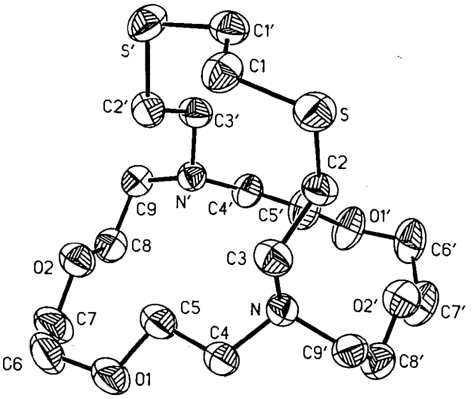


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the $2_S.2_O.2_O$ cryptand. The molecule occupies a twofold rotation axis. Ellipsoids are drawn at 50% probability.

been frequently investigated as a crown complex with a variety of caged ions. A review of these cryptate complexes and their nomenclature is available (Dobler, 1981). The cryptate complex $K^+[2_O.2_O.2_O]$ is very stable and is frequently used as a cationic counterion for anionic complexes in structural determinations (*e.g.* VanAtta, Strouse, Hanson & Valentine, 1987; Bjorgvinsson, Sawyer & Schrobilgen, 1987). A recent review discusses macrocyclic thioethers (Cooper, 1988).

Receipt of the material from Professor R. W. Taylor (Department of Chemistry, University of Oklahoma, Norman, OK 73019, USA) is gratefully acknowledged.

References

Bjorgvinsson, M., Sawyer, J. F. & Schrobilgen, G. J. (1987). *Inorg. Chem.* **26**, 741–749.
Cooper, S. R. (1988). *Acc. Chem. Res.* **21**, 141–146.
Dobler, M. (1981). *Ionophores and Their Structures*, pp. 177–205. New York: John Wiley.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Louis, R., Thierry, J. C. & Weiss, R. (1974). *Acta Cryst.* B**30**, 753–758.
Metz, B., Moras, D. & Weiss, R. (1976). *J. Chem. Soc. Perkin Trans.* 2, pp. 423–429.
Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
VanAtta, R. B., Strouse, C. E., Hanson, L. K. & Valentine, J. S. (1987). *J. Am. Chem. Soc.* **109**, 1425–1434.

Acta Cryst. (1988). C**44**, 2220–2222

Structure of the Pseudomonad Fungal Antibiotic Phenazine-1-carboxylic Acid

By Graham P. Jones,* David G. Lewis† and Max E. Tate‡

Departments of Plant Physiology, Soil Science and Agricultural Biochemistry, Waite Agricultural Research Institute, University of Adelaide, Glen Osmond, South Australia 5064, *Australia*

and Michael R. Snow and Edward R. T. Tiekink

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, South Australia 5001, *Australia*

(*Received* 11 *July* 1988; *accepted* 27 *July* 1988)

Abstract. $C_{13}H_8N_2O_2$, $M_r = 224{\cdot}2$, monoclinic, Cc, $a = 3{\cdot}955\,(1)$, $b = 19{\cdot}278\,(4)$, $c = 13{\cdot}468\,(1)$ Å, $\beta = 98{\cdot}90\,(2)°$, $V = 1015\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}468$ Mg m^{-3}, $\lambda(\text{Mo}\ K\bar{\alpha}) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}061$ mm^{-1}, $F(000) = 464$, $T = 293\,(2)$ K, $R = 0{\cdot}047$ for 571 observed reflections. The crystal-structure determination of the title compound, a phenazine antibiotic from *Pseudomonas fluorescens* 2–79 (NRRL B-15132), confirms its structure as phenazine-1-carboxylic acid. The molecular packing is described by discrete stacks of molecules parallel to the *a* axis with the distance between the essentially planar molecules being *ca* 3·96 Å; there are no significant intermolecular contacts in the lattice.

Experimental. Suitable crystals for X-ray study obtained from the slow evaporation of a dichloromethane/acetonitrile (70/30 *v*/*v*) solution of the compound held at ambient temperature (Brisbane, Janik, Tate & Warren, 1987); yellow needles, m.p. 516–517 K. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochro-

---

[*] Plant Physiology.
[†] Soil Science.
[‡] Agricultural Biochemistry.

