

The Pt—Cl bonds [2.299 (2) and 2.296 (2) Å] and the Pt—N bonds [2.041 (6) and 2.053 (6) Å] are normal and agree well with published values found in *trans*-aminochloroplatinum(II) compounds (Rochon, Kong & Melanson, 1984; Lock & Zvagulis, 1981). The two *trans* organic ligands are arranged so that one ligand is above the platinum plane, while the second is below the plane. The whole molecule has an approximate centre of symmetry (Figs. 1 and 2).

The structure of the organic ligands is normal. All the angles are close to the tetrahedral values (107–112°). The angles around the bonded N atoms [Pt—N—C = 122.9 (5) and 123.1 (5)°] are quite far from the tetrahedral value, but similar values (113–122°) have been observed for these types of compounds (Rochon, Kong & Melanson, 1984; Lock & Zvagulis, 1981; Rochon & Melanson, 1982).

A projection down the *x* axis of the packing of the molecules is shown in Fig. 2. The crystal consists of layers of molecules parallel to the *ac* plane. The layers are held together in the *b* direction by van der Waals forces between the —CH₃ groups. Inside the layers, the structure is stabilized by intermolecular hydrogen bonding between the amino groups and the chlorine atoms. The N...Cl distances vary from 3.335 (6) to 3.504 (6) Å and the C—N...Cl angles from 100.3 (4) to 133.1 (5)°.

Grateful acknowledgments are made to the Natural Sciences and Engineering Research Council of Canada

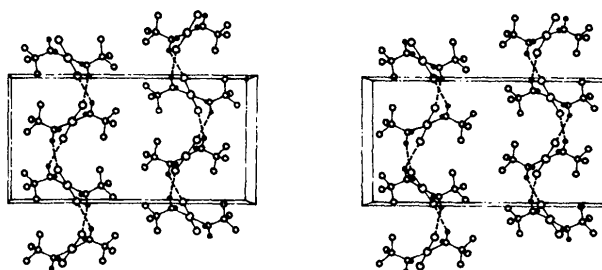


Fig. 2. Stereoscopic diagram of the packing in the *trans*-[Pt(NH₂-C(CH₃)₃)₂Cl₂] crystal (*b* axis horizontal and *c* axis vertical).

and to the Ministère de l'Éducation (FCAC) for financial support.

References

- COPPENS, P. & HAMILTON, W. C. (1970). *Acta Cryst.* **A26**, 71–83.
 CROMER, D. T. (1965). *Acta Cryst.* **18**, 17–23.
 CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.
 LOCK, C. J. L. & ZVAGULIS, M. (1981). *Inorg. Chem.* **20**, 1817–1823.
 MELANSON, R., HUBERT, J. & ROCHON, F. D. (1975). *Can. J. Chem.* **53**, 1139–1143.
 ROCHON, F. D., KONG, P. C. & MELANSON, R. (1984). *Can. J. Chem.* In the press.
 ROCHON, F. D. & MELANSON, R. (1982). *Acta Cryst.* **B38**, 1133–1136.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1985). **C41**, 352–355

1,1,1,2,2,2,3,3,3-Nonacarbonyl- μ_3 -cyclopropylmethylidyne-triangulo-tricobalt, (μ_3 -*c*-C₃H₅C)Co₃(CO)₉

BY PAUL F. SEIDLER, STEPHEN T. MCKENNA, MATTHEW A. KULZICK AND THOMAS M. GILBERT

Department of Chemistry, University of California, Berkeley, California 94720, USA

(Received 29 June 1984; accepted 30 October 1984)

Abstract. $M_r = 481.98$, triclinic, $P\bar{1}$, $a = 7.8772$ (6), $b = 14.3778$ (17), $c = 15.7851$ (16) Å, $\alpha = 103.700$ (9), $\beta = 100.877$ (7), $\gamma = 99.365$ (8)°, $V = 1664.9$ (7) Å³, $Z = 4$, $D_x = 1.923$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 29.93$ cm⁻¹, $F(000) = 944$, $T = 296$ K, $R = 2.7\%$, $R_w = 3.6\%$, $R_{\text{all}} = 3.7\%$, $S = 1.67$ for 3691 observations with $I > 3\sigma(I)$. The final structure clearly shows the staggering of the Co—Co—Co and C—C—C rings in a nearly threefold symmetric environment. There are no intermolecular contacts shorter than 2.70 Å.

