

КРАТКИЕ СООБЩЕНИЯ

UDC 548.73:547.13:546.93

CRYSTAL STRUCTURE
OF (η^5 -PENTAMETHYLCYCLOPENTADIENYL){BIS(PENTAFLUOROPHENYL)THIOMETHYLPHENYL
PHOSPHINE- κ_2S,P }CHLOROIRIDIUM(III) TETRAFLUOROBORATER.M. Bellabarba^{1,2}, M. Nieuwenhuyzen¹, G.C. Saunders³¹The School of Chemistry, Queen's University Belfast, David Keir Building, Belfast, BT9 5BA, United Kingdom²Current address: Sasol Technology (UK) Ltd, Purdie Building, North Haugh, St Andrews, KY16 9ST, United Kingdom³Department of Chemistry, The University of Waikato, Private Bag 3105, Hamilton 3240, New Zealand

E-mail: g.saunders@waikato.ac.nz

Received November, 9, 2011

Revised — January, 31, 2012

The salt (η^5 -pentamethylcyclopentadienyl){bis(pentafluorophenyl)thiomethylphenylphosphine- κ_2S,P }chloroiridium(III) tetrafluoroborate, $[(\eta^5-C_5Me_5)IrCl\{\kappa_2S,P-(C_6F_5)_2PC_6H_4SMe-2\}]BF_4$, crystallizes as a conglomerate in the orthorhombic crystal system in space group $P2_12_12_1$ with unit cell parameters $a = 9.9621(9)$ Å, $b = 16.7793(15)$ Å, $c = 18.5040(16)$ Å, $V = 3093.1(5)$ Å³, $Z = 4$, $d_{calc} = 2.014$ g·cm⁻³. The structure of the S_{Ir} , S_S stereoisomer reveals three-legged piano stool geometry about Ir, with Cp*—Ir, Ir—P, Ir—S and Ir—Cl distances of 1.847(5), 2.2791(14), 2.3451(13) and 2.3840(12) Å respectively.

Keywords: conglomerate, η^5 -pentamethylcyclopentadienyl, iridium, X-ray structure.

Intramolecular dehydrofluorinative coupling of pentamethylcyclopentadienyl and phosphines bearing polyfluoroaryl substituents occurs readily in cationic complexes of rhodium [1, 2] and iridium [2, 3], especially in cases where the phosphine moiety is a component of a chelating ligand. A number of the products are chiral, with some crystallizing as conglomerates, for example the salts comprising cations of tethered ligands $[\{\eta^5,\kappa P,\kappa P-C_5Me_4CH_2-2-C_5F_3N-4-P(C_6F_5)CH_2CH_2PPh_2\} \cdot RhCl][BF_4]$ [4] and $[\{\eta^5,\kappa P-C_5Me_4CH_2-2-C_6F_4P(C_6F_5)CH_2P(C_6F_5)_2\}RhCl_2]$ [5]. Here we report the structure of an iridium complex, $[(\eta^5-C_5Me_5)IrCl\{\kappa P,\kappa S-(C_6F_5)_2PC_6H_4SMe-2\}]BF_4$ (**1**), which undergoes intramolecular dehydrofluorinative coupling [3], and which also crystallizes as a conglomerate.

Experimental. Crystals of compound **1** [3] were grown from dichloromethane. Diffraction data of a single crystal were collected at 153(2) K on a Bruker SMART diffractometer using the SAINT-NT [6] software with graphite-monochromated MoK_α radiation. Crystallographic and refinement data are given in Table 1. Lorentz and polarization corrections were applied. Empirical absorption corrections were applied using SADABS [8]. The structure was solved using direct methods and refined with the programme package SHELXTL [9]. The non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atom positions were added, and idealized positions and a riding model with fixed thermal parameters (U_{ij} $1.2U_{eq}$ for the atom to which they are bonded (1.5 for CH₃)) were used for subsequent refinements. The function minimized was $\sum[w(|F_0|^2 - |F_c|^2)]$. CCDC 221314 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.