

We thank Dr D. D. Hackney and Professor D. E. Koshland for providing the crystals. G. Chapuis acknowledges the Swiss National Funds for financial support.

References

- ALBINATI, A., ZOCCHI, M., GERMAIN, G. & DECLERCQ, J. P. (1973). *Cryst. Struct. Commun.* **2**, 585–587.
- ALTONA, C. & SUNDARALINGAM, M. (1970). *J. Amer. Chem. Soc.* **92**, 1995–1999.
- ALTONA, C. & SUNDARALINGAM, M. (1972). *Acta Cryst.* **B28**, 1806–1816.
- BÜRGI, H. B. (1975). *Angew. Chem. Int. Ed.* **14**, 460–473.
- CHAPUIS, G., ZALKIN, A. & TEMPLETON, D. H. (1973). *Acta Cryst.* **B29**, 2642–2644.
- CHAPUIS, G., ZALKIN, A. & TEMPLETON, D. H. (1977). *Acta Cryst.* **B33**, 560–563.
- CHIANG, J. F., WILCOX, C. F. & BAUER, S. H. (1968). *J. Amer. Chem. Soc.* **90**, 3149–3157.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- DALLINGA, G. & TONEMAN, L. H. (1968). *Rec. Trav. Chim. Pays-Bas*, **87**, 795–804.
- DOYLE, P. A. & TURNER, P. S. (1968). *Acta Cryst.* **A24**, 390–397.
- FLIPPEN, J. L. (1972). *Acta Cryst.* **B28**, 2046–2048.
- FRATINI, A. V., BRITTS, K. & KARLE, I. L. (1967). *J. Phys. Chem.* **71**, 2482–2486.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
- HACKNEY, D. D. (1975). *Models for Chymotrypsin and the Interaction of Lysozyme with Urea*. Thesis, Univ. of California, Berkeley.
- JOHNSON, C. K. (1965). *ORTEP*. Oak Ridge National Laboratory Report ORNL-3794.
- MORIARTY, R. M. & ADAMS, T. (1973). *J. Amer. Chem. Soc.* **95**, 4070–4071.
- MORINO, Y., KUCHITSU, K. & YOKOZEKI, A. (1967). *Bull. Chem. Soc. Japan*, **40**, 1552.
- OLSON, A. J. (1975). *Polar Hydrogen Scattering Factors in X-ray Diffraction Analysis*. Thesis, Univ. of California, Berkeley.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- STORM, D. R. & KOSHLAND, D. E. (1972a). *J. Amer. Chem. Soc.* **94**, 5805–5814.
- STORM, D. R. & KOSHLAND, D. E. (1972b). *J. Amer. Chem. Soc.* **94**, 5815–5825.

Acta Cryst. (1977), **B33**, 1293–1295

Structural Studies of Precursor and Partially Oxidized Conducting Complexes. VI. A Neutron Diffraction Study of Dicesium Tetracyanoplatinate(II) Monohydrate*

BY PAUL L. JOHNSON, TIMOTHY R. KOCH,† AND JACK M. WILLIAMS‡

Chemistry Division, Argonne National Laboratory, Argonne, Illinois 60439, USA

(Received 29 September 1976; accepted 4 December 1976)

Abstract. $\text{Cs}_2[\text{Pt}(\text{CN})_4] \cdot \text{H}_2\text{O}$, hexagonal, $P6_1$ or $P6_5$, (C_6^2 or C_6^3), $a = 9.709$ (4), $c = 19.343$ (8) Å, $Z = 6$, $D_m = 3.66$, $D_c = 3.68$ g cm⁻³. The structure was determined with direct methods. The 1656 reflections collected by neutron diffraction were refined to an agreement index of $R(F_o^2) = 0.101$ for all observed data and $R(F_o) = 0.074$ for 1348 data with $F_o^2 > \sigma(F_o^2)$. The Pt–Pt separation is 3.545 (1) Å and the average C–Pt–Pt–C torsional angle is -32.79 (31)°. The $\text{Pt}(\text{CN})_4^{2-}$ groups comprise an unusual helical Pt chain along c .

Introduction. Preliminary X-ray photographs showed Laue symmetry $6/m$, and the following systematic ab-

sences were observed: $00l$ when $l = 6n$. These conditions establish the space group $P6_1$, or its enantiomorph $P6_5$. Cell constants were established by centering 16 reflections and carrying out a least-squares analysis of the determined angles 2θ , χ , and ϕ .

