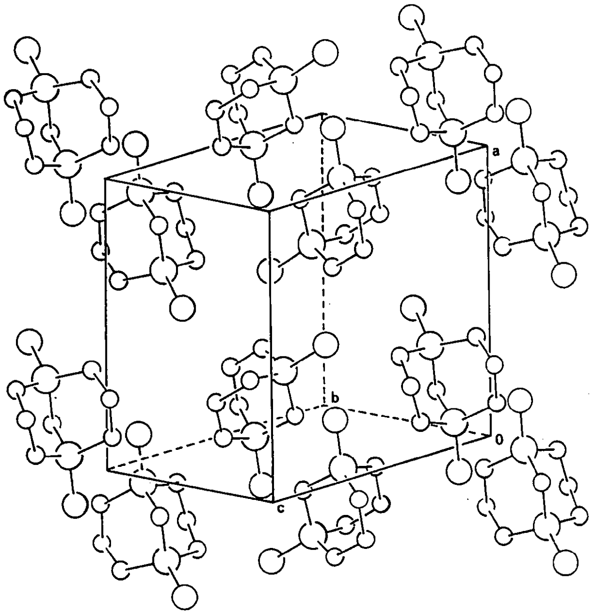

Fig. 2. Molecular packing of 1,5-diphosphabicyclo[3.3.1]nonane 1,5-disulfide.

those obtained for a series of carbabicyclo[3.3.1]-nonanes (*e.g.* Sim, 1983). Since a slight but significant lengthening of these distances has also been observed in 1-phosphabicyclo[3.3.1]nonane 1-sulfide (Baumeister, Hartung & Krech, 1988), the P atoms in the bridge-head positions of the heterobicyclic system seem to be responsible for it.

The coordination around the P atoms is that of a slightly distorted tetrahedron. The C—P—S bond angles are all increased from tetrahedral to 111–114° [mean 113·1 (10)°] whereas most of the C—P—C angles are decreased [mean 105·6 (32)°]. The excellent agreement of the observed bond lengths with the corresponding values in 1-phosphabicyclo[3.3.1]nonane 1-sulfide [given in square brackets] is illustrated by the following data: $\overline{\mathrm{P{-}C}}$ = 1·813 (6) [1·807 (7)], $\overline{\mathrm{P{-}S}}$ = 1·962 (5) [P—S = 1·958 (1)], $\overline{\mathrm{C{-}C}}$ = 1·531 (9) Å [1·530 (5) Å]. The van der Waals radius for P is 1·80 Å (Bondi, 1964) so the nonbonded P(1)···P(2) distance of 2·983 (1) Å is remarkably short. In 1,5-diphosphabicyclo[3.3.0]octane 1,5-disulfide the directly bonded P atoms are separated by 2·200 (5) Å (Hartung, Hickel, Kaiser & Richter, 1979). The C—H distances are in the range 0·85 (3)–1·09 (4) Å [mean 0·95 (6) Å].

The crystal structure (Fig. 2) consists of discrete molecules with all intermolecular contacts between non-H atoms greater than the sums of the corresponding van der Waals radii. Two S···H contacts are slightly shorter than 3·0 Å.

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Structure of 1,3-Diphenyl-4,5-dihydro-1*H*-1,2,4-triazin-6-one

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Abstract. $C_{15}H_{13}N_3O$, $M_r = 251\cdot29$, orthorhombic, $P2_12_12_1$, $a = 11\cdot511\ (3)$, $b = 17\cdot96\ (1)$, $c = 6\cdot118\ (1)$ Å, $V = 1264\cdot8\ (1\cdot2)$ Å^3, $Z = 4$, $D_x = 1\cdot32$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0\cdot93$ cm^{-1}, $F(000) = 528$, $T = 293$ K, final $R(F) = 0\cdot032$ for 960 observed reflections. The six-membered triazine ring is not planar. The phenyl ring in position 3 is nearly coplanar with the mean plane of the heterocycle; the

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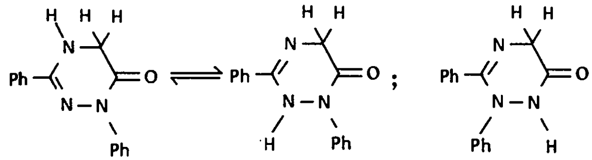
other phenyl ring is twisted 57° out of that plane. Weak N—H···O intermolecular hydrogen bonds of 3·004 (4) Å contribute towards the crystal packing.

