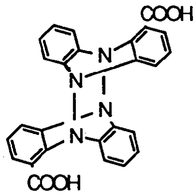
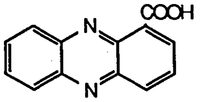
way, Sohns & Jackson, 1956) pseudomonad fungal antibiotic phenazine-1-carboxylic acid, *i.e.* the monomeric structure (II). On the basis of the novelty of the dimeric structure, a US patent (Gurusiddaiah, Jois, Weller & Cook, 1985) has been applied for and licensees for this patent have been called for (*Phytopathology News*, 1988). The crystal structure reported herein provides unequivocal evidence that the antibiotic isolable from *P. fluorescens* 2–79 has the monomeric structure (II), Figs. 1 and 2.



(I)



(II)

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Trichlorotricyanobenzene*–Hexamethylbenzene (1/1)

By Doyle Britton

Department of Chemistry, University of Minnesota, Minneapolis, MN 55455, *USA*

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Abstract. $C_6Cl_3(CN)_3.C_6(CH_3)_6$, $M_r = 418{\cdot}8$, monoclinic, $C2/c$ (No. 15), $a = 13{\cdot}585\,(3)$, $b = 15{\cdot}558\,(3)$, $c = 9{\cdot}750\,(2)$ Å, $\beta = 104{\cdot}73\,(1)°$, $Z = 4$, $V = 1993\,(1)$ Å^3, $D_x = 1{\cdot}396\,(1)$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 42{\cdot}3$ cm^{-1}, $F(000) = 864$, $T = 297\,(2)$ K, $R = 0{\cdot}044$ for 1667 reflections. The bond lengths and angles in the two molecules are normal. The H atoms could not be located and appear to be disordered. The two types of molecules occur in stacks alternating with each other. The molecules are approximately parallel to each other (deviation from parallel 1·6°) and to the (302) plane (deviation of trichlorotricyanobenzene 1·0° from the plane, hexamethylbenzene 1·1°). The two kinds of molecules are at an average distance of 3·58 Å apart.

Experimental. Yellow crystals suitable for X-ray analysis of the 1/1 molecular complex between trichlorotricyanobenzene (Diamond Shamrock Corp.) and hexamethylbenzene (Eastman Organic Chemicals) were prepared by slow evaporation of an acetone–acetonitrile solution containing equimolar amounts of the two compounds. The crystal used was a parallelepiped 0·11 × 0·20 × 0·21 mm bounded by the forms {100} and $\{11\bar{1}\}$. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 24 reflections with $17 < \theta < 38°$ were used to determine the cell parameters. Systematic extinctions (hkl, $h + l$ odd; $h0l$, l odd) showed the space group to be Cc or $C2/c$. The latter was successfully used to determine and refine the structure. Data were collected using ω–2θ scans, in the range $0 < \theta < 78°$ for one quadrant (ranges: h −17 to 17; k 0 to 19; l 0 to 12). Absorption corrections were applied based on the indexed crystal faces, with maximum and minimum transmission factors 0·681 and 0·416, respectively. The intensities of 2015 independent allowed reflections were measured, of which the 1667 with $I > \sigma(I)$ were used in the calculations. Three check reflections, measured every 6000 s of exposure time, showed no systematic change with time. The trial structure, including all non-hydrogen atoms, was found using the *MULTAN*11/82 program (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The H atoms could not be found in difference electron density maps and were included in idealized positions with

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* 2,4,6-Trichloro-1,3,5-benzenetricarbonitrile.

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Cl(1)	0·500	0·05152 (5)	0·250	6·13 (2)
Cl(3)	0·62355 (4)	0·34929 (4)	0·07450 (6)	5·84 (1)
C(1)	0·500	0·1613 (2)	0·250	3·94 (5)
C(2)	0·5554 (1)	0·2054 (1)	0·1699 (2)	3·87 (4)
C(3)	0·5552 (1)	0·2949 (1)	0·1712 (2)	3·87 (4)
C(4)	0·500	0·3404 (2)	0·250	4·03 (5)
C(12)	0·6120 (2)	0·1596 (1)	0·0876 (2)	5·14 (4)
N(2)	0·6560 (2)	0·1235 (1)	0·0207 (2)	7·28 (5)
C(14)	0·500	0·4326 (2)	0·250	5·62 (7)
N(4)	0·500	0·5058 (2)	0·250	8·41 (9)
C(5)	0·2282 (2)	0·1666 (1)	0·0371 (2)	4·04 (4)
C(6)	0·2898 (2)	0·1789 (1)	−0·0567 (2)	4·00 (4)
C(7)	0·3112 (1)	0·2627 (1)	−0·0936 (2)	3·96 (4)
C(15)	0·2048 (2)	0·0765 (2)	0·0776 (3)	6·52 (6)
C(16)	0·3339 (2)	0·1034 (2)	−0·1185 (3)	6·12 (5)
C(17)	0·3778 (2)	0·2757 (2)	−0·1949 (3)	6·13 (5)