Preliminary crystal data on this compound have been published by Otto, Holzapfel, Yersin & Gliemann (1976). There is good agreement of c with their value of 19.336 (6) Å. However, their a [9.687 (2) Å] is 5.5 e.s.d. (this work) shorter.

A crystal weighing 85.1 mg was mounted in a lead-glass capillary for the neutron diffraction studies. The neutron source was the Argonne National Laboratory CP-5 reactor. The diffractometer was a fully automated (Sigma 5 computer control) four-circle instrument which has been described by Petersen, Dahl & Williams (1974).

Data were collected with a θ – 2θ step-scan and an interval of 0.1° in 2θ . The scan widths were varied for

* Work performed under the auspices of the US Energy Research and Development Administration.

† Research participant sponsored by the Argonne Center for Educational Affairs, from Coe College, Cedar Rapids, Iowa.

‡ Author to whom correspondence should be addressed.

different ranges of 2θ according to the observed peak width. The background was established by measuring the intensity at each extremity of the scan. Two standards were measured after every 50 reflections to check for crystal movement or decomposition (maximum variation = 6%).

Data were collected to a minimum d spacing of 0.715 Å [$\lambda = 1.142$ (1) Å]. 1656 unique data were collected. Of these, 1348 had $F_o^2 > \sigma F_o^2$. Structure factors were derived by applying Lorentz and absorption corrections ($\mu = 0.684 \text{ cm}^{-1}$), and the magnitude of the transmission factors ranged from 0.88 to 0.93. Standard errors were assigned to the data with the standard counting-statistics formula with $(0.06 I)^2$ added to the variances to adjust for systematic error. The data were placed on an approximately absolute scale by comparison with a standard NaCl crystal.

Space group $P6_1$ was arbitrarily chosen, and the structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) to locate the Pt, C, N and O atoms. A starting point for each H position was calculated on the assumption that the O—H bond was directed along logical O...N vectors. Refinement was carried out with full-matrix least-squares techniques, first with isotropic and then anisotropic parameters. The final $R(F_o^2) = \Sigma |F_o^2 - F_c^2| / \Sigma F_o^2$ for the 1348 data $> 1.0 \sigma$ was 0.090, while the R based on F_o was 0.074. For all 1656 data, $R(F_o^2) = 0.101$. The standard deviation of an observation of unit weight was 1.14 for all data. The neutron scattering amplitudes were as follows: $b_{\text{Pt}} = 0.95$, $b_{\text{C}} = 0.663$, $b_{\text{N}} = 0.94$, $b_{\text{O}} = 0.575$, $b_{\text{H}} = -0.372$ and $b_{\text{Cs}} = 0.55$ ($\times 10^{-12} \text{ cm}$) (*International Tables for X-ray Crystallography*, 1974). The anomalous scattering of all these atoms is negligible and hence no differentiation could be made between space group $P6_1$ and $P6_5$.

The refined positional parameters are in Table 1. Bond lengths and angles are in Table 2.*

Discussion. The crystal structure consists of nearly square-planar $\text{Pt}(\text{CN})_4^{2-}$ anions stacked in a helical fashion approximately parallel to c . This is in contrast to the linear Pt chains found in $\text{K}_2[\text{Pt}(\text{CN})_4] \cdot 3\text{H}_2\text{O}$ (Washecheck, Peterson, Reis & Williams, 1976). The helix repeat distance is 19.343 Å, and the distance of the Pt atom from the origin is 1.473 Å.

The Pt—Pt—Pt angle is 156.01 (3)°, which is a larger deviation from linearity than that found in $\text{K}_{1.75}[\text{Pt}(\text{CN})_4] \cdot 1.5\text{H}_2\text{O}$ (Williams, Keefer, Washecheck & Enright, 1976). The Pt—Pt distance is 3.545 (1) Å and the average C—Pt—Pt—C torsional angle -32.79 (31)°. A stereoview of the unit-cell contents is in Fig. 1 (*ORTEP*, Johnson, 1965).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32382 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Fractional atomic coordinates ($\times 10^4$) for $\text{Cs}_2[\text{Pt}(\text{CN})_4] \cdot \text{H}_2\text{O}$

