Journal of Organometallic Chemistry, 129 (1977) 145—154

SYNTHESIS OF SOME ORGANOTINS CONTAINING THE CYCLOPENTANE RING

H.G. KUIVILA and P.P. PATNODE

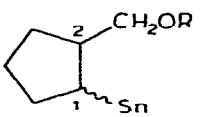
Department of Chemistry, State University of New York at Albany, Albany, N.Y. 12222 (U.S.A.)

(Received August 9th, 1976)

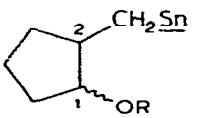
Summary

The synthesis of the *cis* and *trans* isomers of 2-hydroxymethylcyclopentyltrimethylstannane and of *cis*-2-hydroxycyclopentylmethyltrimethylstannane are described, with 2-carbethoxycyclopentanone used as the common source of the carbon skeleton.

To extend our studies on the mechanisms of 1,3-destannoxylations leading to cyclopropanes [1] we wished to examine 3-oxypropylstannyl systems of the type RO—C—C—C—Sn in which the central carbon and one of the terminal carbons constituted parts of the five-membered ring, I—OR and II—OR. In these systems the stereochemistry at the electrofugal center in I—OR and at the nucleofugal



(I-OR)



(II-OR)

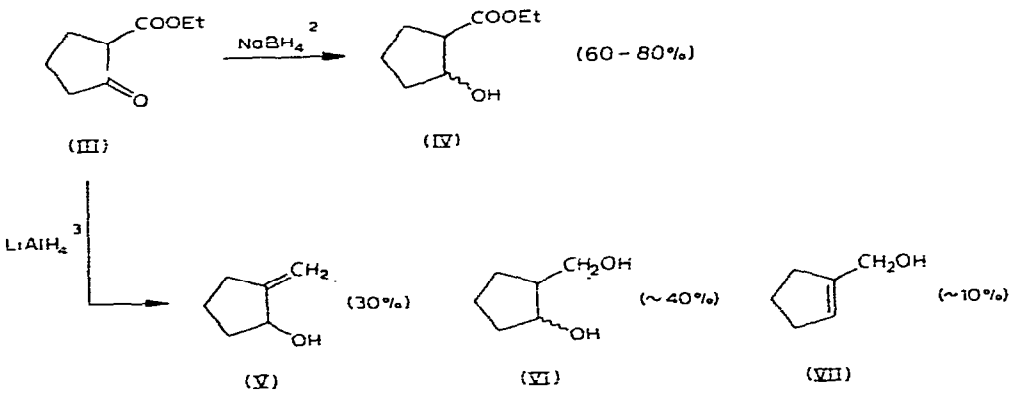
center in II—OR are fixed. Hence the stereochemistry of bicyclohexane formation, if it is to occur is determined by that of the 3-oxypropylstannyl system. We describe the preparation of three of the four isomers in this paper.

The successful syntheses were achieved from compounds IV, V and VI, which could be obtained by reduction of 2-carbethoxycyclopentanone(III), as shown in Scheme 1.

2-Hydroxycyclopentylmethyltrimethylstannane (II—OH)

Hydrostannation of a 1 *M* solution of V in cyclohexane initiated with azobisisobutyronitrile provided a *cis/trans* mixture of the adduct II—OH in 50% yield. Its purity was verified by elemental analysis, and the stereochemical

SCHEME 1



assignments of the *cis* and *trans* isomers were made on the basis of ^{13}C magnetic resonance spectroscopy. The mixture showed thirteen peaks instead of the expected fourteen, but the assignments could be made on the basis of comparison with data for *cis*- and *trans*-2-methylcyclopentanol (VIII) [4], and are shown in Table 1. These assignments for II—OH were also confirmed for the pure stereoisomers. The crucial parameters are the differences between the chemical shifts for C(1), C(2) and C(6). In one isomer each is substantially greater than in the other. This is readily interpretable in terms of compression strain in the *cis* isomer [4], which is reflected in the upfield shifts of all three carbon resonances. It is worth noting, in addition, that the chemical shifts and the geminal coupling constants for the methyl carbons on tin are coincident in the two isomers of II—OH.

