Crystal and Molecular Structure of a Racemic Complex: μ-[1—3,6-η:1,4—6-η-1,3,6-tris(trifluoromethyl)hexa-1,3,5-trien-1,6-diyl]-bis(dicarbonylcobalt)(*Co–Co*)

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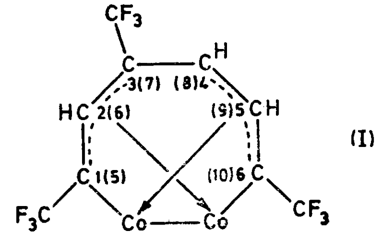
The structure of the title compound (I) has been determined from three-dimensional photographic *X*-ray data. The complex crystallizes as a racemate in the orthorhombic space group $Pna2_1$ (No 33) with $a = 9{\cdot}92$, $b = 11{\cdot}97$, $c = 13{\cdot}70$ Å, $Z = 4$. The structure was refined by least-squares methods to R 0·097 for 757 independent observed reflections. The complex contains a six-carbon atom ligand bridging two $[Co(CO)_2]$ moieties. Trifluoromethyl groups are attached to carbon atoms 1, 3, and 6 within the six-carbon atom ligand. The bridging ligand is composed of two allylic functions joined by a C–C single bond; there is a dihedral angle of 60° between the two allylic planes. The allyl groups are each bonded to both cobalt atoms, which are separated by a normal bonding distance of 2·459(6) Å. The cobalt atoms are seven-co-ordinate if the allyl groups are regarded as tridentate ligands. The two cobalt atoms and the two terminal carbon atoms of the C_6-chain are coplanar; there is a symmetrical arrangement of these four atoms.

THE formation of complexes of the general stoicheiometry $Co_2(CO)_4(alkyne)_3$ has been discussed in detail by Krüerke and Hübel[1] and more recently by Dickson and Fraser.[2] These complexes can be chemically or thermally decomposed to yield substituted benzenes[3] and consequently they can be regarded as possible intermediates in the carbonylcobalt catalysed trimerization of alkynes.[4,5]

Structural data on one of these complexes, $Co_2(CO)_4(HC_2H)(Bu^tC_2H)_2$, has been reported briefly.[6] The complex contains an unusual six-carbon atom bridging group, termed a 'flyover', which is formed by the head-to-tail oligomerization of three alkyne molecules. A similar structure has been suggested[7] for the related complex, $Co_2(CO)_4(PhC_2CO_2Me)_3$, but a detailed discussion of the structure has not been reported.



(I)

(I) Systematic numbering with crystallographic numbering in parentheses

In a previous paper,[8] the formation of the complex $Co_2(CO)_4(CF_3C_2H)_3$, (I) which presumably has an analogous 'flyover' structure, was reported. The probable arrangement of the trifluoromethyl substituents in the 'flyover' fragment of $Co_2(CO)_4(CF_3C_2H)_3$ in solution was deduced[2,9] by analysis of the ^1H and ^{19}F n.m.r. spectra of this and of several closely related complexes.

We now report the crystal structure of (I) determined in order to learn if the solid-state structure would confirm the configuration deduced for the complex in solution. We had recognized the possibility of chirality in such complexes, and were interested to observe the manner in which the anticipated chiral forms were packed in the crystal. In view of the experimental observation that these complexes can be degraded to substituted benzenes, we were interested to study the bond parameters for $Co_2(CO)_4(CF_3C_2H)_3$ together with those previously reported for $Co_2(CO)_4(HC_2H)(Bu^tC_2H)_2$ to seek a better understanding of the factors which control this decomposition.

EXPERIMENTAL

Suitable crystals of the purple complex (I) were grown from a saturated pentane solution at −20 °C. A single crystal (0·2 × 0·2 × 0·15 mm) was mounted inside a sealed glass capillary and integrated data were collected by use of the multifilm pack method, with iron-filtered Co-K_α radiation and a Nonius camera. Unit-cell dimensions were calculated from Weissenberg and oscillation photographs.