Table 2. *Interatomic distances* (Å) *and angles* (°)

Trichlorotricyanobenzene			
C(1)–C(2)	1·395 (2)	C(2)–C(12)	1·434 (2)
C(2)–C(3)	1·392 (2)	C(4)–C(14)	1·433 (3)
C(3)–C(4)	1·395 (2)	C(12)–N(2)	1·140 (2)
C(1)–Cl(1)	1·708 (2)	C(14)–N(4)	1·140 (2)
C(3)–Cl(3)	1·707 (1)		
C(2′)–C(1)–C(2)	121·0 (2)	Cl(3)–C(3)–C(4)	119·7 (1)
C(1)–C(2)–C(3)	119·0 (1)	C(12)–C(2)–C(1)	120·7 (1)
C(2)–C(3)–C(4)	121·1 (1)	C(12)–C(2)–C(3)	120·3 (1)
C(3)–C(4)–C(3′)	118·9 (2)	C(14)–C(4)–C(3)	120·5 (1)
Cl(1)–C(1)–C(2)	119·5 (1)	N(2)–C(12)–C(2)	179·1 (2)
Cl(3)–C(3)–C(2)	119·2 (1)	N(4)–C(14)–C(4)	180·
Hexamethylbenzene			
C(5)–C(6)	1·401 (2)	C(5)–C(15)	1·513 (2)
C(6)–C(7)	1·401 (2)	C(6)–C(16)	1·513 (2)
C(7)–C(5′)	1·396 (2)	C(7)–C(17)	1·514 (2)
C(7′)–C(5)–C(6)	120·1 (1)	C(16)–C(6)–C(5)	121·1 (2)
C(5)–C(6)–C(7)	119·5 (1)	C(16)–C(6)–C(7)	119·4 (2)
C(6)–C(7)–C(5′)	120·4 (1)	C(17)–C(7)–C(6)	119·3 (2)
C(15)–C(5)–C(6)	119·9 (2)	C(17)–C(7)–C(5′)	120·3 (2)
C(15)–C(5)–C7′)	120·1 (2)		

twofold disorder; the positions were based on the approximately D_{3d} arrangements found previously (Hamilton, Edmonds, Tippe & Rush, 1969; Colton & Henn, 1970; Maverick, Trueblood & Bekoe, 1978). All the atoms except H were given anisotropic thermal parameters. All parameters were refined by full-matrix least-squares refinement of F's. Refinement converged with $R = 0.044$, $wR = 0.062$, and $S = 1.849$; $w = 1/\sigma^2(F)$ was calculated from $\sigma^2(I) = \sigma^2(I)_c + (0.05I)^2$, where $\sigma(I)_c$ is the standard deviation in I based on counting statistics alone. $(\Delta/\sigma)_{max}$ in the final cycle was 0·07. The two highest peaks (0·27 e Å^{-3}) in the final difference electron density map were both less than 1 Å from a Cl atom position. Atomic scattering factors and anomalous-dispersion corrections for all atoms were taken from *International Tables for X-ray Crystallography* (1974). The computer programs used were all from the *Enraf–Nonius Structure Determination Package* and have been described by Frenz (1978).

Atomic coordinates are given in Table 1, and interatomic distances and angles in Table 2.* The atom labelling and thermal ellipsoids for both molecules are shown in Fig. 1. The entire complex is shown in Fig. 2.

