ISSN 0036-0236, Russian Journal of Inorganic Chemistry, 2012, Vol. 57, No. 1, pp. 46–51. © Pleiades Publishing, Ltd., 2012.
Original Russian Text © Z.M. Dzhabieva, S.V. Topilin, G.V. Shilov, T.S. Dzhabiev, 2012, published in Zhurnal Neorganicheskoi Khimii, 2012, Vol. 57, No. 1, pp. 51–56.

COORDINATION COMPOUNDS

Crystal Structure of *ansa*-Me_2Si-(2Me-4-*p*-Tol-cyclopenta[*b*]indol-3-yl)$_2ZrCl_2$ and Its Catalytic Properties in the Polymerization of Propylene

Z. M. Dzhabieva, S. V. Topilin, G. V. Shilov, and T. S. Dzhabiev

Institute of Problems of Chemical Physics, Russian Academy of Sciences, pr. Akademika Semenova 1,
Chernogolovka, Moscow oblast, 142432 Russia

Received September 10, 2010

Abstract—A new zirconocene *ansa*-Me_2Si-(2Me-4-*p*-Tol-cyclopenta[*b*]indol-3-yl)$_2ZrCl_2$ complex (**I**), in which the Cp ligand is fused with the indole ring, has been synthesized and studied by X-ray diffraction analysis. Light brown crystals are triclinic, space group $P\bar{1}$; $M = 734.92$, $a = 9.252(2)$ Å, $b = 12.914(3)$ Å, $c = 15.619(3)$ Å, $\alpha = 111.83(3)°$, $\beta = 81.03(3)°$, $\gamma = 117.77(3)°$, $V = 1569(3)$ Å^3, $Z = 2$, $\rho_{calc} = 1.525$ g/cm^3. The structural parameters of complex **I** are compared with the known bis-indenyl zirconium complexes: *rac*-$Me_2Si(Ind)_2ZrCl_2$ (**II**) and *rac*-Me_2Si(2Me-2Ph-1-Ind)$_2ZrCl_2$ (**III**) and analogous substituted *rac*-Me_2Si(2,5-Me_2-3Ph-6-Cp[*b*]Tp)$_2ZrCl_2$ (**IV**) and *rac*-Me_2Si(2,5-Me_2-1Ph-4-Cp[*b*]Py)$_2ZrCl_2$ (**V**). Complex **I** alkylated by the Grignard reagent ($MgMe_2$) in the presence of the Al-*iso*-Bu_3 cocatalyst is an efficient catalyst for the polymerization of propylene into isotactic polypropylene.

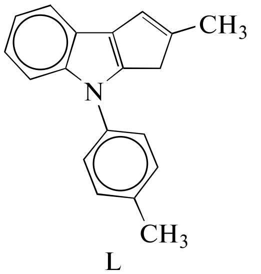
DOI: 10.1134/S0036023612010068

Numerous zirconium *ansa*-complexes of C_2 symmetry with different hapto-bound ligands and substituents in them, including heterocycles, which are excellent catalysts of the polymerization of propylene, have been synthesized by now [1–10]. Different structural modifications of the indenyl $Me_2Si(Ind)_2ZrCl_2$ complex, as well as the use of more available and stable cocatalysts differing from polymethylalumoxane (MAO) [10], make it possible to synthesize higher performance catalysts that afford polypropylene (PP) of higher molecular weight and with stereospecificity attaining that of industrial samples.

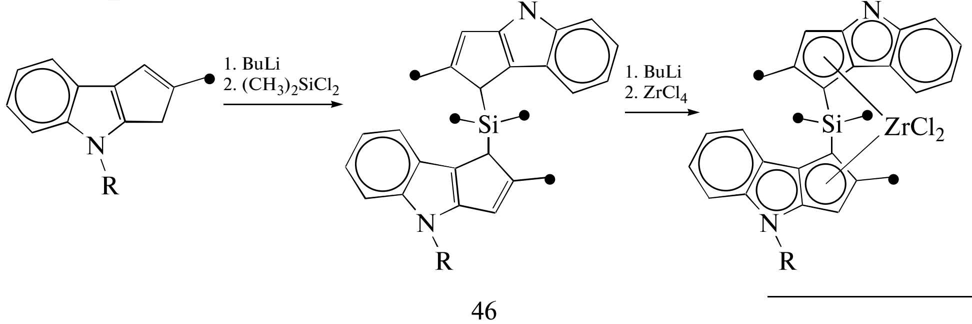
Therefore, the synthesis of new compounds of this type and study of their structural features and properties are of undoubtful interest.