Attempted chromatographic separation (GLPC, dry column, normal column) proved to be impractical methods for separation of the *cis* and *trans* isomers of II—OH. Recrystallization of a mixture of the *p*-nitrobenzoates was partially successful in that the *cis* isomer could be obtained in pure state, but with poor recovery. Fortunately, an alternative pathway to these compounds was successful.

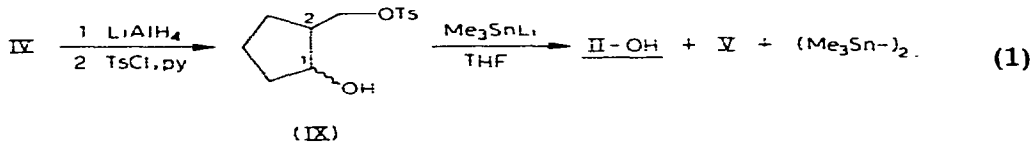
TABLE 1

^{13}CMR PARAMETERS FOR *cis*- *trans*-II—OH USED IN ASSIGNMENT OF STRUCTURE AND STEREOCHEMISTRY (δ, ppm)

C [a]	*cis*	*trans*	Δ [b,c]	Δ [c]
1	76.1	81.5	5.4	4.6
2	43.9	46.1	2.2	2.4
3	34.6	33.8	−0.6	−0.6
4	22.2	21.4	−0.8	−0.6
5	32.2	33.0	−0.8	−0.2
6	10.9 [d]	15.9 [e]	5.0	4.6
CH_3	−9.6 [f]	−9.6 [f]		

[a] Numbering system shown in structure II—OR. [b] Δ values for 2-methylcyclopentanols (VIII), from ref. 4. [c] (δ-*trans* − δ-*cis*) ppm. [d] $J(^{119}Sn—^{13}CH_3)$ 371 Hz. [e] $^{119}Sn—^{13}C$ satellites not visible in spectrum. [f] $J(^{119}Sn—CH_3)$ 314 Hz.

The *cis/trans* mixture of esters of IV could be separated by fractional distillation at reduced pressure using a spinning band column. The esters could be individually reduced to the diols with lithium aluminum hydride, and then converted to the 2-tosyloxymethylcyclopentanols (IX) by selective reaction at the primary hydroxyls with *p*-toluenesulfonyl chloride at low temperature. Moderate yields of the isomers of II—OH could be obtained by reaction of the tosylates individually with trimethylstannyllithium in tetrahydrofuran, eq. 1. The major side reaction products were hexamethyldistannane and 2-methylenecyclopentanol (V), which was the exclusive product when the solvent was a mixture of 40% tetraglyme and 60% tetrahydrofuran. The ^{13}CMR spectra of the

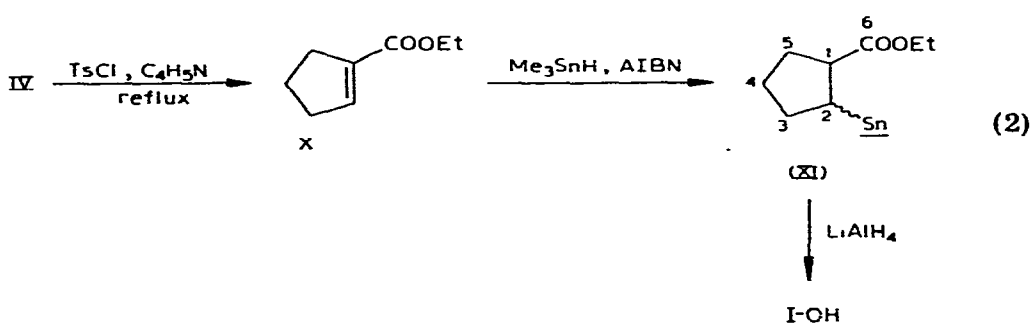


(IX)

