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STEREOCHEMISTRY OF ELECTROPHILIC SUBSTITUTION REACTIONS OF INDENYL-ORGANOTIN COMPOUNDS

A.N. KASHIN *, V.N. BAKUNIN, V.A. KHUTORYANSKII, I.P. BELETSKAYA and O.A. REUTOV

Chemistry Department, Moscow State University, Moscow B-234 (U.S.S.R.)

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Summary

The stereochemistry of the reactions of *S*-(+)-(3-methylindenyl)trimethylstannane (I) with C_6H_5COOH, BrCN and $HgCl_2$ in dimethoxyethane (DME), and of *S*-(+)-(1-methyl-3-phenylindenyl)trimethylstannane (II) with C_6H_5COOH and BrCN in C_6H_6 has been investigated. The reaction products were identified by GLC and ^1H NMR spectroscopy. It was found that the interaction of *S*-(+)-I with C_6H_5COOH yielded a mixture of 2% *R*-(—)-1-methylindene and 98% 3-methylindene. With C_6H_5COOD *S*-(+)-3-methylindene-1-d_1 was formed. The reaction of *S*-(+)-I with BrCN gave *R*-(—)-1-bromo-3-methylindene and *S*-(+)-1-bromo-1-methylindene, the reaction of *S*-(+)-I with $HgCl_2$ yielded *R*-(—)-(3-methylindenyl)mercury chloride. In the reaction of *S*-(+)-II with BrCN *R*-(—)-1-bromo-1-methyl-3-phenylindene was formed, and the reaction with C_6H_5COOH gave an optically inactive mixture of 1-methyl-3-phenyl- and 1-phenyl-3-methylindenes (1/1). The observed stereochemical result is explained by *anti*-attack of the electrophilic agent with reaction centre transfer (S_E2' mechanism).

Introduction

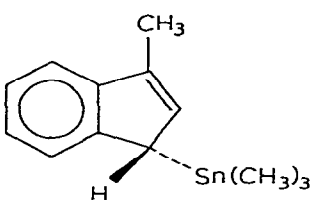
Stereochemical studies of electrophilic substitution reactions in the organomercury series of compounds have allowed formulation of the rule of retention of configuration in S_E2 processes [1]. Extrapolation of this rule to other organometallic compounds has been questioned frequently. Thus, according to Jensen and Davis the bromodestannylation of sec-C_4H_9Sn(neo-C_5H_{11})$_3$, takes place mainly with inversion of configuration but the possibility of retention of configuration in S_E2 porcesses has been shown in some other investigations on

* Author to whom correspondence should be addressed.

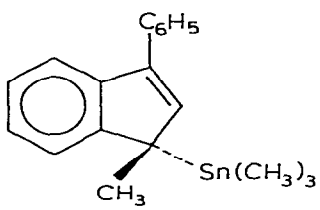
organotin compounds. For instance in the case of organotin derivatives of 1-adamantane [3] and 1-triptycene [4], which are only able to react with retention of configuration, reaction with halogens and acids results in both methyl group elimination and in the elimination of adamantyl and triptycenyl radicals. Retention of configuration is also observed in bromo- [5] and proto-destannylation [6] of cyclopropyl organotin compounds. Rahm and Pereyre [7] have recently overcome this contradiction having shown that bromodestannylation of sec-butyl organotin compounds proceed with retention of configuration. Variation of R in sec-$C_4H_9SnR_3$ has shown inversion of configuration for only the most sterically hindered group (R = neo-C_5H_{11}).

Inversion of configuration in the reactions of transition metal complexes with halogens [8] is apparently due to another mechanism. It is supposed that the complex is oxidized by halogen with subsequent S_N2 attack of the cation-radical formed by the halogen anion. Such a mechanism for non-transition metals (tin in particular) does not seem feasible.

Recently we have obtained *S*-(+)-(3-methylindenyl)trimethylstannane (I) [9] and *S*-(+)-(1-methyl-3-phenylindenyl)trimethylstannane (II) [10]. These com-



(*S*-(+)-I)



(*S*-(+)-II)

pounds react easily with carboxylic acids, BrCN and $HgCl_2$. We have suggested an ion-pair S_E2 mechanism for these reactions [11]. From this point of view an investigation of the stereochemical course of the electrophilic substitution reactions of indenyl organotin compounds is of great interest.