Related literature. The structure of trichlorotricyanobenzene was reported by Britton (1981). The structure of hexamethylbenzene has been reported for the ordinary form [most recently by Hamilton, Edmonds, Tippe & Rush (1969)] and for the high-temperature form (Watanabe, Saito & Chihara, 1949). No structures of other complexes of trichlorotricyanobenzene have been reported. Structures have been reported for complexes of hexamethylbenzene with chloranil (Harding & Wallwork, 1955; Jones & Marsh, 1962), tetracyanobenzene (Niimura, Ohashi & Saito, 1968), hexafluorobenzene (Dahl, 1972, 1973), tetracyanoquinodimethane (Colton & Henn, 1970; Johnson, Reed, Hall & Raaen, 1974), tetracyanoethylene

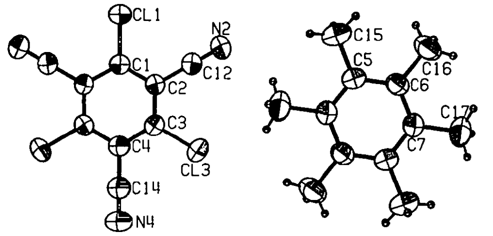

Fig. 1. View of the two molecules in the complex. The independent atoms are labelled. Thermal ellipsoids are shown at the 50% probability level.

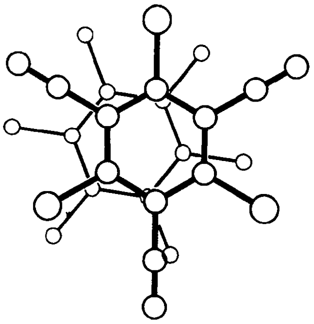

Fig. 2. View of the complex along the perpendicular to the plane of the trichlorotricyanobenzene molecule. Both molecules are shown in the same orientation as in Fig. 1.

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* Lists of anisotropic thermal parameters, H-atom parameters, deviations from planarity, distances between the two molecules, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51231 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Saheki, Yamada, Yoshioka & Nakatsu, 1976; Maverick, Trueblood & Bekoe, 1978), and trinitrofluorenone (Brown, Cheung, Trefonas & Majeste, 1974).

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Structure of 2-Benzyl-2,3-dihydro-3-oxo-4-piperidino-1,2,5-thiazole 1,2-Dioxide

By M. Martínez-Ripoll, F. H. Cano and C. Foces-Foces

UEI de Cristalografía, Instituto Rocasolano, CSIC, Serrano 119, 28006 *Madrid, Spain*

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Abstract. $C_{14}H_{17}N_3O_3S$, $M_r = 307{\cdot}37$, triclinic, $P\bar{1}$, $a = 16{\cdot}3875\,(7)$, $b = 10{\cdot}9715\,(3)$, $c = 9{\cdot}0822\,(3)$ Å, $\alpha = 105{\cdot}757\,(3)$, $\beta = 99{\cdot}465\,(3)$, $\gamma = 72{\cdot}412\,(3)°$, $U = 1492{\cdot}1\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$ Mg m^{-3}, graphite-monochromated Cu $K\alpha$ radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 2{\cdot}009$ mm^{-1}, $F(000) = 648$, $T = 293$ K, $R = 0{\cdot}076$ for 2654 observed reflexions [$I > 3\sigma(I)$]. Bond lengths and angles in molecules *A* and *B* are within 2·5 times the pooled e.s.d.'s. The main difference between the molecules is the conformation of the phenyl rings with respect to the thiazole ring. The N(1) and N(3) atoms are sp^2 hybridized: sums of angles are 359·6 (7), 359·4 (6)° (molecule *A*) and 359·7 (7), 359·9 (8)° (molecule *B*). The piperidine ring exhibits a chair conformation.

Experimental. Colourless plate, 0·30 × 0·20 × 0·03 mm, used for data collection and determination of lattice constants (Cu $K\alpha$, 78 reflexions with $2 < \theta < 45°$). Philips PW 1100 diffractometer, Cu $K\alpha$, graphite monochromator, bisecting geometry, $\omega/2\theta$ scan mode, 5062 independent reflexions up to $\theta = 65°$, *hkl* range −18,18; −12,12; 0,10. Two standard reflexions were measured every 90 min, no decay observed, but the diffraction was rather weak, with only about 50% of the 5062 total recorded reflexions observed. The structure was solved by direct methods (*MULTAN*80, Main *et al.*, 1980). H atoms, located in a difference synthesis, included isotropically in last cycles of refinement. Empirical weights so as to give no trends in $\langle w\Delta F^2\rangle$ *versus* $\langle |F_o| \rangle$ and $\langle (\sin\theta)/\lambda \rangle$ [ΔF was adjusted by

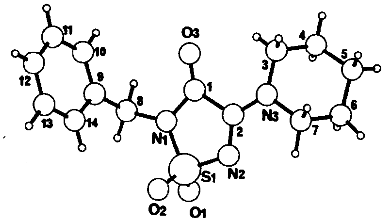

Fig. 1. A view of one of the two nearly identical molecules projected on the molecular least-squares plane showing the atomic numbering.

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