Crystal Data.—$C_{13}H_3F_9O_4Co_2$, $M = 512{\cdot}02$, Orthorhombic, $a = 9{\cdot}92$ (2), $b = 11{\cdot}97$ (2), $c = 13{\cdot}70$ (2) Å, $U = 1627$ (1) Å^3, $D_m = 1{\cdot}97$ (3) (by flotation), $Z = 4$, $D_c = 2{\cdot}09$, $F(000) = 992$. Co-K_α radiation $\lambda = 1{\cdot}790$ Å; $\mu(\text{Co-}K_\alpha) = 34{\cdot}8$ cm^{-1}. Space group $Pnma$ (D_{2h}^{16}, No. 62) or $Pna2_1$ (C_{2v}^{9}, No. 33) from systematic absences: $0kl$ for $k + l$ odd and $h0l$ for h odd; the latter shown to be correct by the subsequent successful refinement. The piezoelectric effect was not investigated. With $Pna2_1$ no crystallographic symmetry is imposed on the molecules. The intensities of the reflections in the levels 0—$7kl$ were estimated visually; 985 independent reflections were measured of which 228 were unobserved; all reflections were corrected for Lorentz and polarization effects; no corrections were made for extinction, absorption, or anomalous dispersion.

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[1] U. Krüeke and W. Hübel, *Chem. Ber.*, 1961, **94**, 2829.

[2] R. S. Dickson and P. J. Fraser, *Austral. J. Chem.*, 1970, **23**, 475.

[3] U. Krüeke, C. Hoogzand, and W. Hübel, *Chem. Ber.*, 1961, **94**, 2817.

[4] C. Hoogzand and W. Hübel, 'Organic Syntheses *via* Metal Carbonyls,' vol. 1, eds. I. Wender and P. Pino, Interscience, New York, 1968, p. 343.

[5] G. M. Whitesides and W. J. Ehmann, *J. Amer. Chem. Soc.*, 1969, **91**, 3800.

[6] O. S. Mills and G. Robinson, *Proc. Chem. Soc.*, 1964, 187.

[7] F. S. Stephens, *Acta Cryst.*, 1966, **21**, *A*, 154.

[8] R. S. Dickson and D. B. W. Yawney, *Austral. J. Chem.*, 1967, **20**, 77.

[9] R. S. Dickson and D. B. W. Yawney, *Austral. J. Chem.*, 1969, **22**, 533.

Structure Solution and Refinement.—The structure was solved by a combination of direct and heavy-atom methods. A modified set of the DP suite of programs[10] was used to calculate normalized structure factors which were sorted in magnitude. The E-statistics indicated almost ideal noncentricity.[11] One set of phases for 208 reflections with normalized structure factors $|E| \geqslant 1{\cdot}30$ was determined from the MULTAN suite of programs.[12] An E-map was computed from this set of phases and revealed the cobalt positions as the two largest peaks. These metal-atom positions were confirmed by a three-dimensional Patterson synthesis. The E-map and initial difference-Fourier syntheses were confused by the presence of a pseudo-mirror plane through the cobalt positions. Careful choice of a few initial atoms subsequently destroyed this false symmetry. Then least-squares refinement and corresponding difference-Fourier electron-density syntheses revealed all other non-hydrogen atom positions. This approximate structure, for which the isotropic temperature factors for cobalt, carbon, fluorine, and oxygen were assigned values of 3, 4, 4, and 4 Å^2 gave R 0·21. Several cycles of full-matrix least-squares refinement using observed reflections only and refining individual scales, all atomic positional parameters except Co(1), $z = \frac{1}{2}$ (in order to define the origin), and isotropic temperature factors resulted in R converging to 0·097. The positions of the three hydrogen atoms in the molecule were calculated assuming C–H 1·05 Å, and these atoms were incorporated in the model with temperature factors equal to those of the carbon atoms to which they were attached [C(6)–H(1), C(8)–H(2), and C(9)–H(3)]. The positional and thermal parameters of these hydrogen atoms were not refined. Incorporation of the 288 unobserved reflections, followed by several cycles of refinement, gave R 0·116. In the final cycle no parameter shifted by $>0{\cdot}3\ \sigma$ and the final difference-Fourier synthesis gave no indication of misplaced atoms or evidence of anisotropic thermal motion of the metal atoms. The final difference-Fourier synthesis revealed no electron density maxima $>0{\cdot}75$ eÅ^{-3}. The atomic scattering factors for cobalt, carbon, fluorine, oxygen, and hydrogen were taken from ref. 13.