0108-2701/88/122220-03$03.00

Table 1. *Fractional atomic coordinates and B_{eq} values* (Å^2)

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
O(1)	0·4026 (13)	−0·4778 (3)	−0·4985 (6)	6·80
O(2)	0·1772 (12)	−0·3776 (3)	−0·4591 (5)	5·35
N(1)	−0·2008	−0·3079 (2)	−0·5981	3·51
N(2)	−0·5203 (12)	−0·3008 (3)	−0·8016 (5)	3·48
C(1)	−0·1624 (13)	−0·3584 (3)	−0·6615 (5)	2·72
C(2)	−0·3249 (13)	−0·3554 (3)	−0·7651 (5)	2·86
C(3)	−0·2867 (13)	−0·4111 (3)	−0·8322 (6)	3·95
C(4)	−0·0936 (16)	−0·4663 (3)	−0·7971 (7)	4·70
C(5)	0·0720 (15)	−0·4696 (3)	−0·6975 (6)	4·31
C(6)	0·0396 (14)	−0·4189 (3)	−0·6306 (6)	3·46
C(7)	−0·3977 (14)	−0·2526 (3)	−0·6338 (6)	3·52
C(8)	−0·5521 (13)	−0·2489 (3)	−0·7364 (6)	3·00
C(9)	−0·7479 (13)	−0·1897 (3)	−0·7707 (6)	4·27
C(10)	−0·7825 (16)	−0·1374 (3)	−0·7029 (6)	4·83
C(11)	−0·6288 (17)	−0·1408 (3)	−0·6009 (7)	5·26
C(12)	−0·4389 (14)	−0·1967 (3)	−0·5656 (7)	4·14
C(13)	0·2186 (15)	−0·4274 (4)	−0·5231 (6)	4·62

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

O(1)—C(13)	1·227 (8)	O(2)—C(13)	1·318 (9)
N(1)—C(1)	1·320 (6)	N(1)—C(7)	1·362 (6)
N(2)—C(2)	1·352 (7)	N(2)—C(8)	1·350 (8)
C(1)—C(2)	1·444 (8)	C(1)—C(6)	1·439 (7)
C(2)—C(3)	1·426 (8)	C(3)—C(4)	1·352 (8)
C(4)—C(5)	1·40 (1)	C(5)—C(6)	1·348 (8)
C(6)—C(13)	1·52 (1)	C(7)—C(8)	1·423 (8)
C(7)—C(12)	1·442 (9)	C(8)—C(9)	1·416 (7)
C(9)—C(10)	1·382 (9)	C(10)—C(11)	1·42 (1)
C(11)—C(12)	1·357 (9)		
C(1)—N(1)—C(7)	117·7 (5)	C(2)—N(2)—C(8)	116·5 (4)
N(1)—C(1)—C(2)	121·2 (4)	N(1)—C(1)—C(6)	121·7 (5)
C(2)—C(1)—C(6)	117·2 (5)	N(2)—C(2)—C(1)	121·7 (5)
N(2)—C(2)—C(3)	117·8 (5)	C(1)—C(2)—C(3)	120·5 (5)
C(2)—C(3)—C(4)	118·7 (7)	C(3)—C(4)—C(5)	121·7 (7)
C(4)—C(5)—C(6)	121·9 (6)	C(1)—C(6)—C(5)	120·0 (6)
C(1)—C(6)—C(13)	121·3 (6)	C(5)—C(6)—C(13)	118·7 (6)
N(1)—C(7)—C(8)	121·2 (5)	N(1)—C(7)—C(12)	118·3 (6)
C(8)—C(7)—C(12)	120·5 (5)	N(2)—C(8)—C(7)	121·7 (5)
N(2)—C(8)—C(9)	119·1 (6)	C(7)—C(8)—C(9)	119·3 (5)
C(8)—C(9)—C(10)	118·6 (6)	C(9)—C(10)—C(11)	122·4 (6)
C(10)—C(11)—C(12)	120·6 (7)	C(7)—C(12)—C(11)	118·8 (7)
O(1)—C(13)—O(2)	122·0 (7)	O(1)—C(13)—C(6)	120·8 (7)
O(2)—C(13)—C(6)	117·2 (6)		