EXPERIMENTAL

All the operations in the synthesis of compound **I** were performed in a dry argon atmosphere. The solvents used in the work were purified by known methods [11]. Compound **I** was synthesized with the use of Schlenk equipment in compliance with the following scheme:



L



The synthesis of the 2-methyl-4-*para*-tolyl-1-4dihydrocyclopenta[b]indole ligand (L) was described in [12].

Synthesis of compound I. A precisely weighed portion of L (4.0 g, 15.6 mmol) was dissolved in toluene (50.0 mL) with a small tetrahydrofuran (THF) additive, and to this solution cooled down to –80°C, BuLi in hexane (9.8 mL, 15.6 mmol, 1.6 M) was slowly added dropwise. To the solution of the resulting lithium salt, $(CH_3)_2SiCl_2$ (1.0 mL, 7.8 mmol) was added, and the reaction mixture was stirred at room temperature for 24 h. Then, the solution was decanted from a LiCl precipitate and concentrated by removing the solvent under vacuum. The product in the form of oil was dissolved in toluene (50 mL), the solution was cooled down to –80°C, and another portion of BuLi in hexane (11.1 mL, 1.6 M) was slowly added. The mixture was slowly heated to room temperature, stirred at this temperature for 2 h, and cooled again. $ZrCl_4$ (1.78 g, 7.8 mmol) was added to the cooled solution, and the mixture was additionally stirred for 8 h at room temperature. After removal of the solvent and recrystallization from diethyl ether, a mixture of the *rac*- and *meso*-forms of compound **I** was obtained as orange-brown crystals (3.7 g, 79%). The fractionation of crystals in an ether/hexane solution gave the pure *rac* form (light brown crystals, 1.21 g, 26%) and the pure *meso* form (orange crystals, 0.71 g, 16%).

For *rac*-$C_{40}H_{36}Cl_2N_2SiZr$ anal. calcd. (%): C, 65.37; H, 4.93; N, 3.81; Cl, 9.64.

Found (%): C, 65.25; H, 4.86; N, 3.94; Cl, 9.51.

X-ray diffraction analysis. The experimental data for a light brown crystal of compound **I** were obtained on an Enraf-Nonius CAD4 automatic four-circle diffractometer (graphite monochromator, MoK_α radiation, θ/2θ scanning). The structure was solved by direct methods with the use of the AREN-88 crystallographic software [13]. Hydrogen atoms were found from a difference Fourier synthesis. The structure was refined by the full-matrix least-squares technique in the anisotropic approximation for non-hydrogen atoms with the use of the SHELXL-97 software [14]. Absorption was not taken into account.

Selected crystal data and characteristics of X-ray diffraction experiment for compound **I** are given in Table 1, and interatomic distances and bond angles are listed in Table 2.

Polymerization of propylene was performed in the presence of complex **I** in a steel high-pressure reactor under vigorous stirring of a reaction mixture.

Table 1. Crystallographic data and characteristics of X-ray diffraction experiment for compound **I**

FW	734.92
Crystal size, mm	0.2 × 0.3 × 0.4
Symmetry system	Triclinic
Space group	$P\bar{1}$
Unit cell parameters:	
a, Å	9.252(2)
b, Å	12.914(3)
c, Å	15.619(3)
α, deg	111.83(3)
β, deg	81.03(3)
γ, deg	117.77(3)
V, Å^3	1569(3)
Z; ρ_{calc}, g/cm^3	2; 1.525
μ, cm^{-1}	60.9
F(000)	724
θ range, deg	1.41–29.60
Index range	$-11 \le h \le 11$, $-16 \le k \le 15$, $0 \le l \le 12$
Total number of measured reflections	5252
Number of reflections with $I \ge 2\sigma(I)$	5242 (R_{int}) = 0.0128
Number of refined parameters	526
GOOF on F^2	0.879
$R[I \ge 2\sigma(I)]$	$R_1 = 0.0364$, $wR_2 = 0.0961$
R (all data)	$R_1 = 0.0438$, $wR_2 = 0.0991$
Residual electron density (max/min), e/Å^3	0.487/–0.487

