

(ZWO). One of us (AM) is indebted to the Department of Crystal and Structure Chemistry, University of Utrecht, for the provision of the crystallographic equipment and the generous support for this research at their laboratory.

References

- BERG, J. B. VAN DEN, DE RUITER, B. & VAN DE GRAMPPEL, J. C. (1976). *Z. Naturforsch. Teil B*, **31**, 1216–1218.
- BERKOVITCH-YELLIN, Z. & LEISEROWITZ, L. (1984). *Acta Cryst.* **B40**, 159–165.
- BOER, J. L. DE & DUISENBERG, A. J. M. (1984). *Acta Cryst.* **A40**, C410.
- BOLHUIS, F. VAN, VAN DEN BERG, J. B. & VAN DE GRAMPPEL, J. C. (1981). *Cryst. Struct. Commun.* **10**, 1031–1035.
- BONDI, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- CREMER, D. & POPLER, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1979). *Acta Cryst.* **B31**, 221–234.
- DUAX, W. L., WEEKS, C. M. & ROHRER, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by N. L. ALLINGER & E. L. ELIEL, pp. 271–383. New York: John Wiley.
- GRAMPPEL, J. C. VAN DE (1981). *Rev. Inorg. Chem.* **3**, 1–28.
- GRAMPPEL, J. C. VAN DE & VOS, A. (1969). *Acta Cryst.* **B25**, 651–656.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- LE PAGE, Y. (1982). *J. Appl. Cryst.* **15**, 255–259.
- MCCANDLISH, L. E., STOUT, G. H. & ANDREWS, L. C. (1975). *Acta Cryst.* **A31**, 245–249.
- PERALES, A., FAYOS, J., VAN DE GRAMPPEL, J. C. & DE RUITER, B. (1980). *Acta Cryst.* **B36**, 838–841.
- SPEK, A. L. (1982). The *EUCLID* package. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.
- STEWART, J. H. & HALL, S. R. (1983). The *XTAL* system. Tech. Rep. TR-1364. Computer Science Center, Univ. of Maryland, College Park, Maryland.
- TUCKER, P. A. & VAN DE GRAMPPEL, J. C. (1974). *Acta Cryst.* **B30**, 2795–2798.

Acta Cryst. (1986). **C42**, 368–371

Structure of *trans*-5-Isopropyl-1,3-diphenyl-5-propyl-1 λ^6 ,3 λ^6 ,2,4,6,5 λ^5 -dithiatriazaphosphorine 1,3-Dioxide

BY A. MEETSMA

Molecular Structure Department, University of Groningen, Nijenborgh 16, 9747 AG Groningen, The Netherlands

A. L. SPEK*

Vakgroep Algemene Chemie, afdeling Kristal- en Structuurchemie, University of Utrecht, Padualaan 8, 3508 TB Utrecht, The Netherlands

AND H. WINTER, J. C. VAN DE GRAMPPEL AND J. L. DE BOER

Department of Inorganic Chemistry, University of Groningen, Nijenborgh 16, 9747 AG Groningen, The Netherlands

(Received 11 July 1985; accepted 23 October 1985)

Abstract. C₁₈H₂₄N₃O₂PS₂, $M_r = 409.5$, monoclinic, $P2_1/n$, $a = 13.432$ (6), $b = 16.727$ (4), $c = 9.602$ (2) Å, $\beta = 110.21$ (3)°, $V = 2025$ (1) Å³, $Z = 4$, $D_x = 1.343$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 3.5$ cm⁻¹, $F(000) = 864$, $T = 295$ K, $R = 0.053$ for 1628 observed reflections. The inorganic ring possesses a twist-boat conformation with the phenyl substituents in *trans* positions. Mean bond lengths are N–P = 1.608 (3), N–S (from segment SNS) = 1.568 (4), N–S (from segment PNS) = 1.544 (3) Å. The average endocyclic angles at S and N are 114.3 (2) and 124.4 (5)° respectively. The endocyclic angle at P amounts to 113.0 (2)°.