mated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters on crystal 0·20 × 0·20 × 0·75 mm from least-squares procedure on 25 reflections ($2 \leq \theta \leq 13°$). No absorption correction applied (Sheldrick, 1976). Total of 1605 reflections ($1 \leq \theta \leq 27·5°$) measured in the range $-5 \leq h \leq 5$, $-25 \leq k \leq 0$, $-17 \leq l \leq 2$; some Friedel pairs also included. No significant variation in the net intensities of three reference reflections ($0\bar{2}2$, $1\bar{3}0$, $2\bar{4}\bar{2}$) measured every 3600 s. 1566 unique reflections and 571 satisfied $I \geq 2·5\sigma(I)$. Structure solved by direct methods (Sheldrick, 1986), full-matrix least-squares refinement of 153 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included at their calculated positions except for the carboxyl H atom which was not located (perhaps owing to disorder in the absence of hydrogen-bonding effects). At convergence $R = 0·047$, $wR = 0·045$, $w = [\sigma^2(F) + 0·0058F^2]^{-1}$, $S = 0·74$, $(\Delta/\sigma)_{max} \leq 0·1$, $\Delta\rho_{max} = 0·27$, $\Delta\rho_{min} = -0·37$ e Å^{-3}; no extinction correction. Scattering factors for all atoms given in *SHELX76* (Sheldrick, 1976). All calculations on VAX11/785 computer system. Atomic parameters given in Table 1, bond distances and angles in Table 2,* the numbering scheme used is shown in Fig. 1 and molecular packing in Fig. 2.

Related literature. Spectroscopic studies have led to a controversy concerning the structure of the fungal antibiotic produced by a fluorescent pseudomonad (*P. fluorescens* 2–79, NRRL B-15132). Gurusiddaiah, Weller, Sarkar & Cook (1986) presented evidence favouring the dimeric structure (I) whereas Brisbane *et al.* (1987) provided evidence in favour of the well documented (Haynes, Stodda, Locke, Pridham, Con-

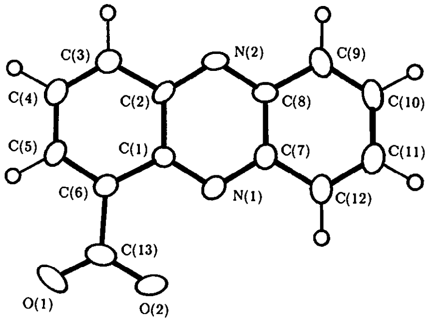


Fig. 1. Molecular structure and numbering scheme for $C_{13}H_8N_2O_2$. H atoms are numbered according to the C atom to which they are bonded (Johnson, 1971).

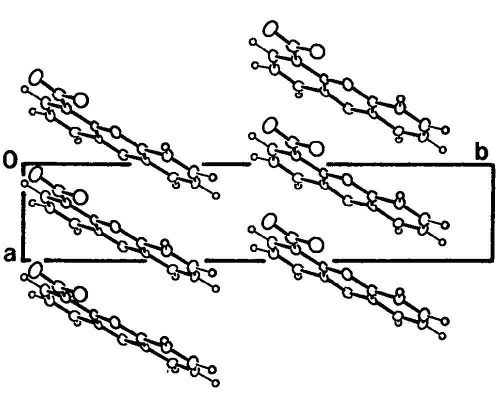
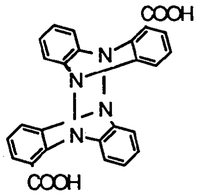


Fig. 2. Unit-cell contents of $C_{13}H_8N_2O_2$ viewed down [001] showing 0 to 0·5c for clarity (Johnson, 1971).

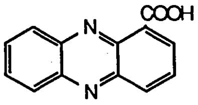
---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51283 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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)

The Australian Research Grants Scheme is thanked for support.

References

Brisbane, P. G., Janik, L. J., Tate, M. E. & Warren, R. F. O. (1987). *Antimicrob. Agents Chemother.* **31**, 1967–1971.

Gurusiddaiah, S., Jois, Y. H., Weller, D. M. & Cook, R. J. (1985). *Chem. Abstr.* **105**, P170629r.

Gurusiddaiah, S., Weller, D. M., Sarkar, A. & Cook, R. J. (1986). *Antimicrob. Agents Chemother.* **29**, 488–495.

Haynes, W. C., Stodda, F. H., Locke, J. M., Pridham, T. G., Conway, H. F., Sohns, V. E. & Jackson, R. W. (1956). *J. Bacteriol.* **72**, 412–417.

Johnson, C. K. (1971). *ORTEPII*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

Phytopathology News (1988). Vol. 22, p. 30. St Paul, Minnesota, USA: American Phytopathological Society.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS86*. A program for the automatic solution of crystal structures. Univ. of Göttingen, Federal Republic of Germany.

Acta Cryst. (1988). C**44**, 2222–2224

Trichlorotricyanobenzene*–Hexamethylbenzene (1/1)

By Doyle Britton

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

(*Received* 6 *April* 1988; *accepted* 12 *July* 1988)

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

---

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

0108-2701/88/122222-03$03.00