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Structure of $[Et_4N][InI\{Co(CO)_4\}_3]$

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Abstract. Tetraethylammonium iodotris(tetracarbonylcobaltio)indate(1−), $C_8H_{20}N^+.C_{12}Co_3IInO_{12}^-$, $M_r = 884.9$, monoclinic, $P2_1/c$, $a = 11.341$ (1), $b = 16.551$ (1), $c = 16.429$ (1) Å, $\beta = 92.110$ (6)°, $V = 3081.7$ (4) Å^3, $Z = 4$, $D_x = 1.91$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 33.6$ cm^{-1}, $F(000) = 1704$, $T = 298$ K, $R = 0.039$ for 3647 unique observed reflections. The anion contains an indium atom coordinated to an iodine and three cobalt atoms in a slightly distorted tetrahedral environment.

Introduction. There are several examples of cobalt carbonyl complexes containing indium (Clarkson, McCrudden, Norman & Farrugia, 1990) and herein we report the structure of $[Et_4N][InI\{Co(CO)_4\}_3]$ (1). Compound (1) completes a series of the general formula $[InX_n\{Co(CO)_4\}_{4-n}]^-$ (*A*), where *X* is a halide. Previous examples are $[PPN][InBr_3\{Co(CO)_4\}]$ (2) ($PPN = Ph_3PNPPh_3^+$] (Burlitch, Leonowicz, Petersen & Hughes, 1979), $[Et_4N][InBr_2\{Co(CO)_4\}_2]$ (3) (Cradwick, 1971), $[Q][InCl_2\{Co(CO)_4\}_2]$ [Q = PPN (4); $Q = Co(CO)_3(PPh_3)_2$ (5)] (Clarkson *et al.*, 1990) and $[Ph_4As][In\{Co(CO)_4\}_4]$ (6) (Robinson & Schussler, 1971). Compounds (3)–(5) have been structurally characterized. A crystal of (1) was obtained from the reaction between $K[Co(CO)_4]$ and $[In\{Co(CO)_4\}_3]$ followed by addition of $[Et_4N]I$ and crystallization from CH_2Cl_2/hexane mixtures, although this procedure afforded $[Et_4N][In\{Co(CO)_4\}_4]$ as the main product. With regard to the formation of (1), we note that the reaction between $[In\{Co(CO)_4\}_3]$ and $[Ph_4As]Cl$ was reported (Robinson & Schussler, 1971) to give a compound $[Cl(In\{Co(CO)_4\}_3)_2]^-$, with no evidence for a 1:1 indium-halide species. Compound (1) is therefore the first of type *A* with $n = 1$.

Experimental. Yellow prisms from dichloromethane/hexane solution: crystal dimensions *ca* 0.4 × 0.4 × 0.5 mm; systematic absences: $k = 2n + 1$ in $0k0$; $l = 2n + 1$ in $h0l$; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters refined by least-squares methods from setting angles of 25 independent reflections with $11 < \theta < 13°$; intensities measured to $\theta = 25.0°$ over hkl range 0 to 13, 0 to 19, −19 to 19; $\bar{2}\bar{7}7$, $\bar{1}$,11,0 and $\bar{3}$,10,$\bar{1}$ measured every 2 h with a 5% decay over 49.5 h data collection; 5398 data measured, 5423 independent data with 3647 having $I > 3.0\sigma(I)$ considered observed and used in structure determination and refinement; R_{int} 0·134 before and 0·036 after absorption correction; corrected for decomposition, Lp and absorption (*DIFABS*; Walker & Stuart, 1983), max., min. values of applied absorption correction 1.27, 0.73. Solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent full-matrix least squares; anisotropic thermal parameters for all non-H atoms, fixed isotropic thermal parameters ($U = 0.08$ Å^2) for H atoms; H atoms included at calculated positions (C—H = 1.0 Å); $\sum w(|F|_o -$

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Table 1. *Fractional coordinates with e.s.d.'s in parentheses and isotropic thermal parameters* ($Å^2$)

