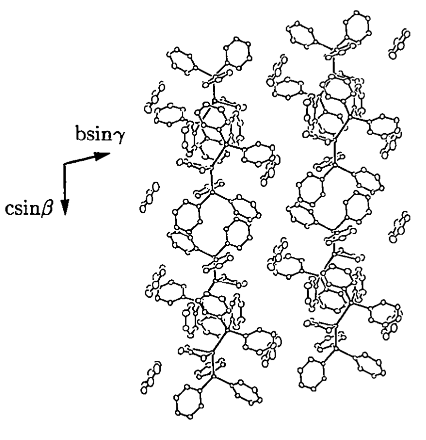

Fig. 2. Crystal lattice for [$Ag(PPh_3)_2NO_3$].$C_6H_6$ viewed down the [100] direction.

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# Structure of Bis($\mu$-diethyllead)-bis(tetracarbonyliron)

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**Abstract.** [$Fe_2(CO)_8\{\mu\text{-}Pb(C_2H_5)_2\}_2$], $M_r = 866{\cdot}4$, orthorhombic, *Pbca*, $a = 9{\cdot}685\,(2)$, $b = 13{\cdot}824\,(6)$, $c = 17{\cdot}940\,(7)$ Å, $V = 2402\,(1)$ Å$^3$, $Z = 4$, $D_x = 2{\cdot}40$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 153{\cdot}0$ cm$^{-1}$, $F(000) = 1584$, $T = 298$ K, $R = 0{\cdot}0526$ for 1038 unique observed reflections. The structure consists of a centrosymmetric planar dimer of $Fe(CO)_4$ and $Pb(C_2H_5)_2$ units, with two crystallographically independent Fe—Pb distances of 2·734 (4) and 2·718 (4) Å. The $Fe(CO)_4$ unit shows a distortion towards tetrahedral geometry, with C(1)—Fe—C(2) and C(3)—Fe—C(4) angles of 158·4 (11) and 98·7 (12) °, respectively.

**Experimental.** Prepared by photolysis of $Fe(CO)_5$ and $PbEt_4$ in hexane. Red prisms from hexane solution: crystal dimensions *ca* 0·6 × 0·4 × 0·2 mm; systematic absences: $0kl$, $k = 2n+1$; $h0l$, $l = 2n+1$; $hk0$, $h = 2n+1$; 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 $\theta$ reflections ($11 < \theta < 13°$); intensities measured to $\theta = 25{\cdot}0°$ over *hkl* range 0 to 11, 0 to 16, −1 to 21; $\bar{6}\bar{1}\bar{3}$ and $1\bar{7}4$ measured every 2 h with a 2% decay over 26 h data collection; 2589 data measured, 2109 independent data with 1038 having $I > 2{\cdot}5\sigma(I)$ considered observed and used in structure determination and refinement; $R_{\rm int}$ before absorption correction 0·057, after correction 0·033; corrected for Lorentz–

Table 1. *Final positional parameters (fractional coordinates) with e.s.d.'s in parentheses, and equivalent isotropic thermal parameters* (Å$^2$)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| Pb | −0·04195 (9) | 0·47058 (7) | 0·58964 (5) | 0·049 |
| Fe | 0·2022 (3) | 0·4441 (2) | 0·5159 (2) | 0·050 |
| O(1) | 0·0580 (19) | 0·2699 (12) | 0·4666 (11) | 0·092 |
| O(2) | 0·2457 (17) | 0·6438 (13) | 0·5655 (10) | 0·084 |
| O(3) | 0·337 (2) | 0·355 (1) | 0·646 (1) | 0·117 |
| O(4) | 0·4368 (18) | 0·4412 (15) | 0·4143 (11) | 0·107 |
| C(1) | 0·116 (2) | 0·341 (2) | 0·485 (1) | 0·060 |
| C(2) | 0·225 (2) | 0·565 (2) | 0·545 (1) | 0·059 |
| C(3) | 0·287 (3) | 0·390 (2) | 0·596 (1) | 0·062 |
| C(4) | 0·343 (2) | 0·442 (2) | 0·452 (1) | 0·061 |
| C(5) | −0·015 (3) | 0·551 (3) | 0·685 (2) | 0·137 |
| C(6) | −0·133 (5) | 0·575 (3) | 0·726 (2) | 0·146 |
| C(7) | −0·127 (3) | 0·332 (2) | 0·627 (2) | 0·095 |
| C(8)† | −0·045 (5) | 0·287 (3) | 0·677 (3) | 0·08 (1) |
| C(8A)‡ | −0·216 (9) | 0·330 (6) | 0·663 (5) | 0·11 (3) |

† Site occupancy 0·6.
‡ Site occupancy 0·4.