Results

Indenyl organotin compounds react easily with moisture and atmospheric oxygen; for this reason all reactions were performed in evacuated sealed glass apparatus with dry solvents. Optically active indenyl organotin compounds racemize slowly in low-polar aprotic solvents, but when neat and on heating the rate of their racemization increases, therefore optically active samples of $RSn(CH_3)_3$ were prepared in each case immediately before the experiment. NMR techniques were used on racemic compounds to show that organotin compounds thus obtained do not contain any impurities.

An investigation of the stereochemistry of S_E reactions of indenyl derivatives presents considerable difficulties. As a rule, two isomeric products with different signs are formed but some reactions give an unstable product, which then converts to another isomer. The configurations of products could not, until recently, be measured and therefore the stereochemical pathway of a reaction was obtained

TABLE 1

ΣR_D [a] AND $[M]_D$ VALUES OF THE SUBSTITUTED 3-METHYLINDENES (OPTICAL PURITY 86%)

Substituent	ΣR_D	$[M]_D$	$[M]_{D, exp}$	$\frac{[M]_{D, exp}}{[M]_D} \times 100$ (%)
D	1.65	—1.7	2.92 [b]	
H	1.68	0	0	
CH_3 [c]	6.3	246	246	
	6.3	695 [b]	—172 [b]	25
Br	9.8	432	—159 [d]	56
$(CH_3)_3Sn$	16.6	794	677	85.5
HgCl	18.4	889	—666	75

[a] Ref. 13. [b] At λ 370 nm. [c] For 1-methylindene. [d] For a mixture of two isomers.

by calculations from the sign of optical rotation of the product mixture. Determination of the absolute configuration of compounds was based on the signs of their optical rotations according to Brewster's rule [12]. Values of molecular rotation were calculated by the method of Jensen and Davis [13], which is based on the linear correlation between the $[M]_D$ (MR) value of the compounds and ΣR_D (BR) value for the sum of the substituents. Since the error in the BR-MR method of calculations for the indenyl system is not known, the values of stereoselectivity obtained are qualitative and only show the absolute degree of inversion of configuration because this method may be slightly inaccurate for the indenyl system.

It is known that in BR-MR [13] calculations linear correlations of the type shown in eq. 1 are used. Coefficients A and B are obtained using known $[M]_D$

$$[M]_D = A\Sigma R_D + B \quad (1)$$

values for two or more compounds. For example, in the calculation of the $[M]_D$ value for (+)-(3-methylindenyl)trimethylstannane the data for *S*-(+)-1-methylindene * ($[\alpha]_D^{18}$ +189°, $[M]_D$ +246°, ΣR_D = 6.3 for CH_3) and 3-methylindene ($[M]_D$ 0°, ΣR_D = 1.68 for H) are used (eq. 2).

$$[M]_D = 53.2\ \Sigma R_D - 89.2 \quad (2)$$

Equation 2 is derived for an *S*-(+)-1-methylindene content of 93% and makes it possible to estimate the stereoselectivity of the stannylation reaction. From the values of $[M]_{D,ob.}$ +677° and $[M]_{D\ calc.}$ +794° it can be concluded that the stereoselectivity of the reaction is 85.5%.

