Studies in the cyclopropanaphthalene series:[1] chemistry of the 1-trimethylsilyl-1*H*-cyclopropa[*b*]naphthalenyl anion

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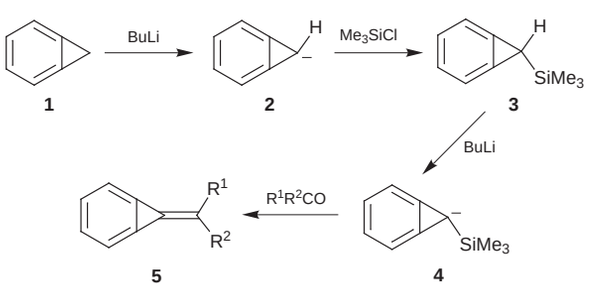
Desilylation of 1,1-bis(trimethylsilyl)-1*H*-cyclopropa[*b*]naphthalene **10** with *tert*-butoxide or hydroxide ion gives anion **9** as a highly reactive species. Formed with *tert*-butoxide **9** can be intercepted by iodomethane to give the 1-methyl-1-trimethylsilyl derivative **13**. With hydroxide ion and water catalytic didesilylation regenerates cyclopropanaphthalene **6** quantitatively. Formation of **9** with hydroxide ion in the presence of benzophenone, fluoren-9-one or 4-(dimethylamino)benzaldehyde provides a simple and economic route to the methylidenecyclopropanaphthalenes **11**, **17** and **18**. With benzaldehyde and 4-fluorobenzophenone this revised protocol (to give **19** and **12**, respectively) is effective only in the presence of 18-crown-6.

Introduction

The class of strained aromatic hydrocarbons known as the cyclopropanaphthalenes and illustrated[2] by the parent compound **1** has provided a wealth of fascinating chemistry in its thirty year history.[3] The number and complexity of the aromatic systems into which a three-membered ring has been fused continue to grow[4] and a wide range of novel and unusual compounds now exists.[3–7] Yet many fundamental properties remain to be addressed and we now report on the behaviour of the 1-trimethylsilylcyclopropa[*b*]naphthalen-1-ide anion **9** with simple electrophiles.

The existence of the cyclopropabenzen-1-ide anion **2** was ably demonstrated by Eaborn and his coworkers[8] who showed that abstraction of a methylene proton from cyclopropabenzene **1** is uneventful, and that the derived benzenide anion **2** can be intercepted by trimethylsilyl chloride as silane **3** (Scheme 1). The study concluded that compound **1** has a pK_a of about 36



Scheme 1

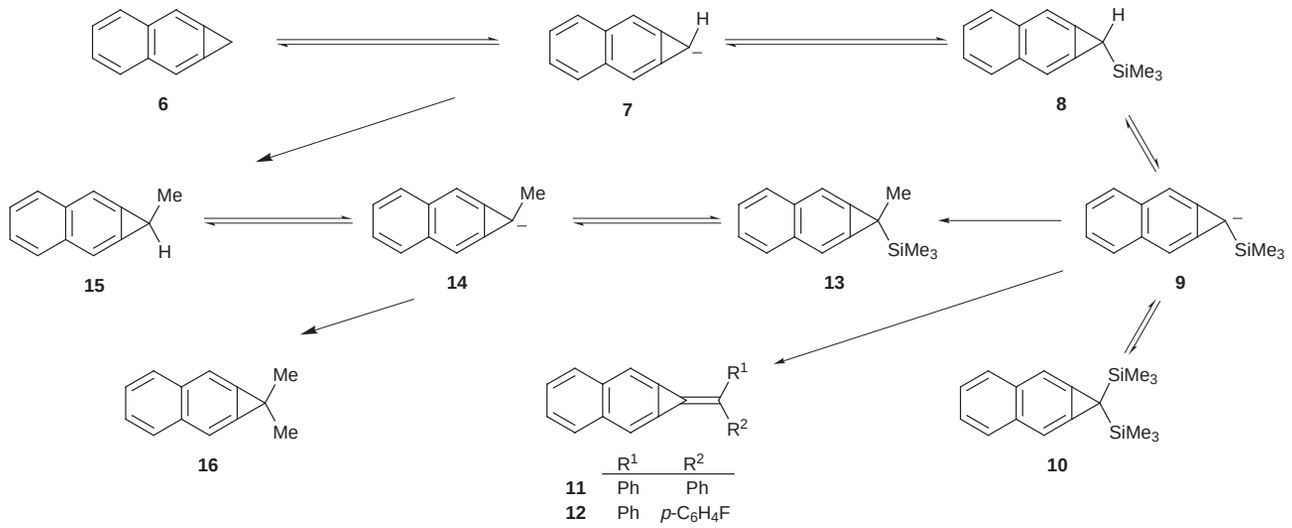
and is more acidic than toluene. However, in the gas phase, the acidity of **1** has now been shown[9] to be somewhat less than that of toluene likely due to enhanced delocalization in the anion in the absence of solvation effects. Subsequent use of lithiated anion **2** (and its homologue **7**) in solution has provided easy access to a large range of stable, crystalline and odour free methylidenecyclopropanaphthalenes, *e.g.* **5** (Scheme 1).[5,6,10–12]