RESULTS AND DISCUSION

The molecular structure of complex **I** in shown in the figure. In the complex, the zirconium atom is placed between two η^5(pentahapto) cyclopentadienyl moieties. The molecule has characteristics typical of *ansa*-metallocene complexes with short bridges. There is no contact between the Zr and N atoms, and this is also a common characteristic of heteroaromatic zirconocenes. The molecule lies on the twofold crystallographic axis passing through the silicon and zirconium atoms. The nitrogen atoms in heterocycles are arranged on the internal ring sides facing the zirconium atom. The N(1)–C(11) bond length in the pyrrole ring (1.384(6) Å) indicates the contribution of a double bond and the presence of a positive charge on the nitrogen atom. This, in its turn, leads to the delocalization of a negative charge over the entire molecule and, in particular, in the Cp ring. The C–C bond lengths in Cp rings vary within 1.411–1.499 Å, whereas in analogous bis-indenyl complexes **II** and **III**, they are within 1.410–1.442 Å [1, 3, 4–9]. The torsion angles between the planes of the Cp and *para*-tolyl rings of 56.1° and 54.1° provide minimum steric and electronic interactions between them. Table 3, in which selected structural parameters of complex **I** are given in comparison with those of complexes **II–V**,

Table 2. Selected bond lengths (*d*) and bond angles (ω) in complex **I**

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
Zr–C(5)	2.184(3)	C(1)–C(2)	1.424(5)	C(12)–C(13)	1.287(4)	C(25)–C(30)	1.349(4)
Zr–C(2)	2.198(3)	C(1)–C(5)	1.499(5)	C(13)–C(14)	1.416(6)	C(26)–C(27)	1.384(6)
Zr–C(3)	2.201(3)	C(2)–C(3)	1.458(4)	C(14)–C(15)	1.328(6)	C(27)–C(28)	1.337(5)
Zr–C(1)	2.218(3)	C(2)–C(18)	1.534(4)	C(15)–C(19)	1.524(5)	C(28)–C(29)	1.360(5)
Zr–C(4)	2.237(3)	C(3)–C(4)	1.411(5)	C(15)–C(16)	1.289(6)	C(29)–C(30)	1.403(5)
Zr–Cl(1)	2.469(9)	C(4)–C(5)	1.423(5)	C(16)–C(17)	1.395(6)	C(30)–N(2)	1.385(4)
Zr–Cl(2)	2.434(11)	C(4)–N(1)	1.424(4)	C(20)–C(21)	1.409(4)	C(31)–N(2)	1.347(4)
Zr–C(21)	2.544(3)	C(5)–C(6)	1.475(4)	C(20)–C(24)	1.439(4)	C(31)–C(32)	1.301(5)
Zr–C(22)	2.638(3)	C(6)–C(7)	1.388(5)	C(20)–Si(1)	1.763(5)	C(31)–C(36)	1.371(6)
Zr–C(20)	2.610(3)	C(6)–C(11)	1.391(5)	C(21)–C(22)	1.375(4)	C(32)–C(33)	1.314(8)
Zr–C(23)	2.624(3)	C(7)–C(8)	1.420(6)	C(21)–C(37)	1.511(5)	C(33)–C(34)	1.365(8)