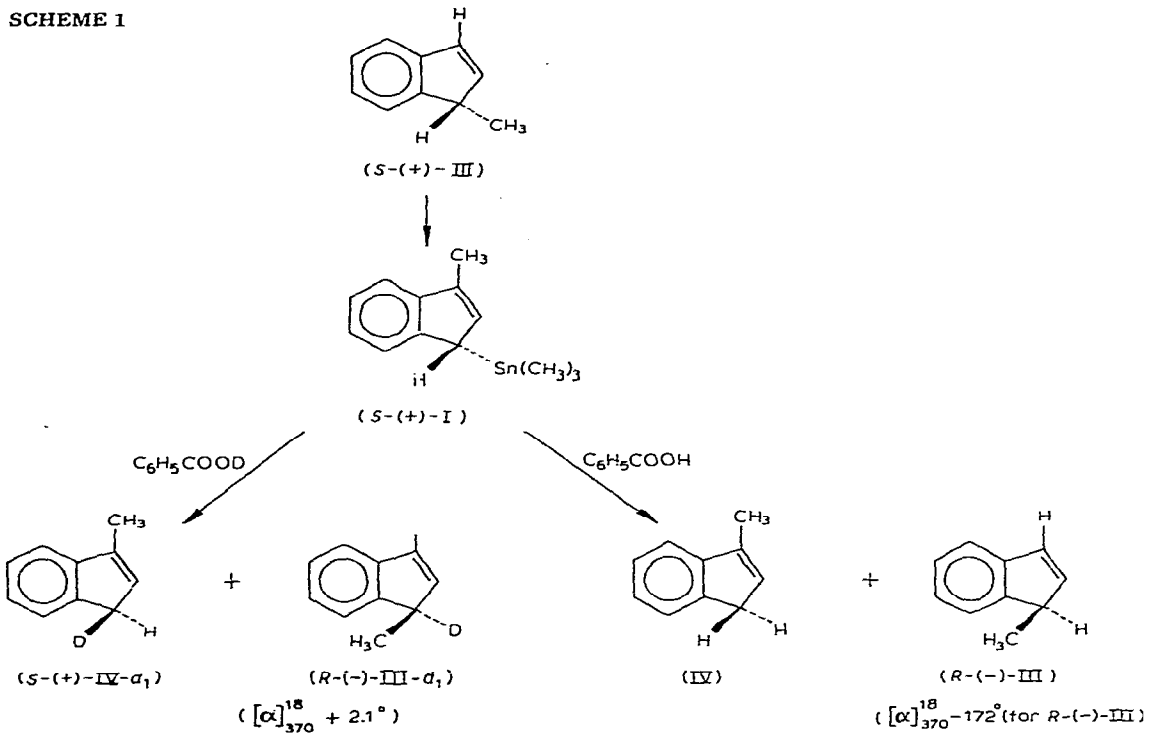
$[M]_D$ values of the electrophilic substitution reaction products, and their absolute configurations were obtained in a similar manner.

The correlation equations used in calculations were obtained taking into consideration the optical purity of the initial organotin compound. Calculated and experimental $[M]_D$ values for some substituted methylindenes are collected in Table 1.

The stereochemistry of protodestannylation was studied in the reaction of

* It is assumed that the lack of a CH_3 substituent in position 3 does not influence the value of $[M]_D$.

SCHEME 1



S-(+)-(3-methylindenyl)trimethylstannane with benzoic acid and its deuterated analogue, in dimethoxyethane (DME). As determined by GLC, the reaction products are 3-methylindene (98%) and 1-methylindene (2%). With C_6H_5COOH the sign of optical rotation is changed. The stereochemistry of 3-methylindene formation was studied in the reaction of *S*-(+)-I with C_6H_5COOD. In this case the sign of optical rotation did not change. The deuterium atom possesses lower polarizability than protium (ΣR_D(D) 1.65, ΣR_D(H) 1.68 [13]), therefore (+)-3-methylindene-1-d_1 must have *S*-configuration. Note, that *R*-1-methylindene-1-d_1 possesses a negative sign of optical rotation: the reaction may be described as in Scheme 1. The stereoselectivity obtained (25%) is a minimum value because it includes two reactions, metallation and protolysis.

The interaction of PhCOOH with (1-methyl-3-phenylindenyl)trimethylstannane in C_6H_6 results in an equimolar mixture of isomeric 1,3-methylphenylindenes. The racemization observed in this reaction may be caused by racemiza-

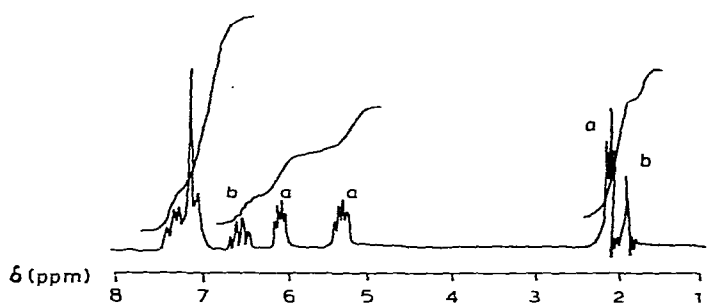
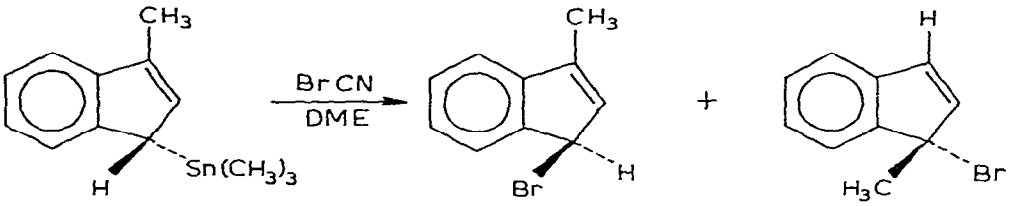