Introduction. A recently developed and important class of organometallic clusters is the alkylidynetricobalt nonacarbonyl series (Seyferth, 1976). Such compounds have been shown to exhibit interesting reactivity (Palyi, Piacenti & Marko, 1970; Seyferth, 1976; Seidler, Bryndza, Frommer, Stuhl & Bergman, 1983; and references therein), and have even found use as catalyst precursors for certain transformations (Seyferth & Withers, 1983).

Sutton & Dahl (1967) first structurally characterized a member of this class using film data; more recently,

Table 1. *Positional parameters, equivalent isotropic thermal parameters and their estimated standard deviations*

$$B_{\text{eq}} = \frac{1}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
Co(1)	0.12261 (5)	-0.30524 (3)	0.46586 (3)	2.809 (9)
Co(2)	0.42814 (5)	-0.22137 (3)	0.47705 (3)	2.865 (9)
Co(3)	0.28864 (5)	-0.17659 (3)	0.60421 (3)	2.797 (9)
Co(4)	0.02925 (5)	0.19022 (3)	-0.11764 (3)	3.21 (1)
Co(5)	0.33304 (6)	0.20011 (3)	-0.10367 (3)	3.36 (1)
Co(6)	0.27906 (6)	0.17298 (3)	-0.00576 (3)	3.37 (1)
O(11)	-0.0802 (3)	-0.1916 (2)	0.3684 (2)	5.87 (7)
O(12)	0.1091 (4)	-0.4782 (2)	0.3221 (2)	6.60 (8)
O(13)	-0.1432 (4)	-0.3984 (2)	0.5424 (2)	7.68 (9)
O(21)	0.3487 (3)	-0.0709 (2)	0.3854 (2)	5.75 (7)
O(22)	0.7938 (3)	-0.1460 (2)	0.5792 (2)	6.35 (8)
O(23)	0.5147 (4)	-0.3663 (2)	0.3384 (2)	7.45 (9)
O(31)	0.1776 (3)	-0.0041 (2)	0.5583 (2)	5.57 (7)
O(32)	0.0605 (3)	-0.2224 (2)	0.7215 (2)	6.16 (7)
O(33)	0.6017 (4)	-0.0770 (2)	0.7488 (2)	6.46 (8)
O(41)	0.0177 (4)	0.0168 (2)	-0.2652 (2)	6.34 (8)
O(42)	-0.1675 (4)	0.3124 (2)	-0.2018 (2)	7.46 (8)
O(43)	-0.2524 (3)	0.1174 (2)	-0.0389 (2)	7.44 (9)
O(51)	0.4417 (4)	0.1407 (2)	-0.2422 (2)	7.09 (8)
O(52)	0.6662 (4)	0.4043 (3)	0.0086 (2)	9.4 (1)
O(53)	0.2309 (4)	0.4224 (2)	-0.1975 (2)	7.46 (9)
O(61)	0.3473 (4)	-0.0102 (2)	-0.1121 (2)	6.27 (8)
O(62)	0.0746 (4)	0.0969 (2)	0.1094 (2)	6.75 (8)
O(63)	0.6143 (4)	0.2487 (3)	0.1285 (2)	7.25 (9)
C(1)	0.3388 (4)	-0.2983 (2)	0.5492 (2)	2.86 (7)
C(2)	0.4043 (4)	-0.3764 (2)	0.5812 (2)	3.87 (8)
C(3)	0.5418 (5)	-0.3555 (3)	0.6666 (3)	5.6 (1)
C(4)	0.3591 (5)	-0.4051 (3)	0.6608 (3)	5.3 (1)
C(5)	0.1907 (4)	0.2865 (2)	-0.0177 (2)	3.23 (7)
C(6)	0.1569 (5)	0.3721 (3)	0.0429 (3)	5.1 (1)
C(7)	0.0896 (6)	0.3691 (4)	0.1219 (3)	7.3 (1)
C(8)	0.2729 (6)	0.4242 (3)	0.1313 (3)	6.8 (1)
C(11)	-0.0027 (4)	-0.2339 (2)	0.4063 (2)	3.66 (8)
C(12)	0.1143 (5)	-0.4108 (3)	0.3775 (2)	4.12 (9)
C(13)	-0.0395 (5)	-0.3616 (3)	0.5136 (2)	4.24 (9)
C(21)	0.3809 (4)	-0.1278 (2)	0.4206 (2)	3.67 (8)
C(22)	0.6519 (4)	-0.1731 (3)	0.5403 (2)	3.89 (8)
C(23)	0.4783 (4)	-0.3102 (3)	0.3914 (2)	4.30 (9)
C(31)	0.2166 (4)	-0.0698 (2)	0.5767 (2)	3.55 (8)
C(32)	0.1476 (4)	-0.2043 (3)	0.6756 (2)	3.81 (8)
C(33)	0.4825 (4)	-0.1158 (3)	0.6923 (2)	4.19 (9)
C(41)	0.0188 (5)	0.0826 (3)	-0.2101 (2)	4.29 (9)
C(42)	-0.0899 (5)	0.2654 (3)	-0.1702 (3)	4.7 (1)
C(43)	0.1418 (4)	0.1454 (3)	-0.0692 (2)	4.46 (9)
C(51)	0.4014 (4)	0.1946 (3)	-0.1898 (2)	4.50 (9)
C(52)	0.5373 (5)	0.3551 (3)	-0.0355 (3)	5.3 (1)
C(53)	0.2697 (5)	0.3672 (3)	-0.1611 (3)	4.9 (1)
C(61)	0.3219 (4)	0.0591 (3)	-0.0712 (2)	4.25 (9)
C(62)	0.1539 (5)	0.1271 (3)	0.0643 (2)	4.48 (9)
C(63)	0.4852 (5)	0.2192 (3)	0.0762 (3)	4.9 (1)