Zr–C(24)	2.613(3)	C(8)–C(9)	1.439(6)	C(22)–C(23)	1.408(4)	C(34)–C(38)	1.522(8)
Si–C(20)	1.763(4)	C(9)–C(10)	1.365(6)	C(23)–C(24)	1.346(4)	C(34)–C(35)	1.313(5)
Si–C(39)	1.830(5)	C(10)–C(11)	1.426(5)	C(23)–N(2)	1.367(5)	C(35)–C(36)	1.312(5)
Si–C(40)	1.870(4)	C(11)–N(1)	1.384(6)	C(24)–C(25)	1.478(4)	C(39)–Si(1)	1.830(5)
Si–C(1)	1.912(4)	C(12)–N(1)	1.540(6)	C(25)–C(26)	1.379(5)	C(40)–Si(1)	1.870(5)
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
C(5)ZrC(2)	63.29(12)	C(21)ZrC(20)	30.9(10)	C(21)ZrC(22)	30.6(12)	C(23)ZrC(24)	32.9(9)
C(5)ZrC(3)	63.22(12)	C(22)ZrC(20)	56.3(9)	Cl(2)ZrC(22)	108.2(9)	C(20)SiC(39)	114.9(3)
C(5)ZrC(1)	39.83(12)	C(1)ZrC(21)	83.3(11)	C(2)ZrC(23)	120.5(11)	C(20)SiC(40)	111.1(3)
C(5)ZrC(4)	37.54(11)	C(2)ZrC(21)	112.2(12)	C(1)ZrC(23)	117.8(11)	C(20)SiC(1)	92.1(10)
C(2)ZrC(3)	38.71(10)	C(3)ZrC(21)	148.2(12)	C(3)ZrC(23)	150.1(11)	C(39)SiC(40)	106.3(2)
C(2)ZrC(1)	37.63(9)	C(4)ZrC(21)	131.0(11)	C(21)ZrC(23)	54.5(12)	C(39)SiC(1)	114.1(2)
C(2)ZrC(4)	62.89(12)	C(5)ZrC(21)	94.5(10)	C(22)ZrC(23)	33.1(11)	C(40)SiC(1)	118.0(2)
C(3)ZrC(1)	64.95(11)	Cl(1)ZrC(21)	93.4(8)	Cl(2)ZrC(23)	81.1(10)	C(2)C(1)C(5)	103.6(3)
C(3)ZrC(4)	37.08(9)	C(1)ZrCl(2)	124.8(9)	Cl(1)ZrC(23)	111.8(10)	C(2)C(1)Si	123.5(3)
C(1)ZrC(4)	64.85(9)	C(2)ZrCl(2)	89.0(10)	C(5)ZrC(23)	148.1(11)	C(2)C(1)Zr	70.4(3)
C(5)ZCl(1)	92.42(9)	C(3)ZrCl(2)	80.7(10)	C(20)ZrC(23)	53.6(11)	SiC(1)Zr	104.1(11)
C(2)ZrCl(1)	145.02(10)	C(4)ZrCl(2)	110.5(10)	C(4)ZrC(23)	175.2(11)	SiC(20)Zr	94.8(3)
C(3)ZrCl(1)	109.06(9)	C(5)ZrCl(2)	143.9(10)	C(2)ZrC(24)	91.3(10)	C(20)C(21)C(22)	106.4(3)
C(1)ZrCl(1)	131.11(9)	Cl(1)ZrCl(2)	99.5(4)	C(1)ZrC(24)	86.9(10)	C(22)C(21)C(37)	123.2(3)
C(4)ZrCl(1)	82.43(7)	C(20)ZrCl(2)	118.2(8)	C(3)ZrC(24)	125.3(10)	C(22)C(21)Zr	78.9(4)
C(1)ZrC(20)	64.1(11)	C(21)ZrCl(2)	135.2(9)	C(21)ZrC(24)	54.8(11)	C(20)C(21)C(37)	130.3(3)
C(2)ZrC(20)	83.7(12)	C(1)ZrC(22)	111.5(11)	C(22)ZrC(24)	53.7(10)	C(20)C(21)Zr	81.1(3)
C(3)ZrC(20)	122.2(11)	C(2)ZrC(22)	130.2(11)	C(2)ZrC(24)	87.4(9)	C(37)C(21)Zr	108.5(4)
C(4)ZrC(20)	127.4(12)	C(3)ZrC(22)	165.8(12)	Cl(1)ZrC(24)	143.1(7)	C(21)C(22)C(23)	108.8(4)
C(5)ZrC(20)	92.3(8)	C(4)ZrC(22)	155.7(12)	C(5)ZrC(24)	123.1(11)	C(21)C(22)Zr	70.4(3)
Cl(1)ZrC(20)	124.3(8)	C(5)ZrC(22)	123.8(11)	C(20)ZrC(24)	32.7(11)	C(23)C(22)Zr	87.3(4)
Cl(2)ZrC(20)	120.4(8)	Cl(1)ZrC(22)	83.7(9)	C(4)ZrC(24)	150.1(9)	C(24)C(23)Zr	79.1(2)

Table 2. (Contd.)

