

C21	0.1760 (5)	1.0348 (5)	0.8555 (4)	3.3 (1)
C22	0.2227 (6)	1.0665 (5)	0.7650 (5)	3.9 (2)
C23	0.2152 (7)	1.0068 (7)	0.6848 (5)	5.6 (2)
C24	0.1645 (7)	0.9121 (7)	0.6929 (6)	6.9 (2)
C25	0.1165 (7)	0.8795 (6)	0.7817 (7)	6.5 (2)
C26	0.1257 (6)	0.9393 (6)	0.8630 (5)	4.6 (2)
C1	0.2910 (5)	1.2205 (5)	1.0172 (4)	3.0 (1)
C2	0.1747 (6)	1.1057 (6)	0.9427 (5)	4.6 (2)
C3	0.5505 (7)	1.3749 (6)	1.0918 (5)	4.9 (2)
C4	0.4959 (7)	1.2698 (7)	0.8089 (5)	5.0 (2)
Cl2	0.1226 (2)	0.5555 (2)	0.9267 (1)	6.68 (6)
P2	0.4439 (2)	0.2835 (2)	0.4959 (1)	4.38 (4)
N2	0.2973 (5)	0.4817 (5)	0.4377 (4)	4.1 (1)
O21	0.5484 (5)	0.3330 (4)	0.5011 (4)	6.1 (1)
O22	0.4467 (6)	0.2061 (5)	0.4020 (4)	6.5 (2)
O23	0.4263 (4)	0.1882 (4)	0.5725 (4)	4.9 (1)
C31	0.2406 (5)	0.4366 (5)	0.6145 (4)	3.2 (1)
C32	0.2834 (6)	0.5246 (6)	0.6521 (5)	4.0 (2)
C33	0.2460 (6)	0.5614 (6)	0.7466 (5)	4.1 (2)
C34	0.1671 (6)	0.5091 (6)	0.8072 (5)	4.2 (2)
C35	0.1226 (6)	0.4234 (6)	0.7718 (5)	5.0 (2)
C36	0.1585 (6)	0.3861 (6)	0.6777 (5)	4.4 (2)
C41	0.1784 (5)	0.6327 (5)	0.3341 (4)	3.4 (1)
C42	0.1248 (6)	0.7554 (6)	0.3265 (5)	4.3 (2)
C43	0.1216 (7)	0.8085 (6)	0.2358 (6)	5.2 (2)
C44	0.1713 (7)	0.7397 (7)	0.1550 (5)	5.1 (2)
C45	0.2241 (7)	0.6200 (6)	0.1611 (5)	4.6 (2)
C46	0.2277 (6)	0.5672 (5)	0.2492 (5)	4.0 (2)
C5	0.2848 (7)	0.3922 (6)	0.5110 (5)	4.2 (2)
C6	0.1771 (7)	0.5722 (6)	0.4291 (5)	5.0 (2)
C7	0.4898 (8)	0.2384 (7)	0.3084 (5)	6.1 (2)
C8	0.5382 (8)	0.0978 (6)	0.5947 (6)	5.9 (2)

Table 2. Selected geometric parameters (Å, °)

Cl1—Cl4	1.733 (6)	Cl2—C34	1.720 (7)
P1—O11	1.425 (4)	P2—O21	1.417 (6)
P1—O12	1.558 (5)	P2—O22	1.602 (6)
P1—O13	1.561 (5)	P2—O23	1.522 (5)
P1—C1	1.806 (6)	P2—C5	1.805 (6)
N1—C1	1.448 (8)	N2—C5	1.432 (8)
N1—C2	1.446 (9)	N2—C6	1.434 (8)
O12—C3	1.430 (9)	O22—C7	1.384 (9)
O13—C4	1.394 (8)	O23—C8	1.420 (8)
C11—C1	1.507 (8)	C31—C5	1.506 (9)
C21—C2	1.49 (1)	C41—C6	1.465 (9)
O11—P1—O12	116.1 (3)	O21—P2—O22	119.3 (3)
O11—P1—O13	114.6 (2)	O21—P2—O23	115.5 (3)
O11—P1—C1	115.5 (3)	O21—P2—C5	115.2 (3)
O12—P1—O13	100.9 (2)	O22—P2—O23	97.8 (3)
O12—P1—C1	102.5 (2)	O22—P2—C5	104.1 (3)
O13—P1—C1	105.5 (3)	O23—P2—C5	102.3 (3)
C1—N1—C2	113.8 (4)	C5—N2—C6	113.6 (5)
P1—O12—C3	120.1 (4)	P2—O22—C7	122.7 (6)
P1—O13—C4	123.6 (4)	P2—O23—C8	118.7 (5)
C12—C11—C1	120.8 (5)	C32—C31—C5	122.5 (6)
C16—C11—C1	121.1 (5)	C36—C31—C5	120.8 (6)
Cl1—C14—C13	118.5 (5)	Cl2—C34—C33	119.4 (6)
Cl1—C14—C15	119.7 (5)	Cl2—C34—C35	121.5 (5)
C22—C21—C2	121.4 (6)	C42—C41—C6	121.1 (6)
C26—C21—C2	121.1 (6)	C46—C41—C6	121.2 (5)
P1—C1—N1	104.6 (3)	P2—C5—N2	103.6 (4)
P1—C1—C11	112.4 (4)	P2—C5—C31	112.0 (5)
N1—C1—C11	118.5 (5)	N2—C5—C31	117.2 (5)
N1—C2—C21	113.1 (5)	N2—C6—C41	112.7 (5)