Interaction of the naphthalen-1-ide anion **7** with chlorotrimethylsilane leads to the disilylcyclopropanaphthalene **10** and not **8**.[12] This is incidental to the synthesis of exocyclic olefins, *e.g.* **11**, as desilylation with *tert*-butoxide generates the essential anion **9** for Peterson olefination.[11,13] The unexpected formation of disilyl **10** occurs even when a stoichiometric quantity of butyllithium is employed, although spectral evidence has been obtained[14] that substantiates formation of monosilyl **8**. We ascribe these latter results to facile deprotonation of silane **8** by unreacted anion **7** thereby providing the stabilized α-silyl anion **9** with simultaneous regeneration of hydrocarbon **6**. The use of one molar equivalent of base strongly argues against the formation of a stationary concentration of anion **9** that desilylates silane **8**. In light of the limited alternative uses to which the α-silyl anion **9** has been put,[15] we elected to assess further its behaviour towards a range of simple electrophiles.

Results and discussion

Disilane **10** is the synthon[6] for a wide range[11] of methylidene derivatives such as **11**. For the present study, treatment of **10** with one molar equivalent of *tert*-butoxide in THF in the presence of 4-fluorobenzophenone at −70 °C gave the hitherto unknown 1-[(4-fluorophenyl)phenylmethylidene]-1*H*-cyclopropa[*b*]naphthalene **12** in 84% yield; the compound provides spectroscopic and analytical data fully in accord with the assigned structure (Experimental section). When anion generation was followed by the addition of an excess of iodomethane, a colourless oil was obtained that consisted of an inseparable four-component mixture. An examination of the ^{1}H NMR spectrum suggested that it consisted of unchanged disilane **10**, cyclopropanaphthalene **6**, 1-methylcyclopropanaphthalene[16] **15** and the expected 1-methyl-1-trimethylsilyl derivative **13** (Scheme 2) in a ratio of *ca.* 4:15:2:42; the last is present to an extent of *ca.* 43%. Although it has not been possible to isolate **13**, the spectroscopic data are fully compatible with the proposed structure. In particular, the NMR spectra show the methyl groups at δ 1.72/22.8 and 0.01/−3.3 in the ^{1}H and ^{13}C modes, respectively [>CMe(SiMe$_3$)]. The C-2/7 protons are displayed as a distinct singlet at δ 7.39 and the carbons are shielded as expected[17] and resonate at δ 109.9. Moreover, the identity of the compound can be regarded as secure since the ion at *m*/*z* 226.1180 in the mass spectrum of the mixture [*m*/*z* 284 (6.7), 227 (3.3), 226 (14.6), 211 (100), 73 (96)] is present in far greater abundance than is observed from independent fragmentation of disilane **10** [*m*/*z* 284 (5.2), 227 (0.8), 226 (0.25), 73 (100)].

The formation of cyclopropanaphthalene **6** as the second most abundant product from desilylation of **10** in the presence of methyl iodide is at first surprising. Removal of the trimethylsilyl function of product **13** by unreacted *tert*-butoxide would provide the monomethyl anion **14** that could then provide the



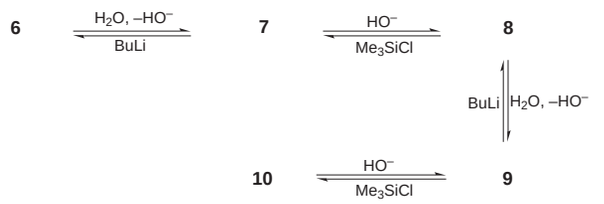
Scheme 2

methylated cyclopropanaphthalenes **15** and **16** from reaction with iodomethane and water, respectively (Scheme 2). The presence of water in a quantity no more than stoichiometric (≤6.5 mg, ≤0.36 mmol) would provide a proton source, with concomitant hydroxide ion formation, and therefore a plausible route to the observed product mixture (Scheme 2). A study of the behaviour of disilane **10** with the bases potassium *tert*-butoxide and potassium hydroxide was thus necessitated.