	x	y	z
Pt	-363 (2)	1304 (2)	0
C(1)	-942 (4)	3007 (4)	13 (3)
C(2)	1888 (3)	2834 (3)	232 (2)
C(3)	143 (4)	-449 (3)	-16 (3)
C(4)	-2611 (3)	-212 (3)	-230 (2)
N(1)	-1342 (3)	3952 (3)	21 (3)
N(2)	3208 (3)	3716 (3)	370 (2)
N(3)	433 (3)	-1469 (3)	-24 (2)
N(4)	-3930 (3)	-1074 (3)	-370 (2)
Cs(1)	-3412 (5)	-4215 (5)	-408 (3)
Cs(2)	-5613 (6)	841 (5)	397 (3)
O(1)	2926 (5)	6685 (6)	4 (4)
H(1)	3993 (10)	7390 (10)	-138 (7)
H(2)	2883 (12)	5689 (10)	73 (7)

Table 2. Interatomic distances (Å) and bond angles (°) for $\text{Cs}_2[\text{Pt}(\text{CN})_4] \cdot \text{H}_2\text{O}$

(a) Distances around Pt atoms

Pt—C(1)	1.996 (3)	Pt—C(4)	1.979 (4)
Pt—C(2)	1.984 (3)	Pt—Pt	3.545 (1)
Pt—C(3)	1.993 (3)		

(b) C—N distances in cyanide groups

C(1)—N(1)	1.161 (4)	C(3)—N(3)	1.158 (3)
C(2)—N(2)	1.162 (4)	C(4)—N(4)	1.158 (4)

(c) Water molecule distances

O(1)—H(1)	0.953 (10)	O(1)—H(2)	0.957 (12)
-----------	------------	-----------	------------

(d) Cs ion interactions

Cs(1)—O(1) ⁱⁱ	3.155 (8)	Cs(2)—O(1) ^j	3.125 (9)
Cs(1)—N(2) ⁱⁱⁱ	3.236 (5)	Cs(2)—N(3) ^{vi}	3.291 (6)
Cs(1)—N(3) ^{iv}	3.243 (6)	Cs(2)—N(2) ^{vii}	3.325 (7)
Cs(1)—N(4) ^j	3.330 (5)	Cs(2)—N(4) ⁱ	3.371 (6)
Cs(1)—N(4) ⁱ	3.332 (6)	Cs(2)—N(1) ⁱ	3.388 (6)
Cs(1)—N(1) ⁱⁱ	3.360 (6)	Cs(2)—N(3) ^{viii}	3.438 (6)
Cs(1)—N(1) ^v	3.387 (5)	Cs(2)—N(2) ^{viii}	3.508 (6)
Cs(1)—N(3) ^j	3.412 (5)		

(e) Angles of bonded atoms

Pt—Pt—Pt	156.01 (3)
C(1)—Pt—C(2)	92.16 (16)
C(2)—Pt—C(3)	89.66 (15)
C(3)—Pt—C(4)	90.73 (15)
C(4)—Pt—C(1)	87.45 (15)
Pt—C(1)—N(1)	177.30 (31)
Pt—C(2)—N(2)	179.25 (29)
Pt—C(3)—N(3)	179.86 (30)
Pt—C(4)—N(4)	178.63 (29)
H(1)—O(1)—H(1)'	104.4 (9)

Symmetry code

(i) x, y, z	(v) $x, -1 + y, z$
(ii) $-1 + y, -1 - x + y, -\frac{1}{6} + z$	(vi) $-1 + x - y, x, \frac{1}{6} + z$
(iii) $-1 + x, -1 + y, z$	(vii) $-1 + y, -x + y, -\frac{1}{6} + z$
(iv) $y, -x + y, -\frac{1}{6} + z$	(viii) $-1 + x, y, z$

