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PREPARATION, RESOLUTION AND DYNAMIC STEREOCHEMISTRY OF THE 7H-7-METHYL-7-SILABENZ[*d,e*]ANTHRACENE SYSTEM

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Summary

(±)-7H-7-Methyl-7-silabenz[*d,e*]anthracene was prepared by pyrolysis of α-naphthylphenylmethylsilane, and was subsequently resolved using the menthoxy diastereomer.

With one exception, the dynamic stereochemistry of this system is similar to that of analogous acyclic α-naphthylphenylmethylsilyl compounds. These stereochemical results are discussed on the basis of steric and angle strain effects at silicon.

Recently, there has been much interest in steric and angle strain effects in silicon chemistry and some surprising results have been obtained in angle strained [11, 12] and non-angle strained [15] compounds. This paper reports on the properties of a silicon compound containing a silicon that is stereochemically in a fairly rigid ring, but not angle strained.

Results

Pyrolysis of (±)-α-naphthylphenylmethylsilane (II-H) in a hot tube at 595°, and vacuum distillation of the pyrolyzate gives a 33% yield of a cyclized dehydrogenated silicon compound; such aromatic dehydrogenations are known reactions [1]. Cyclization could have occurred between an *ortho* phenyl position and either the α or β position of the naphthyl group (eqn. 1), but the infrared spectrum of the pyrolyzate was of no help in deciding between I and III. However, the product's ultraviolet spectrum (Fig. 1) is almost identical to that of 7H-benz-

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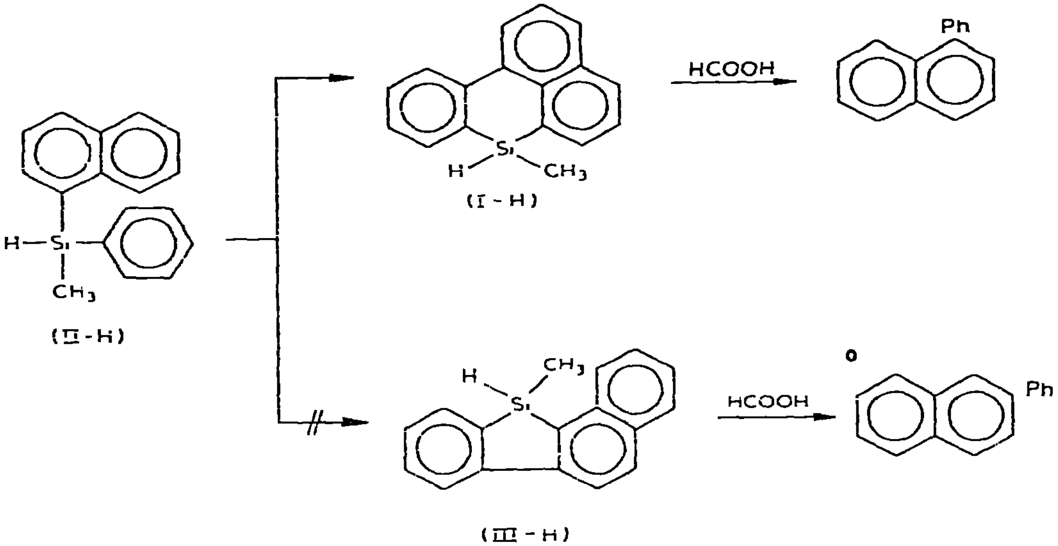


(1)

[*d*,*e*]anthracene, and unlike that of 9-methylbenz[*a*]fluorene [2]. Desilylation with refluxing formic acid [3] (eqn. 1) yielding 1-phenylnaphthalene (III would give 2-phenylnaphthalene) confirms the structure of the pyrolyzate as 7H-7-methyl-7-silabenz[*d*,*e*]anthracene (I-H). (This ring system has recently [4] been prepared by an analogous pyrolytic dehydrochlorination, but there was no structure proof given. The authors said that attempted dehydrogenation of α-naphthylphenyldichlorosilane at 690° did not give any of the desired product.)