Desilylation of **10** at −70 °C in THF with one molar equivalent of *tert*-butoxide does not occur to any significance over 40 min as the major product is unchanged **10** (*ca.* 60%) as a mixture with cycloproparene **6** and monosilyl **8** (<4% each from ^{1}H NMR). However, if water (1 cm^3, 160–200 fold excess) is added *after* the 40 min period and the mixture allowed to reach room temperature over 16 h the product isolated is hydrocarbon **6** (90%). That protonation occurs from the added water was demonstrated from use of D_2O whereby 1,1-dideuteriocyclopropanaphthalene [$^2H_2$]**6** was obtained. More surprising is the formation of hydrocarbon **6** (95%) under these same reaction conditions but with catalytic *tert*-butoxide (6 mol%). Clearly the hydroxide ion generated by protonation (water) of anion **9** (or the *tert*-butoxide) (Scheme 2) must effect desilylation.

One molar equivalent of potassium hydroxide (as a suspension in THF) also brings about didesilylation of **10** when the mixture is allowed to stir with excess added water (1 cm^3) as described above; deuteration is effected again by use of D_2O. Even 6 mol% of hydroxide ion didesilylates **10** to hydrocarbon **6** almost quantitatively. However, pouring the anhydrous reaction mixture into water after 40 min at −70 °C gives 94% of unchanged disilane **10**. Repetition of this last reaction at temperatures from −40 °C to ambient shows (Experimental section) that desilylation does not occur even at room temperature. However, with excess water the use of hydroxide ion (40 min, 22 °C) removes both silyl groups to a level that is easily detected (^{1}H NMR).

These experiments show that *didesilylation* of **10** is easily effected with catalytic base (*tert*-butoxide or hydroxide) after an excess of water is added to the reaction medium. The use of hydroxide ion to remove a trimethylsilyl function is not new as shown by Eaborn and his coworkers[8,18] and is related to the stability of the ensuing carbanion;[8] in the presence of excess water protonation likely will be rapid, *viz.* **9**→**8**. However, such proton abstraction provides hydroxide ion as the conjugate base and the desilylation **8**→**7** (and hence to **6**) can proceed in a catalytic cycle as shown by Scheme 3. The reactions assessed show that **6** is accompanied by traces of unchanged **10** and monosilane **8**. While the precise quantities of these are difficult to assess, the ^{1}H NMR spectra show that the proportion of **10**

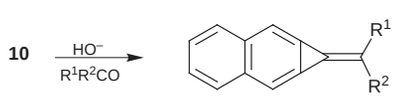


Scheme 3

always exceeds that of **8** by a factor of at least two; base induced desilylation of **8** is likely faster than that of **10**.

In order to provide more substantive evidence for the formation of **8**, disilane **10** was treated with stoichiometric *tert*-butoxide and the anion protonated by addition of excess trifluoroacetic acid on the assumption that the equilibrium would markedly favour **8** and the trifluoroacetate anion. Monosilane **8** is indeed the major product of reaction and it provides distinct proton[14] and carbon NMR signals that show C-2/7 at 110.0, C-1 at 24.3 and the Me_3Si carbons at −2.4 ppm, respectively. It has not been possible yet to separate this compound from either **6** or a ring opened product thought to result from electrophilic addition[19] of the acid across the cycloproparene σ bond (Experimental section).

The ease of desilylation of **10** by hydroxide ion has prompted a re-examination of our Peterson olefination protocol.[6,11,12] We find that a suspension of one molar equivalent of potassium hydroxide in the presence of **10** and benzophenone at −70 °C followed by slow warming to room temperature (16 h) provides the known[12] diphenylmethylidene derivative **11** (Scheme 4) in a yield of 90%—almost the same as that from use of *tert*-butoxide. Even at 0–20 °C **11** is isolated almost quantitatively! Similarly, preparations of the known **17** (from reaction with fluoren-9-one[20]) and **18** [from use of *p*-(dimethylamino)-benzaldehyde[12]] at 0–20 °C proceed to the extent of 70 and 84% that compare with 92 and 94%, respectively, employing *tert*-