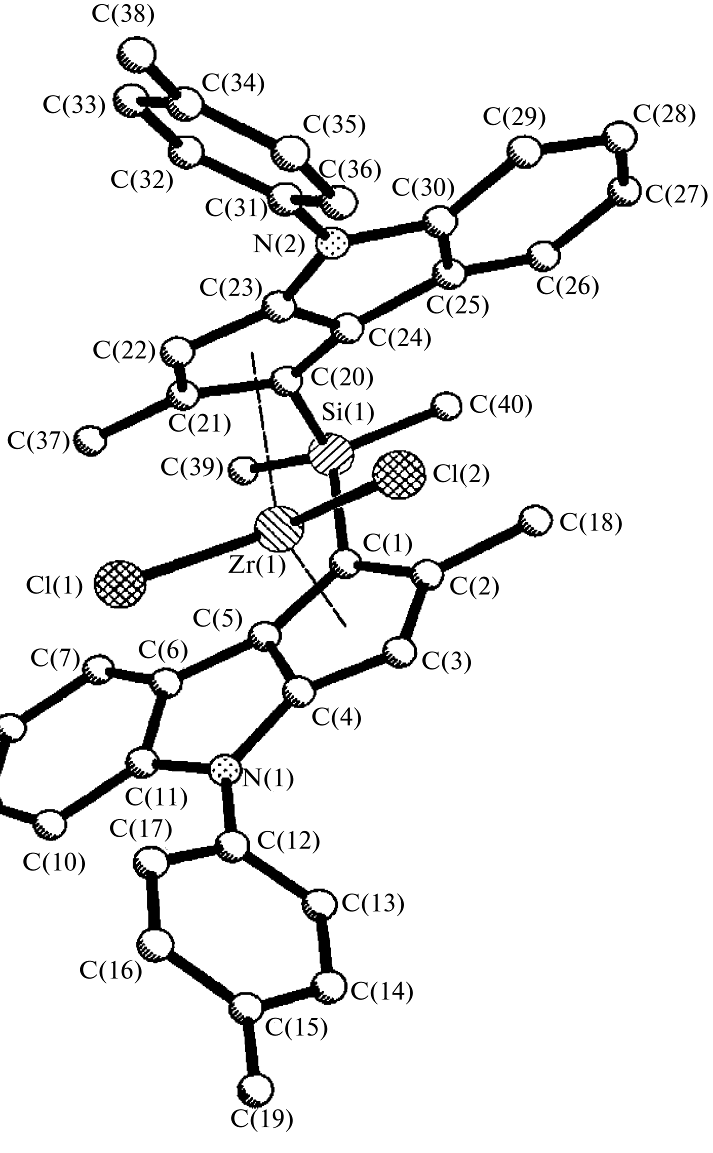
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
C(5)C(1)Si	129.4(3)	C(6)C(5)Zr	123.4(3)	C(16)C(15)C(19)	125.1(4)	C(32)C(33)C(34)	122.6(5)
C(5)C(1)Zr	68.8(3)	C(1)C(5)Zr	71.3(3)	C(15)C(16)C(17)	120.7(4)	N(2)C(23)Zr	125.8(2)
SiC(1)Zr	108.4(12)	C(7)C(6)C(11)	117.6(4)	C(16)C(17)C(12)	124.0(4)	C(22)C(23)Zr	66.4(2)
C(1)C(2)C(3)	110.8(3)	C(7)C(6)C(5)	134.5(4)	C(21)C(20)C(24)	108.4(3)	C(23)C(24)Zr	76.2(2)
C(1)C(2)C(18)	126.0(3)	C(11)C(6)C(5)	107.7(3)	C(21)C(20)Si	119.0(3)	C(20)C(24)Zr	70.3(3)
C(3)C(2)C(18)	124.1(3)	C(6)C(7)C(8)	118.5(4)	C(21)C(20)Zr	68.0(2)	C(25)C(24)Zr	130.5(3)
C(1)C(2)Zr	71.9(2)	C(7)C(8)C(9)	122.2(5)	C(24)C(20)Si	128.3(2)	C(24)C(23)N(2)	108.9(3)
C(3)C(2)Zr	70.7(3)	C(8)C(9)C(10)	120.9(4)	C(24)C(20)Zr	84.4(3)	C(24)C(23)C(22)	109.8(3)
C(18)C(2)Zr	126.2(2)	C(9)C(10)C(11)	116.6(4)	SiC(20)Zr	100.1(10)	N(2)C(23)C(22)	141.2(3)
C(4)C(3)C(2)	107.5(3)	C(10)C(11)N(1)	126.6(4)	C(26)C(27)C(28)	121.9(3)	C(23)C(24)C(20)	106.2(3)
C(4)C(3)Zr	72.8(3)	C(6)C(11)N(1)	109.3(4)	C(27)C(28)C(29)	117.1(4)	C(23)C(24)C(25)	105.5(3)
C(2)C(3)Zr	70.5(10)	C(10)C(11)C(6)	123.9(3)	C(28)C(29)C(30)	120.6(4)	C(20)C(24)C(25)	147.4(3)
C(3)C(4)C(5)	108.3(3)	C(11)N(1)C(4)	106.6(4)	N(2)C(30)C(25)	106.4(3)	C(26)C(25)C(30)	114.7(4)
C(3)C(4)N(1)	138.8(7)	C(11)N(1)C(12)	125.5(4)	C(29)C(30)C(25)	122.9(3)	C(24)C(25)C(30)	108.3(5)
C(3)C(4)Zr	70.0(3)	C(4)N(1)C(12)	127.8(3)	N(2)C(30)C(29)	130.3(3)	C(24)C(25)C(26)	136.6(5)
C(5)C(4)N(1)	112.8(9)	C(13)C(12)C(17)	115.5(4)	C(31)N(2)C(23)	125.1(4)	C(25)C(26)C(27)	122.4(6)
C(5)C(4)Zr	69.1(3)	C(13)C(12)N(1)	120.1(4)	C(31)N(2)C(30)	124.1(3)	C(33)C(34)C(38)	123.1(5)
N(1)C(4)Zr	124.9(3)	C(17)C(12)N(1)	124.2(4)	C(30)N(2)C(23)	110.6(3)	C(33)C(34)C(35)	122.8(5)
C(4)C(5)C(6)	103.3(3)	C(12)C(13)C(14)	119.9(4)	N(2)C(31)C(32)	114.2(4)	C(34)C(35)C(36)	114.3(4)
C(4)C(5)C(1)	109.6(3)	C(15)C(14)C(13)	124.2(5)	C(32)C(31)C(36)	123.2(4)	C(35)C(34)C(38)	129.9(5)
C(4)C(5)Zr	73.2(3)	C(14)C(15)C(19)	123.8(4)	N(2)C(31)C(36)	122.5(3)	C(33)C(36)C(31)	122.4(5)
C(6)C(5)C(1)	146.8(3)	C(16)C(15)C(14)	115.4(4)	C(31)C(32)C(33)	114.4(4)		