Fig. 1. ^{1}H NMR (60 MHz) spectrum of (a) 1-bromo-3-methylindene, and (b) 1-bromo-1-methylindene.

tion of the initial organotin compound [9], because the rate of protolysis under experimental conditions is low. Furthermore, compensation of optical activity may take place, as the isomers may possess opposite configurations.

The products of the reaction of I with BrCN in DME were determined by NMR technique. In the NMR spectrum of the reaction mixture signals may be observed corresponding to 1-bromo-3-methylindene (V): (CCl_4, δ ppm) CH_3 2.10 t, H(1) 5.35 m, H(2) 6.07 m; and 1-bromo-1-methylindene (VI): (CCl_4, δ ppm) CH_3 1.92 s, H(2) 6.37 d, H(3) 6.53 d. The ratio of integral intensities of the methyl group signals is 5/1.

In the same reaction with optically active *S*-(+)-I ($[\alpha]_D^{18}$ +188°) the reaction mixture had $[\alpha]_D^{18}$ —60.6°. Analogously to the protodestannylation reaction the reaction must give *R*-(—)-V and *S*-(+)-VI (eq. 3).



(*S*-(+)-I) (*R*-(—)-V) (*S*-(+)-VI) (3)

The $[M]_D$ value for 1-bromo-3-methylindene was calculated using eq. 4, derived for initial 3-$CH_3C_9H_6Sn(CH_3)_3$ ($[\alpha]_D^{18}$ +188°, optical purity 80%) and 3-methylindene.

$$[M]_D = 36.9\ \Sigma R_D - 62.0 \qquad (4)$$

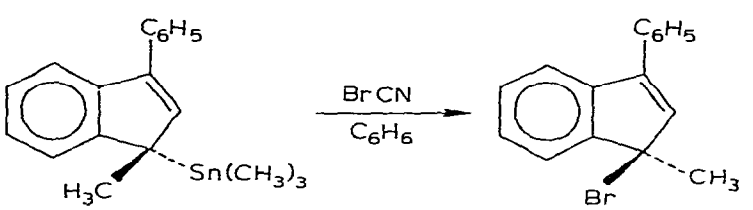
By substitution ΣR_D = 9.8 (Br) one can obtain $[M]_D$ 300° ($[\alpha]_D$ 143°).

To calculate $[M]_D$ for 1-bromo-1-methylindene eq. 5 was derived using known values for initial 1-methylindene [$(M]_D$ —170°, ΣR_D 1.68) and 1,1-dimethylindene ($[M]_D$ 0°, ΣR_D 6.3).

$$[M]_D = 36.9\ \Sigma R_D - 232 \qquad (5)$$

So, for 1-bromo-1-methylindene (VI) we can obtain the value $[M]_D$ 130° ($[\alpha]_D$ 62.2°); and for the mixture of *R*-(—)-V and *S*-(+)-VI in the ratio 5/1, $[\alpha]_D$ 109°. The experimental value $[\alpha]_D$ —60.6° makes it possible to estimate the stereoselectivity of the bromodestannylation reaction to be 56%.

According to its NMR spectrum, the reaction of (1-methyl-3-phenylindenyl)-trimethylstannane with BrCN in C_6H_6 results in only one product, 1-bromo-1-methyl-3-phenylindene (VIII). Alternation of the sign of optical rotation indicates inversion of configuration, because the bromine atom posesses the greatest polarizability (eq. 6).