Partial resolution via fractional crystallization from pentane of the (—)-menthol diastereomers, subsequent reduction by $LiAlH_4$ (eqns. 2 and 3

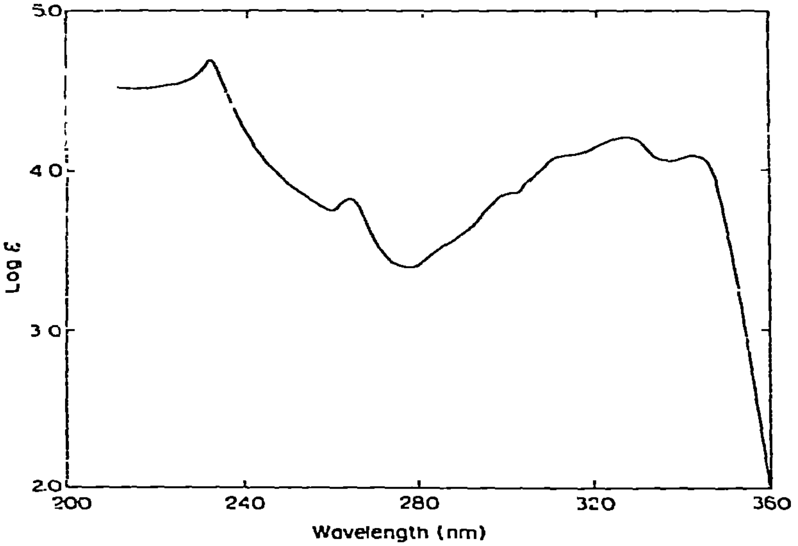


Fig. 1. Ultraviolet spectrum of (±)-7H-7-methyl-7-silabenz[*d*,*e*]anthracene in cyclohexane (conc.: 360-260 nm, 5 × $10^{-4}M$; < 260 nm, 1.7 × $10^{-4}M$).

where the rotations of crude I-H are given), and subsequent recrystallization gave pure (+)- or (—)-I-H, $[\alpha]_D$ 31.0° (*c* 0.76, pentane). By procedures similar

$$\begin{array}{lll}(+)\text{-I-}(-)\text{-OMen} + \text{LiAlH}_4 \rightarrow & (-)\text{-I-H} & \\ \quad [\alpha]_D + 94.5^\circ & [\alpha]_D -26.7^\circ & (2)\end{array}$$

$$\begin{array}{lll}(-)\text{-I-}(-)\text{-OMen} + \text{LiAlH}_4 \rightarrow & (+)\text{-I-H} & \\ \quad [\alpha]_D -96.2^\circ & [\alpha]_D + 12.7^\circ & (3)\end{array}$$

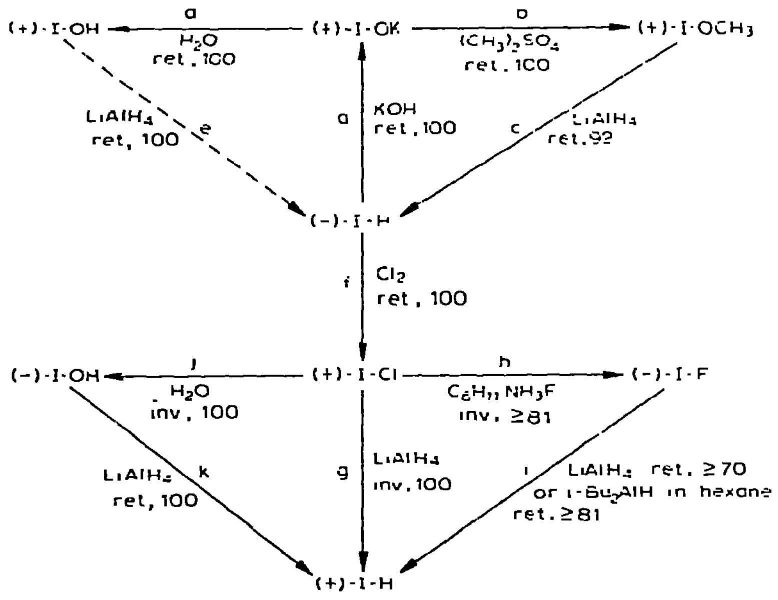
to those used for system II [5], the reactions shown in Scheme 1 were run and their stereochemical assignments are discussed below.