Table 3. Comparison of the selected structural parameters of compound **I** and compounds **II–V**

Parameter	**I**	**II**	**III**	**IV**	**V**
Zr–Cl, Å	2.4420^a	2.4314(2)	2.419(1)	2.437(3)	2.443(4)
ClZrCl, deg	99.58(4)	98.76(1)	96.8(1)	98.00(11)	97.86(3)
Zr–Cp^b, Å	2.148^a	2.2413(1)	2.243(4)	2.251(6)	2.230(4)
Zr–C(Cp) min, Å	2.184(3)	2.470(1)	2.478(3)	2.484(10)	2.463(3)
Zr–C(Cp) max, Å	2.638(3)	2.659(1)	2.640(4)	2.663(9)	2.650(3)
CpZrCp, deg	126.51(1)	127.81(1)	128.5(3)	128.4(6)	128.7(4)
E1–E2^c	58.1	61.94	59.2	59.8	59.9
θ^d, deg	139.1(9)	132.8	131.4	139.3(9)	141.2(8)
Φ^e, deg	56.1		44.4	30.7(5)	35.5(34)

Note: **II** is *rac*-$Me_2Si(Ind)_2ZrCl_2$ [1], **III** is *rac*-$Me_2Si(2Me\text{-}4Ph\text{-}1\text{-}Ind)_2ZrCl_2$ [2], **IV** is *rac*-$Me_2Si(2,5\text{-}Me_2\text{-}3Ph\text{-}6\text{-}Cp[b]Tp)_2ZrCl_2$, where Tp is thiophene [3], and **V** is *rac*-$Me_2Si(2,5\text{-}Me_2\text{-}1Ph\text{-}4\text{-}Cp[b]Py)_2ZrCl_2$, where Py is pyrrole [3].

[a] Average value. [b] Centroid of the Cp rings. [c] Angle between the averaged planes of the Cp rings. [d] Exo angle between the fused 5/5 and 5/6 rings. [e] Torsion angle between the planes of the *para*-tolyl and Cp rings.