(*S*-(+)-II) (*R*-(—)-VIII) (6)

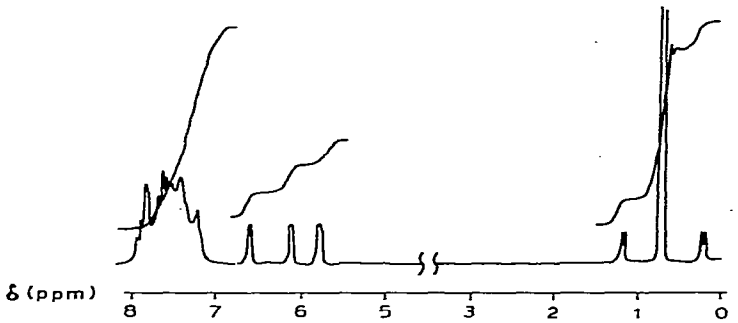
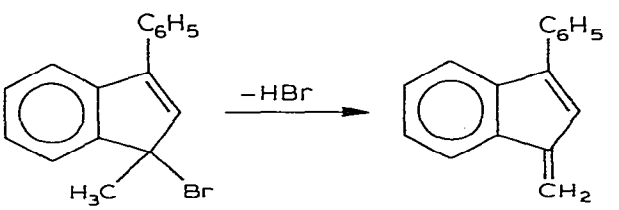


Fig. 2. ^{1}H NMR (60 MHz) spectrum of the reaction mixture of (1-methyl-3-phenylindenyl)trimethylstannane and BrCN, in benzene, after 30 min.

It is interesting to note that VIII formed in this reaction is not the final product; after 15 min a decrease in intensity and finally the disappearance of the CH_3-group signal (δ 2.0 ppm) and the vinyl proton signal from the second C-atom (δ 6.6 ppm), and the appearance of three new signals at δ 5.8, 6.2 and 6.6 ppm of approximately equal intensity (Fig. 2) may be obtained in the NMR spectrum of the reaction mixture.

This reaction was not studied specially * but one possible explanation may be the formation of 1-methylene-3-phenylindene as the result of HBr elimination (eq. 7).



(7)

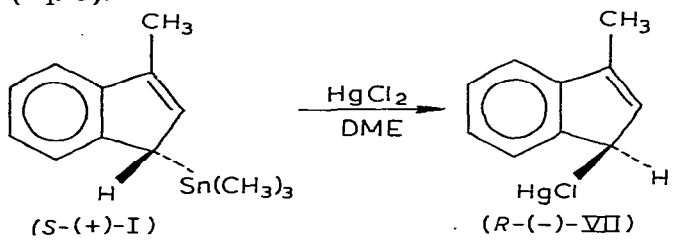
In NMR experiments, when greater concentrations of reagents were used (*c* 0.36 *M*), conversion of the product was already completed after 30 min, whereas in polarimetric experiments (*c* 0.012 *M*) complete racemization was observed only after 12 h. These facts suggest that the reaction is not a monomolecular process, and it appears that $(CH_3)_3SnCN$ formed in the reaction acts as an electrophilic catalyst. The formation of HBr is indicated by a gradual shift of the methyl proton signal in $(CH_3)_3SnCN$ to lower field; this is caused by coordination of the bromide ion with the tin atom.

(3-Methylindenyl)trimethylstannane reacts very rapidly with $HgCl_2$ in DME. The product, (3-methylindenyl)mercury chloride, is only slightly soluble in DME and is precipitated at high concentrations of reagents.

In the reaction of *S*-(+)-I with $HgCl_2$ a change of sign of optical rotation takes place. This is testimony of inversion of configuration in the reaction

* An attempt to synthesise VIII from 1-methyl-1-phenylindene and *N*-bromosuccinimide under radical conditions failed: the reaction led to a complicated mixture of products.

because the ΣR_D value of HgCl (as of $(CH_3)_3Sn$) is the greatest in the molecule (eq. 8).



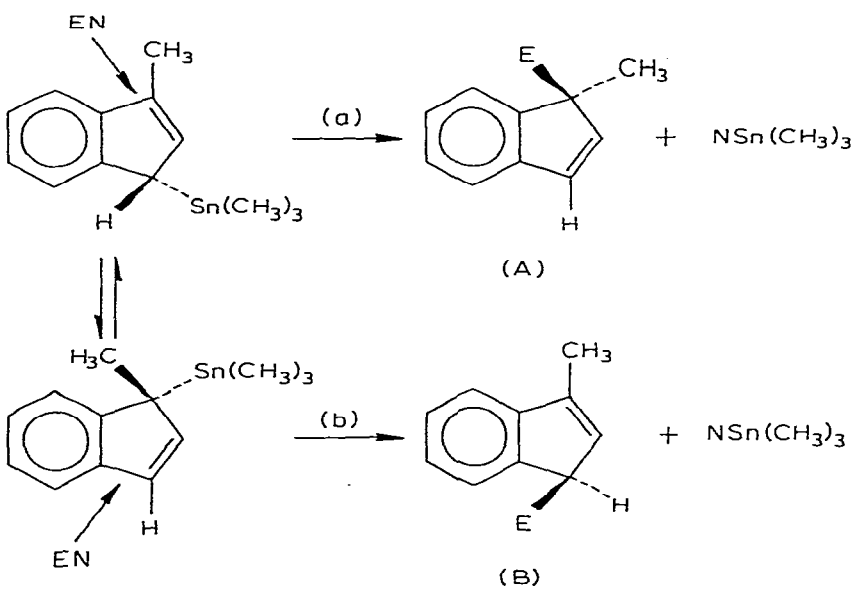
(S-(+)-I) (R-(−)-VII) (8)