	R^1	R^2
11	Ph	Ph
12	Ph	*p*-C_6H_4F
17	9-fluorenylidene	
18	*p*-$C_6H_4NMe_2$	H
19	Ph	H

Scheme 4

butoxide. Reactions with 4-fluorobenzophenone and benzaldehyde provide the methylidene derivatives **12** and **19**, but only in trace quantities. However, the efficacy of the nucleophile in these last two hydroxide ion-induced desilylations is improved by use of 18-crown-6 whereupon **12** and **19** are formed in yields of 65 and 37%, respectively. These experiments show that exocyclic olefin derivatives of **6** can be obtained from **10** by employing hydroxide ion with 18-crown-6 in a reaction that not only employs less expensive reagents but also is easy to perform at ambient temperature.

Experimental

Melting points were determined using a Reichert Thermovar hot-stage melting point apparatus and are uncorrected. Microanalyses were performed by the microanalytical facility of the University of Otago, Dunedin, New Zealand. Low-resolution mass spectra were recorded on a Hewlett-Packard 5595 instrument at 70 eV, and accurate mass data were from a Kratos MS80 RFA instrument. IR spectra were recorded for KBr disks on a BIORAD FTS-7, and UV–VIS spectra on a Hewlett-Packard 8452A Diode Array Spectrophotometer in quartz cells. ^{1}H and ^{13}C NMR spectra were recorded on either a Brucker AC 300E or a Varian UNITY Inova instrument operating at 300 and 75 MHz in [^{2}H]chloroform solution with (and without) tetramethylsilane as internal standard; resonances were assigned with the aid of ^{1}H COSY, ^{1}H–^{13}C HETCOR, HMQC, HMBC and DEPT experiments; *J* values are in Hz and chemical shifts in ppm.

TLC analyses were performed using DC-Alufolien $60F_{254}$ (layer thickness: 0.2 mm) and components detected by UV at 254 or 350 nm. Kieselgel 60 (230–400 mesh ASTM) was used for flash column chromatography and radial chromatography plates were coated with silica gel DGF_{254} to a thickness of 2 mm. Chromatographic separations employed the solvent gradients: light petroleum–dichloromethane–ethyl acetate–acetone.

Freshly powdered potassium hydroxide pellets were used immediately and potassium *tert*-butoxide sublimed using a Büchi GKR-51 ball oven in sublimation mode and stored in a dry flask under a nitrogen atmosphere for no more than five days prior to use. Other reagents were purified according to the procedures given in Perrin, Armarego and Perrin,[21] and solid reagents were stored over diphosphorus pentoxide in vacuum overnight before use. Butyllithium concentrations were determined by the method of Winkle, Lansinger and Ronald.[22] THF was distilled from potassium–benzophenone immediately before use.

1,1-Bis(trimethylsilyl)-1*H*-cyclopropa[*b*]naphthalene 10

Disilane **10** was prepared in four steps from naphthalene according to the literature procedures.[7,23,24]

Reactions of 1,1-bis(trimethylsilyl)-1*H*-cyclopropa[*b*]-naphthalene 10 with potassium *tert*-butoxide

General method. Unless stated to the contrary, a solution of potassium *tert*-butoxide (1.0–1.1 mol equiv.) in tetrahydrofuran (THF) (5 cm^3) was added dropwise to a stirred solution of disilylcycloproparene **10** (93–110 mg, 0.33–0.39 mmol) in the same solvent (10 cm^3) at −70 °C under an inert nitrogen atmosphere and in a cooling/heating bath. The stirred solution was warmed to −40 °C for 90 min (except as stated) and then returned to −70 °C whereupon the electrophile was added. The mixture was warmed to room temperature over 16 h, water (15 cm^3) and dichloromethane (15 cm^3) were added and the organic phase separated. The aqueous phase was re-extracted with dichloromethane (until the extracts were transparent to TLC) and the combined organic phases were washed (water, 50 cm^3), dried ($MgSO_4$) and concentrated in vacuum. The crude product mixture thus obtained was subjected to radial chromatography whereupon the major fraction was eluted with light petroleum.

Control reaction. In the absence of base, water (1 cm^3, 55.55 mmol) in THF (5 cm^3) was added dropwise to the stirred solution of disilane **10** (40 mg, 0.14 mmol). Unchanged **10** (39 mg, 97%) was recovered as colourless prisms; mp, mmp 95–97 °C.