Discussion

Of the reactions in Scheme 1, the stereochemistry is known for two, b and d, both of which, because the silicon center is not affected, are retention. The cycles abc and ade have an even number (0 or 2) of inversions, while cycles fg, fjk, and fhi have an odd number (1 or 3) of inversions. With the exception of fhi, these conditions are simply met by assigning retention or inversion by analogy with system II [5]. To assign the opposite stereochemistries would mean reactions f, a, c, and h must be inversions, which has never been observed for these types of reactions.

SCHEME 1

The numbers below the reagents used indicate the stereoselectivity, assuming that optically pure starting materials were used. A stereoselectivity of, say, 80%, means that the product was 60% optically pure, and 40% racemic.



Reaction sequence fhi must involve either 1 or 3 inversions; f has already been assigned as a retention, so either h or i must be an inversion. Since reactions j and g are both nucleophillic displacements of chloride and proceed with inversion of configuration, the analogous displacement by fluoride ion is probably also an inversion; thus reaction i is a retention (a change from system II). The retention stereochemistry of i is confirmed by the reduction with i-Bu_2AlH in hexane, a reaction that in all previous cases has proceeded with retention of configuration [6]. The probable reason for this stereochemistry crossover in the $LiAlH_4$ reduction is that fluoride is displaced with retention or inversion at asymmetric silicon, depending upon the particular reaction [7, 8]. For instance, while II-F is reduced by $LiAlH_4$ with inversion, it is displaced by II-OK with retention of configuration at asymmetric silicon [5, 8]. Furthermore, reduction of II-F by i-Bu_2AlH in hexane proceeds with retention, the same result as with I-F [6].

Chauviere et al. have reported [9] that when compared to acyclic systems the 2-silatetrahydronaphthalene ring system appears to favor retention mechanisms for reactions with organolithiums and Grignard reagents (other factors must be considered in these reactions); but the $LiAlH_4$ reduction of 2-(α-naphthyl)-2-silatetrahydronaphthalene gave racemic silane [10].

In Table 1 are all of the compounds of the (+)-I-H configuration together with their rotations. No deliberate experimentation on variation of rotation with solvent was undertaken, but I-OMe changes sign of rotation on changing from pentane to benzene solvent.

The significance of these results is related to three properties of the 7H-7-methyl-7-silabenz[*d,e*]anthracene system that seem apparent when molecular models are studied: the molecule (except for the methyl group) is almost planar and relatively rigid; the "front face" of the silicon is relatively open, compared to acyclic silanes such as II; there is little angle strain at the silicon. The recently reported results on the 1-phenyl-1-silaacenaphthalene system (IV) [11] and silacyclobutanes (V) [12] are in direct contrast to those obtained here — in those compounds (almost) all of the reactions (including nucleophilic chloride displacement) proceed via retention. Although the front face of the silicon in IV and V is somewhat more accessible than that of I, it is difficult to rationalize

TABLE 1

COMPOUNDS HAVING THE (+)-I-H CONFIGURATION

Compound	Rotation (°)
(—)-I-(—)-OMen[a]	—235[b] (*c* 0.88, pentane)
(—)-I-Cl	—86.9 (*c* 0.72, benzene)
(—)-I-F	—70.6 (*c* 1.4, benzene)
(—)-I-OK	—57 (*c* 1.5, *p*-xylene)
(—)-I-OCH_3	—15.6 (*c* 0.56, benzene)
(—)-I-OH	—14.6 (*c* 1.0, $CHCl_3$)
(+)-I-H	+31.0 (*c* 0.76, pentane)
(+)-I-(—)-OMen[a, c]	+117[b] (*c* 2.0, pentane)

[a] OMen is menthoxy. [b] Calculated from the reduction by $LiAlH_4$, assuming it is 100% retention. [c] This compound has the (—)-I-H configuration.

the large stereochemical difference on this basis. The large amount of angle strain must favor a retention over inversion. These differences can be explained by transition states proposed by Sommer for the S_Ni-*Si* (retention) and S_N2-*Si* (inversion) mechanisms [13].

