# 1,3,5-Triphenyl-1*H*-1,2,4-benzotriazepine

BY C. FOCES-FOCES, F. H. CANO AND S. GARCÍA-BLANCO

*Departamento de Rayos X, Instituto de Química-Física 'Rocasolano', Serrano 119, Madrid-6, Spain*

**Abstract.** $C_{26}H_{19}N_3$, orthorhombic, *Pbnm*, $a = 18\cdot271$ (6), $b = 10\cdot717$ (1), $c = 10\cdot112$ (1) Å, $Z = 4$, $D_x = 1\cdot25$ g cm$^{-3}$. The structure was solved by direct methods and refined to an $R$ of $0\cdot066$ for 904 observed reflexions. Molecular disorder is present.

**Introduction.** The title compound (I) was prepared by Conde, Corral & Madroñero (1974) in order to obtain new heterocyclic compounds with biological activity and to investigate the synthesis of the hitherto unknown 1,2,4-benzotriazepine system. The purpose of the present work is to establish the geometry of the trisubstituted N atom and the conformation of the molecule.



(I)
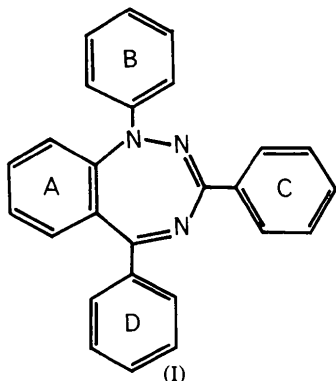
Intensity data from a single crystal ($0\cdot33 \times 0\cdot33 \times 0\cdot20$ mm) were recorded on a Philips PW 1100 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation [$\lambda$(Mo $K\alpha$) = $0\cdot7107$ Å]. 1803 independent reflexions were measured to $\theta \leq 25°$; 904 obeyed the condition $I \geq 4\sigma(I)$ and were considered as observed; the crystal diffracted poorly at high $\theta$. No absorption correction was applied ($\mu = 0\cdot81$ cm$^{-1}$). Systematic absences were consistent with space groups *Pbnm* and $P2_1cn$. The statistical distribution of intensities indicated the centrosymmetric space group *Pbnm*. Since $Z = 4$ the molecule has to be located at a special position. The structure was solved by direct methods with *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974); the *E* map of highest probability contained all the non-hydrogen atoms. The refinement was carried out by full-matrix least-squares techniques. A difference synthesis permitted the location of all H atoms. Because (I) does not show any

symmetry several unsuccessful attempts at refinement in the non-centrosymmetric space group were carried out. Attempts to separate the convoluted peaks also failed; we have tried to refine N(3) with a population of $0\cdot5$ and assignment of C and N in several ways, but no improvement in the description was obtained. The final thermally mixed refinement (C, N, O anisotropic; H isotropic) converged to $R = 0\cdot066$ and $R_w = 0\cdot081$. Weights were chosen as functions of $|F_o|$ and sin $\theta/\lambda$ to give approximately constant values of $\langle w\Delta^2 \rangle$. No significant peaks were found in a final difference map. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final atomic coordinates are given in Table 1.*

Table 1. *Final positional parameters with their standard deviations in parentheses*

