Crystal structure of 1-(9-fluorenyl)-2-(1-indenyl)ethane, $C_{24}H_{20}$

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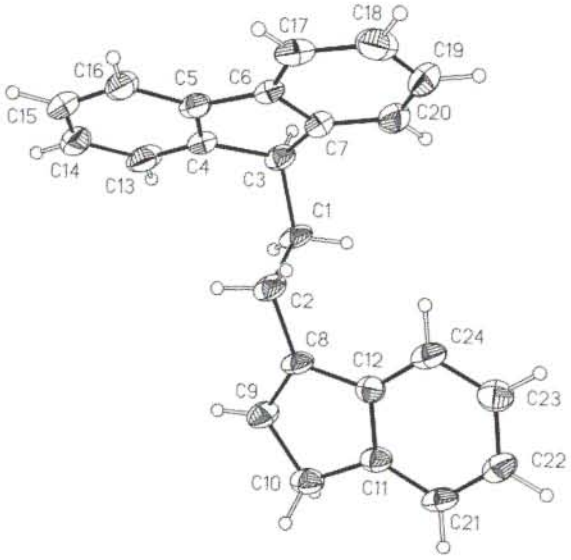

Abstract

$C_{24}H_{20}$, monoclinic, $P12_1/n1$ (No. 14), a = 9.623(5) Å, b = 8.477(4) Å, c = 21.064(4) Å, β = 93.17(3)°, V = 1715.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.062, $wR_{ref}(F^2)$ = 0.129, T = 193 K.

Source of material

1-(9-Fluorenyl)-2-(1-indenyl)ethane was prepared via nucleophilic ring opening of oxirane according to literature procedures [1] by reacting 2-(9-fluorenyl)ethyltrifluoromethanesulfonate with indenyllithium in dioxane. Crystals suitable for X-ray studies were obtained by crystallization from a mixture of toluene/hexane (2:3).

Discussion

Racemic group 4 *ansa*-metallocene dichlorides have found widespread application as homogeneous catalyst systems in sterospecific α-olefin polymerization when activated with methylaluminoxane (MAO) [2]. In this context the C_2-symmetric *rac*-[1,2-bis(3-indenyl)ethane]zirconium dichloride [3], can be regarded as one of the key structures in metallocene catalyzed olefin polymerization. Although ethylene bridged ligand precursors like 1,2-bis(3-indenyl)ethane [4] or 1,2-bis(9-fluorenyl)ethane [5] are plainly available from 1,2-dibromo ethane as starting material, the unsymmetric title compound is difficult to synthesize that way. To introduce two different cyclopentadienyl fragments in the ethylene bridge a ring opening reaction of oxirane with fluorenyllithium was performed, followed by a functionalization of the resulting alcohol, into the trifluoromethanesulfonate derivative, which was further treated with indenyllithium to obtain the title compound [1,6].

Bond lengths and angles are very similar to them found in 1-(9-fluorenyl)-1(*R,S*)-phenyl-2-(1-indenyl)ethane [6]. Aromatic C—C distances vary between 1.365(6) Å and 1.416(6) Å and the single bond lengths are between 1.463(5) Å and 1.557(5) Å, respectively. The double bond C8—C9 distance is 1.331(5) Å.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.25 × 0.30 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.67 cm^{-1}
Diffractometer, scan mode:	AFC7S, ω/2θ
$2\theta_{max}$:	50.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2394, 2394
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 992
$N(param)_{refined}$:	217
Programs:	TEXSAN [7], SHELXTL [8], SHELX-97 [9]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.2366	0.9574	−0.0084	0.061
H(1B)	4*e*	0.0750	0.9443	−0.0298	0.061
H(2A)	4*e*	0.1163	0.6681	−0.0554	0.064
H(2B)	4*e*	0.2688	0.6877	−0.0220	0.064
H(3)	4*e*	0.2109	1.0559	−0.1127	0.058
H(9)	4*e*	0.2845	0.6703	0.1021	0.069
H(10A)	4*e*	0.1066	0.5272	0.1681	0.068
H(10B)	4*e*	0.1025	0.7131	0.1846	0.068
H(13)	4*e*	0.4898	0.9488	−0.0652	0.081
H(14)	4*e*	0.6688	0.7949	−0.1073	0.092
H(15)	4*e*	0.6221	0.6327	−0.1935	0.083
H(16)	4*e*	0.3979	0.6080	−0.2405	0.068
H(17)	4*e*	0.1253	0.6309	−0.2760	0.073
H(18)	4*e*	−0.1124	0.6986	−0.2910	0.092
H(19)	4*e*	−0.2127	0.8688	−0.2199	0.094
H(20)	4*e*	−0.0826	0.9830	−0.1364	0.079
H(21)	4*e*	−0.1908	0.6015	0.1833	0.066
H(22)	4*e*	−0.3849	0.6214	0.1128	0.068
H(23)	4*e*	−0.3615	0.6702	0.0053	0.072
H(24)	4*e*	−0.1387	0.7121	−0.0327	0.065