RESULTS AND DISCUSSION

The final atomic co-ordinates and isotropic thermal parameters are given in Table 1. Observed and calculated structure factors are listed in Supplementary Publication No. SUP 20439 (4 pp., 1 microfiche). Details of intramolecular distances, interatomic angles, dihedral angles, and least-square planes are given in Tables 2—5.

Discussion of the Structure.—A representation of the molecular structure of $Co_2(CO)_4(CF_3C_2H)_3$ is shown in Figure 1 which also indicates the labelling of the atoms. Figure 2 is a crystal packing diagram which shows the right- and left-handed molecules related by the glide planes n and a. These chiral forms pack together to form a crystal racemate. There are no unusual intermolecular contacts, the shortest being 2·50 Å between the idealized hydrogen position H(1) and F(7) transformed by $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} + z$.

The basic molecular structure consists of a six-carbon atom ligand bridging two $[Co(CO)_2]$ moieties. The bridging ligand is composed of two allyl functions, defined by C(5), C(6), C(7) and C(8), C(9), C(10) and joined by a single bond between C(7) and C(8). The

TABLE 1

Final fractional co-ordinates and thermal parameters (Å^2) with their estimated standard deviation in parentheses

Atom	x/a	y/b	z/c	B/Å^2
Co(1)	0·4249(5)	0·0520(3)	0·5000	4·061(6)
Co(2)	0·1947(5)	−0·0220(3)	0·4842(5)	3·619(6)
C(1)	0·505(4)	0·011(3)	0·393(2)	4·10(4)
C(2)	0·573(4)	0·057(2)	0·568(2)	3·86(4)
C(3)	0·208(4)	−0·108(2)	0·381(2)	3·53(4)
C(4)	0·016(4)	0·003(2)	0·471(2)	3·84(4)
C(5)	0·348(3)	−0·085(2)	0·555(2)	2·65(3)
C(6)	0·246(3)	−0·072(2)	0·623(2)	3·15(4)
C(7)	0·205(3)	0·040(2)	0·621(2)	2·53(3)
C(8)	0·314(3)	0·131(2)	0·612(2)	2·43(3)
C(9)	0·306(3)	0·191(2)	0·519(2)	2·23(3)
C(10)	0·207(3)	0·122(2)	0·438(2)	2·55(3)
C(11)	0·423(4)	−0·210(3)	0·543(2)	4·08(5)
C(12)	0·100(3)	0·074(2)	0·685(2)	2·46(3)
C(13)	0·245(4)	0·173(3)	0·336(3)	4·52(4)
F(1)	0·466(3)	−0·224(2)	0·456(2)	7·28(4)
F(2)	0·337(3)	−0·284(2)	0·569(2)	7·30(4)
F(3)	0·522(3)	−0·211(2)	0·609(2)	6·90(3)
F(4)	0·030(2)	0·165(2)	0·647(2)	6·21(3)
F(5)	−0·003(2)	0·000(2)	0·692(1)	5·65(3)
F(6)	0·123(2)	0·104(2)	0·773(1)	5·74(3)
F(7)	0·316(2)	0·268(2)	0·328(2)	6·00(3)
F(8)	0·273(3)	0·105(2)	0·271(2)	7·14(3)
F(9)	0·114(3)	0·205(2)	0·332(2)	6·85(3)
O(1)	0·556(3)	−0·007(2)	0·319(2)	6·60(4)
O(2)	0·668(3)	0·067(2)	0·618(2)	6·65(4)
O(3)	0·207(3)	−0·166(2)	0·312(2)	5·06(3)
O(4)	−0·091(3)	0·022(2)	0·457(2)	4·66(3)
H(1)	0·203	−0·130	0·669	
H(2)	0·387	0·153	0·666	
H(3)	0·326	0·275	0·507	

TABLE 2

Intramolecular bonding and selected non-bonding distances (Å), with estimated standard deviations in parentheses