|  | $x$ | $y$ | $z$ |
|---|---|---|---|
| C(1) | 0·25 | 0·0343 (5) | 0·4388 (5) |
| N(2) | 0·1855 (2) | 0·0693 (4) | 0·3929 (5) |
| N(3) | 0·1751 (2) | 0·1750 (3) | 0·3299 (3) |
| C(4) | 0·2116 (2) | 0·2851 (3) | 0·3817 (4) |
| C(5) | 0·1740 (3) | 0·3853 (4) | 0·4366 (4) |
| C(6) | 0·2123 (3) | 0·4843 (4) | 0·4893 (4) |
| C(7) | 0·1091 (2) | 0·1864 (3) | 0·2532 (4) |
| C(8) | 0·0599 (3) | 0·0882 (4) | 0·2443 (5) |
| C(9) | −0·0005 (3) | 0·0959 (6) | 0·1639 (5) |
| C(10) | −0·0141 (3) | 0·2034 (7) | 0·0924 (6) |
| C(11) | 0·0344 (3) | 0·3010 (5) | 0·0980 (4) |
| C(12) | 0·0953 (2) | 0·2938 (4) | 0·1785 (4) |
| C(13) | 0·25 | −0·0654 (5) | 0·5400 (5) |
| C(14) | 0·1841 (3) | −0·1121 (4) | 0·5884 (5) |
| C(15) | 0·1836 (7) | −0·2051 (6) | 0·6810 (8) |
| C(16) | 0·25 | −0·2529 (9) | 0·7278 (9) |
| H(5) | 0·120 (4) | 0·386 (6) | 0·436 (6) |
| H(6) | 0·182 (6) | 0·553 (6) | 0·530 (6) |
| H(8) | 0·070 (3) | 0·011 (5) | 0·293 (5) |
| H(9) | −0·037 (4) | 0·028 (7) | 0·165 (6) |
| H(10) | −0·059 (5) | 0·196 (6) | 0·037 (7) |
| H(11) | 0·034 (3) | 0·380 (5) | 0·040 (5) |
| H(12) | 0·128 (2) | 0·363 (4) | 0·182 (4) |
| H(14) | 0·160 (5) | −0·141 (6) | 0·524 (6) |
| H(15) | 0·133 (4) | −0·221 (6) | 0·714 (7) |
| H(16) | 0·25 | −0·342 (9) | 0·788 (10) |
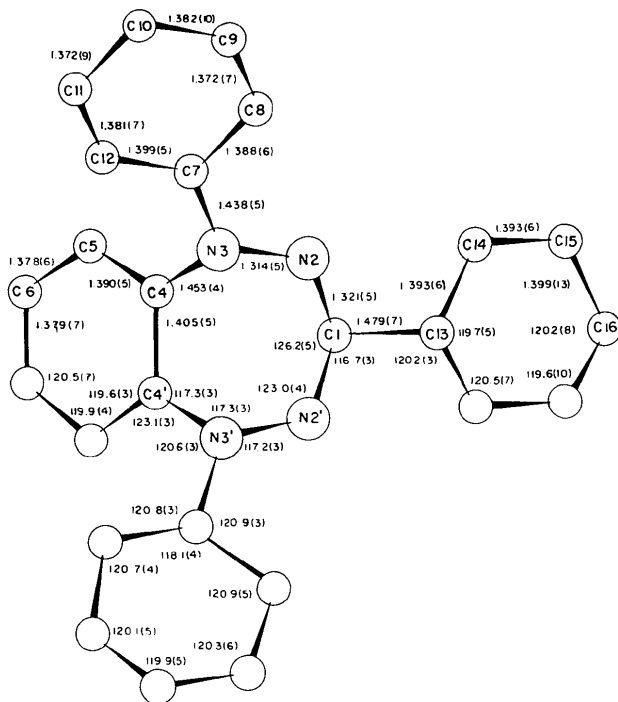
Fig. 1. A perspective view of the molecule showing the numbering system.

## Table 2. Main torsion angles (°)

| | |
|---|---|
| N(2)—N(3)—C(7)—C(8) | −1·5 (5) |
| C(4)—N(3)—C(7)—C(12) | 28·2 (4) |
| N(2)—C(1)—C(13)—C(14) | −4·0 (5) |
| N(2′)—C(1)—N(2)—N(3) | −25·9 (6) |
| C(1)—N(2)—N(3)—C(4) | −41·8 (4) |
| N(2)—N(3)—C(4)—C(4′) | 63·3 (4) |
| N(3)—C(4)—C(4′)—N(3′) | 0 |

angle at N(3) is 118·3°. The molecular conformation is described in terms of torsion angles (Table 2), the seven-membered ring having the boat conformation. Rings *A*, *B* and *A*, *C* and *B*, *D* make angles of 59·7, 70·3 and 63·1° respectively.

No intermolecular contacts shorter than the sum of the van der Waals radii were found.

**Discussion.** The molecule including atom labels, bond distances and angles is shown in Fig. 1. Atoms C(1), C(13) and C(16) lie on the mirror plane at $x = 0.25$. Because the molecule cannot possess symmetry [see (I)] a disordered structure has to be assumed, the present model being the average of two orientations. The disorder is also suggested by some high temperature parameters, mainly in ring *C*.

The C(1)—N(2) and N(2)—N(3) distances are longer than the corresponding double bond and much shorter than the corresponding single bond, as might be expected from the molecular disorder. The mean bond

## References

Conde, S., Corral, C. & Madroñero, R. (1974). *Tetrahedron*, **30**, 195–200.

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.

Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J. P. (1974). *MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Stewart, J. M., Kundell, F. A. & Baldwin, J. C. (1970). *The XRAY 70 system.* Computer Science Center, Univ. of Maryland, College Park, Maryland.

# Salicylideneaniline

By R. Destro, A. Gavezzotti and M. Simonetta

*Istituto di Chimica Fisica e Centro CNR, Università, Via Golgi 19, 20133 Milano, Italy*

**Abstract.** $C_{13}H_{11}NO$, orthorhombic, space group *Fd2d*; $a = 27.968$ (16), $b = 5.941$ (2), $c - 12.882$ (3) Å, $Z = 8$, $D_m = 1.22$, $D_c = 1.22$ g cm$^{-3}$. The number of equivalent positions being 16, orientational disorder simulates symmetry around a twofold axis, and prevents the determination of accurate molecular