pure isomers of II—OH thus obtained agreed with those reported in Table 1. Although the reaction product from *cis*-IX in THF contained from one-half to one-third of the elimination product V, this compound was formed in only minor amounts from *trans*-IX. These observations may be rationalized on the basis that, in *cis*-IX the hydroxyl group provides some steric obstruction to an S_N2 displacement of the tosylate group, and the proton to be abstracted from C(2) is relatively unencumbered. In the *trans* isomer the reverse is true: the tosyloxymethyl group is more free, and the proton on C(2) is more encumbered by the neighboring hydroxyl. If the trimethylstannyllithium reacted with the hydroxylic proton faster than it does in the S_N2 reaction, the product-forming step would involve the alkoxide ion of IX, in which case one should consider the steric bulk of the oxyanionoid, the degree of negative charge which it bears, and its interactions with the approaching trimethylstannylanionoid.

2-Hydroxymethylcyclopentyltrimethylstannanes (I—OH).

The first route to this structure involved tosylation and dehydrotosylation [5] of the hydroxyester IV in pyridine to provide ethyl cyclopentene-1-carboxylate (X), which was then subjected to hydrostannation with trimethylstannane to form ethyl 2-trimethylstannylcyclopentanecarboxylate (XI), in good yield, eq. 2. Reduction of the ester with lithium aluminum hydride provided alcohol I—OH. Compound XI showed the expected spectral properties and elemental



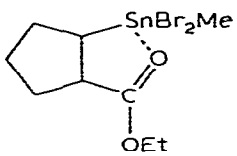
X

(XI)

analysis, and its ^{13}CMR spectrum (Table 2) was in satisfactory agreement with that calculated on the basis of the additivity principle [6], but did not permit the assignment of stereochemistry because only a single isomer was found.

A plausible assignment of stereochemistry was achieved as follows. Compound XI was treated with two molar equivalents of bromine, resulting in the replacement of two methyl groups on tin by bromines. Simple esters of this type have been shown to display intramolecular coordination between the carbonyl carbon of the ester group and the tin atom [8]. Evidence for such coordination may be obtained from infrared, electronic, proton magnetic resonance, carbon magnetic resonance, and Mössbauer spectra [9].

A carbonyl stretching frequency found at 1730 cm^{-1} in XI was also present



(XII)

in XII along with another band of twice its intensity at 1665 cm^{-1}, thus implicating the coordination depicted in XII. The coupling constant $^2J(^{119}Sn—H)$ for the methyl protons in dibromodimethylstannane and dibromodiethylstannane are 66 and 54 Hz, respectively [10]. Similarly, the value for bromotrimethylstannane and cyclohexyldimethylbromostannane are 58 and 49 Hz, respectively; i.e., the substitution of other alkyls for methyl decreases ^2J(Sn—H). Hence the unperturbed coupling constant in XII would be expected to lie between 66 and 54 Hz. The observed value for XII of 66 Hz, is larger than predicted, as has been observed in other systems involving intramolecular coordination [9]. The observation of intramolecular coordination leads to the conclusion that the isomer in question has the *cis* configuration. This is supported by examination of Dreiding models which shows that the distance of closest approach of the carbonyl oxygen and the tin atom in the *trans* isomer is far too large (ca. 4.0 vs. 2.4 Å for the *cis* isomer) for significant bonding to occur. The observation of two carbonyl bands in the IR spectrum, but only one tin methyl resonance in