The structure was solved by direct methods and refined by full-matrix least-squares calculation on  $F$ , with anisotropic displacement parameters for non-H atoms and fixed isotropic displacement parameters for H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *MolEN* (Fair, 1990). Data reduction: *MolEN*. Program(s) used to solve structure: *MolEN*; *MULTAN11/82* (Main *et al.*, 1982). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *MolEN*.

This work was supported by the Key Discipline Fund of Tianjin Higher Education, People's Republic of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1259). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Li, Y.-G., Wang, G.-H., Zhang, D.-K., Miao, F.-M., Liu, X.-L., Cao, J.-H. & Guo, H.-Z. (1988). *Sci. China*, **B11**, 1137–1146.
- Li, Y.-G., Wang, J.-J., Han, T., Liu, Y.-S., Cao, J.-H., Jiang, X.-C., Miao, F.-M. & Liu, X.-L. (1988). *Acta Chim. Sin.* **46**, 679–685.
- Liu, X.-L., Miao, F.-M., Liu, L.-J. & Chen, R.-Y. (1991). *Chem. J. Chin. Univs.* **12**, 1483–1485.
- Liu, X.-L., Sun, M., Miao, F.-M., Feng, K.-S. & Chen, R.-Y. (1992). *Acta Cryst.* **C48**, 947–949.
- Liu, X.-L., Sun, M., Miao, F.-M., Li, Y.-G., Wang, J.-J., Han, Y.-Z. & Xu, X.-J. (1992). *Acta Phys. Chim. Sinica*, **8**, 100–108.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Marre, M.-R., Sanchez, M., Wolf, R., Jaud, T. & Galy, J. (1984). *Can. J. Chem.* **62**, 2186–2191.
- Perales, A. & Garcia-Blanco, S. (1977). *Acta Cryst.* **B33**, 1935–1939.

*Acta Cryst.* (1995). **C51**, 2352–2354

## 1,3,5-Tricyanobenzene

D. SHEKHAR REDDY, KALIYAMOORTHY PANNEERSELVAM  
AND GAUTAM R. DESIRAJU

*School of Chemistry, University of Hyderabad,  
PO Central University, Hyderabad 500 046, India*

H. L. CARRELL AND C. J. CARRELL

*Fox Chase Cancer Center, 7701 Burholme Avenue,  
Philadelphia, PA 19111, USA*

(Received 9 December 1994; accepted 25 May 1995)

## Abstract

The crystal of the title compound (1,3,5-benzenetricarbonitrile, C<sub>6</sub>H<sub>3</sub>N<sub>3</sub>) consists of C—H···N hydrogen-bonded helices which when projected down the helix axis result in a quasi-hexagonal network.

### Comment

The study of intermolecular interactions is important in structural chemistry and crystal engineering because these interactions are directly responsible for molecular association (Desiraju, 1989). As a result, much work has been carried out using conventional or strong hydrogen bonds. However, the incorporation of weak intermolecular interactions such as C—H···O, C—H···N, C—H···halogen, halogen···halogen, halogen···heteroatom,  $\pi$ ··· $\pi$  and hydrophobic interactions into supramolecular architecture can greatly extend the scope of systematic crystal engineering. Here we describe the role of C—H···N hydrogen bonds in the crystal structure of the title compound, (I).

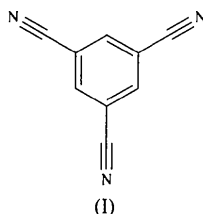


Fig. 1 is an *ORTEPII* (Johnson, 1976) drawing of the title compound showing the atomic numbering scheme. There are 12 C—H···N hydrogen bonds per molecule, details of which are given in Table 3. Each H atom forms bifurcated (three centred) hydrogen bonds to two N atoms and each N atom is involved in bifurcated hydrogen bonds to two H atoms. These bifurcated hydrogen bonds result in infinite C—H···N mediated helices which when combined give quasi-hexagonal networks (Fig. 2). Such networks have been observed by us previously in the 1:1 molecular complex formed by 1,3,5-tricyanobenzene with hexamethylbenzene (Reddy, Goud,