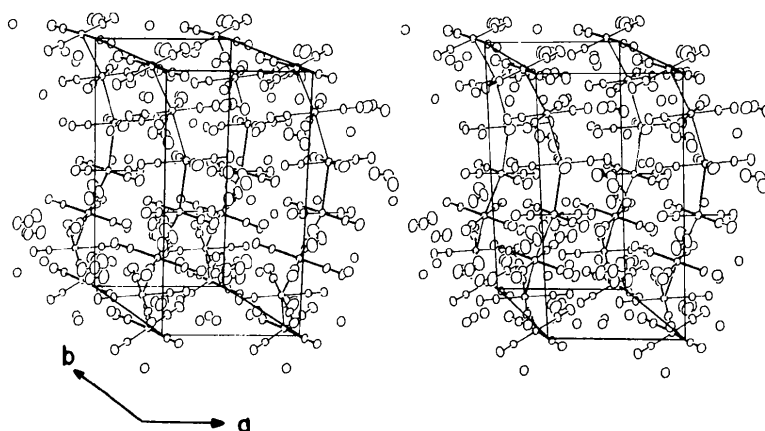


Fig. 1. Stereoscopic drawing of the unit cell of $\text{Cs}_2[\text{Pt}(\text{CN})_4] \cdot \text{H}_2\text{O}$.

The average Pt—C bond length is 1.988 (7) Å, and the average C—N bond length is 1.160 (8) Å. The Pt—C—N angle is nearly linear with a 178.76 (60)° average. The four C atoms surrounding the Pt atom and the attached N atoms are planar with a maximum deviation of 0.006 Å, while the Pt atom is exactly in this plane. The dihedral angle between adjacent $\text{Pt}(\text{CN})_4^{2-}$ groups is 13.0 (2)°.

The coordination sphere around each Cs atom is listed in Table 2. The coordination is sevenfold around Cs(2) and eightfold around Cs(1). All Cs—N distances are longer than the van der Waals sum (~3.1 Å). Apparently, the $\text{Cs}^+ \cdots \text{N} \equiv \text{C}$ interactions are not of sufficient strength to distort the $\text{Pt}(\text{CN})_4^{2-}$ groups. The O of the water molecule is 3.155 (8) Å from Cs(1) and 3.125 (9) Å from Cs(2).

The water molecule has an intramolecular bond angle [H(1)—O(1)—H(2)] of 104.4 (9)°. The O(1)—H(1) distance is 0.953 (10) Å and O(1)—H(2) is 0.957 (12) Å. If a riding-motion correction is made, O—H distances become 0.965 (11) and 0.985 (13) Å respectively.

Both H(1) and H(2) form hydrogen bonds to N(4) and N(2) respectively. The distances are 1.866 (9) and 2.168 (10) Å for H(1)—N(4) and H(2)—N(2). O(1)—H(1)—N(4) is 174.5 (10)° and O(1)—H(2)—N(2) is 168.0 (12)°.

References

- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 270–272. Birmingham: Kynoch Press.
 JOHNSON, C. K. (1965). *ORTEP*. Oak Ridge National Laboratory Report ORNL-3794.
 OTTO, H. H., HOLZAPFEL, W., YERSIN, H. & GLIEMANN, G. (1976). *Z. Naturforsch.* **31b**, 528–529.
 PETERSEN, J. L., DAHL, L. F. & WILLIAMS, J. M. (1974). *J. Amer. Chem. Soc.* **96**, 6610–6620.
 WASHECHECK, D. M., PETERSON, S. W., REIS, A. H. JR & WILLIAMS, J. M. (1976). *Inorg. Chem.* **15**, 74–78.
 WILLIAMS, J. M., KEEFER, K. D., WASHECHECK, D. M. & ENRIGHT, N. P. (1976). *Inorg. Chem.* **15**, 2446–2455.

Acta Cryst. (1977). **B33**, 1295–1297

N,N'-Bis(β -chloroethyl)pimelamide*

BY M. R. CIAJOLO, V. PAVONE AND E. BENEDETTI

Laboratorio di Chimica Generale ed Inorganica, Istituto Chimico, Università di Napoli, Via Mezzocannone 4, 80134 Napoli, Italy

(Received 17 November 1976; accepted 14 December 1976)

Abstract. $\text{C}_{11}\text{H}_{20}\text{N}_2\text{O}_2\text{Cl}_2$, $M_r = 283.15$, monoclinic, $P2_1$, $a = 4.941$ (5), $b = 32.425$ (30), $c = 4.817$ (5) Å, β

$= 113^\circ 37$ (5)' at 25°C, $V = 707.07$ Å³, $D_x = 1.331$ g cm⁻³, $Z = 2$. The molecular conformation is not fully extended. Each molecule forms hydrogen bonds along two directions (almost the *a* and *c* directions). A com-

* Model Compounds of Nylons. III.