TABLE 2

^{13}CMR PARAMETERS, CALCULATED AND OBSERVED FOR XI

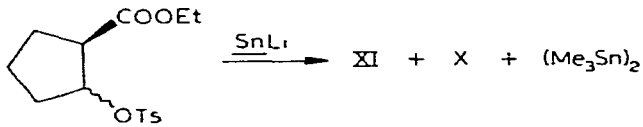
C	$C_5H_9CH_3$	Δ(COOR) [a]	Δ($SnMe_3$) [b]	δ(calcd)	δ(obsd)	$J(^{119}Sn—^{13}C)$
1	34.8	+10.0	+3.6	48.4	47.3	C
2	34.9	—1.0	—1.9	32.0	28.7	404
3	25.5	—1.0	+3.6	28.1	31.0	30
4	25.5	—1.0	—1.2	23.3	26.2	44
5	34.9	—1.0	—1.2	32.7	30.5	
6					177.0	25
CH_3					—9.08	332

[a] On replacement of CH_3 of methylcyclopentane by COOEt (ppm). [b] On replacement of H on C(2) of ethyl cyclopentanecarboxylate by $SnMe_3$ (ppm) [7]. [c] Not determined.

the PMR spectrum indicates that the coordinated and uncoordinated structures of XII are present, and interconvert rapidly on the NMR time scale [9].

Attempts were made to epimerize XI at the carbon bearing the ethoxycarbonyl group using the following reagents: sodium ethoxide in ethanol, potassium t-butoxide in t-butyl alcohol, sodium hydride in refluxing THF and in 1,2-dimethoxyethane; and lithium cyclohexylamide in cyclohexylamine. In no case was any significant amount of isomeric product observed by ^{13}CMR. Consequently, an alternative synthetic approach was tried.

The hydroxyesters *cis*- and *trans*-IV were converted to the tosylates XIII, and treated with trimethylstannyllithium in the hope that the tosylate group would be replaced by the trimethylstannyl group with inversion to provide XI, eq. 3.



(3)

(XIII)

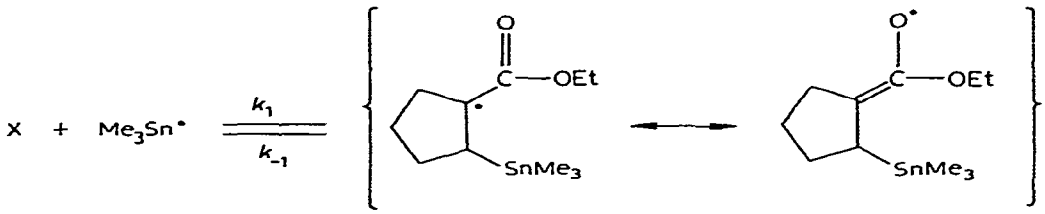
In each case both substitution and elimination occurred, along with the formation of small amounts of hexamethylditin. Most importantly, however, the substitution product obtained from the *cis*-tosylate was indistinguishable from that obtained from the *trans*-tosylate as indicated by their ^{13}CMR parameters. This could be due to at least two circumstances: one isomeric tosylate reacts with inversion and the other with retention; or a common intermediate is formed from each tosylate, which then reacts further to provide the single substitution product. A likely pathway is shown in eq. 4. In the first step of this

$$\text{XIII} \xrightarrow[(-\text{HOTs})]{\text{Me}_3\text{SnLi}} \text{X} \xrightarrow{\text{Me}_3\text{SnLi}} \textit{cis}\text{-XI} \qquad (4)$$

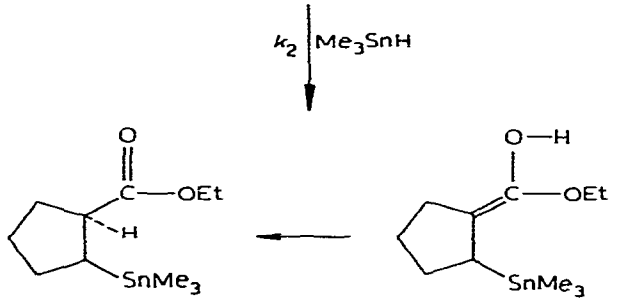
scheme the trimethylstannyllithium functions as a base in promoting E_2 elimination of toluenesulfonic acid, and in the second step in functions in a Michael addition to the unsaturated ester *. Thus, if the Michael reaction is stereoselective *trans* as might be expected on thermodynamic grounds rather than stereoselective *cis*, the course of the reaction can be understood. *Trans* addition to form the *cis* adduct would be the result of preferential transfer of a proton to the intermediate enolate anion from the side opposite to the bulky trimethylstannyl group in the product-determining step.