Introduction. To gather insights into the characteristics of cyclothiaphosphazenes we prepared a series of compounds, *trans*-NPRR'(NSOPh)₂. The N–S and N–P bonds within the ring skeleton are supposed to be strengthened by d_π – p_π interactions (van de GramppeL, 1981). The substituents R and R' most probably influence the contributions of 'π bonding' and hence the overall binding properties of these ring systems. Therefore it would be interesting to determine the bond lengths and angles of *trans*-NPRR'(NSOPh)₂ while varying R and R' along the electronegativity scale. The crystal and molecular structure of *trans*-NPRR'(NSOPh)₂ with two electron-withdrawing substituents ($R = R' = \text{Cl}$) is known (van Bolhuis, van den Berg & van de GramppeL, 1981), whereas the structure

* Author to whom correspondence should be addressed.

of *trans*-NPIⁱPr(NSOPh)₂ has been investigated recently (Meetsma, Spek, Olthof-Hazekamp, Winter, van de Grampsel & de Boer, 1985). In order to extend this series we now report the crystal and molecular structure of the title compound, which incorporates two electron-releasing substituents. This compound was prepared by reaction of *trans*-NPCl₂(NSOPh)₂ with (Bu₃PCuI)₄/ⁱPrMgCl and PrI according to a published procedure (Allcock, Harris & Connolly, 1981).

Experimental. Colorless crystals obtained by recrystallization from diethyl ether. Enraf-Nonius CAD-4F diffractometer, graphite-monochromated Mo K α radiation; crystal 0.25 × 0.23 × 0.12 mm glued on top of a glass fiber. Total 5051 reflections; $\omega/2\theta$ scan; $\Delta\omega = (0.90 + 0.35 \text{ tg } \theta)^\circ$; $1.2 < \theta < 22^\circ$; $h 0 \rightarrow 14$, $k -17 \rightarrow 17$, $l -9 \rightarrow 10$. Three reference reflections measured every 2 h ($1\bar{3}\bar{1}$: r.m.s.d. 0.4%; $0\bar{2}\bar{2}$: r.m.s.d. 0.6%; $0\bar{2}\bar{2}$: r.m.s.d. 0.4%) showed no decay during the 40.5 h of X-ray exposure time. Cell dimensions from setting angles of 15 reflections ($12.0 < \theta < 22^\circ$). A 360° ψ scan for the reflection $5\bar{1}\bar{1}$ showed an intensity variation up to 10% about the mean. Intensities corrected for Lorentz and polarization effects, but not for absorption. Variance $\sigma^2(I)$ calculated based on counting statistics plus a term $(PI)^2$, where $P (= 0.005)$ is the instability constant as derived from the excess variance in the reference reflections (McCandlish, Stout & Andrews, 1975). 2475 unique reflections ($R_{\text{int}} = 0.046$). Structure solved by direct methods using GENTAN (Stewart & Hall, 1983) and Fourier methods. Full-matrix least-squares refinement procedures minimizing the function $\sum w(|F_o| - |F_c|)^2$ used. Anisotropic thermal parameters for non-H atoms. H atoms introduced at positions 1.0 Å from their carrier atoms, assuming sp^2 or sp^3 hybridization, and refined in the riding mode with one overall isotropic temperature factor. Convergence reached at $R = 0.053$, $wR = 0.031$; $w = 1/\sigma^2(F)$; $S = 0.86$; 1628 observed reflections with $I > 2.5\sigma(I)$; 236 refined parameters; maximum $\Delta/\sigma = 0.02$. Minimum and maximum residual densities in final difference Fourier map -0.41 and $0.44 \text{ e } \text{Å}^{-3}$ in neighborhood of C(17) and C(18), indicating some disorder. The final values of the refined parameters are given in Table 1.* Scattering factors from Cromer & Mann (1968). Anomalous dispersion from Cromer & Liberman (1970). Calculations carried out on the Cyber 180-855 of the University of Utrecht Computer Center with program packages XTAL (Stewart & Hall, 1983) and EUCLID (Spek, 1982;

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H and an ORTEP plot (Johnson, 1965) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42593 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic coordinates and equivalent isotropic thermal parameters and their e.s.d.'s in parentheses