Comparison of experimental $[M]_D$ values and those calculated according to the BR-MR [13] method (Table 1), shows the stereoselectivity of mercury-destannylation to be 75%.

Discussion

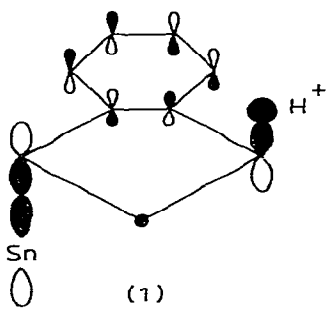
As can be seen from the above, in proto- and bromo-destannylation reactions of 3-methylindenyl derivatives of trimethyltin two isomers are formed, and in the reaction with $HgCl_2$ only one product arises. In all cases the electrophile attacks the organotin molecule from the side opposite to the leaving $(CH_3)_3Sn$ group. This stereochemical result needs some explanation. Theoretically, indenyl organotin compounds can undergo S_E reactions with reaction centre transfer (allylic rearrangement) but the stereochemistry of this process has not yet been investigated. Also, the possibility of a rapid metallotropic rearrangement which, as has been shown for *S*-(+)-(1-methyl-3-phenylindenyl)trimethylstannane [10], occurs suprafacially with retention of optical activity must be considered. Thus, the mechanism of electrophilic substitution reactions of indenyl organotin compounds may be presented as in Scheme 2.

SCHEME 2

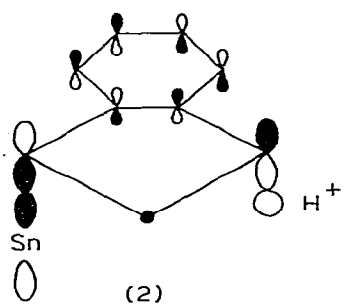


According to Scheme 2 the unrearranged product B is formed by two consecutive reactions: metallotropic rearrangement followed by substitution with reaction centre transfer. Previously Kuivila et al. [14] proposed such a mechanism for protolysis of crotyl organotin compounds. Furthermore, this does not contradict the results obtained for the base protolysis of *trans*-cinnamyl organotin compounds in methanol [15].

Apparently, the reaction of *S*-(+)-3-$CH_3C_9H_6Sn(CH_3)_3$ with $HgCl_2$ is just as complicated; the unstable 1-$CH_3C_9H_6HgCl$ formed initially undergoes a rapid metallotropic rearrangement resulting in *R*-(—)-3-$CH_3C_9H_6HgCl$. As in the case of organotin compounds this rearrangement must proceed intramolecularly and with retention of optical activity. In the mechanism discussed (Scheme 2) for the reactions of indenyl organotin compounds the stereochemistry of S_E2' processes includes *anti*-interaction with the electrophilic substrate. This does not contradict orbital symmetry rules [16]: the electrophilic molecule can, in fact, interact with the highest occupied molecular orbital (HOMO) of the indenyl group from *anti*-(1) and *syn*-(2) positions, on condition that electrophilic attack and elimination of $(CH_3)_3Sn$-group are simultaneous in the transitional state of the reaction. For the reaction with a proton these two events may be represented as follows:



(1)



(2)

In the second case the phases of terminal lobes coincide, in the first they do not. Since the number of participating electrons is 4, the transition state 1 must be more favourable and reaction must proceed by *anti*-attack of H^+.

In the case of BrCN the transition state involves the lowest unoccupied molecular orbital (LUMO) of BrCN and 8 electrons:

(LUMO)

σ^*

Since in a type 1 transition state the phases of terminal lobes do not coincide, the reaction with BrCN must also proceed via *anti*-attack.

