.8040 (7)	-0.0184 (4)	0.050 (2)
C(30)	0.1309 (8)	0.6910 (9)	-0.1535 (4)	0.068 (3)
C(31)	0.3473 (7)	1.0662 (7)	0.1261 (4)	0.039 (2)
C(32)	0.4419 (7)	1.0653 (8)	0.0954 (4)	0.054 (3)
C(33)	0.5330 (9)	1.0663 (8)	0.1314 (4)	0.066 (3)
C(34)	0.5290 (8)	1.0644 (8)	0.1940 (4)	0.061 (3)
C(35)	0.4352 (8)	1.0642 (8)	0.2230 (4)	0.059 (3)
C(36)	0.3445 (7)	1.0653 (7)	0.1876 (4)	0.050 (2)
C(37)	0.2852 (7)	1.1969 (7)	0.0815 (4)	0.042 (2)
C(38)	0.3078 (7)	1.2321 (8)	0.0247 (4)	0.057 (3)
C(39)	0.3484 (8)	1.2350 (9)	0.0200 (4)	0.068 (3)
C(40)	0.3663 (8)	1.4289 (9)	0.0719 (4)	0.063 (3)
C(41)	0.3445 (8)	1.3956 (8)	0.1282 (4)	0.065 (3)
C(42)	0.3036 (7)	1.2771 (8)	0.1339 (4)	0.053 (2)

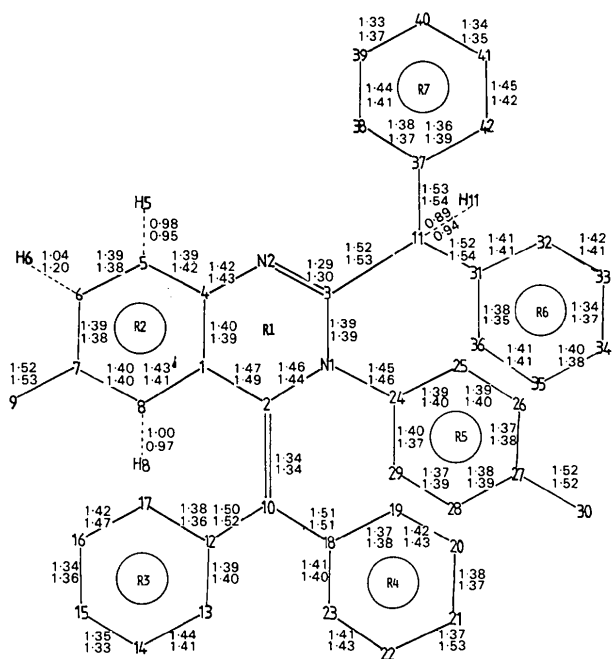


Fig. 3. Bond lengths (Å) for molecules A (upper) and B (lower) with average e.s.d. 0.015 Å.

As the final dimensions of molecules A and B are generally identical, most discrepancies being covered by the e.s.d.'s, and as the most significant variation is a greater degree of twisting ( $\sim 10^\circ$ ) along bond N(1)–C(3) in A compared with B, the following discussion does not differentiate between the two molecules, any parameters being presented in the form A,B;A,B;....

The bond lengths and angles for essentially planar R2 (see Figs. 3 and 4) are appropriate for a completely delocalized ring; however, bulky substituents attached to C(2), N(1) and C(3) have distorted R1 into a boat conformation, flattened at N(2) owing to the double bond. Even though the puckering of R1 has projected

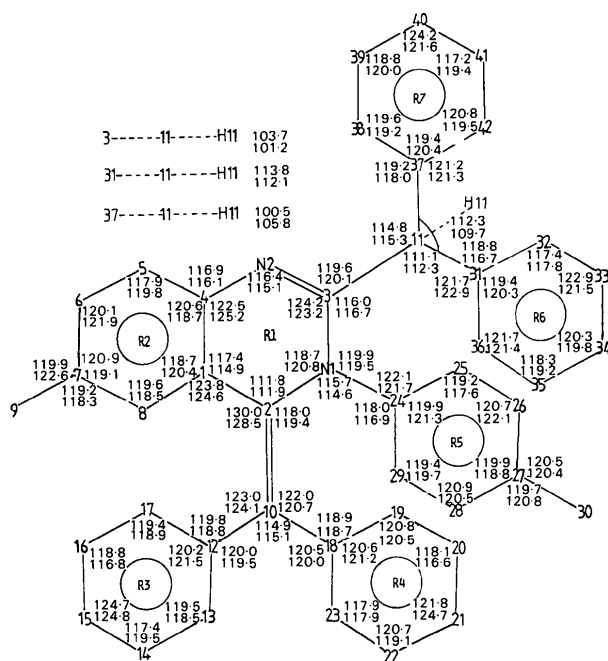


Fig. 4. Bond angles ( $^{\circ}$ ) for molecules *A* (upper) and *B* (lower) with average e.s.d.  $1.0^{\circ}$ .

C(24) away from C(10) and C(11) and the substituents have, where possible, attained a staggered conformation, several relatively short intramolecular non-bonded distances still persist. There does not appear to be any intramolecular non-bonded  $\pi$  interaction except, possibly, between R4 and R6, which overlap and approach fairly closely [C(31)–C(19) 3.70 (4), 4.00 (5) Å], but as the rings are far from parallel [61 (2), 56 (2) $^{\circ}$ ] this seems unlikely. R1 bond lengths indicate little delocalization and apart from C(1)–C(4), which is common to R2, the most significant deviation from integral bonding is N(1)–C(3) [1.39 (1), 1.39 (1) Å]. These bond lengths contrast with those in the planar quinazoline structure (Huiszoon, 1976), where C–N range from 1.31 to 1.37 Å and C–C are 1.40 and 1.41 Å. However, the difference is not so great where C(2) has achieved  $sp^2$  hybridization by

double bonding externally to the quinazoline frame, e.g. in tricycloquinazoline (Iball & Motherwell, 1969), where the quinazoline forms part of a larger planar system, the bonds vary from those of the title molecule by less than  $2\sigma$ . Incomplete delocalization is also seen in planar quinazolinone derivatives. In the case of 2-phenyl-4(3*H*)-quinazolinone (Holm, Christophersen, Ottersen, Hope & Christensen, 1977), C(2)–N(1) at 1.36 Å is the only significant difference from the title molecule [1.46 (1), 1.44 (1) Å]. However, in 2-phenyl-3-[*p*-(2,2,4-trimethylchroman-4-yl)phenyl]-4(3*H*)-quinazolinone (Gilmore, Hardy, MacNicol & Wilson, 1977), this bond has lengthened to 1.40 Å, possibly because of the substituent on N(1), but at 1.37 Å C(4)–N(2) is significantly reduced [title compound: 1.42 (1), 1.43 (1) Å]. The shortest heavy-atom intermolecular contacts are 3.46 (4), 3.39 (4) Å between C(28) and C(5) at  $1-x, 2-y, 1-z$  (molecule *A*) and  $-x, 2-y, -z$  (molecule *B*).

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### *cis*-1,4,9-Trimethylthioxanthene 10-Oxide, C<sub>16</sub>H<sub>16</sub>OS

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**Abstract.**  $M_r = 256.36$ , orthorhombic,  $P2_12_12_1$ ,  $a = 1307.1$  (6) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.303$  Mg m<sup>-3</sup>, graphite-monochromated Cu  $K\alpha$ ,  $\lambda = 1.5418$  Å,  $\mu =$