We thank the SERC for support and for a research studentship (PWF).

References

BAU, R., CARROLL, W. E., HART, D. W., TELLER, R. G. & KOETZLE, T. F. (1978). *Adv. Chem. Ser.* **167**, 73–92.

BAUDRY, D., EPHRITIKHINE, M. & FELKIN, H. (1980). *J. Chem. Soc. Chem. Commun.* pp. 1243–1244.

BAUDRY, D., EPHRITIKHINE, M. & FELKIN, H. (1982). *J. Organomet. Chem.* **224**, 363–376.

CHAUDRET, B., COMMENGES, G. & POILBLANC, R. (1982). *J. Chem. Soc. Chem. Commun.* pp. 1388–1390.

CHAUDRET, B., DEVILLERS, J. & POILBLANC, R. (1983). *J. Chem. Soc. Chem. Commun.* pp. 641–643.

CONNELLY, N. G., HOWARD, J. A. K., SPENCER, J. L. & WOODLEY, P. K. (1984). *J. Chem. Soc. Dalton Trans.* In the press.

CRABTREE, R. H. & HLATKY, G. G. (1982). *J. Organomet. Chem.* **238**, C21–C23.

DOUGLAS, P. G. & SHAW, B. L. (1970). *J. Chem. Soc.* (*A*), pp. 334–338.

GINSBERG, A. P., ABRAHAMS, S. C. & JAMIESON, P. B. (1973). *J. Am. Chem. Soc.* **95**, 4751–4752.

GIROLAMI, G. S., SALT, J. E., WILKINSON, G., THORNTON-PETT, M. & HURSTHOUSE, M. B. (1983). *J. Am. Chem. Soc.* **105**, 5954–5956.

GREEN, M. A., HUFFMAN, J. C. & CAULTON, K. E. (1983). *J. Organomet. Chem.* **243**, C78–C82.

GREGSON, D., HOWARD, J. A. K., NICHOLLS, J. N., SPENCER, J. L. & TURNER, D. G. (1980). *J. Chem. Soc. Chem. Commun.* pp. 572–573.

HOARD, J. L. & SILVERTON, J. V. (1963). *Inorg. Chem.* **2**, 235–243.

HOWARD, J. A. K., GREGSON, D., SPENCER, J. L. & TURNER, D. G. (1981). *Acta Cryst.* A**37**, C240.

HOWARD, J. A. K., MEAD, K. A. & SPENCER, J. L. (1983). *Acta Cryst.* C**39**, 555–559.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

KEPERT, D. L. (1982). *Inorganic Stereochemistry.* Berlin: Springer-Verlag.

LOBKOVSKII, E. B., BORISOV, A. P., MAKHAEV, V. D. & SEMENENKO, K. N. (1980). *Zh. Strukt. Khim.* **21**, 126–130; Engl. Transl: *J. Struct. Chem.* **21**, 100–103.

LOBKOVSKII, E. B., MAKHAEV, V. D., BORISOV, A. P. & SEMENENKO, K. N. (1979). *Zh. Strukt. Khim.* **20**, 944–946; Engl. Transl: *J. Struct. Chem.* **20**, 812–813.

MEAKIN, P., GUGGENBERGER, L. J., PEET, W. G., MUETTERTIES, E. L. & JESSON, J. P. (1973). *J. Am. Chem. Soc.* **95**, 1467–1474.

ORIOLI, P. L. & VASKA, L. (1962). *Proc. Chem. Soc.* p. 333.

SHELDRICK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.

TELLER, R. G., CARROLL, W. E. & BAU, R. (1983). Private communication.

TOLMAN, C. A. (1977). *Chem. Rev.* **77**, 313–348.

Acta Cryst. (1984). C**40**, 949–951

Structure of Bis(2-methyl-1,2-propanediamine-*N*,*N*′)bis(trichloroacetato-*O*)nickel(II), $[Ni(C_2Cl_3O_2)_2(C_4H_{12}N_2)_2]$

BY S. GARCÍA-GRANDA AND F. GÓMEZ-BELTRÁN

Departamento de Química Física, Facultad de Química, Universidad de Oviedo, Calvo Sotelo S/N, Oviedo, Spain

(*Received* 24 *November* 1983; *accepted* 31 *January* 1984)

Abstract. $M_r = 559{\cdot}8$, monoclinic, $P2_1/n$, $a = 7{\cdot}3163\ (8)$, $b = 16{\cdot}198\ (1)$, $c = 9{\cdot}943\ (4)$ Å, $\beta = 95{\cdot}44\ (1)°$, $V = 1173\ (1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}58$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}54$ mm^{-1}, $F(000) = 572$, $T = 298$ K, final $R = 0{\cdot}060$ for 1481 reflexions. The central Ni atom is octahedrally coordinated by four N atoms in a plane and two trichloroacetate ions in *trans* positions. The bond distances and angles of the ligands are as expected. The large anisotropy in the Cl-atom thermal parameters indicates some disorder in the $-CCl_3$ group.