Using the principle of least motion of the non-reacting groups [13], compounds with silicon bond angles $\leqslant$ 109° (but close to 90°) such as IV or V are already "partially in the S_Ni-*Si* transition state" and require little movement of the non-reacting groups. On the other hand, it would be very difficult for compounds such as IV or V to assume an S_N2-*Si* transition state, since it is probable that the more electronegative groups are favored in the apical position [14]. Thus for compounds IV, V and 1-silabicyclo[2.2.1]heptane the S_Ni-*Si* activation energy is relatively low, while the S_N2-*Si* activation energy is relatively high, the exact opposite result as obtained in non-angle strained systems such as I and II.

The hypotheses regarding non-angle strained silicon such as in I and II are confirmed by the unique properties reported [15] for the 1,3,5,7-tetrasilaadamantane system. This system, which is quite rigid, has a relatively open front face at silicon, and little or no angle strain, also cannot undergo an S_N2-*Si* inversion mechanism. Its structure is such that it is impossible for the R—Si—R bond angles to decrease to 90°, so an S_Ni-*Si* retention mechanism is unlikely. The fact that compounds VI are, in many cases, extraordinarily unreactive gives confidence that the above hypotheses are correct.

Experimental

Preparation of 7H-methyl-7-silabenz[d,e]anthracene (I-H)

The reaction zone was a quartz tube, 2.2 cm ID × 30 cm long heated to 595 ± 10° by a tube oven, and the tube was flushed with nitrogen (flow rate was about 1 l/h). α-NpPhMeSiH was vaporized slowly and swept through (residence time ~ 9 min); about 12 g/day was reacted.

A typical run is as follows. Over the course of three days, 32 g of (±)-α-NpPhMeSiH was passed through the tube. The resulting pyrolyzate (30.3 g) was a mobile dark red liquid, which when heated under vacuum gave off some solid material. The liquid was distilled on an 18 × 1/4″ spinning band column. The first fraction, 6.0 g, b.p. 135-144.5°/0.4 torr was α-NpPhMeSiH and the second fraction, 1.2 g, b.p. 147-157.5°/0.4 torr, was a mixture of α-NpPhMeSiH and I-H. The last two fractions, b.p. 160-162.5°/0.35 torr were I-H. The recovery of starting material was 19%, and the yield of I-H was 33%. Crystallization from 15 ml of pentane yielded 8.2 g of (±)-I-H.

From a previous run, (±)-I-H, m.p. 60.5-65.5°, was obtained by chromatography (75% CCl_4—25% PhH, v/v) over silica gel. The sample was subjected to UV, IR and mass spectral analysis (Varian M66 instrument). Peaks in the mass spectrum of relative abundance > 10 were: 247, 19; 246, 100.0; 245, 49; 232, 20; 231, 86; 229, 14; 205, 12; 193, 11; 192, 22.

Analysis found: C, 83.12; H, 5.81%; mol. wt., 246.0791 (vs. $C_5H_6{}^{35}Cl^{37}Cl$). $C_{17}H_{14}Si$ calcd.: C, 82.87; H, 5.73%; mol. wt. 246.0864.

Preparation of (±)-I-(—)-OMen

Into a 3-necked round bottomed flask was placed 5.28 g of (—)-menthol.

The flask was equipped with a magnetic stirrer and addition funnel, and under nitrogen. To the addition funnel was added 7.95 g of (±)-I-H, and then dry toluene was added to both the flask (10 ml) and the addition funnel (15 ml) and sparged with N_2. A small piece of potassium was added to the flask, and was dissolved while heating and stirring. While the flask contents were being refluxed, the silane solution was added over a period of 10 min; a gas was given off (H_2?) as the silicon compound was added. About 30 sec after the addition was complete gas evolution ceased, and the dark tan solution was refluxed an additional 5 min and then allowed to cool.