	x	y	z	$U_{\text{eq}}^*(\text{Å}^2)$
S(1)	0.2032 (1)	0.1245 (1)	0.9649 (2)	0.0590 (6)
S(2)	0.2104 (1)	0.1116 (1)	0.6794 (2)	0.0558 (6)
P(1)	0.0928 (1)	0.0024 (1)	0.7790 (2)	0.0563 (6)
O(1)	0.1468 (3)	0.1881 (3)	1.0056 (5)	0.099 (2)
O(2)	0.3016 (3)	0.0875 (2)	0.6439 (4)	0.081 (2)
N(1)	0.1475 (3)	0.0422 (3)	0.9402 (4)	0.062 (2)
N(2)	0.2433 (4)	0.1511 (3)	0.8369 (5)	0.077 (2)
N(3)	0.1244 (3)	0.0464 (2)	0.6515 (4)	0.056 (2)
C(1)	0.3217 (4)	0.1110 (4)	1.1173 (6)	0.053 (3)
C(2)	0.3542 (5)	0.1724 (4)	1.2170 (7)	0.077 (3)
C(3)	0.4497 (6)	0.1651 (6)	1.3330 (8)	0.111 (5)
C(4)	0.5048 (7)	0.0958 (6)	1.349 (1)	0.135 (5)
C(5)	0.4744 (7)	0.0353 (5)	1.248 (1)	0.141 (5)
C(6)	0.3805 (5)	0.0440 (4)	1.1285 (8)	0.102 (3)
C(7)	0.1495 (4)	0.1886 (3)	0.5530 (6)	0.045 (2)
C(8)	0.1713 (4)	0.1954 (3)	0.4252 (7)	0.055 (2)
C(9)	0.1190 (5)	0.2521 (4)	0.3196 (7)	0.070 (3)
C(10)	0.0464 (6)	0.3010 (4)	0.3459 (8)	0.080 (3)
C(11)	0.0237 (5)	0.2933 (4)	0.4737 (9)	0.088 (4)
C(12)	0.0755 (5)	0.2369 (4)	0.5817 (7)	0.072 (3)
C(13)	-0.0486 (4)	0.0036 (4)	0.7300 (6)	0.068 (3)
C(14)	-0.0961 (4)	0.0865 (4)	0.7001 (7)	0.084 (3)
C(15)	-0.2155 (4)	0.0807 (4)	0.6601 (7)	0.114 (4)
C(16)	0.1124 (5)	-0.1448 (4)	0.9157 (6)	0.101 (3)
C(17)	0.1270 (5)	-0.1024 (4)	0.7826 (8)	0.092 (3)
C(18)	0.2237 (6)	-0.1164 (4)	0.7624 (8)	0.157 (5)

$$* U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

calculation of geometric data and preparation of illustrations including an extended version of the program PLUTO).

Discussion. Fig. 1 gives a view of the title compound including the adopted numbering scheme. Selected data on the geometry are given in Table 2. The monoclinic unit cell contains four discrete molecules (Fig. 2). The observed conformation of the PNS ring approaches that of a twist-boat (Cremer & Pople, 1975) with torsion angles ranging from -23.4 (4) to 5.3 (5) $^\circ$ and lowest asymmetry-parameter value $\Delta C_2 = 2.5$ (5) $^\circ$ for the bonds S(1)–N(1) and N(3)–S(2) (Duax, Weeks & Rohrer, 1976). The puckering parameters are: $Q = 0.242$ (3) Å, $\theta = 82.5$ (9), $\varphi = 27.0$ (9) $^\circ$. P and S atoms show distorted tetrahedral geometry. The P–C bond lengths, P(1)–C(13) = 1.792 (6) and P(1)–C(17) = 1.809 (7) Å, are in agreement with the values reported on related structures; the exocyclic angle C(13)–P(1)–C(17) of 104.8 (3) $^\circ$ is comparable with the values observed in related cyclophosphazenes (Ritchie, Harris & Allcock, 1980). The C–C bonds in the propyl substituents are in the range to be expected for sp^3 – sp^3 bonds (Lide, 1962), except for the C(17)–C(18) distance of 1.40 (1) Å. The shortening of this bond is probably due to some positional disorder of the C(18) atom. Both phenyl rings display normal geometry; average C–C distances are 1.373 (6)* and

* Throughout this paper the mean values and their standard errors have been calculated using the formulae given by Domenicano, Vaciago & Coulson (1979).

1.368(7) Å respectively. The angles between the phenyl planes and the related NSN-segment plane are 83.1(4) and 85.8(4)° respectively. The angle between the bisector of the angle O(1)–S(1)–C(1) and the plane segment N(1)–S(1)–N(2) is 9.8(3)°; for the S(2)O(2)C(7) moiety this angle equals 8.5(3)°. The torsion angle O(1)–S(1)–C(1)–C(2) is –19.2(6)° whereas the torsion angle O(2)–S(2)–C(7)–C(8) equals 20.0(5)°. Short C–H...O bonds (Berkovitch-Yellin & Leiserowitz, 1984) are O(1)...H(9) ($x, y, 1+z$) = 2.460(8), O(2)...H(5)($1-x, -y, 2-z$) = 2.58(1) Å (sum of the van der Waals radii is 2.72 Å) and N(3)...H(131)($-x, -y, 1-z$) = 2.66(1) Å [sum of the van der Waals radii is 2.75 Å (Bondi, 1964)]. As found in the related structures NPIⁿPr(NSOPh)₂ (Meetsma *et al.*, 1985) and *cis*-NPCI₂(NSOPh)₂ (Meetsma, Spek, Winter, Cnossen-Voswijk, van de Grampel & de Boer, 1986), the sequence of the bond lengths of the inorganic ring skeleton reflects the difference in electronegativity of the phosphorus and sulfur centers. All endocyclic bond lengths observed are smaller than their single-bond values (Sass, 1960; Cruickshank, 1964).