Introduction. As a continuation of our investigations of the structure of the Ni and Co transition-metal complexes of ethylenediamines and C-substituted ethylenediamines (Alcalá Aranda, Fernández, Gómez Beltrán & Larena, 1977; Gómez Beltrán, Valero Capilla & Alcalá Aranda, 1978, 1979; Valero Capilla, Alcalá Aranda & Gómez Beltrán, 1980), the crystal structure of another compound of this series is communicated herein.

Experimental. Blue crystal 0·1 × 0·17 × 0·35 mm, Nonius CAD-4-F automatic diffractometer, cell dimensions by least-squares fitting of the θ values of 25 reflexions, intensities collected by $\omega/2\theta$ scans, no appreciable drop in intensity of 3 standard reflexions checked every hour; 3451 reflexions within $0 < \theta < 29°$ and hkl range from $-10,0,0$ to $10,22,13$; 1481 with $I > 3\sigma(I)$ used in subsequent calculations; intensities corrected for Lorentz and polarization effects, not for absorption ($\mu R = 0{\cdot}54$); scattering factors for neutral atoms and anomalous-dispersion corrections for Ni and Cl from *International Tables for X-ray Crystallography* (1974); statistical test showed a centric intensity distribution; structure solved using

CRYSTAN (Burzlaff, Böhme & Gomm, 1977) by three-dimensional Patterson and standard Fourier techniques; H atoms located by ΔF synthesis; full-matrix least-squares refinement on F using anisotropic thermal parameters for all non-hydrogen atoms and isotropic ones for H atoms; final $R = 0{\cdot}060$, $R_w = 0{\cdot}051$, $w = 1/\sigma^2(F_o)$; av. and max. Δ/σ in final LS cycle 0·17 and 0·31, final difference synthesis had no electron density $> 0{\cdot}45$ and $< -0{\cdot}42$ e Å^{-3}. Atomic parameters are listed in Table 1.*

Discussion. Interatomic distances and angles for the title compound are listed in Table 2. Fig. 1 (Johnson, 1965) shows the geometry of the molecule and the atom labelling. As was expected, the Ni environment is pseudo-octahedral, with the trichloroacetate ligands in *trans* positions. The Ni—N distances are 2·080 (4) and 2·063 (5) Å. The N(1)—Ni—N(2) angle is 82·5 (2)°.

The large anisotropy of the thermal parameters of the Cl atoms suggests a slight disorder in the $-CCl_3$ group.

Angles between the best least-squares planes for various portions of the molecule, principal torsion angles and geometrical features of the hydrogen bonds, computed by *PARST* 5 (Nardelli, Musatti, Domiano & Andreetti, 1965), have been deposited.*

Most of the calculations were carried out on the HP-1000-F minicomputer of the Computing Center of the University of Oviedo.

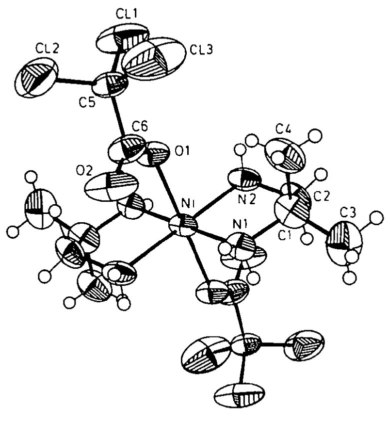


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule, showing the atom numbering.

Table 1. *Coordinates and isotropic thermal parameters* (Å^2 × 10^3; *for* H Å^2 × 10^2)