The toluene solution was washed 3 times with water (neutrality), the green organic layer was dried over anhydrous Na_2SO_4 and filtered. Solvent was removed first on a steam bath and then under vacuum on a rotary evaporator. The yield of viscous oil was 12.6 g (95%). An infrared spectrum indicated there were no large amounts of impurities present.

Analyses were performed after resolution. Found for (+)-I-(−)-OMen (m.p. 95-101°): C, 80.86; H, 8.16. Found for (−)-I-(−)-OMen (m.p. 62.2-70.5°); C, 81.19; H, 8.18. $C_{27}H_{32}OSi$ calcd.: C, 80.94; H, 8.05%.

Resolution of the menthoxydiastereomers

This procedure was not optimized, but a typical experiment is given below.

The viscous oil from the previous experiment was dissolved in 18 ml of pentane and placed in a refrigerator (2°). A solid (4.9 g) was recovered and recrystallized slowly from pentane. The second crystallization yielded 2.0 g, and the mother liquor another 0.8 g of solid. This 2.8 g was recrystallized from pentane (lowest temperature about −15°), yielding 2.5 g of (+)-I-(−)-OMen, $[\alpha]_D$ + 94.5° (*c* 2.0, pentane), m.p. 95-101°.

The filtrate from the first crystallization, which contained the lower melting diastereomer, was crystallized from the pentane (−15°) yielding about 1.8 g of crystals. These were recrystallized twice to give 0.5 g of (−)-I-(−)-OMen, $[\alpha]_D$ −96.2° (*c* 0.88, pentane), m.p. 62.2-70.5°.

Reduction of (+)-I-(−)-OMen

To a 50 ml 3-necked round bottomed flask equipped with a thermometer, condenser, and magnetic stirrer were added 0.5 g of $LiAlH_4$, 10 ml of n-butyl ether, and 5 ml of ethyl ether. To this was added a solution of 3.1 g of (+)-I-(−)-OMen, $[\alpha]_D$ + 94.5° (*c* 2.0, pentane), in 10 ml of ethyl ether. The mixture was stirred and heated, and the ethyl ether distilled until the pot temperature reached 82°.

After approximately 16 h at 82-93° the mixture was cooled and the excess $LiAlH_4$ was destroyed with acetone. The mixture was poured onto an ice/10 ml conc. HCl mixture, shaken, washed four times with water (neutral) and dried over anhydrous Na_2SO_4. After filtration the solvent was removed, first on a steam bath and then on a rotary evaporator at 1 torr. The material was placed on an 18 × 1/2" silica gel column and eluted with 50% PhH/50% pentane (v/v) solvent. The first fraction was (−)-I-H contaminated with a small amount of menthol. After removing the menthol under vacuum, 1.7 g (89%) of product, $[\alpha]_D$−26.7° (*c* 1.5, pentane) was obtained. After crystallization from 20 ml of hexane, 1.0 g of crystals, m.p. 91.6-94.1°, $[\alpha]_D$−31.0° (*c* 0.76, pentane) was obtained.

Apparently due to solvent adhering to the crystals, it was necessary to sublime a small sample for analysis. An infrared spectrum had an absorption at 2118 cm^{-1} due to Si—H.

Analysis found: C, 82.71; H, 6.02; Si, 11.40. $C_{17}H_{14}Si$ calcd.: C, 82.87; H, 5.73; Si, 11.40%.

Reduction of (—)-I-(—)-OMen with $LiAlH_4$

By a procedure similar to that in the preceding experiment, (—)-I-(—)-OMen, $[\alpha]_D$ —96.2° (*c* 0.88, pentane), was reduced to (+)-I-H, $[\alpha]_D$ +12.7° (*c* 0.36, pentane).

Preparation of (+)-I-Cl

A solution of 0.72 g of (—)-I-H, $[\alpha]_D$ —31.0° (*c* 0.76, pentane), in 10 ml of CCl_4 in a 25 ml filter flask was sparged with N_2, and then Cl_2 was bubbled in until a faint green color appeared (4 min). During addition the solution warmed, and was cooled in a water bath. The solvent was removed under vacuum and 0.82 g (100% yield) of a solid remained. The material had a rotation of $[\alpha]_D$ +86.9° (*c* 0.72, benzene). After removing 0.18 g, the solid was recrystallized from 15 ml of hot heptane to yield 0.50 g of thick needle-like crystals, m.p. 126.0-133.0°, $[\alpha]_D$ +85.5° (*c* 0.72, benzene). The infrared spectrum had a strong absorption at 535 cm^{-1} (Si—Cl).