The hydrostannation of 2-hydroxymethylcyclopentene (VII), (a minor product in the reduction of ester III with lithium aluminum hydride, and also obtained in 40% yield by similar reduction of the unsaturated ester X) [11] could not be carried out successfully using either photoinitiation or initiation by azobisisobutyronitrile. The low reactivity of VII in hydrostannation, in contrast with the higher reactivity and stereospecificity in the hydrostannation of X merit comment. It has been shown that the initial step in the hydrostannation of mono olefins is reversible [12], as shown in eq. 5 with X for illustration. The overall

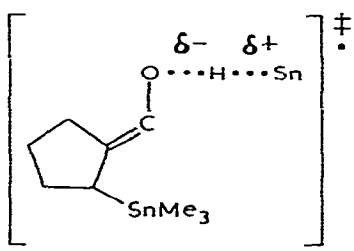
* Current work by G. Lein indicates that such addition does indeed occur, and that *cis*-XI is formed along with small amounts of another tin-containing product, which may be *trans*-XI.



(XIV)



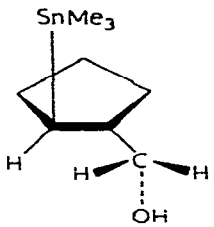
(XI) (XV)



(XVI)

rate of addition will depend on the relative magnitudes of the rate constants shown and on the concentration of trimethylstannane. The resonance stabilized intermediate radical XIV formed from X can abstract hydrogen from the tin atom either at the ring carbon to form XI directly, or at the oxygen atom to form the enol of the ester, XV which then ketonizes to form XI. The latter possibility is particularly attractive for two reasons. First, the oxygen atom is relatively electron deficient compared with an ordinary ester carbonyl, and a transition state such as XVI with the kind of charge separation indicated would be stabilized relative to one involving hydrogen transfer to carbon. (Both reactions would have about the same energetics because the O—H and C—H bonds have about the same dissociation energies, ca. 102 kcal, in simple systems.) The resonance stabilization and enhanced reactivity of XIV would account for the greater rate of hydrostannation of X as compared to VII. Furthermore, in the conversion of XV to XI proton transfer to the ring carbon would occur faster from the side opposite to the trimethylstannyl group, thus accounting for the formation of only the *cis* isomer of XI. The intermediate radical formed

from VII has a radical center bearing the non-planar hydroxymethyl group vicinal to the bulky trimethylstannyl group (dihedral angle ca. 55°). The result is that the more stable conformations of the hydroxymethyl group as in XVII are such as to raise the activation energy of hydrogen atom transfer to the radical center to such a degree that the product-forming step cannot compete effectively with loss of the trimethylstannyl radical: attack from above the plane of the ring is obstructed by the Me_3Sn group, and from below that plane by the hydroxyl group.



(XVII)

Experimental

Proton NMR spectra were obtained with a Varian Associates A60-A or HA-100 spectrometers. Carbon-13 spectra were obtained with the latter instrument interfaced with a Digilab FTS-3 pulse and data system. Spectra were 8K or 16K Fourier transformed and recorded at bandwidths of 2000 Hz. Coupling constants involving tin are given for ^{119}Sn or for the center of unresolved satellites when the coupling constants were small. Infrared (IR) spectra were recorded with a Beckman IR-10 instrument.