published reviews have described the interesting structural features of these compounds (Palyi, Piacenti & Marko, 1970; Penfold & Robinson, 1973; Schmidt, 1978). In order to add to the available data, and also to investigate the possibility of interaction between the strained cyclopropyl ring and the tricobalt ring, we chose to determine the molecular structure of the title compound, (μ_3 -*c*-C₃H₅C)Co₃(CO)₉.

Experimental. Material prepared from butyl cyclopropanecarbodithioate and Co₂(CO)₈ (Patin, Mignani & van Hulle, 1979), crystallized from hot methanol under argon as oblong burgundy flakes; data crystal 0.12 × 0.15 × 0.50 mm, mounted on a glass fiber with polycyanoacrylate cement with the major crystal axis approximately perpendicular to the fiber axis; Enraf-Nonius CAD-4 diffractometer with graphite-

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

Co(1)-Co(2)	2.464 (1)	Co(4)-Co(5)	2.471 (1)
Co(1)-Co(3)	2.474 (1)	Co(4)-Co(6)	2.471 (1)
Co(2)-Co(3)	2.470 (1)	Co(5)-Co(6)	2.471 (1)
Co(1)-C(1)	1.918 (2)	Co(4)-C(5)	1.912 (3)
Co(2)-C(1)	1.913 (3)	Co(5)-C(5)	1.911 (3)
Co(3)-C(1)	1.900 (2)	Co(6)-C(5)	1.911 (3)
C(1)-C(2)	1.463 (4)	C(5)-C(6)	1.466 (4)
C(2)-C(3)	1.494 (4)	C(6)-C(7)	1.452 (5)
C(2)-C(4)	1.497 (4)	C(6)-C(8)	1.465 (5)
C(3)-C(4)	1.473 (5)	C(7)-C(8)	1.492 (5)
Co-C(axial carbonyl) (average)			1.834 (3)
Co-C(equatorial carbonyl) (average)			1.735 (3)
C-O(carbonyl) (average)			1.128 (4)
Co(1)-C(11)-Co(2)	80.1 (1)	Co(4)-C(5)-Co(5)	80.5 (1)
Co(1)-C(1)-Co(3)	80.8 (1)	Co(4)-C(5)-Co(6)	80.6 (1)
Co(2)-C(1)-Co(3)	80.8 (1)	Co(5)-C(5)-Co(6)	80.6 (1)
C(2)-C(3)-C(4)	60.6 (2)	C(6)-C(7)-C(8)	59.7 (3)
C(3)-C(2)-C(4)	59.0 (2)	C(7)-C(6)-C(8)	61.5 (3)
C(1)-C(2)-H(2)	104.0 (2)	C(5)-C(6)-H(6)	100.6 (3)
C(1)-C(2)-C(3)	122.2 (3)	C(5)-C(6)-C(7)	124.0 (3)
C(1)-C(2)-C(4)	122.6 (3)	C(5)-C(6)-C(8)	123.7 (3)
C(1)-Co(1)-C(12)	101.9 (1)	C(5)-Co(5)-C(52)	100.7 (1)
C(1)-Co(1)-C(13)	102.0 (1)	C(5)-Co(5)-C(53)	102.9 (1)
C(1)-Co(2)-C(22)	100.9 (1)	C(5)-Co(4)-C(42)	101.3 (1)
C(1)-Co(2)-C(23)	102.8 (1)	C(5)-Co(4)-C(43)	102.8 (1)
C(1)-Co(3)-C(32)	105.2 (1)	C(5)-Co(6)-C(62)	102.9 (1)
C(1)-Co(3)-C(33)	104.5 (1)	C(5)-Co(6)-C(63)	105.1 (1)
Co-C-O (average)	178.7 (3)		

