triphenyltin halide–phosphine oxide complexes is large, typically about 156° (Rheingold, Ng & Zuckerman, 1984).

We thank the University of Malaya (PJP 152/91) and the National Science Council for Research and Development (grant No. 2-04-07-06) for supporting this work.

References

Bokii, N. G., Zakharova, G. N. & Struchkov, Yu. T. (1970). *J. Struct. Chem.* (*Engl. Transl.*) **11**, 895–902.

Fair, C. K. (1990). *MolEN Structure Determination System.* Delft Instruments, X-ray Diffraction B. V., Röntgenweg 1, 2624 BD Delft, The Netherlands.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Pelizzi, C. & Pelizzi, G. (1980*a*). *Inorg. Nucl. Chem. Lett.* **16**, 451–454.

Pelizzi, C. & Pelizzi, G. (1980*b*). *J. Organomet. Chem.* **202**, 411–419.

Rheingold, A. L., Ng, S. W. & Zuckerman, J. J. (1984). *Inorg. Chim. Acta*, **86**, 179–193.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1992). C**48**, 1841–1842

Carbonatobis(1,10-phenanthroline)cobalt(III) Perchlorate

By C. A. McAuliffe and R. G. Pritchard

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

and M. R. Bermejo, A. Garcia-Vazquez, A. Macias, J. Sanmartín, J. Romero and A. Sousa

Departamento de Quimica Inorganica, Universidad de Santiago de Compostela, 15706 Santiago, Spain

(*Received* 25 *May* 1991; *accepted* 12 *February* 1992)

Abstract. $[Co(CO_3)(C_{12}H_8N_2)_2](ClO_4)$, $M_r = 578.81$, monoclinic, $P2_1/n$, $a = 11.732\,(5)$, $b = 12.404\,(4)$, $c = 16.194\,(5)$ Å, $\beta = 108.80\,(3)°$, $V = 2231\,(1)$ Å^3, $Z = 4$, $D_x = 1.723$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.944$ mm^{-1}, $F(000) = 1176$, $T = 293$ K, $R = 0.063$, 2547 unique reflexions [$I \geq 2\sigma(I)$]. The cation has typical non-crystallographic twofold symmetry with the Co environment approximating a *cis* octahedron. There is a tendency for Co—N bonds *trans* to O atoms to be slightly longer than the *cis* bonds [1.936 (5), 1.951 (5) Å *cf* 1.922 (5), 1.931 (5) Å]; however, both coordinated carbonate O atoms are equidistant from Co [1.889 (4), 1.886 (4) Å].

Experimental. The compound was obtained during the synthesis of Co complexes of 2(1*H*)-pyridone by use of the electrochemical method proposed by Tuck (Habeeb, Tuck & Walters, 1978). The electrochemical oxidation of a Co anode in an acetonitrile solution (70 ml) containing 1,10-phenanthroline (0.25 g), 2(1*H*)-pyridone (0.20 g) and tetramethylammonium perchlorate (*ca* 10 mg) led to the formation of a brown solid. Crystals of the title compound, suitable for X-ray studies, were obtained by slow evaporation of the mother liquor at room temperature.

A crystal of dimensions 0.3 × 0.3 × 0.1 mm was used for data collection on a Rigaku AFC-6*S* diffractometer, with graphite-monochromated Mo $K\alpha$ radiation. Unit-cell dimensions were determined from the setting angles of 25 accurately centered reflexions ($12.4 \leq 2\theta \leq 28.3°$). Reflexions were measured using the ω–2θ scan mode with ω-scan width $(1.26 + 0.30\tan\theta)°$ and scan speed of 8° min^{-1}, with up to two additional scans of weak reflexions [$I < 10\sigma(I)$]; $0 \leq h \leq 13$, $0 \leq k \leq 14$, $-18 \leq l \leq 18$, $0 \leq \theta \leq 25°$. 4354 reflexions were measured, of which 4140 were unique ($R_{\rm int} = 0.072$) and 2547 observed [$I \geq 2\sigma(I)$]. Intensity standards (025, 033, 132) measured every 150 reflexions showed no decline. Lp and absorption (maximum/minimum transmission 0.86/1.0) corrections were applied. *MITHRIL* (Gilmore, 1984) was used to solve the phase problem. All non-H atoms were found in Fourier maps, H atoms from ΔF synthesis. Full-matrix least-squares refinement was based on F using *TEXSAN* (Molecular Structure Corporation, 1985) crystallographic software. Final $R = 0.063$ $\{wR = 0.062$, $w = 1/[\sigma^2(F_o) + (0.03F_o)^2]\}$, $S = 2.01$. Anisotropic thermal parameters were refined for heavier atoms, isotropic for H atoms. Maximum fluctuation in the final ΔF map was in the range -0.41 to 0.71 e Å^{-3}. Maxi-