All operations involving trimethylstannylalkali derivatives and trimethylstannane were conducted under a nitrogen or argon atmosphere.

cis- and trans-2-Carbethoxycyclopentanols (IV)

To 63 g (0.4 mol) of 2-carbethoxycyclopentanone in 200 ml anhydrous ethyl ether at 0°C was added 3.8 g (0.1 mol) of sodium borohydride and the mixture stirred overnight. It was treated with 300 ml of water and stirred until the precipitate which had formed dissolved. The layers were separated, the aqueous layer extracted with additional ether and the combined ether layers dried over anhydrous sodium sulfate. The ether was stripped off and the hydroxy-esters distilled; b.p. 57—75°C/0.23 Torr; 52 g (80%); and then fractionated through a spinning band column (Nester Faust) providing the *cis* isomer, b.p. 44—48°C/0.01—0.02 Torr; lit., b.p. 54—56.5°C/0.1—0.2 Torr [14]. When 90% of this isomer had been distilled the residue was distilled through a Vigreux column in order to minimize polymerization providing a major cut of the *trans* isomer, b.p. 57—60°C/0.28 Torr; lit., b.p. 57.5—60°C/0.1—0.2 Torr [14]. The isomers were shown to be pure by GLPC analysis on a 6 ft × 1/8 in column of 10% Carbowax 20M on Chromosorb W. At 200°C the retention time of the *cis* isomer was 1.6 min; that of the *trans* isomer was 2.4 min.

2-Methylenecyclopentanol (V), 2-hydroxymethylcyclopentanol (VI) and 1-hydroxymethylcyclopentene (VII) by reduction of 2-carbethoxycyclopentanone (III)

Using the procedure of Dreiding and Hartman [3] in a typical reduction 30 g (0.20 mol) of 2-carbethoxycyclopentanone in 500 ml of ether was reduced with 15.2 g (0.40 mol) of lithium aluminum hydride. The yield of 2-methylenecyclopentanol was 30%; b.p. 68°C/20 Torr; lit. b.p. 85—86°C/68 Torr; IR (neat; cm^{-1}) 3350s, 3095w, 1665m, 1100s, 890s; NMR (CCl_4 δ ppm): 5.13(m) 1H, 4.97(m), 1H; 4.37(broad s), 1H; 3.90 (broad), 1H; 2.33(m), 2H; 1.70(m), 4H. About 40% of 2-hydroxymethylcyclopentanol b.p. 87—90°C/1.0 Torr; lit. 127.5°C/9 Torr was the second product. The third was about 10% of 1-hydroxymethylcyclopentene b.p. 60—62°C/10 Torr; lit. 95—96°C/68 Torr.

2-Carbethoxycyclopentyltrimethylstannane (XI)

1-Carbethoxycyclopentene (40 g, 0.30 mol) was hydrostannated with 4.3 g (0.26 mol, 10% deficiency) of trimethylstannane at 60°C using azobisisobutyronitrile as a catalyst. This gave 64 g of the adduct (70% yield), b.p. 71—74°C/0.5 Torr; NMR (CCl_4, δ ppm) 4.1 (q, 2/H J 7 Hz), 1.5(m, 7H), 1.3(t, 3H, J 7 Hz), and 0.0 (s, 9H, $J(^{119}Sn—C—H)$ 53 Hz). IR (neat) 1730s, 1190s, 1100w, 1025w, and 760s cm^{-1}. Analysis: Found: C, 43.29; H, 7.13. $C_{11}H_{22}O_2Sn$ calcd.: C, 43.31; H, 7.28%.

2-Carbethoxycyclopentylmethyldibromostannane (XII)

To 1 g (3.3 mmol) of XI in 3 ml of CCl_4 was added 1.06 g (6.6 mmol) of bromine in 7 ml of CCl_4. When the addition was complete some of the orange color of the bromine remained but disappeared on warming. Removal of most of the solvent and examination of the IR spectrum revealed a carbonyl band at 1730 cm^{-1} (free C=O) and another of about twice its intensity at 1665 cm^{-1} (coordinated C=O); NMR (CCl_4, δ ppm) 0.67 $^2J(^{119}Sn—H)$, 66 Hz, 3 H.

