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{[μ,η^2-Bis(trimethylstannyl)]acetylene}hexacarbonyl-dicobalt

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.034
wR factor = 0.091
Data-to-parameter ratio = 21.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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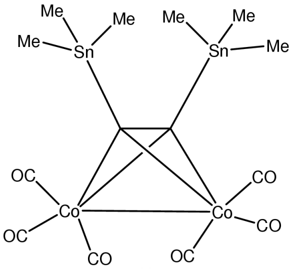
The structure of the title compound, [$Co_2(C_8H_{18}Sn_2)(CO)_6$], a distannyl-substituted dicobaltatetrahedrane, shows geometrical parameters noticeably different from those observed in the only monostannyl-substituted dicobaltatetrahedrane structurally studied so far. Thus, the complexed acetylenic bond in the title compound is shorter, and the cobalt–cobalt and cobalt–carbon bonds are longer, while the Sn—C—C angles are much smaller than in the complex with monostannyl-substituted ligand.

Comment

The title compound, (I), was prepared thirty years ago, but the nature of the bonding in the crystal remained unknown until now. The tin–carbon bond in stannyl alkynes is relatively labile, though these can be used in Stille-type palladium-catalysed cross-coupling reactions with organyl halides (Mitchell, 1998). However, the reactivity of the carbon–carbon triple bond is such that oligomeric and polymeric side-products are obtained in large amounts. Complexation of the carbon–carbon triple bond by the dicobalt hexacarbonyl moiety to give a dicobaltatetrahedrane structure does not hinder cross-coupling with activated organyl halides (Zavgorodnii *et al.*, 2000). The title compound is air- and moisture-insensitive, whereas the uncomplexed bis(trimethylstannyl)acetylene is easily hydrolysed by atmospheric moisture.



(I)

Only one dicobaltatetrahedrane derived from a stannyl alkyne has so far been studied by X-ray crystallography (Wrackmeyer *et al.*, 1997) but this compound (*A*) could be atypical for steric reasons, as it is prepared by reacting diphenyldiethynyltin $Ph_2Sn(C{\equiv}CH)_2$ with dicobalt octacarbonyl, both acetylenic bonds being converted to dicobaltatetrahedrane moieties.

The structure of (I) is shown in Fig. 1. The length of the complexed acetylenic triple bond in this structure is 1.309 (5) Å, as compared with 1.238 Å in the uncomplexed

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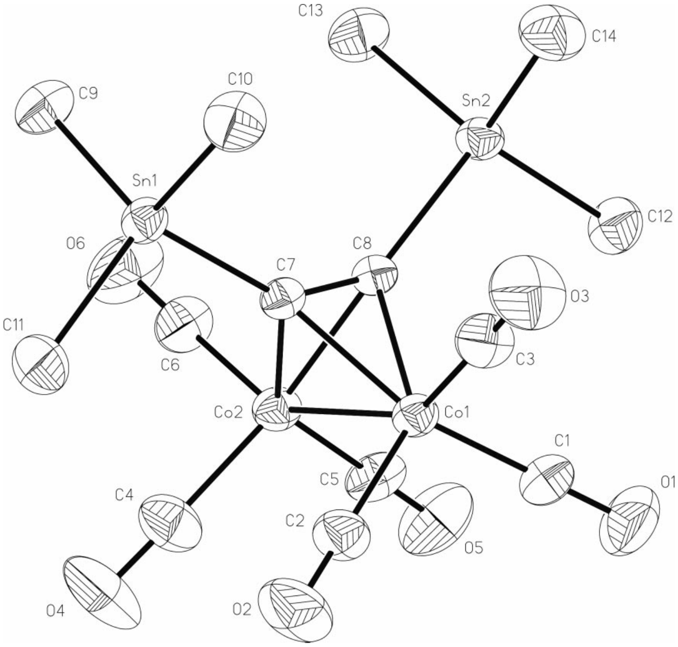

Figure 1
View of the title compound showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 30% probability level. H atoms are omitted for clarity.

stannyl acetylene and 1.212 (1) Å in acetylene itself (Khaikin *et al.*, 2000); it is markedly shorter than in (*A*) [1.322 (9) and 1.335 (9) Å]. The cobalt–cobalt distance (2.5043(9) Å) is considerably longer than in (*A*) [2.4788 (2) and 2.487 (2) Å]. While in (*A*) the cobalt–carbon bond lengths in the dicobaltatetrahedrane moiety lie between 1.944 and 1.990 Å (the authors describe these as 'normal' values), all four Co—C bonds in the title compound are equal or longer than 2.000 Å (up to 2.007 Å). The originally linear Sn—C—C—Sn arrangement is naturally considerably bent, the angles Sn—C—C being 144.7 (3)° and 145.7 (3)°; in (A), however, the bond angles are much larger, *viz*. 150.5 (5) and 151.5 (5)°.

Experimental

The compound was prepared in 66% yield as a dark red solid, decomposing at 373 K, according to the procedure described previously (Seyferth & White, 1971).

Crystal data

$[Co_2(C_8H_{18}Sn_2)(CO)_6]$
$M_r = 637.52$
Triclinic, $P\overline{1}$
$a = 8.659$ (4) Å
$b = 10.0360$ (17) Å
$c = 13.292$ (3) Å
$\alpha = 91.731$ (16)°
$\beta = 91.17$ (3)°
$\gamma = 109.09$ (2)°
$V = 1090.5$ (6) Å^3

$Z = 2$
$D_x = 1.942$ Mg m^{-3}
Ag *K*α radiation
Cell parameters from 25 reflections
$\theta = 12.1$–15.4°
$\mu = 1.98$ mm^{-1}
$T = 293$ (1) K
Plate, red–brown
0.40 × 0.30 × 0.05 mm

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*WinGX*; Farrugia, 1999; North *et al.*, 1968)
$T_{min} = 0.620$, $T_{max} = 0.997$
9426 measured reflections
4713 independent reflections
3813 reflections with $I > 2\sigma(I)$

$R_{int} = 0.019$
$\theta_{max} = 21.0°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.091$
$S = 1.03$
4713 reflections
224 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.15$ e Å^{-3}
$\Delta\rho_{min} = -1.54$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0075 (11)

H atoms were placed in calculated positions and included in the refinement in the riding motion approximation with U_{iso} constrained to be 1.5 times U_{eq} of the carrier atom for the methyl–H and 1.2 times U_{eq} for the remaining H atoms. The highest peak in the final difference map is at a distance of 0.84 Å from Sn2.

Data collection: *CAD*-4 *Software* (Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms, 1996); program(s) used to solve structure: *SHELXS*-97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997), *PARST*95 (Nardelli, 1995).

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