0108-2701/92/101841-02\$06.00

Table 1. *Positional parameters and equivalent isotropic thermal parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$B_{\rm eq}$
Co(1)	0.74501 (8)	0.05831 (7)	0.36288 (6)	2.25 (3)
O(1)	0.8373 (4)	−0.0682 (3)	0.3989 (3)	2.8 (2)
O(2)	0.6457 (4)	−0.0645 (4)	0.3318 (3)	2.9 (2)
O(3)	0.7382 (5)	−0.2266 (3)	0.3676 (3)	4.0 (2)
N(1)	0.7814 (5)	0.0502 (4)	0.2555 (3)	2.7 (2)
N(2)	0.8763 (5)	0.1609 (4)	0.3962 (4)	2.5 (2)
N(3)	0.7066 (5)	0.0655 (4)	0.4701 (3)	2.5 (2)
N(4)	0.6243 (5)	0.1718 (4)	0.3249 (4)	2.4 (2)
C(1)	0.8870 (6)	0.0998 (5)	0.2615 (4)	2.5 (3)
C(2)	0.7261 (8)	−0.0034 (6)	0.1827 (5)	3.4 (3)
C(3)	0.7726 (9)	−0.0101 (7)	0.1157 (5)	4.1 (4)
C(4)	0.8783 (9)	0.0365 (6)	0.1209 (5)	4.1 (4)
C(5)	0.9405 (7)	0.0963 (6)	0.1960 (5)	3.2 (3)
C(6)	1.0480 (8)	0.1561 (7)	0.2095 (6)	4.3 (4)
C(7)	1.0954 (7)	0.2181 (7)	0.2805 (6)	4.3 (4)
C(8)	1.0401 (6)	0.2251 (6)	0.3468 (5)	3.3 (3)
C(9)	1.0780 (7)	0.2906 (7)	0.4224 (6)	4.2 (4)
C(10)	1.0169 (7)	0.2905 (6)	0.4800 (6)	4.0 (4)
C(11)	0.9164 (7)	0.2243 (6)	0.4670 (5)	3.2 (3)
C(12)	0.9371 (6)	0.1633 (5)	0.3377 (4)	2.3 (3)
C(13)	0.6107 (6)	0.1306 (5)	0.4635 (4)	2.3 (3)
C(14)	0.7510 (7)	0.0087 (6)	0.5436 (5)	3.1 (3)
C(15)	0.7015 (8)	0.0138 (6)	0.6099 (5)	3.4 (3)
C(16)	0.6057 (7)	0.0775 (6)	0.6036 (5)	3.4 (3)
C(17)	0.5578 (6)	0.1405 (5)	0.5282 (4)	2.6 (3)
C(18)	0.4592 (7)	0.2137 (6)	0.5134 (5)	3.3 (3)
C(19)	0.4212 (7)	0.2744 (6)	0.4400 (5)	3.4 (3)
C(20)	0.4748 (6)	0.2653 (5)	0.3735 (5)	2.7 (3)
C(21)	0.4445 (7)	0.3263 (6)	0.2949 (5)	3.5 (3)
C(22)	0.5013 (7)	0.3085 (6)	0.2379 (5)	3.3 (3)
C(23)	0.5938 (7)	0.2307 (6)	0.2526 (5)	3.0 (3)
C(24)	0.5679 (6)	0.1916 (5)	0.3848 (4)	2.2 (3)
C(25)	0.7387 (7)	−0.1279 (6)	0.3662 (5)	3.1 (3)
Cl(1)	0.3110 (2)	0.0775 (2)	0.1331 (1)	3.61 (8)
O(4)	0.4125 (5)	0.0577 (5)	0.1077 (4)	6.4 (3)
O(5)	0.3414 (6)	0.0853 (6)	0.2239 (4)	8.4 (4)
O(6)	0.2607 (7)	0.1784 (6)	0.0974 (5)	8.7 (4)
O(7)	0.2248 (6)	−0.0017 (6)	0.1041 (5)	9.1 (4)