(*a*) Bonding distances

Co(1)–Co(2)	2·459(6)	C(5)–C(6)	1·39(3)
Co(1)–C(1)	1·73(3)	C(6)–C(7)	1·39(4)
Co(1)–C(2)	1·74(4)	C(7)–C(8)	1·54(4)
Co(2)–C(3)	1·76(3)	C(8)–C(9)	1·47(3)
Co(2)–C(4)	1·80(4)	C(9)–C(10)	1·42(4)
C(1)–O(1)	1·16(4)	C(5)–C(11)	1·69(4)
C(2)–O(2)	1·17(4)	C(7)–C(12)	1·42(4)
C(3)–O(3)	1·16(3)	C(10)–C(13)	1·54(4)
C(4)–O(4)	1·11(3)	C(11)–F(1)	1·27(3)
Co(1)–C(5)	1·96(3)	C(11)–F(2)	1·28(4)
Co(1)–C(10)	1·95(3)	C(11)–F(3)	1·34(4)
Co(2)–C(5)	1·95(3)	C(12)–F(4)	1·40(3)
Co(2)–C(10)	1·98(3)	C(12)–F(5)	1·35(3)
Co(1)–C(8)	2·11(3)	C(12)–F(6)	1·28(3)
Co(1)–C(9)	2·05(3)	C(13)–F(7)	1·34(3)
Co(2)–C(6)	2·06(3)	C(13)–F(8)	1·24(4)
Co(2)–C(7)	2·02(3)	C(13)–F(9)	1·36(4)

(*b*) Non-bonding distances

C(5) ··· C(10)	3·05(4)	F(4) ··· F(6)	2·09(3)
F(1) ··· F(2)	2·13(3)	F(5) ··· F(6)	2·08(3)
F(1) ··· F(3)	2·17(3)	F(7) ··· F(8)	2·15(3)
F(2) ··· F(3)	2·11(3)	F(7) ··· F(9)	2·15(3)
F(4) ··· F(5)	2·09(3)	F(8) ··· F(9)	2·16(3)

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[10] S. R. Hall, Crystallographic programs for the PDP 6 Computer, University of Western Australia, 1968.

[11] G. H. Stout and L. H. Jensen, '*X*-Ray Structure Determination,' Macmillan, London, 1968, p. 321.

[12] P. Main, M. Woolfson, and G. Germain, MULTAN series of programs.

[13] 'International Tables for *X*-Ray Crystallography', vol. III, Kynoch Press, Birmingham, 1962, p. 202.

allyl groups are each bonded to both cobalt atoms. This bonding can be described in terms of σ-bonds between Co(1) and C(5) and between Co(2) and C(10), and π-bonds between Co(1) and the allyl group C(8), C(9), C(10) and between Co(2) and the other allyl group C(5), C(6), C(7). The mean Co–C(allyl) π-bond is 2·02 Å and the σ-bond 1·97 Å. The distinction between metal–carbon π and σ bonds is made only on the basis of metal–ligand geometry (Figure 3); note the symmetrical arrangement of atoms within the Co(1), C(5), C(10), Co(2) segment of the molecule (Figure 4).

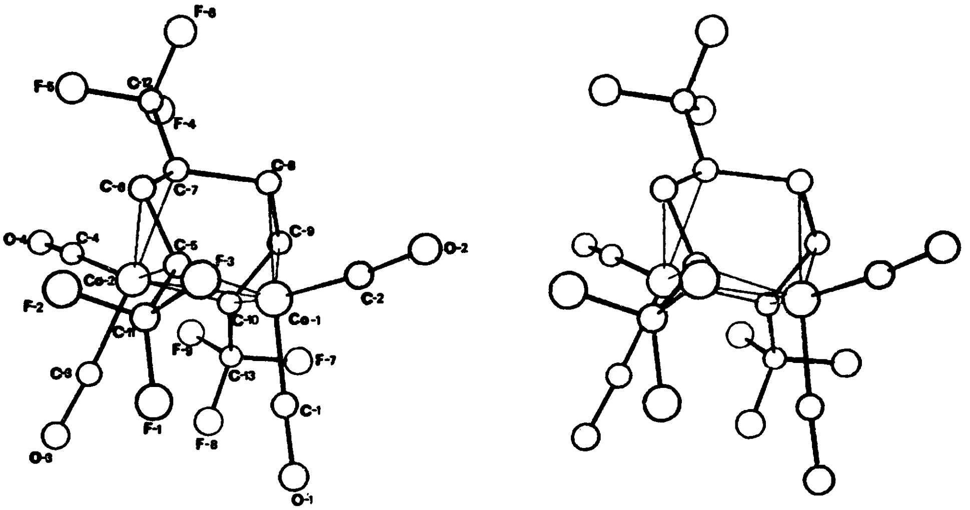

Figure 1 A stereoscopic view of the molecular structure indicating the atom labelling used in the analysis