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

Primed atoms are related by the symmetry operation: $-x, 1-y, 1-z$.

| | | | |
|---|---|---|---|
| Fe—Pb | 2·734 (4) | Fe—Pb′ | 2·718 (4) |
| Pb···Pb′ | 3·416 (2) | Fe···Fe′ | 4·249 (4) |
| Pb—C(5) | 2·06 (4) | Pb—C(7) | 2·19 (4) |
| Fe—C(1) | 1·75 (3) | Fe—C(2) | 1·76 (3) |
| Fe—C(3) | 1·81 (3) | Fe—C(4) | 1·78 (3) |
| C(1)—O(1) | 1·18 (3) | C(2)—O(2) | 1·17 (4) |
| C(3)—O(3) | 1·12 (4) | C(4)—O(4) | 1·14 (3) |
| C(5)—C(6) | 1·39 (6) | C(7)—C(8) | 1·36 (6) |
| C(7)—C(8A) | 1·08 (10) | | |

| | | | |
|---|---|---|---|
| Fe—Pb—Fe′ | 102·4 (2) | C(5)—Pb—C(7) | 105·1 (14) |
| Pb—Fe—Pb′ | 77·6 (1) | C(1)—Fe—C(2) | 158·4 (11) |
| C(3)—Fe—C(4) | 98·7 (12) | C(1)—Fe—C(3) | 97·5 (12) |
| C(1)—Fe—C(4) | 98·4 (12) | C(2)—Fe—C(3) | 95·7 (12) |
| C(2)—Fe—C(4) | 96·4 (11) | Mean Fe—C—O | 177 (2) |

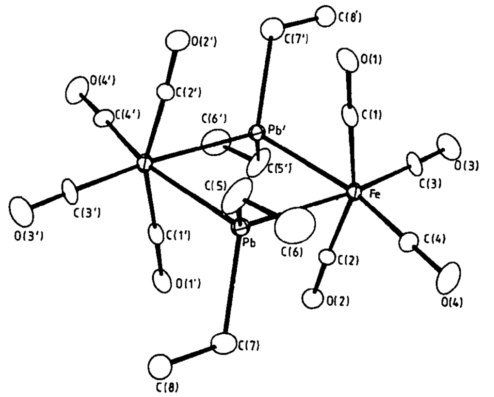

Fig. 1. Molecular structure and atomic labelling scheme. Only the major component of the disordered methyl C(8) atom is shown.

polarization and absorption (*DIFABS*: Stuart & Walker, 1983); solved by Patterson function and subsequent full-matrix least squares; anisotropic thermal parameters for all non-H atoms except the disordered methyl C(8) atom of one ethyl group; two positions determined for disordered C(8) atom with occupancies 0·6 and 0·4; H atoms included at calculated positions, C—H = 1·0 Å, for ordered ethyl group only; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max. $\Delta/\sigma$ 0·037, av. 0·004; $(\Delta\rho)_{\max}$ 0·94, $(\Delta\rho)_{\min}$ − 1·20 e Å$^{-3}$; $R = 0{\cdot}0526$, $wR = 0{\cdot}047$, $S = 1{\cdot}59$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); calculations carried out on a Gould-SEL 32/27 minicomputer using the *GX* suite of programs (Mallinson & Muir, 1985). Final positional parameters are given in Table 1, with selected bond distances and angles in Table 2.* The atomic labelling scheme and molecular structure are shown in Fig. 1.

**Related literature.** Related complexes have been reviewed (Bonny, 1978; Whitmire, 1988). The title complex is isostructural with the germanium analogue [(CO)$_4$Fe($\mu$-GeEt$_2$)$_2$Fe(CO)$_4$] (Zimmer & Huber, 1968), and similar to the tin compound [(CO)$_4$Fe($\mu$-SnMe$_2$)$_2$Fe(CO)$_4$] (Gilmore & Woodward, 1972). The distortion of the Fe(CO)$_4$ unit towards tetrahedral geometry has been discussed (Elian & Hoffmann, 1975). Previous structures with Fe—Pb distances are [Pb{Fe(CO)$_4$}$_2${Fe$_2$(CO)$_8$}]$^{2-}$, Fe—Pb = 2·651 (5)–2·832 (4) Å (Lagrone, Whitmire, Churchill & Fettinger, 1986) and Pb[Fe$_2$(CO)$_8$]$_2$, Fe—Pb = 2·606 (3)–2·635 (3) Å (Whitmire, Lagrone, Churchill, Fettinger & Robinson, 1987).

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* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positional parameters, and complete bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52066 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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