Molecular structure of complex **I**.

shows that the structural parameters of compound **I** insignificantly differ from the literature data. Major distinctions are observed in the Zr–Cp distances of bis-indenyl and bis-heterocene zirconium complexes. Complex **I** has the shortest Zr–Cp distance of 2.148 Å in comparison with those of indenyl compounds **II** and **III** (2.241 and 2.243 Å, respectively). This seems to be a result of the fact that the bis-indenyl complexes are more sterically strained as compared to fused bis-heterocene complexes. Complexes **II** and **III** exemplify that the dihedral angle between the planes of the indenyl ligands is slightly larger (59.2°–61.94°) than the pyrrole–Cp dihedral angle in complex **I** (58.1°). Complex **I** has the largest torsion angle φ between the planes of the *para*-tolyl and Cp rings (56.1°) among the considered compounds. The other structural characteristics are similar and typical of such complexes.

Complex **I** is an efficient catalyst for the synthesis of highly isotactic PP. Synthesis conditions and some characteristics of the isotactic PP obtained in the presence of compound **I** are given in Table 4. The data of Table 4 show that the presented characteristics of PP synthesized over the $MgMe_2$ + Al · *iso*-Bu_3 cocatalyst are highly competitive with the analogous characteristics of PP obtained with the use of MAO at low Al/Zr molar ratios (500–2000).

Thus, synthesized complex **I** with a short Me_2Si bridge joining two ligands with the methyl group located in the 2-position near the silicon bridge and the tolyl group in the 4-position, which is close to the

Table 4. Catalytic properties of the system based on complex **I**

No.	Complex **I**, mol $\times 10^6$	Cocatalyst	Al/Zr	*A*, kg PP/mol Zr h	$M_\eta \times 10^{-3}$	*mmmm*, %
1	1.128	MAO	2000	5513	224	96
2	1.269	MAO	500	937	144	79
3	2.8	AliBu$_3$ + MgMe$_2$	500	2814	185	94
4	9.94	AliBu$_3$ + MgMe$_2$	300	2208	215	90

Note: Polymerization conditions are $p_{C_3H_6} = 7.0$ atm, toluene, $T = 70.0$°C, $\tau = 1.0$ h, M_η is the viscous molecular mass, and *mmmm* is the number of isotactic pentads in a polymer chain and determined by the ^{13}C NMR method.

Cp ring and comes outside the boundary of the complex, provide the high activity and stereospecificity in the polymerization of propylene.

ACKNOWLEDGMENTS

We are grateful to Academician S.M. Aldoshin for valuable advices and helpful discussions.

REFERENCES

1. W. A. Hermann, J. Rohrmann, E. Hardtweeck, et al., Angew. Chem. **101**, 1536 (1989).
2. W. A. Hermann, J. Rohrmann, E. Hardtweeck, et al., Angew. Chem., Int. Ed. Engl. **29**, 1511 (1989).
3. W. Spaleck, F. Kuber, A. Winter, et al., Organometallica **13**, 954 (1994).
4. J. A. Ewen, M. J. Elder, R. L. Jones, et al., J. Am. Chem. Soc. **123**, 4763 (2001).
5. W. Spaleck, M. Antberg, J. Rohmann, et al., Angew. Chem. **104**, 1373 (1992).
6. Angew. Chem., Int. Ed. Engl. **31**, 1347 (1992).
7. Sh. G. Mkoyan, Z. G. Aliev, L. O. Atovmyan, and R. V. Ivchenko, Izv. Akad. Nauk, Ser. Khim., No. 2, 305 (1995).
8. U. Stehling, J. Diebold, R. Kirsten, et al., Organometallica **13**, 964 (1994).
9. T. Saito, H. Ayukawa, N. Sumizawa, et al., J. Chem. Soc., Perkin Trans., No. 4, 1405 (1991).
10. Z. M. Dzhabieva, S. V. Topilin, S. A. Kuznetsova, et al., RF Patent No. 2275380, Byull. Izobret., No. 12 (2006).
11. A. J. Gordon and R. A. Ford, *The Chemist's Companion. A Handbook of Practical Data, Techniques, and References* (Wiley, New York-London-Sydney-Toronto, 1972), p. 443.
12. S. V. Topilin and Z. M. Dzhabieva, *Proceedings of XXI International Chugaev Conference on Coordination Chemistry, Kiev 2003*, p. 378.
13. V. I. Andrianov, Kristallografiya **32**, 228 (1987).
14. G. M. Sheldrick, SHELXL-97, Program for Crystal Structure Refinement, Univ. of Göttingen, Germany, 1997.