2-Hydroxymethylcyclopentyltrimethylstannane (I—OH)

2-Carbethoxycyclopentyltrimethylstannane (XI) (44.6 g, 145 mmol) was reduced with 5.6 g (145 mmol) of lithium aluminum hydride according to the general procedure. Distillation of the residual liquid gave 29.6 g (77% yield) of I—OH b.p. 80—90°C/0.05—1.10 Torr. NMR (CCl_4, δ ppm) 3.3 (d, 1H, J 7 Hz), 2.0 (broad, s, 1H), 1.9—1.0 (broad m, 8H) and 0.05 (s, 9H, $J(^{119}Sn—C—H)$ 52 Hz). Analysis: Found: C, 41.26; H, 7.68. $C_9H_{20}OSn$ calcd.: C, 41.13; H, 7.61%.

2-Trimethylstannylmethylcyclopentanol (II—OH), cis/trans mixture

2-Methylenecyclopentanol (9.8 g, 0.1 mol) was hydrostannated in 100 ml cyclohexane with 18.2 g (0.11 mol, 10% excess) trimethylstannane according to Procedure B. The amount of adduct obtained was 12.7 g (50% yield); b.p. 110°C/0.1 Torr. NMR (CCl_4, δ ppm) 3.9 (broad, 1H), 2.0—1.5 (m, 8H), 1.0 (d, J 7 Hz), and 0.0 (s, 9H, $J(^{119}Sn—C—H)$ 53 Hz). IR (neat) 3400s, 1190m, 1100m, 1050m, 1000m and 760s cm^{-1}. Analysis: Found: C, 41.33; H, 7.76. $C_9H_{20}OSn$ calcd.: C, 41.13; H, 7.61%.

The alcohol was converted to the *para*-nitrobenzoate derivative according to

the method already described. Better than 90% yields were obtained. The ester was recrystallized from 1 : 1 benzene/hexane; m.p. after one recrystallization 59—62°C; after four recrystallizations, m.p. 68—70°C. CMR data are given in Table 1. Analysis: Found: C, 46.31; H, 5.52. $C_{16}H_{23}NO_4Sn$ calcd.: C, 46.63; H, 5.64%.

cis-2-Hydroxymethylcyclopentanol (cis-VI)

cis-2-Carbethoxycyclopentanol (*cis*-IV) (28.5 g, 0.18 mol) was reduced in 400 ml ether with 7.6 g (0.18 mol plus 10%) lithium aluminum hydride in ethyl ether [13]. Distillation of the residual liquid after removal of solvent gave 15 g (71%), b.p. 70—74°C/0.4 Torr. IR (neat) 3400—3250s, 1450m, 1350m, 1150w, 1050—1000s, and 930m cm^{-1}.

trans-2-Hydroxymethylcyclopentanol (trans-VI)

trans-2-Carbethoxycyclopentanol (21.2 g, 0.13 mol) was reduced in 300 ml ether with 6.4 g (0.13 mol plus 25%) lithium aluminum hydride. Distillation gave 9.3 g (60%) of the *trans* diol; b.p. 85—88°C/0.4 Torr. IR (neat) 3400—3250s, 1450m, 1350m, 1150w, 1050—1000s, and 960m cm^{-1} (shoulder on band at 1000 cm^{-1}).

cis-2-Trimethylstannylmethylcyclopentanol (cis-II—OH)

cis-2-Hydroxymethylcyclopentanol (18 g, 155 mmol) was converted to 2-tosyloxymethylcyclopentanol by treatment with *p*-toluenesulfonyl chloride in pyridine at 0°C. 30 g (71%) were obtained. This was treated as a 1 *M* solution in tetrahydrofuran with a 0.5*M* tetrahydrofuran solution of trimethylstannyllithium prepared from 46 g (230 mmol) chlorotrimethylstannane and 3.5 g (460 mmol plus 10% excess) lithium.