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

	x	y	z	U_{eq}
In	0.78230 (4)	0.06806 (3)	0.23449 (3)	0.051
Co(1)	0.73654 (10)	0.22235 (6)	0.19311 (6)	0.065
Co(2)	0.60500 (9)	−0.03652 (6)	0.19276 (6)	0.061
Co(3)	0.99580 (9)	0.01570 (6)	0.18884 (6)	0.057
I	0.79366 (7)	0.07058 (4)	0.40511 (3)	0.094
N	0.2692 (5)	0.2991 (3)	−0.0075 (3)	0.057
O(11)	0.5006 (7)	0.1942 (4)	0.2492 (5)	0.125
O(12)	0.9056 (8)	0.2665 (4)	0.3236 (5)	0.121
O(13)	0.8117 (7)	0.1677 (4)	0.0362 (4)	0.102
O(14)	0.7033 (8)	0.3898 (4)	0.1472 (5)	0.144
O(21)	0.4287 (6)	−0.1531 (3)	0.1341 (4)	0.097
O(22)	0.6014 (6)	0.0533 (4)	0.0405 (4)	0.105
O(23)	0.4791 (9)	0.0277 (5)	0.3294 (6)	0.178
O(24)	0.7726 (6)	−0.1617 (4)	0.2377 (4)	0.102
O(31)	1.0745 (6)	0.1810 (4)	0.2132 (5)	0.123
O(32)	0.8696 (6)	−0.0342 (4)	0.0405 (4)	0.098
O(33)	0.9965 (6)	−0.0894 (4)	0.3318 (4)	0.105
O(34)	1.2262 (7)	−0.0362 (5)	0.1396 (5)	0.130
C(1)	0.1665 (9)	0.3145 (6)	0.0430 (6)	0.097
C(2)	0.0547 (10)	0.2706 (8)	0.0103 (7)	0.126
C(3)	0.2461 (11)	0.3331 (7)	−0.0928 (6)	0.118
C(4)	0.2068 (12)	0.4171 (7)	−0.0999 (7)	0.122
C(5)	0.3708 (11)	0.3469 (7)	0.0335 (6)	0.113
C(6)	0.4858 (11)	0.3402 (10)	−0.0067 (8)	0.159
C(7)	0.2991 (11)	0.2132 (6)	−0.0114 (7)	0.116
C(8)	0.3250 (11)	0.1681 (6)	0.0661 (7)	0.114
C(11)	0.5940 (9)	0.2009 (5)	0.2268 (6)	0.083
C(12)	0.8431 (9)	0.2451 (5)	0.2741 (6)	0.087
C(13)	0.7820 (8)	0.1875 (5)	0.0991 (6)	0.077
C(14)	0.7160 (9)	0.3240 (6)	0.1637 (6)	0.090
C(21)	0.4946 (7)	−0.1065 (5)	0.1570 (5)	0.070
C(22)	0.6073 (7)	0.0195 (5)	0.1027 (6)	0.073
C(23)	0.5321 (10)	0.0060 (6)	0.2755 (7)	0.113
C(24)	0.7128 (8)	0.1088 (5)	0.2208 (5)	0.070
C(31)	1.0379 (7)	0.1170 (6)	0.2051 (5)	0.074
C(32)	0.9153 (7)	−0.0136 (5)	0.1005 (5)	0.071
C(33)	0.9917 (7)	−0.0474 (5)	0.2767 (5)	0.072
C(34)	1.1358 (9)	−0.0178 (6)	0.1562 (6)	0.086