Analysis found: C, 72.81; H, 4.82; Cl, 12.61. $C_{17}H_{13}ClSi$ calcd.: C, 72.70; H, 4.66; Cl, 12.63%.

Preparation of (—)-I-F

To a solution of 0.40 g of (+)-I-Cl, $[\alpha]_D$ +85.5° (*c* 0.72, benzene), in 25 ml of freshly purified $CHCl_3$ was added 0.17 g of cyclohexylammonium fluoride, which dissolved immediately. After 3 min 200 ml of pentane was added and a white precipitate formed. After 45 min at —15°, the mixture was filtered, and the solvent was removed by an aspirator and then a vacuum pump. The residual oil gradually solidified (0.34 g, 90%), and the solid had a rotation of $[\alpha]_D$ —69.3° (*c* 1.4, benzene). After recrystallization from 10 ml of hexane the (—)-I-F had a m.p. of 98.2-106.3° and $[\alpha]_D$ —70.6° (0.81, benzene). The infrared spectrum had a strong Si—F absorption at 857 cm^{-1}.

Analysis found: C, 77.47; H, 4.99; F, 7.07. $C_{17}H_{13}FSi$ calcd.: C, 77.23; H, 4.96; F, 7.19%.

Preparation of (—)-I-OCH_3

To a flask containing 0.36 g of (+)-I-H, $[\alpha]_D$ +29.5° (*c* 0.66, pentane) in 30 ml of *p*-xylene was added about 0.5 g of powdered KOH. The flask was placed on a steam bath for 1 h, during which time a gas (H_2?) was evolved. A rotation taken of the solution indicated $[\alpha]_D$ —54.2° (*c* 1.5, *p*-xylene), assuming all of the silane had been converted to the silanolate.

Approximately 25 ml of the above solution was added to a flask containing 2 ml of methyl sulfate and 10 ml of *p*-xylene. While shaking vigorously for 3 min a precipitate formed, and the mixture was poured into a separatory funnel containing 100 ml of 5% K_2CO_3 solution and 100 ml of pentane. After washing the organic layer three times with 5% K_2CO_3 solution, it was dried over anhydrous

K_2CO_3. After filtration the solvent was stripped under vacuum and the residual oil solidified. The material, 0.31 g (90%), had $[\alpha]_D$ +5.1° (*c* 1.2, pentane).

After recrystallization from hexane, 0.19 g m.p. 75.7-84.5°, $[\alpha]_D$ −15.6° (*c* 0.56, benzene), of (−)-I-OCH_3 was obtained. A strong absorption band at 1090 cm^{-1} characteristic of Si—OCH_3 was present in the infrared spectrum.

Preparation of (−)-I-OH

A solution of 0.31 g of (+)-I-Cl, $[\alpha]_D$ +81.6° (*c* 1.2, benzene), in 50 ml of ether was added to a separatory funnel containing 50 ml of water and the mixture was shaken for 1 min. After washing the organic layer twice with water, it was dried over anhydrous K_2CO_3, filtered, the solvent stripped under vacuum, and a rotation was taken on the solid remaining, $[\alpha]_D$ −13.7° (*c* 1.0, $CHCl_3$). The yield was 0.26 g (90%) of (−)-I-OH, m.p. 141.0-163.8°, and the compound had a strong OH absorption band at 3350 cm^{-1} (KBr pellet). Some disiloxane (1050 cm^{-1}) was also present.

The silanol was also prepared by hydrolysis of the silanolate. An approximately 0.049 *M* *p*-xylene solution (4 ml) of (−)-I-OK, $[\alpha]_D$ −54.2° (*c* 1.5, *p*-xylene), was added to a separatory funnel containing 25 ml of water and 25 ml of ether and the mixture was shaken for 1 min. After washing twice more with water and drying over anhydrous Na_2SO_4, a rotation was taken of the 0.032 g (63%) of (−)-I-OH obtained, $[\alpha]_D$ −17° (*c* 0.22, $CHCl_3$).