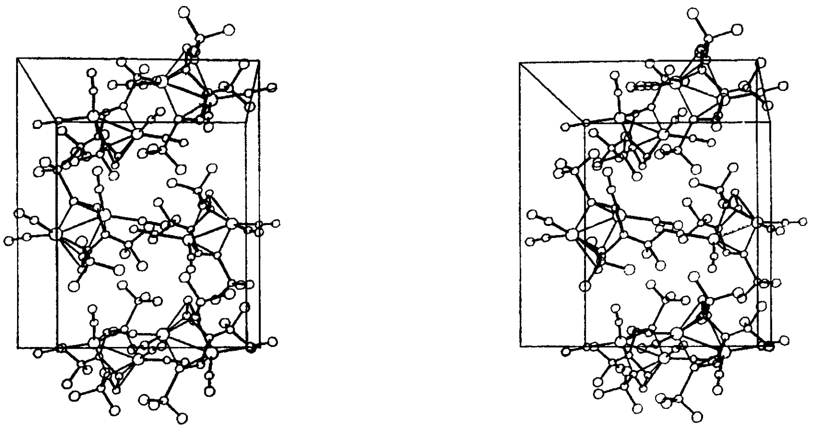

Figure 2 A stereoscopic view of the molecular packing in the unit cell viewed down the *c* axis

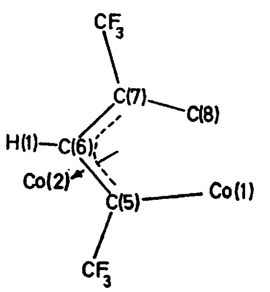

Figure 3 A representation of σ and π contributions to the metal–ligand bonding for one allyl group. The Co–Co bond is omitted for clarity

The two cobalt atoms are separated by 2·459 Å and this is typical of metal–metal bond distances in binuclear carbonylcobalt complexes.[14] Consequently, each cobalt atom is seven-co-ordinate if the allyl groups

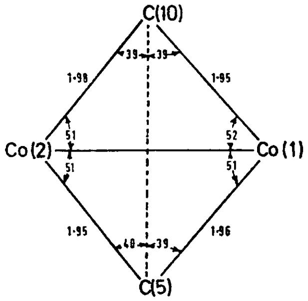

Figure 4 The symmetrical arrangement of atoms within the Co(1), C(5), C(10), Co(2) (Plane 3) segment of the molecule

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[14] W. Harrison and J. Trotter, *J. Chem. Soc.* (*A*), 1971, 1607.

TABLE 3

Selected intramolecular angles (°) with estimated standard deviations in parentheses