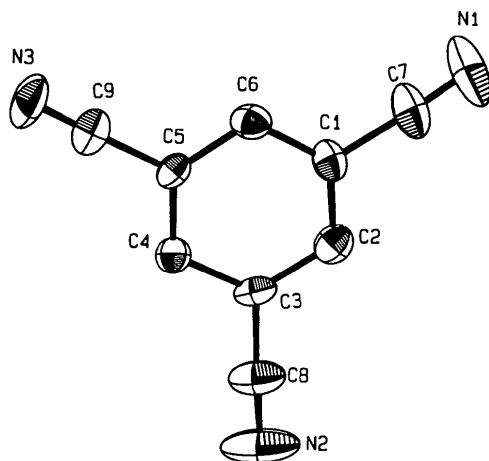


Fig. 1. *ORTEPII* (Johnson, 1976) drawing of the title molecule showing atom labelling. Displacement ellipsoids are plotted at the 50% probability level.

Panneerselvam & Desiraju, 1993). However, the hexagonal network in the 1:1 complex is two-dimensional while in (I) it is three-dimensional. Curiously, the space group is non-centrosymmetric.

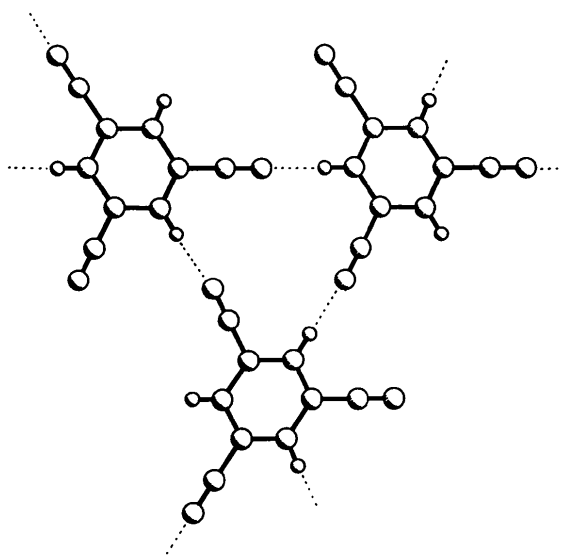


Fig. 2. Three-dimensional C—H···N hydrogen bonding in the crystal structure of 1,3,5-tricyanobenzene.

### Experimental

The title compound was prepared from mesitylene according to literature procedures (Skala, Hlavaty & Kuthan, 1970) and single crystals of X-ray diffraction quality were obtained from ethanol.

#### Crystal data

C<sub>9</sub>H<sub>3</sub>N<sub>3</sub>  
*M<sub>r</sub>* = 153.14  
 Monoclinic  
*P*2<sub>1</sub>  
*a* = 13.719 (2) Å  
*b* = 7.726 (1) Å  
*c* = 3.875 (1) Å  
 $\beta$  = 95.50 (2)°  
*V* = 408.8 (2) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.244 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.7107 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 10–20°  
 $\mu$  = 0.08 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Needle  
 0.2 × 0.1 × 0.1 mm  
 Colourless

#### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 1416 measured reflections  
 1416 independent reflections  
 1128 observed reflections  
 $[I > 2\sigma(I)]$

$\theta_{\max}$  = 32.0°  
 $h = 0 \rightarrow 20$   
 $k = 0 \rightarrow 11$   
 $l = -5 \rightarrow 5$   
 3 standard reflections monitored every 97 reflections  
 intensity decay: not significant

## Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\max} = 2.747$
$R[F^2 > 2\sigma(F^2)] = 0.0428$	$\Delta\rho_{\max} = 0.215 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.1328$	$\Delta\rho_{\min} = -0.145 \text{ e } \text{\AA}^{-3}$
$S = 0.628$	Extinction correction: none
1416 reflections	Atomic scattering factors
120 parameters	from <i>International Tables</i>
H atoms refined isotropically	for <i>Crystallography</i> (1992,
$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$	Vol. C, Tables 4.2.6.8 and
where $P = (F_o^2 + 2F_c^2)/3$	6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j \cdot a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
C1	0.33209 (12)	-0.1833 (4)	0.0931 (5)	0.0434 (4)
C2	0.33233 (13)	-0.0144 (4)	0.2138 (5)	0.0433 (4)
C3	0.24702 (13)	0.0826	0.1607 (4)	0.0406 (4)
C4	0.16236 (12)	0.0130 (3)	-0.0110 (4)	0.0398 (4)
C5	0.16440 (12)	-0.1561 (3)	-0.1302 (4)	0.0382 (4)
C6	0.24880 (14)	-0.2560 (3)	-0.0801 (4)	0.0418 (4)
C7	0.4200 (2)	-0.2871 (5)	0.1493 (7)	0.0704 (8)
C8	0.2467 (2)	0.2570 (4)	0.2919 (6)	0.0619 (6)
C9	0.0773 (2)	-0.2309 (4)	-0.3094 (5)	0.0560 (6)
N1	0.4893 (2)	-0.3663 (7)	0.1975 (8)	0.1145 (14)
N2	0.2465 (2)	0.3933 (4)	0.4012 (7)	0.0959 (10)
N3	0.0093 (2)	-0.2902 (5)	-0.4519 (7)	0.0875 (9)

