

## КРАТКИЕ СООБЩЕНИЯ

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CRYSTAL STRUCTURE  
OF ( $\eta^5$ -PENTAMETHYLCYCLOPENTADIENYL){BIS(PENTAFLUOROPHENYL)THIOMETHYLPHENYL  
PHOSPHINE- $\kappa_2S,P$ }CHLOROIRIDIUM(III) TETRAFLUOROBORATER.M. Bellabarba<sup>1,2</sup>, M. Nieuwenhuyzen<sup>1</sup>, G.C. Saunders<sup>3</sup><sup>1</sup>The School of Chemistry, Queen's University Belfast, David Keir Building, Belfast, BT9 5BA, United Kingdom<sup>2</sup>Current address: Sasol Technology (UK) Ltd, Purdie Building, North Haugh, St Andrews, KY16 9ST, United Kingdom<sup>3</sup>Department of Chemistry, The University of Waikato, Private Bag 3105, Hamilton 3240, New Zealand

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The salt ( $\eta^5$ -pentamethylcyclopentadienyl){bis(pentafluorophenyl)thiomethylphenylphosphine- $\kappa_2S,P$ }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,  $V = 3093.1(5)$  Å<sup>3</sup>,  $Z = 4$ ,  $d_{calc} = 2.014$  g·cm<sup>-3</sup>. 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.

**Key words:** conglomerate,  $\eta^5$ -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  $[\{\eta^5, \kappa P, \kappa P-C_5Me_4CH_2-2-C_6F_5N-4-P(C_6F_5)CH_2CH_2PPh_2\} \cdot RhCl][BF_4]$  [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  $MoK_\alpha$  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 ( $U_{ij}$  1.2  $U_{eq}$  for the atom to which they are bonded (1.5 for  $CH_3$ )) were used for subsequent refinements. The function minimized was  $\sum[w(|F_o|^2 - |F_c|^2)]$ . 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](http://www.ccdc.cam.ac.uk/data_request/cif), or by emailing [data\\_request@ccdc.cam.ac.uk](mailto: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.