Co(1)–C(1)–O(1)	173·9(6)	Co(2)–C(5)–C(10)	39·7(7)
Co(1)–C(2)–O(2)	174·6(5)	Co(1)–C(10)–C(5)	38·9(8)
Co(2)–C(3)–O(3)	175·4(5)	Co(2)–C(10)–C(5)	38·8(8)
Co(2)–C(4)–O(4)	174·9(5)	Co(1)–C(5)–Co(2)	78·2(1·0)
Co(1)–Co(2)–C(3)	102·4(1·1)	Co(1)–C(10)–Co(2)	77·6(9)
Co(2)–Co(1)–C(1)	104·2(1·3)	Co(1)–C(5)–C(11)	122·5(2·0)
Co(1)–Co(2)–C(4)	149·5(8)	Co(2)–C(10)–C(13)	125·4(2·0)
Co(2)–Co(1)–C(2)	147·4(9)	Co(2)–C(5)–C(11)	129·9(1·9)
C(1)–Co(1)–C(2)	94·5(1·7)	Co(1)–C(10)–C(13)	133·5(2·2)
C(3)–Co(2)–C(4)	95·1(1·5)	C(10)–C(5)–C(11)	141·9(1·6)
Co(1)–Co(2)–C(5)	51·0(8)	C(5)–C(10)–C(13)	147·0(1·6)
Co(1)–Co(2)–C(10)	50·6(9)	C(6)–C(5)–C(11)	119·5(2·4)
Co(2)–Co(1)–C(5)	50·8(9)	C(9)–C(10)–C(13)	121·1(2·1)
Co(2)–Co(1)–C(10)	51·8(8)	C(6)–C(7)–C(12)	118·8(2·4)
C(5)–Co(1)–C(10)	102·5(1·2)	C(8)–C(7)–C(12)	111·6(2·2)
C(5)–Co(2)–C(10)	101·4(1·2)	Co(1)–C(5)–C(6)	116·2(1·9)
Co(1)–C(5)–C(10)	38·7(8)	Co(2)–C(10)–C(9)	109·9(1·7)
Co(1)–C(10)–C(9)	72·7(1·6)	C(10)–C(13)–F(7)	110·3(2·8)
Co(2)–C(5)–C(6)	73·8(1·8)	C(10)–C(13)–F(8)	111·3(2·6)
C(5)–C(6)–C(7)	108·7(2·5)	C(10)–C(13)–F(9)	107·9(2·9)
C(8)–C(9)–C(10)	114·5(2·0)	F(1)–C(11)–F(2)	113·7(3·2)
C(6)–C(7)–C(8)	117·9(2·4)	F(1)–C(11)–F(3)	112·8(3·1)
C(7)–C(8)–C(9)	112·6(2·3)	F(2)–C(11)–F(3)	107·0(2·8)
C(5)–C(11)–F(1)	110·3(2·7)	F(4)–C(12)–F(5)	98·8(2·3)
C(5)–C(11)–F(2)	107·1(2·6)	F(4)–C(12)–F(6)	102·8(2·2)
C(5)–C(11)–F(3)	105·5(2·5)	F(5)–C(12)–F(6)	104·7(2·3)
C(7)–C(12)–F(4)	111·5(2·2)	F(7)–C(13)–F(8)	111·7(3·1)
C(7)–C(12)–F(5)	114·4(2·2)	F(7)–C(17)–F(9)	104·9(2·7)
C(7)–C(12)–F(6)	121·7(2·6)	F(8)–C(13)–F(9)	110·5(3·2)

TABLE 4

Dihedral angles (°) between planes each defined by three atoms

[C(5), C(6), C(7)] and [C(8), C(9), C(10)]	60·0(1·5)
[Co(1), C(1), C(2)] and [Co(2), C(3), C(4)]	154·0(8)

TABLE 5

Least-squares planes defined by atomic positions,* and in square brackets, distances (Å) of the atoms from these planes

	A	*B*	*C*	*D*
Plane (1):				
C(5)—(7)	−0·6788	−0·2203	−0·7005	7·4445
[Co(1) −0·35, Co(2) 1·55, C(8) −0·89, C(11) −0·06, C(12) 0·00]				
Plane (2):				
C(8)—(10)	0·9669	−0·1919	−0·1684	−1·2991
[Co(1) 1·50, Co(2) −0·50, C(7) −0·86, C(13) −0·12]				
Plane (3):				
Co(1), Co(2), C(5), C(10)	0·2610	−0·4655	−0·8457	5·0182
[Co(1) 0·04, Co(2) 0·04, C(5) −0·04, C(6) −1·16, C(7) −1·87, C(8) −1·99, C(9) −1·27, C(10) −0·04, C(11) 0·99, C(13) 0·80]				
Plane (4):				
Co(1), Co(2), C(1), C(3), O(1), O(3)	−0·3637	0·7838	−0·5035	4·3812
[Co(1) −0·11, Co(2) 0·13, C(1) −0·05, C(3) −0·01, O(1) 0·11, O(3) −0·07]				

* Equations are in the form $AX + BY + CZ + D = 0$ where X, Y, and Z are orthogonal co-ordinates in Å relative to the crystal axes *a*, *b*, *c*.

are regarded as tridentate ligands. The bond description is complicated and it would be informative therefore to undertake a MO study of the system. Calculations to generate localized orbitals [15] are under way, and these will give a valence-bond type picture of the electron distribution which could enable σ and π-type interactions to be distinguished more clearly.