The investigation was supported in part (ALS, HW) by the Netherlands Foundation for Chemical Research (SON) with financial aid from the Netherlands Organization for Advancement of Pure Research (ZWO). One of us (AM) is indebted to the Department

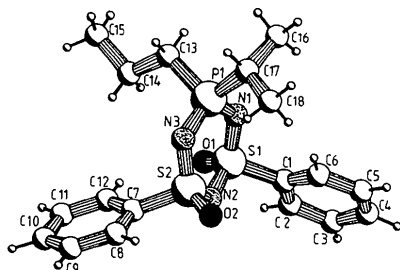


Fig. 1. PLUTO (EUCLID version) drawing of *trans*-NPⁿPr^mPr(NSOPh)₂ illustrating the puckering and the adopted numbering scheme.

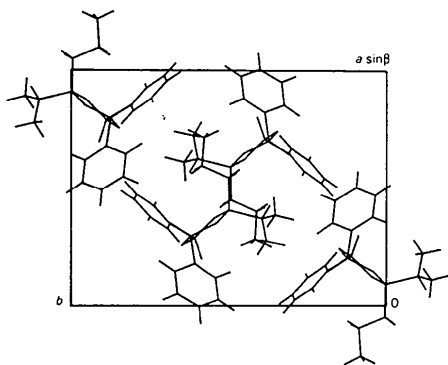


Fig. 2. Projection of the structure down the *c* axis.

Table 2. Data on the geometry of *trans*-NPⁿPr(NSOPh)₂

Bond distances (Å)			
P(1)–N(1)	1.610 (4)	C(2)–C(3)	1.38 (1)
P(1)–N(3)	1.606 (4)	C(3)–C(4)	1.36 (1)
P(1)–C(13)	1.792 (6)	C(4)–C(5)	1.36 (1)
P(1)–C(17)	1.809 (7)	C(5)–C(6)	1.39 (1)
S(1)–N(1)	1.545 (5)	C(7)–C(8)	1.361 (8)
S(1)–N(2)	1.567 (5)	C(7)–C(12)	1.381 (9)
S(1)–O(1)	1.436 (5)	C(8)–C(9)	1.389 (9)
S(1)–C(1)	1.765 (6)	C(9)–C(10)	1.36 (1)
S(2)–N(2)	1.568 (5)	C(10)–C(11)	1.37 (1)
S(2)–N(3)	1.543 (4)	C(11)–C(12)	1.40 (1)
S(2)–O(2)	1.438 (4)	C(13)–C(14)	1.512 (9)
S(2)–C(7)	1.765 (6)	C(14)–C(15)	1.518 (8)
C(1)–C(2)	1.369 (9)	C(16)–C(17)	1.532 (9)
C(1)–C(6)	1.354 (9)	C(17)–C(18)	1.40 (1)
Bond angles (°)			
O(1)–S(1)–N(1)	115.4 (3)	S(1)–C(1)–C(2)	117.5 (5)
O(1)–S(1)–N(2)	111.1 (3)	S(1)–C(1)–C(6)	120.4 (5)
O(1)–S(1)–C(1)	106.2 (3)	C(2)–C(1)–C(6)	122.0 (6)
N(1)–S(1)–N(2)	114.3 (3)	C(1)–C(2)–C(3)	118.7 (7)
N(1)–S(1)–C(1)	105.3 (3)	C(2)–C(3)–C(4)	119.0 (8)
N(2)–S(1)–C(1)	103.3 (3)	C(3)–C(4)–C(5)	122.5 (9)
O(2)–S(2)–N(2)	111.6 (3)	C(4)–C(5)–C(6)	118.3 (8)
O(2)–S(2)–N(3)	114.1 (2)	C(1)–C(6)–C(5)	119.4 (7)
O(2)–S(2)–C(7)	105.8 (3)	S(2)–C(7)–C(12)	119.0 (4)
N(2)–S(2)–N(3)	114.2 (3)	S(2)–C(7)–C(8)	119.2 (4)
N(2)–S(2)–C(7)	105.4 (3)	C(8)–C(7)–C(12)	121.7 (5)
N(3)–S(2)–C(7)	104.8 (2)	C(7)–C(8)–C(9)	120.2 (5)
N(1)–P(1)–N(3)	113.0 (2)	C(8)–C(9)–C(10)	119.3 (6)
N(1)–P(1)–C(13)	109.8 (3)	C(9)–C(10)–C(11)	120.4 (7)
N(1)–P(1)–C(17)	110.7 (3)	C(10)–C(11)–C(12)	121.4 (7)
N(3)–P(1)–C(13)	109.3 (3)	C(7)–C(12)–C(11)	117.1 (6)
N(3)–P(1)–C(17)	109.0 (3)	P(1)–C(13)–C(14)	113.6 (4)
C(13)–P(1)–C(17)	104.8 (3)	C(13)–C(14)–C(15)	109.2 (5)
S(1)–N(1)–P(1)	123.5 (3)	P(1)–C(17)–C(16)	111.1 (5)
S(1)–N(2)–S(2)	125.3 (4)	C(16)–C(17)–C(18)	115.5 (6)
S(2)–N(3)–P(1)	124.8 (3)	P(1)–C(17)–C(18)	113.7 (5)
Torsion angles (°)			
N(2)–S(1)–N(1)–P(1)	–23.4 (4)	N(3)–P(1)–N(1)–S(1)	13.1 (4)
N(1)–S(1)–N(2)–S(2)	13.9 (5)	N(1)–P(1)–N(3)–S(2)	8.8 (4)
O(1)–S(1)–C(1)–C(2)	–19.2 (6)	P(1)–C(13)–C(14)–C(15)	–179.9 (4)
N(3)–S(2)–N(2)–S(1)	5.3 (5)	C(13)–P(1)–C(17)–C(18)	156.9 (5)
N(2)–S(2)–N(3)–P(1)	–17.2 (4)	C(17)–P(1)–C(13)–C(14)	–172.1 (4)
O(2)–S(2)–C(7)–C(8)	20.0 (5)	C(13)–P(1)–C(17)–C(16)	–70.8 (5)