monochromated Mo K α radiation; 24 well centered reflections with 27° < 2 θ < 36° used to obtain lattice parameters; 4543 data collected at 296 (2) K over 86 h using θ -2 θ scans from 3 < 2 θ < 45° with (sin θ / λ)_{max} = 0.538 Å⁻¹; $h \pm 8$, $k \pm 15$, l 0 to + 16; intensity standards 423, $\bar{5}41$, $\bar{4}2\bar{6}$ every 2 h of exposure; data corrected for isotropic decay due to radiation damage to a maximum of 21.7%; orientation standards checked every 250 reflections with no reorientation necessary; data reduction through Frenz (1981) *Structure Determination Package* providing scattering factors and corrections for anomalous dispersion (*International Tables for X-ray Crystallography*, 1974) and all other programs; data analytically corrected for absorption, with correction based on crystal shape; $\mu = 29.93$ cm⁻¹, maximum transmission coefficient 0.73, minimum 0.30, average 0.60; correction for secondary extinction, $g = 1.443 \times 10^{-7}$ (refined); phasing *via MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982); balance of non-hydrogen atoms located through combination of difference Fourier and *SEARCH* techniques and refined anisotropically with full-matrix non-linear least squares; function minimized in least squares was $\sum w(F_o - F_c)^2$ where $w = 1/\sigma(F_o^2)$, and $\sigma(F_o^2) = \{(\sigma_f)^2 + [0.035 \times (F_o^2)]^2\}^{1/2}$, where σ_f is based on counting statistics; all H-atom positions calculated (not refined) based on the attached C at fixed distances (0.95 Å); all H atoms given fixed isotropic thermal parameters based on the attached C; largest feature in final difference Fourier map 0.59 e Å⁻³ near C(6); refining 452 variables against 4352 unique data, 3691 with $I > 3\sigma(I)$, gave $R = 0.027$, $R_w = 0.036$, $R_{\text{all}} = 0.037$, $S = 1.67$, $(A/\sigma)_{\text{max}} = 0.15$.

Discussion. Data appropriate to discussion of the structure are given in Tables 1 and 2.*

The data clearly demonstrate that $(\mu_3\text{-}c\text{-C}_3\text{H}_5\text{C})\text{Co}_3(\text{CO})_9$ exists as discrete molecules in the solid state; no intermolecular contacts shorter than 2.70 Å were observed.

As may be seen from Table 2 and Fig. 1, the three Co atoms form an equilateral triangle within experimental error, with average bond distances of 2.470 (2) Å. The structure of the nonacarbonyltricobalt fragment of the molecule is what would be expected intuitively for such a species. The carbonyl ligands have quite normal bonding distances and the Co–C–O angles are essentially linear. One interesting result is that the average Co–C(equatorial carbonyl) bond distance, e.g. Co(1)–C(12), is approximately 0.05 Å shorter than the average Co–C(axial carbonyl) bond distance, e.g. Co(1)–C(11), although all C–O carbonyl bond lengths are experimentally equal. This result has been observed previously (Leung, Coppens, McMullan & Koetzle, 1981) for $(\mu_3\text{-HC})\text{Co}_3(\text{CO})_9$ in both neutron and X-ray studies.

The average Co–C(alkylidyne) bond distance is 1.911 (4) Å, with the C lying 1.271 (3) Å above the tricobalt plane. These distances agree well with the respective values of 1.90 (2) and 1.25 (3) Å determined for $(\mu_3\text{-CH}_3\text{C})\text{Co}_3(\text{CO})_9$ (Sutton & Dahl, 1967).