Table 2. Selected bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

C1—C2	1.386 (3)	C4—C5	1.387 (3)
C1—C6	1.387 (3)	C5—C6	1.390 (3)
C1—C7	1.447 (3)	C5—C9	1.444 (3)
C2—C3	1.388 (3)	C7—N1	1.131 (4)
C3—C4	1.390 (2)	C8—N2	1.135 (3)
C3—C8	1.440 (3)	C9—N3	1.134 (3)
C2—C1—C6	121.3 (2)	C6—C5—C4	121.3 (2)
C2—C1—C7	119.8 (2)	C6—C5—C9	119.0 (2)
C6—C1—C7	118.9 (2)	C4—C5—C9	119.7 (2)
C3—C2—C1	118.9 (2)	C5—C6—C1	118.6 (2)
C2—C3—C4	121.1 (2)	N1—C7—C1	178.8 (4)
C2—C3—C8	118.9 (2)	N2—C8—C3	178.8 (3)
C4—C3—C8	120.0 (2)	N3—C9—C5	179.5 (3)
C5—C4—C3	118.8 (2)		

Table 3. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D—H...A	D—H	H...A	D...A	D—H...A
C2—H2...N1 <sup>i</sup>	0.96 (3)	2.45 (3)	3.381 (4)	164 (2)
C2—H2...N1 <sup>ii</sup>	0.96 (3)	2.89 (3)	3.253 (4)	103 (2)
C4—H4...N3 <sup>iii</sup>	0.93 (3)	2.85 (3)	3.447 (4)	124 (2)
C4—H4...N3 <sup>iv</sup>	0.93 (3)	2.55 (3)	3.354 (4)	145 (2)
C6—H6...N2 <sup>v</sup>	0.91 (3)	2.85 (3)	3.291 (4)	111 (2)
C6—H6...N2 <sup>vi</sup>	0.91 (3)	2.52 (3)	3.372 (4)	156 (2)

Symmetry codes: (i)  $1 - x, \frac{1}{2} + y, 1 - z$ ; (ii)  $1 - x, \frac{1}{2} + y, -z$ ; (iii)  $-x, \frac{1}{2} + y, -z$ ; (iv)  $-x, \frac{1}{2} + y, -1 - z$ ; (v)  $x, y - 1, z$ ; (vi)  $x, y - 1, z - 1$ .

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

Financial support from the DST and UGC, Government of India, and from the American Cancer Society (grant CN-10) is acknowledged.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: SE1078). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Desiraju, G. R. (1989). In *Crystal Engineering. The Design of Organic Solids*. Amsterdam: Elsevier.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Reddy, D. S., Goud, B. S., Panneerselvam, K. & Desiraju, G. R. (1993). *J. Chem. Soc. Chem. Commun.* pp. 663–664.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
- Skala, V., Hlavaty, J. & Kuthan, J. (1970). *Collect. Czech. Chem. Commun.* **35**, 350–354.

*Acta Cryst.* (1995). **C51**, 2354–2356

## 2,3-Diethyl-5,6,7,8-tetrahydro-2a $\lambda^4$ -thia-2,3,4a,8a-tetraazacyclo[cd]azulene-1(2H),4(3H)-dithione

DAVID G. BILLING, JAN C. A. BOEYENS, LONG-LI LAI AND DAVID H. REID

*Center for Molecular Design, Department of Chemistry, University of Witwatersrand, Wits 2050, Johannesburg, South Africa*

(Received 5 May 1994; accepted 8 March 1995)

## Abstract

The title compound, C<sub>11</sub>H<sub>18</sub>N<sub>4</sub>S<sub>3</sub>, contains elongated N—S bonds [1.877 (1) and 1.903 (1)  $\text{\AA}$ ] which are greater by 7.9 and 9.4%, respectively, than the two-centre two-electron N—S bond [1.74  $\text{\AA}$ ; Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19]. The lengths of corresponding bonds and the size of the corresponding bond angles in the two halves of the triheteropentalene framework are very similar, differing from one another at most by 0.027  $\text{\AA}$  and 0.83 $^\circ$ , respectively.

## Comment

The structure determination of the title compound, (1), was carried out to establish whether the product from the reaction of the salt (2) with ethyl isothiocyanate in dichloromethane in the presence of triethylamine contains