Data obtained in the present work seem to be the first example of confirmation of *anti*-substitution in S_E2' processes, which had been predicted earlier by orbital symmetry rules [16]. It is interesting to note, that in the case of S_N2' reactions the opposite result is observed: *syn*-displacement by a nucleophilic reagent is allowed by symmetry. This conclusion has been confirmed experimentally [17,18]. Retention of configuration in S_E2 reactions of organometallic compounds is also well founded using this approach, and our results agree with data obtained earlier.

An alternative explanation of the obtained stereochemical results is possible. According to ideas which we have developed recently [11], indenyl organotin compounds react with electrophilic substrates in the form of ion pairs. The existence of ion pairs was shown by an investigation of the racemization of *S*-(+)-(3-methylindenyl)trimethylstannane in DME [9]. Racemization of *S*-(+)-(1-methyl-3-phenylindenyl)trimethylstannane [10] is particularly fast. It thus seems possible for an electrophilic reagent to attack an indenyl organotin compound from the side opposite to the $(CH_3)_3Sn$-group because in the ion pair the electrons become more accessible than in the covalent form: steric hindrances and electrostatic interaction of the electrophile with the leaving group are absent. In this case the only consequence of an ion pair mechanism would be the decreased stereoselectivity of the reaction. The correctness of such a hypothesis can be verified only by further stereochemical investigations on organotin compounds which do not exhibit an S_E2' mechanism, but which react via ion pair participation.

In conclusion we wish to note the importance of chiral organotin compounds for synthetic purposes in obtaining other optically active substances. The present work shows the route to bromo and mercury derivatives in quantitative yields, starting from optically active indenes via organotin compounds. The relative accessibility of organotin compounds, their sufficient stability, high reactivity in electrophilic substitution reactions, and ability to retain configuration make them irreplaceable in the synthesis of chiral compounds, containing delocalized π-electron systems or electron-withdrawing groups.

Experimental

Polarimetric measurements were carried out on a Hilger and Watts polarimeter using the sodium D-line (instrument error ±0.01°) and on a Jusco J-20 spectropolarimeter (instrument error ±0.001°). NMR spectra were obtained on Varian T-60 and XL-100 instruments (cyclohexane internal standard, 1.42 ppm).

Chromatographic analyses were carried out on a Tsvet-2 chromatograph. Column: *l*, 1.5 m; *d*, 2.8 mm; Chromosorb W-DMS, 100—120 mesh, 5% E-301, N_2 30 ml/min. At 90°C (evaporator 190°C) retention time: 1-methylindene, 5.1 min; 3-methylindene, 8.4 min.

S-(+)-(3-Methylindenyl)trimethylstannane (I)

S-(+)-I was obtained from 0.0671 g (0.52×10^{-3} mol) of *S*-(+)-1-methylindene, ($[\alpha]_D^{18}$ +189°), and 0.1868 g (0.79×10^{-3} mol) $(C_2H_5)_2NSn(CH_3)_3$ in 4.18 ml of C_6H_6. On reaching a maximum value of specific rotation (after 2 h), excess $(C_2H_5)_2NSn(CH_3)_3$, benzene and diethylamine were evaporated under vacuum and the residue was dissolved in 3.99 ml of C_6H_6. The organotin compound obtained had an optical rotation $[\alpha]_D^{18}$ +231°. The NMR spectrum of (±)-I, which was synthesized in a similar manner, proved that the substance contained no impurities.

S-(+)-(1-Methyl-3-phenylindenyl)trimethylstannane (II)

S-(+)-II was obtained from 0.1274 g (0.62×10^{-3} mol) of *R*-(—)-1-methyl-3-phenylindene ($[\alpha]_D^{18}$ —55.6°) and 0.3112 g (1.3×10^{-2} mol) of $(C_2H_5)_2NSn(CH_3)_3$

in 4.22 ml of benzene using the same procedure as for *S*-(+)-I. The residue was dissolved in 4.16 ml of benzene and had an optical rotation $[\alpha]_D^{18}$ +24°.