Introduction. The reaction of phenylhydrazine with *N*-(ethoxycarbonylmethyl) benzimidate could lead to the formation of imidazole or to derivatives of 1,2,4-triazine. Elemental analysis, IR and ^{1}H NMR spectroscopy confirm the second hypothesis (Mathis, Mathis, Chihaoui & Baccar, 1982; Hajjem, 1984) but two stereoisomers, 1,3-diphenyl-4,5-dihydro-1*H*-1,2,4-triazin-6-one (I) with its tautomer (I′) and 2,3-diphenyl-1,2-dihydro-5*H*-1,2,4-triazin-6-one (II), may be obtained as illustrated schematically below.



(I) (I′) (II)

X-ray diffraction was carried out in order to determine the actual conformation and the crystal and molecular structure.

Experimental. Prismatic yellow crystals were obtained by evaporation of an ethanol solution. X-ray photographs taken with Mo *Kα* radiation showed the crystals to be orthorhombic. Systematic absences permitted the assignment of the space group to this compound. A crystal with dimensions 0·20 × 0·14 × 0·12 mm was mounted on a computer-controlled Philips PW 1100 single-crystal diffractometer, using graphite-monochromatized Mo *Kα* radiation. The cell dimensions and the orientation matrix were determined by least-squares calculations based on the setting angles of 25 reflections with 2θ angles ranging between 17·62 and 22·75°. The intensities were collected at room temperature up to $2\theta = 50°$ corresponding to $(\sin\theta/\lambda) = 0{\cdot}59\ \text{Å}^{-1}$ with $\omega/2\theta$ step scanning mode. The scan range was 1·4° and the speed 0·03° s^{-1}. The background was measured on either side of the reflection during 5 s. A total of 1503 *hkl* and $hk\bar{l}$ independent reflections (h 0→13, k 0→20, l −4→7) were measured. Some reflections (224) of the *hkl* octant were measured twice and averaged to give an agreement of 0·01. Then 1239 independent reflections were obtained of which 279 having $I < 3\sigma(I)$ were considered unobserved and excluded from the last cycle of refinement. Three standard reflections 242, $\bar{2}42$ and $2\bar{4}2$ were measured every 90 min and showed no apparent variation during data collection. The intensities were corrected for Lorentz–polarization but for neither absorption nor extinction.

The structure was solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined with *SHELX*76 (Sheldrick, 1976).

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{\text{eq}} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} \mathbf{a}_i . \mathbf{a}_j a_i^* a_j^*.$$

	x	y	z	B_{eq}(Å^2)
O	0·3796 (2)	0·2256 (1)	−0·1465 (4)	4·8 (1)
C(11)	0·6780 (2)	0·1322 (2)	0·4956 (5)	2·9 (1)
C(12)	0·7911 (3)	0·1570 (2)	0·5180 (6)	3·8 (2)
C(13)	0·8568 (3)	0·1339 (2)	0·6953 (6)	4·7 (2)
C(14)	0·8128 (3)	0·0869 (2)	0·8491 (8)	5·0 (2)
C(15)	0·6994 (3)	0·0606 (2)	0·8264 (7)	4·4 (2)
C(16)	0·6331 (3)	0·0843 (2)	0·6529 (6)	3·7 (2)
C(21)	0·6006 (2)	0·1577 (2)	0·3162 (5)	3·1 (1)
N(1)	0·5021 (2)	0·1241 (1)	0·2982 (4)	3·7 (1)
N(2)	0·4250 (2)	0·1520 (2)	0·1403 (5)	3·6 (1)
C(24)	0·4510 (3)	0·2035 (2)	−0·0109 (6)	3·3 (1)
C(25)	0·5723 (3)	0·2341 (2)	−0·0087 (6)	3·5 (1)
N(3)	0·6324 (3)	0·2162 (2)	0·1918 (5)	4·1 (1)
C(31)	0·3091 (2)	0·1242 (2)	0·1702 (6)	3·1 (1)
C(32)	0·2519 (3)	0·0862 (2)	0·0075 (7)	4·1 (2)
C(33)	0·1407 (3)	0·0595 (2)	0·0495 (8)	4·7 (2)
C(34)	0·0890 (3)	0·0706 (2)	0·2469 (8)	4·7 (2)
C(35)	0·8475 (3)	0·1083 (2)	0·4072 (7)	4·7 (2)
C(36)	0·2575 (3)	0·1358 (2)	0·3719 (6)	3·9 (2)

