Location of Hydrogen Atoms in Trimethyl(nonane-4,6-dionato)platinum(IV) by Neutron Diffraction

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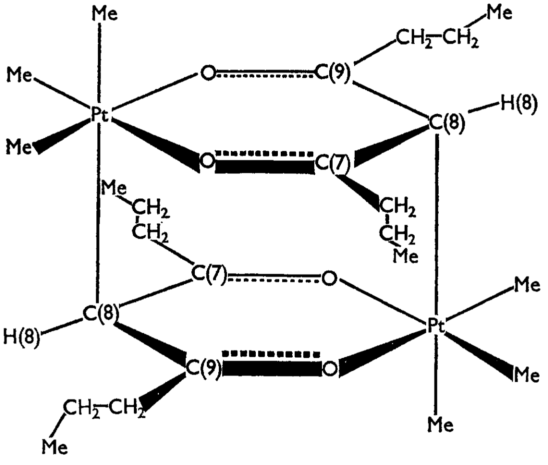
TRIMETHYL (NONANE-4, 6-DIONATO) PLATINUM (IV), (I), was the first complex shown[1] by *X*-ray crystal structure analysis to contain a β-diketone coordinated to a metal atom *via* the γ-carbon atom. The hydrogen atom on this carbon atom was not located. Attempts to prepare γ-carbon-bonded β-diketo-complexes with a substituent other than hydrogen have been unsuccessful.[2,3] It seemed possible that the presence of the hydrogen atom was vital to the formation of complexes of this type and its unequivocal location is the subject of the present work.

The compound is monoclinic with two centrosymmetrical dimeric molecules in the unit cell. Platinum, oxygen, and carbon atom co-ordinates and isotropic vibration parameters of the asymmetric unit $Me_3(Pr^nCO{\cdot}CH{\cdot}COPr^n)$ were known.[1]

Neutron-diffraction facilities at A.E.R.E. Harwell were used. Intensity measurements were made at room temperature, on a four-circle diffractometer, with $\lambda = 1{\cdot}0415$ Å. Limitations of crystal size, 3 mm.^{3}, and neutron flux, 1500 counts per second have restricted us to 282 unique reflections in the time available so far.

For the heavy atoms only, R was 0·37. Addition of the 8 hydrogen atoms on the CH$_2$ groups of the side-chains in calculated positions reduced R to 0·29. Successive Fourier syntheses phased with known atoms revealed the other 16 hydrogen atoms, inclusion of which reduced R to 0·14 after isotropic refinement of the hydrogen parameters only. The hydrogen atoms on the methyl groups bound to the platinum atom and at the ends of the side-chains complete the expected tetrahedra. The hydrogen atom designated H(8) in (I) completes a distorted tetrahedron about C(8). From the



parameters determined by neutron diffraction for H(8) and by *X*-ray diffraction for the other atoms we found:—

C(8)–H(8) = 1·10(6) Å
Pt′–C(8)–C(7) = 102(1)° C(7)–C(8)–H(8) = 110(4)°
Pt′–C(8)–H(8) = 98(4)° C(7)–C(8)–C(9) = 122(2)°
Pt′–C(8)–C(9) = 103(1)° C(9)–C(8)–H(8) = 118(4)°

Although the standard deviations are still too large to justify detailed discussion of the nature of the bonding, it is clear that the hydrogen atom occupies a site which is sterically suitable also for larger atoms.

If the difficulties in synthesis can be overcome γ-carbon-bonded β-diketo-complexes with other substituents than hydrogen should be stable.

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