Within the allyl groups, the C–C distances and the C–C–C angles are within the ranges normally found for π-allyl–metal complexes.[16,17] Deviations of the allyl substituents from the allyl planes (Table 5) presumably result from strain imposed by the requirements of Co(1)–Co(2) and C(7)–C(8) bonding. The dihedral angle between the three-carbon atom planes defined by the two allyl groups is *ca.* 60°. This differs significantly from the value (90°) obtained [6] for the related complex $Co_2(CO)_4(HC_2H)(Bu^tC_2H)_2$.

There is no particular reason why this angle should be 60 or 90°. The angle actually adopted in each case is presumably controlled both by the unusual bonding arrangement and by intermolecular packing forces. In the complex $Co_2(CO)_4(CF_3C_2H)_3$, the vector C(7) · · · C(8) makes an angle of 23° with the vector Co(1) · · · Co(2); this can be compared to the value of 28° observed [6] in $Co_2(CO)_4(HC_2H)(Bu^tC_2H)_2$.

The parameters for the four Co–CO moieties in the molecule are similar to those in other carbonylcobalt complexes. The atoms Co(1), Co(2), C(1), O(1), C(3), O(3) define a plane from which the maximum deviation is only 0·13 Å (see Table 5) so that two of the carbonyl groups are effectively eclipsed when the molecule is viewed along the metal–metal bond axis. The mean Co–Co–C(CO) angle for these two carbonyl groups is 103°. In contrast, the mean Co–Co–C(CO) angle for the other two carbonyl groups [C(2)–O(2). C(4)–O(4)] is 148°. The dihedral angle between the planes defined by Co(1), C(1), O(1), C(2), O(2) and Co(2), C(3), O(3), C(4), O(4) is 154°. The two CO–M–CO angles are 95° [C(3)–Co(2)–C(4)] and 96° [C(1)–Co(1)–C(2)].

Parameters associated with the trifluoromethyl groups are typical of such organometallic complexes.[18,19] The 1,3,6-arrangement of the trifluoromethyl substituents within the bridge confirms the earlier deduction based on n.m.r. studies.[2]

We have indicated (*vide supra*) that the complexes $Co_2(CO)_4(alkyne)_3$ are intermediates in the formation of substituted benzenes. It is therefore pertinent to discuss the non-bonded distance between the two carbon atoms at the ends of the six-carbon atom bridge. C(5) and C(10) are separated by 3·05 Å in the present structure while Mills and Robinson report [6] a distance of 3·2 Å between the corresponding atoms in the compound $Co_2(CO)_4(HC_2H)(Bu^tC_2H)_2$. To achieve ring closure, it is necessary to move these atoms so that they are separated by only 1·4—1·5 Å, and this can be accomplished only if there is considerable distortion of

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the Co(1), C(5), C(10), Co(2) plane. It has been suggested[6] that this could proceed by initial Co–Co bond rupture; however, our recent preparative investigations[20] indicate that benzene formation sometimes occurs under conditions where the Co–Co bond is known to be stable. Consequently, benzene formation may occur as a result of initial Co–C σ-bond rupture with concomitant loose co-ordination of the C(5)–C(6)–C(7)–C(8)–C(9)–C(10) fragment on the Co–Co site. The free ends of the C_6 chain could then ' knit ' together and the benzene ring so formed could be released subsequently.

This degradation pathway requires that the tendency for $Co_2(CO)_4(alkyne)_3$ complexes to decompose to benzenes will be lowest when the Co–C σ-bonds are most stable. Fluorocarbon–transition-metal σ-bonds are generally more stable than corresponding hydrocarbon–transition-metal bonds. Comparisons between our observed Co–C σ-bond lengths with those for other such systems[17] are not possible owing to differences in metal oxidation state and stereochemistry. However, we are encouraged to believe that the complex $Co_2(CO)_4(CF_3C_2H)_3$ incorporates stable Co–C σ-bonds by a comparison of our data [mean Co–C(CF) σ 1·96(3); mean Co–CO 1·76 (4) Å] with that for the stable compound *cis*-$(HCF_2CF_2)_2Fe(CO)_4$[21] [mean Fe–CF_2 (σ) 2·068(14); mean Fe–CO 1·845(14) Å].

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