of Crystal and Structure Chemistry, University of Utrecht, for the support of this research at their laboratory.

References

- ALLCOCK, H. R., HARRIS, P. J. & CONNOLLY, M. S. (1981). *Inorg. Chem.* **20**, 11–16.
- BERKOVITCH-YELLIN, Z. & LEISEROWITZ, L. (1984). *Acta Cryst.* **B40**, 159–165.
- BOLHUIS, F. VAN, VAN DEN BERG, J. B. & VAN DE GRAMPSEL, J. C. (1981). *Cryst. Struct. Commun.* **10**, 1031–1035.
- BONDI, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- CREMER, D. & POPLI, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- CRUICKSHANK, D. W. J. (1964). *Acta Cryst.* **17**, 671–672.
- DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1979). *Acta Cryst.* **B31**, 221–234.
- DUAX, W. L., WEEKS, C. M. & ROHRER, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by N. L. ALLINGER & E. L. ELIEL, pp. 271–383. New York: John Wiley.
- GRAMPSEL, J. C. VAN DE (1981). *Rev. Inorg. Chem.* **3**, 1–28.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

- LIDE, D. R. (1962). *Tetrahedron*, **17**, 125–134.
 MCCANDLISH, L. E., STOUT, G. H. & ANDREWS, L. C. (1975). *Acta Cryst.* **A31**, 245–249.
 MEETSMA, A., SPEK, A. L., OLTHOF-HAZEKAMP, R., WINTER, H., VAN DE GRAMPPEL, J. C. & DE BOER, J. L. (1985). *Acta Cryst.* **C41**, 1801–1804.
 MEETSMA, A., SPEK, A. L., WINTER, H., CNOSSEN-VOSWIJK, C., VAN DE GRAMPPEL, J. C. & DE BOER, J. L. (1986). *Acta Cryst.* **C42**, 365–368.
 RITCHIE, R. J., HARRIS, P. J. & ALLCOCK, H. R. (1980). *Inorg. Chem.* **19**, 2483–2486.
 SASS, R. L. (1960). *Acta Cryst.* **13**, 320–323.
 SPEK, A. L. (1982). The *EUCLID* package. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.
 STEWART, J. H. & HALL, S. R. (1983). The *XTAL* system. Tech. Rep. TR-1364. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1986). **C42**, 371–373

