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The Structure of Cryptand $2_S.2_O.2_O$*

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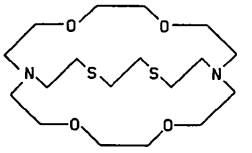
Abstract. $C_{18}H_{36}N_2O_4S_2$, $M_r = 408{\cdot}63$, orthorhombic, *Pbcn*, $a = 13{\cdot}745$ (8), $b = 9{\cdot}105$ (2), $c = 17{\cdot}992$ (4) Å, $V = 2251{\cdot}7$ (1·5) Å^3, $Z = 4$, $D_x = 1{\cdot}205$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 2{\cdot}17$ cm^{-1}, $F(000) = 888$, $T = 298$ (2) K, $R = 0{\cdot}046$ for 1193 unique observed reflections. The molecule possesses an internal crystallographic twofold rotation axis and adopts the *endo–endo* conformation of the nitrogen electron lone pairs. The molecule exhibits the shortest N···N nonbonding distance of any of the [2.2.2] cryptates examined: N···N = 4·613 (3) Å. As for the O and S atoms, only a single lone pair on O2 is directed toward the cavity; all others are directed outward.

Experimental. Colorless thin plates, 0·58 × 0·50 × 0·10 mm, Nonius CAD-4 diffractometer, monochromated Mo $K\alpha$, $\theta/2\theta$ scans, $2 \le 2\theta \le 50°$, lattice parameters from 48 high-angle reflections ($2\theta > 20°$) measured at $\pm 2\theta$, no absorption corrections or corrections for extinction; $0 \le h \le 16$, $0 \le k \le 10$, $0 \le l \le 21$, three standard reflections fluctuated 6%, 2007 total reflections, 1961 unique, 1193 observed with $I_o \ge 2{\cdot}5\sigma(I)$. Direct methods, full-matrix refinement *via* *SHELX*76 (Sheldrick, 1976) on F's minimizing $\sum w(F_o - \|F_c\|)^2$; all nonhydrogen atoms anisotropic, H atoms were placed in calculated geometries and all isotropic H temperature factors were tied to a single variable. For observed reflections $R = 0{\cdot}046$, $wR = 0{\cdot}052$, $S = 1{\cdot}56$, $w = (\sigma_F)^{-2}$. In the final cycle: $(\Delta/\sigma)_{max} = 0{\cdot}01$, $(\Delta\rho)_{max} = 0{\cdot}18$, $(\Delta\rho)_{min} = -0{\cdot}23$ e Å^{-3}. Neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Table 1† gives the atomic coordinates and Table 2 lists interatomic distances and angles. Fig. 1 illustrates the geometry and labeling scheme. A diagram of the title molecule is shown below.



Related literature. The only other $2_S.2_O.2_O$ cryptate structure published contains a Pd^{II} ion coordinated to the S atoms of the cryptate and external to the ligand cavity (Louis, Thierry & Weiss, 1974). The related oxygen-donor cryptand $2_O.2_O.2_O$ has been examined as a free molecule (Metz, Moras & Weiss, 1976) and has

* IUPAC name: 1,10-diaza-4,7,13,16-tetraoxa-21,24-dithiabicyclo[8.8.8]hexacosane.

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51263 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

	x	y	z	U_{eq}* (Å^2)
S	0·45445 (8)	0·14810 (9)	0·13318 (5)	0·0567 (3)
N	0·3973 (2)	0·5881 (3)	0·1486 (1)	0·0391 (9)
O1	0·2745 (2)	0·7586 (3)	0·3082 (1)	0·066 (1)
O2	0·4279 (2)	0·7016 (3)	0·4293 (1)	0·0570 (9)
C1	0·4496 (2)	0·1127 (3)	0·2321 (2)	0·052 (1)
C2	0·4714 (2)	0·3462 (3)	0·1338 (2)	0·048 (1)
C3	0·3770 (2)	0·4291 (4)	0·1489 (2)	0·046 (1)
C4	0·3260 (2)	0·6724 (4)	0·1916 (2)	0·048 (1)
C5	0·3399 (3)	0·6550 (4)	0·2746 (2)	0·059 (1)
C6	0·2660 (3)	0·7482 (5)	0·3860 (2)	0·070 (2)
C7	0·3512 (3)	0·8069 (4)	0·4282 (2)	0·071 (1)
C8	0·5210 (3)	0·7652 (4)	0·4336 (2)	0·053 (1)
C9	0·5958 (2)	0·6457 (4)	0·4274 (2)	0·046 (1)

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

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

S–C1	1·809 (3)	O2–C7	1·426 (4)
S–C2	1·819 (3)	O2–C8	1·407 (4)
N–C3	1·474 (4)	C1–C1′	1·528 (6)
N–C4	1·466 (4)	C2–C3	1·525 (4)
N–C9′	1·466 (4)	C4–C5	1·513 (4)
O1–C5	1·436 (4)	C6–C7	1·494 (5)
O1–C6	1·409 (4)	C8–C9	1·501 (4)
C1–S–C2	100·1 (1)	C2–C3–N	108·9 (3)
C3–N–C4	112·7 (2)	N–C4–C5	112·4 (3)
C3–N–C9′	111·5 (2)	C4–C5–O1	105·5 (3)
C4–N–C9′	110·4 (2)	O1–C6–C7	114·6 (3)
C5–O1–C6	115·3 (3)	C6–C7–O2	110·3 (3)
C7–O2–C8	113·4 (3)	O2–C8–C9	108·7 (3)
C1′–C1–S	112·4 (2)	C8–C9–N′	111·9 (3)
S–C2–C3	112·5 (2)		

Primed atoms are transformed through the twofold rotation operation by $1-x, y, 0{\cdot}5-z$.

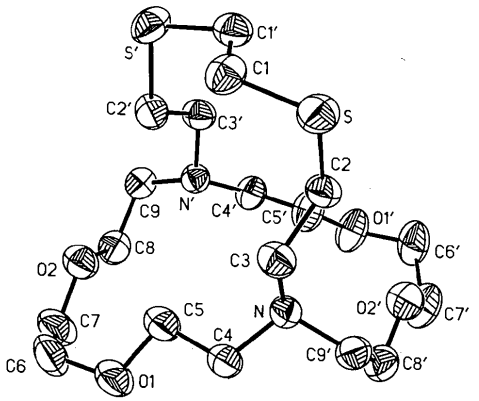

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the $2_S.2_O.2_O$ cryptand. The molecule occupies a twofold rotation axis. Ellipsoids are drawn at 50% probability.

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).

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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.

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

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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-

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