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Crystal structure of 1-ferrocenyl-6-bromopyrene, C_{26}H_{17}BrFe

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Abstract
C_{26}H_{17}BrFe, monoclinic, C2/c (no. 15), a = 30.486(10) Å, b = 7497(3) Å, c = 18.669(6) Å, β = 120.184(5)°, V = 3689(2) Å³, Z = 8, R(gt)(F) = 0.0407, wR(ref)(F²) = 0.1015, T = 296(2) K.

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The asymmetric unit of the crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material
The title compound was obtained by Suzuki coupling reaction between 1,6-dibromopyrene and ferroceneboronic acid. Ferroceneboronic acid was prepared according to the literature [3]. Under the atmosphere of nitrogen, a mixture of 1,6-dibromopyrene (0.72 g, 2 mmol), ferroceneboronic acid (0.51 g, 2.2 mmol), Pd(PPh₃)₄ (0.05 g, 0.05 mmol) and 2 M potassium carbonate (2 mL) in toluene (40 mL) was heated to reflux for 24 h. After cooling, water and dichloromethane were added and the organic layer was separated. The organic layer was dried over MgSO₄ and the solvent was removed under vacuum. Finally, the crude products were purified by silica gel column chromatography using hexane/dichloromethane as eluent to afford 1-ferrocenyl-6-bromopyrene as a red solid (Yield: 65%). Needle-shaped crystals of the title compound were obtained by the slow evaporation of a mixture of dichloromethane/methanol at room temperature. Elemental analysis calculated: C, 66.84%; H, 4.10%; found: C, 66.56%, H, 4.19%.

Experimental details
All H atoms bond to C atoms were introduced using the HFIX command in the SHELXL-97 program [1], with the value of 0.93 Å for C—H bonds distances. Their Uiso(C) were set to 1.2 Ueq(C). The structure was checked using PLATON [2].

Discussion
Pyrene is a well-known chromophore and has been a typical candidate for organic fluorescent materials [4]. Since pyrene exhibits pure blue fluorescence with high quantum yield, long fluorescence lifetime, excellent thermal stability and high charge carrier mobility, it has been attracted much attention as an important organic semiconductor for the application in organic light emitting diodes (OLEDs) [5, 6], organic solar cells (OSCs) [7], organic field-effect transistors (OFETs) [8], etc. To this purpose, efforts have been made in order to enhance the electronic and optical properties of pyrene derivatives by modifying its molecular structure. The photoelectric properties can be fine-tuned by introducing specific
electron-donating or -accepting groups into the pyrene ring, resulting in improvement in the hole- or electron-transporting abilities of the materials [9, 10]. The electronic properties aren’t reported. Herein, we report the synthesis and crystal structure of the title compound which involves a ferrocenyl group at the pyrene core.

The X-ray diffraction analysis shows that the ferrocenyl and Br atom at the 1- and 6-positions of pyrene core. The central iron atom in the ferrocenyl segment is coordinated by ten carbons atoms and the bond distances of Fe–C are in the narrow range of 2.005(5)–2.050(4) Å. All the sixteen carbon atoms of the pyrene ring are located on a nearly strict plane with the largest derivation of 0.034(4) Å for C6 atom from the mean plane. The C8–C17 bond distance linking the pyrene core and ferrocenyl group is 1.473(4) Å. The C1–Br1 bond distance is 1.898(4) Å. The dihedral angle between the pyrene ring and cyclopentadiene is 40.42(2)°. There exist weak intermolecular π–π interactions between two adjacent pyrene rings and C–H–π interactions between the pyrene ring and cyclopentadiene ring, which connect the complexes of the title compound to a two-dimensional supramolecular structure.

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References