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

	x	y	z	U_{eq}/U_{iso}
Ni	0·00000 (1)	0·00000 (1)	0·00000 (1)	384 (4)
Cl(1)	−0·1840 (3)	0·27160 (12)	0·1365 (3)	1038 (12)
Cl(2)	−0·4301 (4)	0·20133 (13)	0·3051 (3)	1184 (13)
Cl(3)	−0·5366 (4)	0·22222 (17)	0·0291 (3)	1396 (15)
O(1)	−0·1354 (5)	0·1045 (2)	0·0673 (4)	509 (16)
O(2)	−0·3897 (6)	0·0524 (2)	0·1388 (6)	824 (23)
N(1)	0·2038 (6)	0·0300 (3)	0·1525 (5)	449 (19)
N(2)	−0·0949 (6)	−0·0714 (3)	0·1506 (5)	500 (20)
C(1)	0·1953 (7)	−0·0211 (4)	0·2753 (7)	583 (28)
C(2)	−0·0094 (8)	−0·0417 (4)	0·2829 (7)	578 (27)
C(3)	0·2659 (10)	0·0254 (5)	0·3989 (7)	833 (36)
C(4)	0·3045 (9)	−0·0989 (5)	0·2598 (8)	750 (33)
C(5)	−0·3510 (9)	0·1965 (4)	0·1475 (7)	589 (27)
C(6)	−0·2873 (7)	0·1078 (4)	0·1132 (6)	488 (24)
H1[N(1)]	0·186 (7)	0·076 (3)	0·184 (6)	26 (18)†
H2[N(1)]	0·301 (11)	0·036 (5)	0·126 (8)	89 (35)†
H1[N(2)]	−0·231 (9)	−0·071 (4)	0·147 (6)	58 (23)†
H1[C(2)]	−0·074 (6)	0·019 (4)	0·313 (5)	30 (19)†
H2[N(2)]	−0·088 (6)	−0·117 (3)	0·140 (5)	19 (12)†
H2[C(2)]	−0·019 (7)	−0·080 (3)	0·348 (5)	21 (14)†
H1[C(3)]	0·183 (9)	0·079 (4)	0·419 (7)	81 (25)†
H2[C(3)]	0·269 (8)	−0·012 (4)	0·476 (6)	44 (18)†
H3[C(3)]	0·353 (10)	0·045 (4)	0·415 (7)	79 (27)†
H1[C(4)]	0·323 (8)	−0·131 (5)	0·333 (7)	65 (21)†
H2[C(4)]	0·263 (7)	−0·123 (4)	0·179 (6)	25 (16)†
H3[C(4)]	0·446 (9)	−0·094 (5)	0·238 (7)	80 (26)†

† U_{iso}.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Ni—O(1)	2·103 (4)	O(2)—C(6)	1·212 (7)
Ni—N(1)	2·080 (4)	N(1)—C(1)	1·481 (8)
Ni—N(2)	2·063 (5)	N(2)—C(2)	1·482 (8)
Cl(1)—C(5)	1·734 (6)	C(1)—C(2)	1·542 (8)
Cl(2)—C(5)	1·723 (8)	C(1)—C(3)	1·491 (9)
Cl(3)—C(5)	1·760 (7)	C(1)—C(4)	1·507 (9)
O(1)—C(6)	1·242 (7)	C(5)—C(6)	1·558 (8)
N(1)—Ni—N(2)	82·5 (2)	C(2)—C(1)—C(3)	109·3 (5)
O(1)—Ni—N(2)	91·1 (2)	C(3)—C(1)—C(4)	111·1 (5)
O(1)—Ni—N(1)	84·7 (2)	Cl(1)—C(5)—Cl(2)	108·9 (4)
Ni—O(1)—C(6)	128·0 (4)	Cl(1)—C(5)—Cl(3)	107·2 (3)
Ni—N(1)—C(1)	112·8 (3)	Cl(2)—C(5)—Cl(3)	107·0 (4)
Ni—N(2)—C(2)	108·6 (3)	Cl(1)—C(5)—C(6)	113·9 (4)
N(1)—C(1)—C(2)	106·1 (5)	Cl(2)—C(5)—C(6)	111·9 (4)
N(1)—C(1)—C(3)	111·1 (5)	Cl(3)—C(5)—C(6)	107·4 (4)
N(1)—C(1)—C(4)	108·6 (5)	O(1)—C(6)—C(5)	114·6 (4)
N(2)—C(2)—C(1)	110·9 (5)	O(2)—C(6)—C(5)	115·7 (5)
C(2)—C(1)—C(4)	110·5 (5)	O(1)—C(6)—O(2)	129·7 (5)

The authors wish to thank Dr E. Gutiérrez-Puebla, Inorganic Chemistry Department, Complutense University of Madrid, for the measurement of the intensities and Miss R. Diaz-Fernández for providing the crystals.

References

Alcalá Aranda, R., Fernández, J., Gómez Beltrán, F. & Larena, A. (1977). *Cryst. Struct. Commun.* **6**, 635–638.

Burzlaff, H., Böhme, R. & Gomm, M. (1977). *CRYSTAN*. Univ. of Erlangen, German Federal Republic.

---

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving hydrogen, least-squares-planes' calculations, principal torsion angles and hydrogen-bonding parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39243 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Gómez Beltrán, F., Valero Capilla, A. & Alcalá Aranda, R. (1978). *Cryst. Struct. Commun.* **7**, 153–157, 255–258.
Gómez Beltrán, F., Valero Capilla, A. & Alcalá Aranda, R. (1979). *Cryst. Struct. Commun.* **8**, 87–91.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
Nardelli, M., Musatti, A., Domiano, P. & Andreetti, G. D. (1965). *Ric. Sci. Parte* 2 *Sez. A*. **8**, 807–810.
Valero Capilla, A., Alcalá Aranda, R. & Gómez Beltrán, F. (1980). *Cryst. Struct. Commun.* **9**, 147–150.