* Lists of structure factors, anisotropic thermal parameters, positional and isotropic thermal parameters for calculated H atoms, raw bond distances and angles, and selected least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39874 (43 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

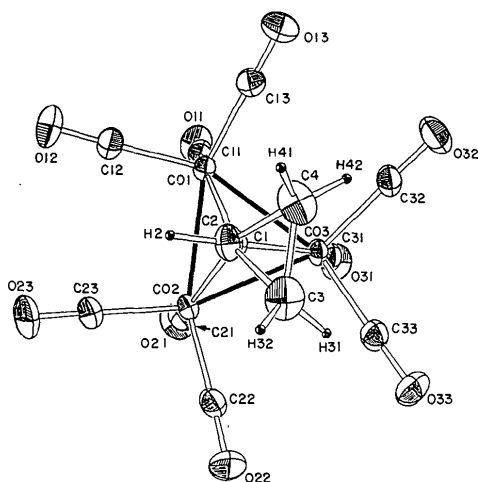


Fig. 1. Atomic labeling (ORTEP, Johnson, 1965) for $(\mu_3\text{-}c\text{-C}_3\text{H}_5\text{C})\text{Co}_3(\text{CO})_9$, viewed approximately along the methylidyne axis.

The effect of increased C *p* orbital contribution to the C–C bonding (Streitwieser & Heathcock, 1981) may be seen by considering the C–C bond distances of the cyclopropyl fragment. The average C(alkylidyne)–C(cyclopropyl) bonding distance is 1.465 (4) Å, significantly shorter than the average $sp^3\text{-}sp^3$ C–C bond length of 1.54 Å (*International Tables for X-ray Crystallography*, 1968) and shorter even than the analogous bond in methylcyclopropane, observed to be 1.517 (2) Å (Klein & Schrupf, 1981), and the analogous bond in $(\mu_3\text{-CH}_3\text{C})\text{Co}_3(\text{CO})_9$, reported as 1.53 (3) Å (Sutton & Dahl, 1967). In fact, this distance approximates the $sp\text{-}sp^3$ C–C bond length of 1.460 (3) Å observed for propyne, $\text{CH}_3\text{C}\equiv\text{CH}$ (*International Tables for X-ray Crystallography*, 1968). Unusually short bonds to the alkylidyne C have been previously observed in this class of tricobalt clusters (Penfold & Robinson, 1973). For example, a 1.37 (1) Å carbon(alkylidyne)–carbon(alkylidyne) bond is found in $(\text{CO})_9\text{Co}_3\text{C-CCo}_3(\text{CO})_9$ (Brice & Penfold, 1972).

The cyclopropyl C atoms have bonding distances and angles which suggest, within experimental error, that the atoms form an equilateral triangle, but the bonding distances, which range from 1.452 (5) to 1.497 (4) Å, are again significantly shorter than the $sp^3\text{-}sp^3$ value of 1.54 Å quoted above, and in fact are considerably shorter than the distance of 1.510 Å observed for free cyclopropane (Streitwieser & Heathcock, 1981) and the 1.509 (1) Å observed for methylcyclopropane (Klein & Schrupf, 1981).

The three-membered rings adopt a staggered conformation, apparently to minimize nonbonding contacts (Fig. 1). As evidence of slight steric repulsion, the data show that the cyclopropyl ligand 'pushes' the equatorial carbonyl C atoms on the eclipsed Co atom [Co(3)] 0.07–0.10 Å closer to the Co_3 plane than the other equatorial carbonyl C atoms. However, this push represents the only interaction between the two rings which we could detect.

We thank Drs F. J. Hollander, K. N. Raymond, and Sung-Hou Kim, and Mr Robert Scarrow for their patience, tutelage and assistance. Funds for the structure were provided by the College of Chemistry as part of the CHM 295 series of courses; partial funding for the X-ray diffraction equipment was provided by NSF Grant CHE 79-07027. STM and PFS are grateful for NSF predoctoral fellowships.

References

- BRICE, M. D. & PENFOLD, B. R. (1972). *Inorg. Chem.* **11**, 1381–1384.
 FRENZ, B. A. (1981). *Structure Determination Package*, 4th ed. B. A. FRENZ & Associates, College Station, Texas 77840.
International Tables for X-ray Crystallography (1968). Vol. III, pp. 275–276. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- KLEIN, A. W. & SCHRUMPF, G. (1981). *Acta Chem. Scand. Ser. A*, **35**, 425–430.
- LEUNG, P., COPPENS, P., McMULLAN, R. K. & KOETZLE, T. F. (1981). *Acta Cryst. B37*, 1347–1352.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- PALYI, G., PIACENTI, F. & MARKO, L. (1970). *Inorg. Chim. Acta Rev.* **4**, 109–121.
- PATIN, H., MIGNANI, G. & VAN HULLE, M. (1979). *Tetrahedron Lett.* **26**, 2441–2442.
- PENFOLD, B. R. & ROBINSON, B. H. (1973). *Acc. Chem. Res.* **6**, 73–80.
- SCHMIDT, G. (1978). *Angew. Chem. Int. Ed. Engl.* **17**, 392–400.
- SEIDLER, P. F., BRYNDZA, H. E., FROMER, J. E., STUHL, L. S. & BERGMAN, R. G. (1983). *Organometallics*, **2**, 1701–1705.
- SEYFERTH, D. (1976). *Adv. Organomet. Chem.* **14**, 97–144.
- SEYFERTH, D. & WITHERS, H. P. (1983). *Inorg. Chem.* **22**, 2931–2936.
- STREITWEISER, A. & HEATHCOCK, C. H. (1981). *Introduction to Organic Chemistry*, second edition, p. 84. New York: Macmillan.
- SUTTON, P. W. & DAHL, L. F. (1967). *J. Am. Chem. Soc.* **89**, 261–268.