Structure of 6,14,17,22,25-Pentaoxa-1,3,9,11-tetraazabicyclo[9.8.8]heptacosane-2,10-dithione

BY YU. A. SIMONOV, A. A. DVORKIN AND T. I. MALINOWSKY

Institute of Applied Physics, Academy of Sciences of the Moldavian SSR, Kishinev-277028, Grosul str. 5, USSR

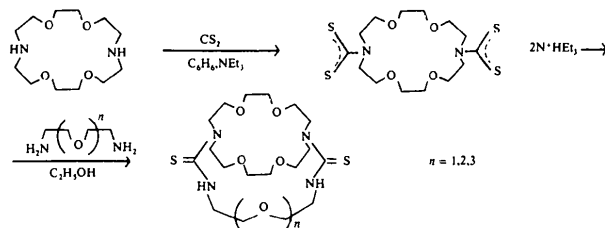
AND A. V. BOGATSKY, N. G. LUKYANENKO, T. I. KIRITCHENKO AND S. V. SCHERBAKOV

Physico-Chemical Institute, Academy of Sciences of the Ukrainian SSR, Odessa-2770080, Chernomorskaya doroga 86, USSR

(Received 2 July 1985; accepted 5 November 1985)

Abstract. $C_{18}H_{34}N_4O_5S_2$, $M_r = 450.61$, orthorhombic, *Pbca*, $a = 16.714(4)$, $b = 17.254(4)$, $c = 15.526(3)$ Å, $V = 4477.4(4)$ Å³, $Z = 8$, $D_x = 1.337$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 2.28$ cm⁻¹, $F(000) = 1936$, $T = 293$ K. Final $R = 0.058$ for 1947 observed reflections. In the cryptand [3_{NON},2_O,2_O] the conformation of the diaza-18-crown-6 fragment is described by the sequence of $g^-,g^+,g^-,t,g^+,t,g^+,g^-,g^-,g^+,g^+,g^-,g^-,t,g^+,g^-$ rotamers. Both thiourea fragments have a *cis* configuration, which is stabilized by N–H...O intramolecular hydrogen bonds of 2.830 and 2.825 Å. Distances between neighbouring molecules are equal to or exceed van der Waals radii.

Introduction. The reaction of 1,10-diaza-18-crown-6 with carbon disulfide in benzene gives dithiocarbamate salts in the presence of triethylamine. Boiling the salt with 1,5-diamino-3-oxapentane in ethanol gave 6,14,17,22,25-pentaoxa-1,3,9,11-tetraazabicyclo[9.8.8]-heptacosane-2,10-dithione (cryptand [3_{NON},2_O,2_O]) (Lukyanenko, Bogatsky, Kiritchenko, Scherbakov & Nazarova, 1984).



Cationic selectivity of this cryptand in relation to alkaline metal ions is unusual, if it is compared with the traditional cryptands. It can be explained by the effect of the thiourea fragments (Lukyanenko *et al.*, 1984). The study of cryptand [3_{NON},2_O,2_O], investigated by X-ray methods, is presented in this paper. Colourless crystals of this compound were obtained by slow crystallization of a diluted acetone solution.

Experimental. Full set of integral intensity data obtained within angle range $2 < 2\theta < 50^\circ$ from a single crystal of prismatic habit with dimensions $0.4 \times 0.5 \times 0.5$ mm. Equi-inclined DAR-UMB automatic diffractometer operated by M-6000 computer, graphite-monochromated Mo *K* α radiation, used to obtain experimental data by combined ω - $\theta/2\theta$ scanning with constant rate 8° min^{-1} . 2346 reflections recorded, 1947 independent with $I(hkl) > 3\sigma(I)$ used for calculations (h 0–20, k 0–20, l 0–16, 17 standard reflections, 5% intensity variation, three reflections used for measuring lattice parameters). Corrections made for Lorentz and polarization effects. Absorption and extinction corrections neglected. Structure solved by direct methods with *YANX* (Gerr, Yanovsky & Struchkov, 1983). On first *E* map all non-H atoms located. Refinement of atomic coordinates by full-matrix LS carried out taking into account both anisotropy for non-H atoms and isotropic vibrations for H atoms, located on difference Fourier map. Final *R* index of 0.058 obtained with unit weights. Remaining peaks within 0.45 and $-0.28 \text{ e } \text{Å}^{-3}$, $(\Delta/\sigma)_{\text{max}} = 0.01$;