$LiAlH_4$ reductions

I-Cl. A solution of 0.18 g of (+)-I-Cl, $[\alpha]_D$ +86.9° (*c* 0.72, benzene), in 25 ml of benzene was added to a solution of 0.3 g of $LiAlH_4$ in 25 ml of ethyl ether. After 15 min the excess hydride was decomposed with acetone and the mixture was poured onto an ice/8 ml conc. HCl mixture. After the usual workup 0.15 g (96%) of (+)-I-H was obtained, $[\alpha]_D$ +30.4° (*c* 0.54, pentane).

I-F. To a solution of 0.16 g of (−)-I-F, $[\alpha]_D$ −70.6° (*c* 0.81, benzene), in 10 ml of ethyl ether was added 0.2 g of $LiAlH_4$. After refluxing 65 min and standing an additional 2 h, the excess hydride was decomposed with acetone and the reaction worked up in the normal manner. The product was 0.11 g (75%) of (+)-I-H, $[\alpha]_D$ +12.2° (*c* 0.70, pentane).

I-OCH_3. A mixture of 0.14 g (−)-I-OCH_3, $[\alpha]_D$ −15.6° (*c* 0.56, benzene), 10 ml of ethyl ether, and 0.2 g of $LiAlH_4$ was allowed to stand overnight (about 17 h). After working up in the usual way, 0.10 g (84%) of (+)-I-H, $[\alpha]_D$ +26.3° (*c* 0.40, pentane), was obtained.

I-OH. A mixture of 0.10 g of (−)-I-OH, $[\alpha]_D$ −13.1° (*c* 0.40, $CHCl_3$), 5 ml of ethyl ether, 10 ml of n-butyl ether, and 0.2 g of $LiAlH_4$ was heated and the ethyl ether was allowed to escape until the pot temperature reached 90°. After refluxing at 90° for 30 min, the remaining ethyl ether was distilled, and the mixture was refluxed at 140° for 1 h. Subsequent to workup 0.079 g (86%) of (+)-I-H, $[\alpha]_D$ +32.5° (*c* 0.32, pentane), was obtained.

Reduction of (−)-I-F with i-Bu_2AlH

To a solution of 0.19 g of (−)-I-F, $[\alpha]_D$ −34.7° (*c* 0.52, benzene), in 4 ml of hexane was added 0.30 ml (1.6 mmol) of i-Bu_2AlH. After 23 h the solution was hydrolyzed in ice/3 ml conc. HCl, washed three times with water, and dried over anhydrous Na_2SO_4. After stripping the solvent under vacuum, 0.14 g (62%) of (+)-I-H was obtained, $[\alpha]_D$ +9.3° (c 0.87, pentane).

When the reduction was attempted in ethyl ether solvent, the rotation quickly (less than ca. 2 min) fell to zero and remained there.

Reaction of (±)-I-H with formic acid

A mixture of 0.14 g of (±)-I-H and 4 ml of 97% formic acid was refluxed under N_2 for four days. The mixture was then extracted with water and pentane, and then the organic layer was washed until neutral, dried over anhydrous Na_2SO_4, and filtered.

After stripping the solvent, the residue was dissolved in hexane and analyzed by GLPC using a 5 foot 20% SE-30 on Chromosorb P column, with He as the carrier gas. There was only one major component of b.p. > 225°, and its retention time coincided with that of the authentic sample (K & K Lab.) of 1-phenylnaphthalene. The unknown material was isolated and its infrared and mass spectra were identical to those of the known compound. The relative intensities of 15 most abundant ions in the mass spectrum of the unknown are given below (mass, abundance): 205, 16.5; 204 (*P*), 100.0; 203, 70.4; 202, 49.6; 201, 9.2; 200, 8.2; 102, 7.3; 101.5, 7.4; 101, 27.0; 100, 7.6; 89, 8.7; 88, 8.7; 63, 6.5; 51, 8.3; 50, 7.9.

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