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.1685(5)	0.9023(5)	–0.0375(2)	0.076(4)	0.039(3)	0.039(3)	0.005(3)	0.030(2)	–0.004(2)
C(2)	4*e*	0.1717(5)	0.7261(5)	–0.0220(2)	0.075(4)	0.046(4)	0.041(3)	0.009(3)	0.029(3)	–0.001(3)
C(3)	4*e*	0.2027(4)	0.9391(6)	–0.1074(2)	0.066(3)	0.040(3)	0.041(3)	0.005(3)	0.021(3)	–0.002(3)
C(4)	4*e*	0.3377(5)	0.8604(6)	–0.1265(2)	0.052(3)	0.054(4)	0.037(3)	0.000(3)	0.013(2)	0.009(3)
C(5)	4*e*	0.3085(5)	0.7636(6)	–0.1788(2)	0.055(3)	0.050(4)	0.036(3)	0.000(3)	0.012(2)	0.005(3)
C(6)	4*e*	0.1608(5)	0.7730(6)	–0.1989(2)	0.050(3)	0.054(4)	0.036(3)	–0.002(3)	0.013(3)	0.009(3)
C(7)	4*e*	0.0980(5)	0.8766(6)	–0.1562(2)	0.048(3)	0.058(4)	0.042(3)	0.003(3)	0.013(2)	0.008(3)
C(8)	4*e*	0.1141(5)	0.6920(5)	0.0419(2)	0.069(4)	0.043(3)	0.031(3)	0.002(3)	0.019(2)	0.001(3)
C(9)	4*e*	0.1859(5)	0.6673(6)	0.0969(2)	0.066(3)	0.072(4)	0.036(3)	0.003(3)	0.019(2)	–0.001(3)
C(10)	4*e*	0.0905(4)	0.6340(6)	0.1501(2)	0.074(4)	0.062(4)	0.035(3)	0.002(3)	0.008(3)	0.006(3)
C(11)	4*e*	–0.0504(5)	0.6466(6)	0.1164(2)	0.064(3)	0.047(4)	0.034(3)	0.002(3)	0.009(3)	0.001(3)
C(12)	4*e*	–0.0356(5)	0.6784(5)	0.0528(2)	0.056(3)	0.039(3)	0.041(3)	0.008(3)	0.010(2)	–0.002(3)
C(13)	4*e*	0.4705(5)	0.8785(7)	–0.0997(2)	0.065(4)	0.101(5)	0.037(3)	–0.013(4)	0.013(3)	–0.002(3)
C(14)	4*e*	0.5765(5)	0.7884(7)	–0.1255(2)	0.043(3)	0.130(6)	0.057(4)	0.003(4)	0.011(3)	0.026(4)
C(15)	4*e*	0.5481(5)	0.6911(7)	–0.1768(2)	0.064(4)	0.093(5)	0.052(3)	0.015(4)	0.028(3)	0.019(4)
C(16)	4*e*	0.4159(5)	0.6754(6)	–0.2050(2)	0.065(3)	0.058(4)	0.048(3)	0.004(3)	0.019(3)	0.004(3)
C(17)	4*e*	0.0837(5)	0.7033(6)	–0.2483(2)	0.078(4)	0.069(4)	0.036(3)	–0.013(4)	0.011(3)	0.006(3)
C(18)	4*e*	–0.0572(6)	0.7422(7)	–0.2566(2)	0.068(4)	0.104(6)	0.057(4)	–0.030(4)	–0.014(3)	0.027(4)
C(19)	4*e*	–0.1164(5)	0.8448(8)	–0.2142(3)	0.052(4)	0.104(6)	0.082(4)	0.003(4)	0.026(4)	0.033(4)
C(20)	4*e*	–0.0401(5)	0.9124(7)	–0.1645(3)	0.062(4)	0.069(5)	0.068(4)	0.009(4)	0.022(3)	0.025(3)
C(21)	4*e*	–0.1805(5)	0.6240(6)	0.1396(2)	0.080(4)	0.049(4)	0.039(3)	–0.001(3)	0.022(3)	0.004(3)
C(22)	4*e*	–0.2946(5)	0.6350(6)	0.0976(2)	0.062(4)	0.045(4)	0.064(4)	–0.007(3)	0.026(3)	0.002(3)
C(23)	4*e*	–0.2812(5)	0.6655(6)	0.0336(2)	0.070(4)	0.054(4)	0.054(3)	0.000(3)	0.001(3)	–0.001(3)
C(24)	4*e*	–0.1494(5)	0.6891(6)	0.0109(2)	0.072(4)	0.052(4)	0.042(3)	–0.001(3)	0.021(3)	0.004(3)

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