Crystal Structure of *p*-Chlorobenzoylpentacarbonylrhenium

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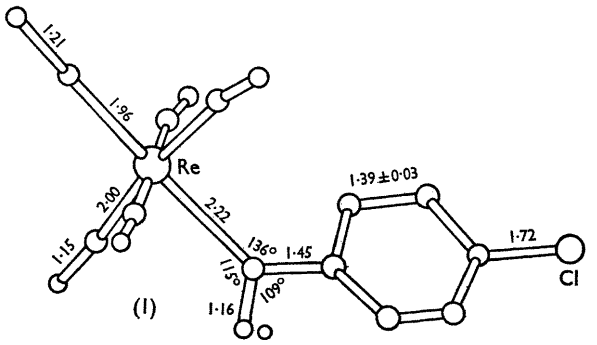
Summary The σ-bond Re–C(benzoyl) length 2·22 Å coincides with the covalent radii sum 2·25 Å.

RECENTLY we reported[1,2] on the synthesis and properties of halogen-containing benzoyl derivatives of manganese and rhenium carbonyls, and now we present the results of an *X*-ray study of *p*-chlorobenzoylpentacarbonyl rhenium, *p*-Cl·C_6H_4·CORe$(CO)_5$ (I). The crystals are yellow needles, triclinic, with $a = 10{\cdot}15 \pm 0{\cdot}02$, $b = 11{\cdot}16 \pm 0{\cdot}02$, $c = 6{\cdot}10 \pm 0{\cdot}03$ Å, $\alpha = 97 \pm 1$, $\beta = 95 \pm 1$, $\gamma = 81 \pm 1°$, $U = 677$ Å^3, $D_m = 2{\cdot}18$, $D_c = 2{\cdot}28$, $Z = 2$, $M = 465{\cdot}84$. Intensities of *ca.* 800 non-zero independent reflexions were estimated visually without regard to the absorption correction (an equi-inclination Weissenberg goniometer, unfiltered copper radiation). The structure was solved by the usual heavy-atom technique and refined by the full-matrix least-squares method with isotropic temperature factors, to R 0·13 with the overall temperature-factor $B = 5{\cdot}2$ Å^2. Standard deviations in bond lengths are 0·01 for Re–C, 0·02 for C–O, 0·03 Å for C–C, and in bond angles 2—3° depending on the atomic numbers.



(I)

FIGURE

The Re atom co-ordination is that of a slightly distorted octahedron (see Figure). The mean Re–CO (equatorial) bond-length is 2·00 Å with C–O 1·15 Å, R–CO (axial) is 1·96 Å with C–O 1·15 Å. These differences in bond lengths are analogous with, although less than, those found in other pentacarbonyl metal derivatives, $XM(CO)_5^{3-}$,[5] which suggest greater back-donation $d_\pi(M) \rightarrow \pi^*(CO)$ to the apical CO-group. The mean value of the C(equatorial)–Re–C(axial) angles is 90·7 ± 2·4°; and the Re–C–O angles are somewhat distorted (mean value 172°). The Re–C σ-bond length is 2·22 Å.

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