All H atoms were found in the difference Fourier map and refined with the same overall isotropic thermal parameter [$B = 4{\cdot}9\ (2)\ \text{Å}^2$]. The weighting scheme used was $w(F) = 1/[\sigma^2(F) + 0{\cdot}0049F^2]$. The $\Delta\rho_{\max}$ and the $\Delta\rho_{\min}$ in the last difference Fourier map were 0·16 and −0·28 e Å^{-3} respectively. $(\Delta/\sigma)_{\max} = 0{\cdot}03$. Final $R(F) = 0{\cdot}032$ and $wR = 0{\cdot}042$. $S = 0{\cdot}62$. Scattering factors, f' and f'' were taken from *International Tables for X-ray Crystallography* (1974). The calculations were carried out on a VAX 11/780 at the Khawarezmi Computer Center of Tunis.

Discussion. The atomic coordinates are listed in Table 1 and the bond lengths and angles in Table 2.* A projection of the molecule with thermal ellipsoids of vibration (50% probability) and the packing of the molecules in the unit cell are shown in Figs. 1 and 2 respectively.

The NMR spectra of the title compound dissolved in $CDCl_3$ show three signals which can be attributed unambiguously to the protons of the phenyl rings (multiplet centred at 7·5 p.p.m.). From the IR study it was shown that the heterocycle is a six-membered triazine (Mathis *et al.*, 1982) but the relative positions of the phenyls could not be deduced unambiguously. This X-ray diffraction study determined the structure of the title compound as that of tautomer (I) as shown in Fig. 1.

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* Lists of structure factors, H-atom coordinates, anisotropic thermal parameters and bond lengths and angles involving H atoms have been deposited with British Library Document Supply Centre as Supplementary Publication No. SUP 44922 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°)

C(11)–C(12)	1·382 (4)	C(24)–C(25)	1·501 (5)
C(12)–C(13)	1·386 (5)	C(25)–N(3)	1·445 (4)
C(13)–C(14)	1·362 (5)	N(3)–C(21)	1·348 (4)
C(14)–C(15)	1·394 (5)	N(2)–C(31)	1·436 (4)
C(15)–C(16)	1·374 (5)	C(31)–C(32)	1·376 (5)
C(16)–C(11)	1·390 (4)	C(32)–C(33)	1·390 (5)
C(11)–C(21)	1·486 (4)	C(33)–C(34)	1·361 (6)
C(21)–N(1)	1·289 (4)	C(34)–C(35)	1·370 (5)
N(1)–N(2)	1·405 (3)	C(35)–C(36)	1·376 (5)
N(2)–C(24)	1·342 (4)	C(36)–C(31)	1·385 (5)
C(24)–O	1·233 (3)		
C(11)–C(12)–C(13)	119·7 (4)	N(3)–C(21)–C(11)	119·6 (3)
C(12)–C(13)–C(14)	121·5 (4)	N(3)–C(21)–N(1)	123·7 (3)
C(13)–C(14)–C(15)	119·3 (4)	N(2)–C(24)–O	122·4 (3)
C(14)–C(15)–C(16)	119·4 (4)	O–C(24)–C(25)	120·6 (3)
C(15)–C(16)–C(11)	121·3 (3)	N(1)–N(2)–C(31)	112·1 (2)
C(16)–C(11)–C(12)	118·7 (3)	C(24)–N(2)–C(31)	122·3 (3)
C(16)–C(11)–C(21)	118·6 (3)	N(2)–C(31)–C(32)	121·7 (3)
C(12)–C(11)–C(21)	122·6 (3)	C(31)–C(32)–C(33)	118·6 (4)
C(11)–C(21)–N(1)	116·5 (3)	C(32)–C(33)–C(34)	121·1 (4)
C(21)–N(1)–N(2)	116·6 (2)	C(33)–C(34)–C(35)	119·5 (3)
N(1)–N(2)–C(24)	125·4 (2)	C(34)–C(35)–C(36)	121·1 (4)
N(2)–C(24)–C(25)	117·0 (3)	C(35)–C(36)–C(31)	118·7 (3)
C(24)–C(25)–N(3)	111·8 (3)	C(36)–C(31)–C(32)	120·9 (3)
C(25)–N(3)–C(21)	121·5 (3)	C(36)–C(31)–N(2)	117·4 (3)