Acta Cryst. (1985). **C41**, 355–358

Structure of Monoclinic Sodium (Ethylenediaminetetraacetato)holmate(III) Octahydrate, $\text{Na}[\text{Ho}(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_8)].8\text{H}_2\text{O}$: Disorder Induced by Radiation

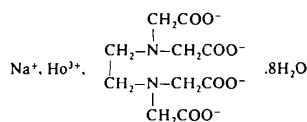
BY LIESELOTTE K. TEMPLETON, DAVID H. TEMPLETON AND ALLAN ZALKIN

Materials and Molecular Research Division, Lawrence Berkeley Laboratory and Department of Chemistry, University of California, Berkeley, California 94720, USA

(Received 25 September 1984; accepted 1 November 1984)

Abstract. $M_r = 620.3$, monoclinic, $Fd\bar{1}1$, $a = 19.333$ (3), $b = 35.366$ (6), $c = 12.106$ (2) Å, $\alpha = 90.40$ (10)°, $V = 8277.0$ Å³, $Z = 16$ [conventional setting: Cc , $a = 12.106$ (2), $b = 19.333$ (3), $c = 18.650$ (3) Å, $\beta = 108.54$ (10)°, $Z = 8$], $D_x = 1.991$ g cm⁻³, $\text{Mo K}\alpha$, $\lambda(\alpha_1) = 0.70930$ Å, $\mu = 39.7$ cm⁻¹, $F(000) = 4924$, $T = 294$ K, $R = 0.026$ for 4843 reflections. The atomic positions are very similar to those in the analogous compounds of lighter lanthanoid elements which crystallize in the orthorhombic space group $Fdd2$. The monoclinic symmetry comes from ordering of water molecules in alternate sites which are occupied randomly in the orthorhombic structure. Exposure to X-rays induces disorder which makes the crystals more nearly orthorhombic. The Dy compound of the same composition is isomorphous with $a = 19.358$ (3), $b = 35.408$ (3), $c = 12.103$ (5) Å, $\alpha = 90.34$ (3)°.

Introduction. Crystal structure parameters were desired for the title salt,



so that it could be used to measure anomalous-scattering terms for Ho at various wavelengths

(Chapuis, Templeton & Templeton, 1985) by analysis of diffraction intensities. We had expected the crystals to have the orthorhombic structure with space group $Fdd2$ which has been reported for the analogous salts of Pr, Nd, Sm, Gd, Tb, Dy and Er (Hoard, Lee & Lind, 1965; Koetzle & Hamilton, 1975; Nassimbeni, Wright, van Niekerk & McCallum, 1979; Templeton, Templeton, Zalkin & Ruben, 1982). Indeed, the diffraction patterns are very similar to those of the orthorhombic crystals, but close inspection reveals monoclinic symmetry with twice as large an asymmetric unit. We describe the structure in space group $Fd\bar{1}1$ (an unconventional setting of Cc) because that gives cell dimensions and atomic coordinates which are nearly the same as for $Fdd2$. We find the same monoclinic structure for crystals of the Dy compound, contrary to the findings of Nassimbeni *et al.* (1979). A recent neutron-diffraction study by Engel, Takusagawa & Koetzle (1984) of the orthorhombic Sm compound found disorder of water molecules among two sets of sites. The monoclinic symmetry involves an ordering of the occupancy of these same sites.

Experimental. The compound was made from Ho_2O_3 (Research Chemical Corporation) dissolved in HCl and mixed with a hot solution of $\text{Na}_2\text{H}_2(\text{edta})\cdot 2\text{H}_2\text{O}$ (Aldrich Chemical Company); pH adjusted to about 5 with NaOH; slow evaporation, then recrystallization