Reaction of S-(+)-I with C_6H_5COOH and C_6H_5COOD

S-(+)-I was obtained from 0.1124 g (0.87×10^{-3} mol) of *S*-(+)-1-methylindene ($[\alpha]_{370}^{18}$ +695°) and 0.390 g (1.7×10^{-3} mol) of $(C_2H_5)_2NSn(CH_3)_3$ and dissolved in 6.22 ml of DME ($[\alpha]_D^{18}$ +207°). To one portion (3 ml) of the solution 0.0953 g (0.78×10^{-3} mol) of C_6H_5COOH was added and to another 3 ml portion was added C_6H_5COOD (0.0915 g, 0.74×10^{-3} mol). After 14 h all volatile components were distilled off under high vacuum. In the case of C_6H_5COOH the resulting 1-methylindene showed $[\alpha]_{370}^{18}$ —172°; for C_6H_5COOD the mixture of isomers showed $[\alpha]_{370}^{18}$ —2.1°. The GLC analysis of these mixtures showed 3-methylindene 98% and 2% 1-methylindene in both solutions.

Reaction of (±)-I with BrCN in DME

To 1.590 g (5.08×10^{-3} mol) of 3-$CH_3C_9H_6Sn(CH_3)_3$ in 4 ml of DME was added a solution of 0.5916 g (5.59×10^{-3} mol) BrCN in 2 ml of DME (under an argon atmosphere). After 2—3 min crystals of $(CH_3)_3SnCN$ appeared. 24 h later the sediment was filtered off and the solvent was evaporated. The residue was distilled under vacuum (b.p. 58—60°C/0.1 mm; yield 1.01 g, 90%). NMR spectrum (CCl_4, δ ppm): 1-bromo-3-methylindene: CH_3 2.10 t, H(1) 5.35 m, H(2) 6.07 m; 1-bromo-1-methylindene: CH_3 1.95 s, H(2) 6.37 d, H(3) 6.53 d. Integration of methyl groups signals gave a ratio of isomers of 4/3. Yield of $(CH_3)_3SnCN$ was 0.64 g (62%); m.p. 182°C (lit. [19]: 184°C).

Reaction of S-(+)-I with BrCN in DME

S-(+)-I was obtained from 0.0860 g (0.66×10^{-3} mol) of *S*-(+)-1-methylindene ($[\alpha]_D^{18}$ +164°) and dissolved in 6.4 ml of DME ($[\alpha]_D^{18}$ +188°). 24 hours after the addition of 0.1711 g (1.61×10^{-3} mol) of BrCN the mixture exhibited $[\alpha]_D^{18}$ —60.6 ($t_{1/2}$ ~4 h). Integration of methyl groups signals gave V/VI 5/1.

Reaction of (±)-I with $HgCl_2$ in DME

0.7723 g (2.64×10^{-3} mol) of 3-$CH_3C_9H_6Sn(CH_3)_3$ in 2 ml of DME were added to 0.8626 g (3.17×10^{-3} mol) of $HgCl_2$, suspended in 2 ml of DME under Ar. After 15 min 3-$CH_3C_9H_6HgCl$ was separated by filtration, washed with $(C_2H_5)_2O$ and dried under high vacuum. Yield 0.85 g (94.5%); m.p. 137°C (lit. [20]: 138°C); NMR spectrum (DMSO-d_6, δ ppm): CH_3 2.22 t, H(1) 4.30 m, H(2) 6.47 m.

Reaction of S-(+)-I with $HgCl_2$ in DME

S-(+)-I was obtained from 0.0079 g (0.61×10^{-4} mol) of *S*-(+)-1-methylindene (α_D +0.0225°, $[\alpha]_D^{18}$ +231°). The amount of (+)-$RSn(CH_3)_3$ was reduced from 8.85 mg to 4.21 mg (α_D +0.012). After addition of 0.0082 g (0.3×10^{-4} mol) of $HgCl_2$ the reaction was over in 17 min, the solution showed α_D —0.0118 ($[\alpha]_D^{18}$ —182°).

Reaction of S-(+)-II with BrCN in C_6H_6

S-(+)-II was obtained from 0.1314 g (0.64×10^{-3} mol) of *R*-(—)-1-methyl-3-

phenylindene ($[\alpha]_D^{18}$ −55°) and 0.2712 g (1.18×10^{-3} mol) of $(C_2H_5)_2NSn(CH_3)_3$ and dissolved in 3.94 ml of C_6H_6 ($[\alpha]_D^{18}$ +19.8°). After addition of BrCN (0.0695 g, 0.65×10^{-3} mol) the highest observed value of specific rotation, $[\alpha]_D^{18}$ −21.2°, was observed 1 h later. After 24 h the compound has racemized.

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