1-[(4-Fluorophenyl)phenylmethylidene]-1*H*-cyclopropa[*b*]-naphthalene 12

The base, disilane **10** (102 mg, 0.36 mmol) and 4-fluorobenzophenone (80 mg, 0.40 mmol) at −70 °C without warming gave a bright yellow oil which on trituration (diethyl ether) afforded *compound* **12** (97 mg, 84%) as bright yellow needles (diethyl ether); mp 105–106 °C (Found: C, 89.16; H, 4.69. $C_{24}H_{15}F$ requires C, 89.42; H, 4.69; F, 5.89%); ν_{max}/cm^{-1} 1744, 1636, 1597, 1499, 1400, 1223, 1157, 1136, 835, 760, 746 and 695; λ_{max}(hexane)/nm 228 (3.65), 248 (3.41), 288 (3.21), 408 (3.55) and 434 (log ε 3.59); λ_{max}(acetonitrile)/nm 228 (3.64), 246sh (3.40), 286 (3.21), 406 (3.55) and 432 (log ε 3.57); δ_H 7.04 (broad t, *J* 8.7, 11/13-H), 7.26 (t, *J* 7.3, 18-H), 7.30–7.38 (m, 4H, 4/5-H + 16/20-H or 17/19-H), 7.41 (d, *J* 1.8, 2-H or 7-H), 7.43 (d, *J* 1.8, 7-H or 2-H), 7.57–7.65 (m, 4H, 10/14-H + 16/20-H or 17/19-H), 7.73–7.79 (AA′, 3/6-H); δ_C 107.0 (C-2/7), 111.6 (C-1), 115.2 (d, $^2J_{C-F}$ 21.4, C-11/13), 118.6 (C-8), 128.6 (C-3/6), 126.6 (C-4/5), 126.9 (C-1a or C-7a), 127.1 (C-7a or C-1a), 127.3 (C-18), 127.8 (C-16/20 or C-17/19), 128.4 (C-17/19 or C-16/20), 129.6 (d, $^3J_{C-F}$ 7.7, C-10/14), 135.4 (C-9), 138.6 (C-2a/7a), 139.2 (C-15), 162.0 (d, $^1J_{C-F}$ 247, C-12); *m/z* 323 (26, M + 1), 322 (100, M), 321 (42, M − H), 320 (62, M − 2H), 318 (21).

1-Methyl-1-trimethylsilyl-1*H*-cyclopropa[*b*]naphthalene 13

With the base, disilane **10** (102 mg, 0.36 mmol) and iodomethane (0.025 cm^3, 0.40 mmol) gave a colourless oil (49 mg) that contained (^{1}H NMR) disilane **10**, cyclopropanaphthalene **6**, 1-methylcyclopropanaphthalene **15** and *1-methyl-1-trimethylsilyl-1H-cyclopropa[b]naphthalene* **13** in a 4:15:2:42 ratio; the components eluded separation; (Found for **13**: $M^{\bullet+}$ *m/z* 226.11803. $C_{15}H_{18}Si$ requires 226.11778); abstracted NMR data for **13** δ_H 0.01 (s, $SiMe_3$), 1.72 (s, Me), 7.39 (s, 2/7-H), 7.41–7.44 (BB′, 4/5-H), 7.81–7.84 (AA′, 3/6-H); δ_C −3.3 ($SiMe_3$), 22.8 (Me), 31.9 (C-1), 109.9 (C-2/7), 124.9 (C-4/5), 128.0 (C-3/6), 134.2 (C-1a/7a), 135.9 (C-2a/6a).

1-Trimethylsilyl-1*H*-cyclopropa[*b*]naphthalene 8

With the base, disilane **10** (104 mg, 0.36 mmol) and trifluoroacetic acid (0.03 cm^3, 0.36 mmol) gave an inseparable yellow oil (60 mg) that contained disilane **10**, cyclopropanaphthalene **6**, a component thought to be a 2-substituted naphthalene [δ_H 8.10 (dd, 1H, *J* 8.6, 1.7), 8.61 (broad s, 1H)] and *1-trimethylsilyl-1H-cyclopropa[b]naphthalene*[14] **8** in a 4:9:6:17 ratio. Abstracted NMR data for **8** are: δ_H 0.06 (s, $SiMe_3$), 3.27 (s, 1-H), 7.41 (s, 2/7-H), 7.44–7.47 (BB′, 4/5-H), 7.84–7.87 (AA′, 3/6-H); δ_C −2.4 ($SiMe_3$), 24.3 (C-1), 110.0 (C-2/7), 125.0 (C-4/5), 127.1 (C-1a/7a), 127.9 (C-3/6), 136.3 (C-2a/6a).