Distillation of the extraction residue gave 7.1 g (25%) of *cis*-2-trimethylstannylmethylcyclopentanol; b.p. 74—80°C (0.15—0.30 Torr). The IR and NMR spectra were the same as observed for the mixture of isomers; the *cis* isomer was identified by its proton decoupled ^{13}CMR spectrum: (CHOH) 76 ppm (*J*-(^{119}Sn—C—C—C) 43 Hz). The IR spectrum showed weak bands at 1160, 985, and 860 cm^{-1} which were absent in the spectrum of the *trans* isomer.

trans-2-Trimethylstannylmethylcyclopentanol (trans-II—OH)

trans-2-Hydroxymethylcyclopentanol (15.6 g, 134 mmol) was converted to *trans*-2-tosyloxymethylcyclopentanol according to the previously described procedure for the *cis* isomer. 29 g (80%) were obtained. Unlike the *cis* isomer, the *trans* compound did not crystallize on storing in the refrigerator. This was treated as a 1.0 *M* solution in tetrahydrofuran with a 0.5 *M* tetrahydrofuran solution of trimethylstannyllithium prepared from 47 g (235 mmol) of chlorotrimethylstannane and 3.6 g (470 mmol plus 10% excess) of lithium metal.

The procedure and work-up was as before and gave 8.5 g (30%) of *trans*-2-trimethylstannylmethylcyclopentanol b.p. 80—85°C/0.2—0.3 Torr. Its ^{13}CMR spectrum identified it as the *trans*-isomer (Table 1). The IR spectrum showed a weak band at 910 cm^{-1} which was absent in the spectrum of the *cis* isomer.

Acknowledgement

Acknowledgement is made to the donors of The Petroleum Research Fund, administered by the American Chemical Society, for support of this research, and to K.R. Wursthorn for the CMR data.

References

1 H.G. Kuivila and N.M. Scarpa, J. Amer. Chem. Soc., 92 (1970) 699.
2 H.O. House, Modern Synthetic Reactions, W.A. Benjamin, Menlo Park, CA, 2nd. ed., 1972, p. 47.
3 A.S. Dreiding and J.A. Hartman, J. Amer. Chem. Soc., 75 (1953) 939.
4 M. Christl, H.J. Reich and J.D. Roberts, J. Amer. Chem. Soc., 93 (1971) 3463.
5 R.L. Kronenthal and E.I. Becker, J. Amer. Chem. Soc., 79 (1967) 1095.
6 G.C. Levy and G.L. Nelson, Carbon-13 Nuclear Magnetic Resonance for Organic Chemists, John Wiley & Sons, Inc., New York, 1972, p. 45.
7 H.G. Kuivila, J.L. Considine, R.J. Mynott and R.H. Sarma, J. Organometal. Chem., 55 (1973) C11; 111 (1976) 179.
8 S. Matsuda and N. Nomura, J. Organometal. Chem., 25 (1970) 101; 71 (1970) 1526. For a recent review see I. Omae, Rev. Silicon, Germanium, Tin Lead Compds., 1 (1972) 59.
9 H.G. Kuivila, J.E. Dixon, P.L. Maxfield, N.M. Scarpa, T.M. Topka, K-H. Tsai and K.R. Wursthorn, J. Organometal. Chem., 86 (1975) 89.
10 J. Lorberth and H. Vahrenkamp, J. Organometal. Chem., 11 (1968) 111.
11 R.S. Davidson, W.H.H. Gunther, S.M. Waddington-Feather and B. Lythgoe, J. Chem. Soc., (1964) 4907.
12 H.G. Kuivila and R. Sommer, J. Amer. Chem. Soc., 89 (1967) 5616.
13 J.P. Vila and J. Costella, J. Amer. Chem. Soc., 74 (1952) 2899.
14 L.F. Fieser and M. Fieser, Reagents for Organic Synthesis, Vol. I, J. Wiley & Sons, New York, 1967, p. 594.