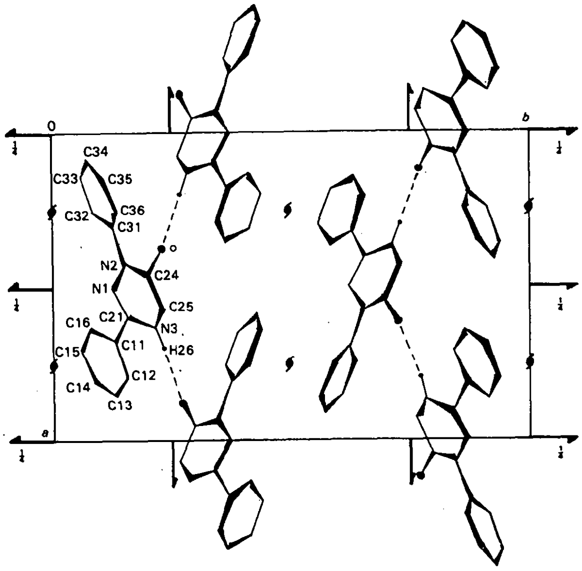

Fig. 2. Projection of $C_{15}H_{13}N_3O$ along the *c* axis. Dashed lines join the hydrogen-bonded atoms.

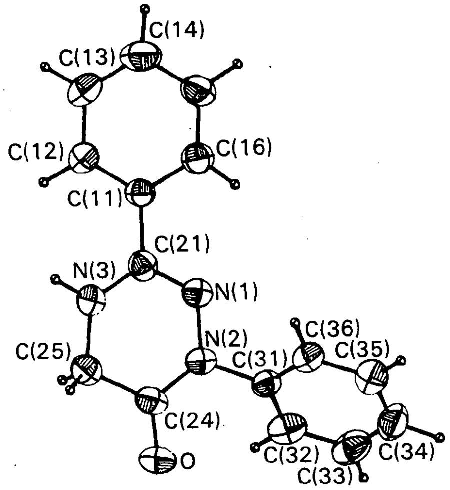

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule showing the atom numbering. Except for the H atoms thermal ellipsoids are shown with 50% probability.

The bond lengths in the two phenyl rings are in the range 1·361–1·390 Å. The distances C(24)–O [1·233 (3) Å] and C(21)–N(1) [1·289 (4) Å] are typical for a C=O and C=N double bond respectively while C(21)–N(3) [1·348 (4) Å] and C(24)–N(2) [1·342 (4) Å] are shorter than the single bond C(25)–N(3) [1·445 (4) Å]. This can be interpreted in terms of conjugation in the heterocycle.

The heterocycle is not planar as previously found in 3,4-diphenyl-4*H*-1,2,4-oxadiazol-5-one (Bel Hadj Amor, Kallel, Baccar, Driss & Gilmore, 1987). The planes of the N(1), N(3) and C(11) and C(25), N(2) and O atoms form an angle of 9° with each other. The two atoms C(21) and C(24) are 0·021 (24) and 0·001 (1) Å respectively out of these planes as expected since these two C atoms are in the sp^2-hybridization state. The mean plane of the heterocycle is nearly coplanar with the phenyl ring in position 3 but forms an angle of 57° with the phenyl ring in position 1.

A weak intermolecular hydrogen bond with distances N(3)–H(26)···O = 3·004 (4), H(26)···O = 2·21 (4) Å and an angle N(3)–H(26)···O = 167·5 (5)° contributes to the cohesion of the crystalline state [O is related to N(3) by $\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$].

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