Table 2. *Selected intramolecular distances* (Å) *and angles* (°) *involving the non*-H *atoms*

Co(1)—O(1)	1.889 (4)	Co(1)—N(4)	1.951 (5)
Co(1)—O(2)	1.886 (4)	O(1)—C(25)	1.331 (8)
Co(1)—N(1)	1.922 (5)	O(2)—C(25)	1.315 (8)
Co(1)—N(2)	1.936 (5)	O(3)—C(25)	1.224 (7)
Co(1)—N(3)	1.931 (5)		
O(1)—Co(1)—O(2)	69.8 (2)	O(2)—Co(1)—N(3)	88.5 (2)
O(1)—Co(1)—N(1)	87.9 (2)	O(2)—Co(1)—N(4)	100.1 (2)
O(1)—Co(1)—N(2)	97.8 (2)	N(1)—Co(1)—N(2)	84.1 (2)
O(1)—Co(1)—N(3)	92.1 (2)	N(3)—Co(1)—N(4)	83.9 (2)
O(2)—Co(1)—N(1)	90.8 (2)		

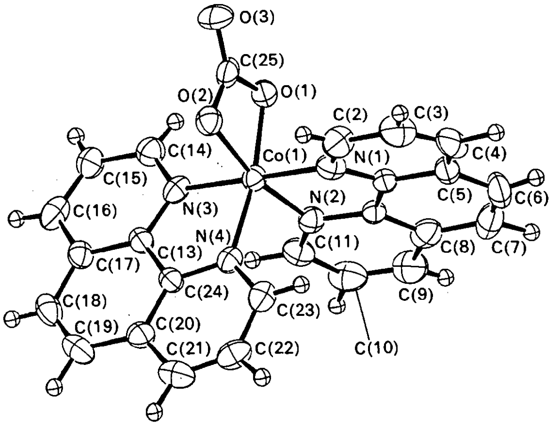

Fig. 1. The title structure, excluding the perchlorate anion, drawn using *ORTEP*II (Johnson, 1976)

mum $\Delta/\sigma = 0.01$. Scattering factors were obtained from Cromer & Waber (1974). Computation was performed on a Digital VAX station 3520. A literature survey was performed *via* the Cambridge Structural Database using the crystal structure search and retrieval interactive system (CSSR, 1984). Fractional atomic coordinates and vibrational parameters for non-H atoms are presented in Table 1* and selected bond lengths and angles in Table 2. The title molecular cation, including atomic labelling, is displayed in Fig. 1.

Related literature. The structure of the cation of the title compound has been reported in the form of a triclinic (Hennig, Sieler, Benedix, Kaiser, Sjolin & Lindqvist, 1980) and monoclinic (Niederhoffer, Martell, Rudolf & Clearfield, 1982) bromide tetrahydrate and as a chloride trihydrate (Guild, Hayden & Brennan, 1980). Although all four structures are very similar, the present case exhibits the most extreme ligand bowing, with one peripheral C atom deviating from the ligand least-squares plane by 0.119 (8) Å.

We thank the Xunta de Galicia for a grant.

References

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2A. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual.* SERC Daresbury Laboratory, Warrington, England.

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

Guild, B. C., Hayden, T. & Brennan, T. F. (1980). *Cryst. Struct. Commun.* **9**, 371–376.

Habeeb, J. J., Tuck, D. G. & Walters, F. H. (1978). *J. Coord. Chem.* **27**, 8–12.

Hennig, H., Sieler, J., Benedix, R., Kaiser, J., Sjolin, L. & Lindqvist, O. (1980). *Z. Anorg. Allg. Chem.* **464**, 151–158.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.

Niederhoffer, E. C., Martell, A. E., Rudolf, P. & Clearfield, A. (1982). *Inorg. Chem.* **21**, 3734–3741.

---

* Lists of structure factors, H-atom coordinates, anisotropic vibrational parameters, bond distances and angles, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55187 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0481]