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

In—Co(1)	2.688 (1)	In—Co(2)	2.722 (1)
In—Co(3)	2.704 (1)	In—I	2.802 (1)
Co(1)—C(11)	1.764 (11)	Co(1)—C(12)	1.804 (11)
Co(1)—C(13)	1.745 (10)	Co(1)—C(14)	1.764 (10)
Co(2)—C(21)	1.788 (9)	Co(2)—C(22)	1.747 (9)
Co(2)—C(23)	1.764 (12)	Co(2)—C(24)	1.760 (9)
Co(3)—C(31)	1.761 (10)	Co(3)—C(32)	1.754 (9)
Co(3)—C(33)	1.784 (9)	Co(3)—C(34)	1.782 (10)
N—C(1)	1.477 (12)	N—C(3)	1.524 (11)
N—C(5)	1.533 (13)	N—C(7)	1.463 (13)
O(11)—C(11)	1.139 (14)	O(12)—C(12)	1.117 (13)
O(13)—C(13)	1.147 (12)	O(14)—C(14)	1.131 (12)
O(21)—C(21)	1.129 (11)	O(22)—C(22)	1.165 (12)
O(23)—C(23)	1.146 (16)	O(24)—C(24)	1.136 (11)
O(31)—C(31)	1.144 (12)	O(32)—C(32)	1.148 (11)
O(33)—C(33)	1.141 (11)	O(34)—C(34)	1.113 (13)
C(1)—C(2)	1.541 (16)	C(3)—C(4)	1.463 (17)
C(5)—C(6)	1.487 (18)	C(7)—C(8)	1.495 (16)
Co(1)—In—Co(3)	113.6 (1)	Co(1)—In—Co(2)	114.0 (1)
Co(2)—In—Co(3)	112.8 (1)	Co(1)—In—I	103.9 (1)
Co(3)—In—I	105.9 (1)	Co(2)—In—I	105.5 (1)

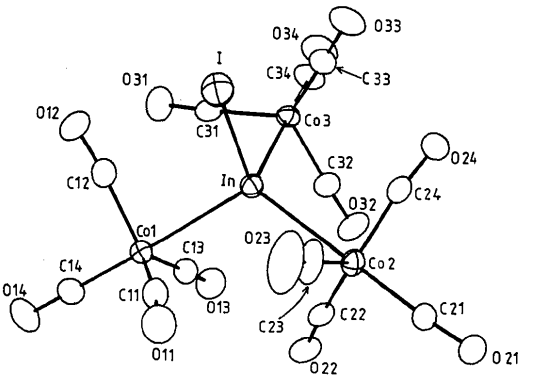

Fig. 1. Molecular structure and atomic labelling scheme for the anion $[InI\{Co(CO)_4\}_3]^-$.

$|F|_c)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max. Δ/σ 0.06, av. 0.01; $\Delta\rho_{max}$ + 0.79, $\Delta\rho_{min}$ − 1.07 e $Å^{-3}$ in vicinity of Et_4N cation; final $R = 0.039$, $wR = 0.050$, using 343 parameters; $S = 2.20$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71–151); calculations carried out on a MicroVAX 3600 computer using the Glasgow *GX* suite of programs (Mallinson & Muir, 1985).

Discussion. Final positional parameters are given in Table 1, with selected bond distances and angles in Table 2.* The structure consists of NEt_4^+ cations and $[InI\{Co(CO)_4\}_3]^-$ anions separated by normal van der Waals distances. The atomic labelling scheme and molecular structure of the anion is shown in Fig. 1. This anion contains an In atom in a tetrahedral environment, which is slightly distorted owing to the steric bulk of the $Co(CO)_4$ substituents. Thus the I—In—Co angles are in the range 103.9 (1)–105.9 (1)°, while the Co—In—Co angles range from 112·8 (1)–114·0 (1)°. The In—Co distances average 2·705 Å, which is ~0·1 Å longer than found in other related In–Co compounds (Clarkson *et al.*, 1990). The overall geometry is very similar to that recently reported for the isoelectronic species $[SnCl\{Co(CO)_4\}_3]$ (Klüfers, 1991). The In atom occupies an axial site with respect to the trigonal bipyramidal geometry around each cobalt, a feature observed in all other related species.

* Lists of structure factors, anisotropic thermal parameters, full bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54409 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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