Acta Cryst. (1984). C**40**, 951–955

The Structures of 15,18,34,37-Tetramethoxy-1,4,7,10,13,20,23,26,29,32-decaoxa-[13.13]parabenzenophane, $C_{32}H_{48}O_{14}$, and its 1:2:2 Complex with Sodium Thiocyanate and Water, $C_{32}H_{48}O_{14}.2(NaSCN.H_2O)$*

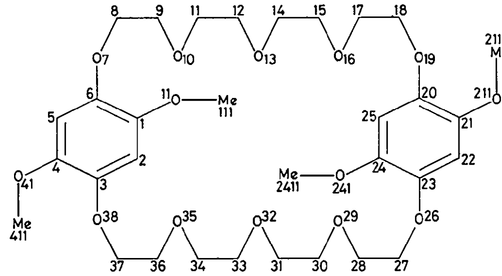
By John D. Owen

Molecular Structures Department, Rothamsted Experimental Station, Harpenden, Hertfordshire AL5 2JQ, England

(*Received* 3 *January* 1984; *accepted* 22 *February* 1984)

Abstract. $C_{32}H_{48}O_{14}$ (I): $M_r = 656{\cdot}7$, $C2/c$, $a = 32{\cdot}705\,(4)$, $b = 7{\cdot}637\,(2)$, $c = 13{\cdot}645\,(3)$ Å, $\beta = 98{\cdot}469\,(16)°$, $V = 3370{\cdot}9\,(12)$ Å^3, $Z = 4$, $D_x = 1{\cdot}294$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}1$ cm^{-1}, $F(000) = 1408$, $T = 293$ K, $R = 0{\cdot}044$ for 1684 observations. The crystals are formed by packing of discrete molecules separated by normal van der Waals contacts. The oxygen atoms of each half of the centrosymmetrical molecule are almost coplanar with the benzene ring. $2(NaSCN.H_2O).(I)$: $M_r = 854{\cdot}9$, $P\bar{1}$, $a = 13{\cdot}178\,(3)$, $b = 13{\cdot}708\,(2)$, $c = 23{\cdot}567\,(4)$ Å, $\alpha = 83{\cdot}054\,(13)$, $\beta = 89{\cdot}816\,(14)$, $\gamma = 94{\cdot}162\,(20)°$, $V = 4214{\cdot}5\,(12)$ Å^3, $Z = 4$, $D_x = 1{\cdot}347$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}2$ cm^{-1}, $F(000) = 1808$, $T = 293$ K, $R = 0{\cdot}089$ for 2727 observations. One sodium cation is six-coordinated, the other is eight-coordinated. The molecules form dimers, joined by hydrogen-bonded waters and anions and the benzene rings are stacked within the molecule, within the dimer and between dimers forming infinitely stacked chains along $[1\bar{1}1]$.

Introduction. 'Crown ether' compounds containing benzene rings were among the first to be prepared (Pedersen, 1967). These contained benzenes with two oxygen atoms in the *ortho* position, and many more have been prepared since (Gokel & Korzeniowski, 1982). These molecules form cavities which can accept small organic molecules or alkali-metal cations. Benzocrowns with oxygens in the *meta* position are much rarer (Van Keulen, Kellogg & Piepers, 1979; Elben, Fuchs, Frensch & Vögtle, 1979) and no structures of complexes have been reported. *para* substitution is also rare (Helgeson, Timko & Cram, 1974; Helgeson, Tarnowski, Timko & Cram, 1977; Kawashima, Kawashima, Otsubo & Misumi, 1978) and again no complexes have been reported. Model building indicated that the presence of methoxy groups on the benzene ring should aid complexation with alkali-metal cations, since a cavity surrounded by several oxygens can be formed at each end of a molecule such as (I). The preparation and characterization of (I) will be reported elsewhere (Parsons, 1983). The structures of (I) and its complex with $2(NaSCN.H_2O)$ are the first reported for a *para*-substituted benzo-crown molecule, and were undertaken to compare the conformations of the free and complexed molecules.



(I)

Experimental. Data for both structures collected on a CAD-4 diffractometer, intensities of 2 standard reflections changed insignificantly during data collections; no absorption corrections; scattering factors calculated from published analytical coefficents (*International Tables for X-ray Crystallography,* 1974); structure solution and refinement with *SHELX* (Sheldrick, 1976); data reduction and final geometry with local programs on a Prime 550 computer.

---

* Crystal Structures of Complexes between Alkali-Metal Salts and Cyclic Polyethers. 14. Part 13: Owen (1983).

0108-2701/84/060951-05$01.50