1*H*-Cyclopropa[*b*]naphthalene 6

A. The solution of disilane **10** (96 mg, 0.34 mmol) and base (38 mg, 0.34 mmol) was stirred at −70 °C for 40 min then water (1 cm^3, 55.55 mmol) was added. Work up gave *compound* **6** (42 mg, 89%) as white needles; mp, mmp 86–87 °C.

B. With a catalytic amount of base (2.2 mg, 0.02 mmol) under the same conditions as **A** above, disilane **10** (93 mg, 0.33 mmol) gave a pale yellow solid (45 mg) that contained unchanged **10**, **8** and **6** in a 2:1:90 ratio (*ca.* 94% of **6**). Separation of the three components was not attempted. Upon changing water for [$^2H_2$]water, [1,1-$^2H_2$]cyclopropa[*b*]naphthalene was obtained (see below) that had >95% of label incorporated (^{1}H NMR).

C. The solution of **10** (102 mg, 0.36 mmol) with 1 mol equiv. of *tert*-butoxide was stirred at −70 °C for 40 min and then poured into water (50 cm^3). Work up gave three fractions, the major of which, a pale yellow solid (64 mg), contained unchanged **10** (*ca.* 58%), **8** and **6** in a ratio of 30 : 2 : 2. When the reaction was repeated at −40 °C more uncharacterisable material was obtained but unchanged **10** was the major component of the product mixture.

Reactions of 1,1-bis(trimethylsilyl)-1*H*-cyclopropa[*b*]-naphthalene 10 with potassium hydroxide

General method. A solution of **10** (96–194 mg, 0.34–68 mmol) in THF (5 cm^3) was added dropwise to a stirred suspension of freshly powdered KOH (0.05–1.10 mol equiv.) in THF (10 cm^3) at the specified temperature and under an inert nitrogen atmosphere in a cooling/heating bath. Work up, including warming to RT, was as for the *tert*-butoxide reactions described above.

A. Disilane **10** (100 mg, 0.35 mmol) and KOH (20 mg, 0.35 mmol) were stirred at −70 °C for 40 min then water (1 cm^3, 55.55 mmol) was added. Work up gave *1H-cyclopropa[b]-naphthalene* **6** (41 mg, 84%) as white needles; mp, mmp 86–87 °C. In like manner use of deuterium oxide (1 cm^3, 55.42 mmol), **10** (194 mg, 0.68 mmol) and KOH (38 mg, 0.68 mmol) gave white needles of [*1,1-*$^2H_2$*]-1H-cyclopropa[b]naphthalene* [$^2H_2$]**6** (95.5 mg, 70%). Two recrystallisations (light petroleum) gave an analytical sample (12 mg, 13%); mp 85–86 °C (Found: C, 92.93; H, 5.53; the H value equates to 6.95% for 3H:1D. $C_{11}H_6D_2$ requires C, 92.92; H, 4.25; D, 2.82; 7.07% H for 3H:1D); ν_{max}/cm^{-1} 3067, 3046, 1669, 1593, 1522, 1364, 1250, 1175, 1142, 945, 874, 847, 762, 727 and 467; δ_H 7.45–7.49 (BB′, 4/5-H), 7.60 (s, 2/7-H), 7.88–7.91 (AA′, 3/6-H); δ_C C-1 not observed, 112.3 (C-2/7), 123.3 (C-1a/7a), 125.4 (C-4/5), 128.4 (C-3/6), 136.6 (C-2a/6a); *m/z* 143 (11, M + 1), 142 (100, M), 141 (83, M − 1), 140 (28, M − 2H).

With **10** (149 mg, 0.52 mmol), catalytic KOH (1.5 mg, 0.027 mmol) and water at −70 °C as above, a white solid (69 mg) that contained unchanged disilane **10**, **8** and **6** in a 3 : 1 : 130 ratio (^{1}H NMR) was obtained.

B. The suspension of 1 mol equiv. of KOH with **10** (98 mg, 0.35 mmol) at −70 °C in THF was stirred at −70 °C for 40 min and then poured into water (50 cm^3). Work up gave colourless prisms of unchanged **10** (92 mg, 94%), mp, mmp 95–97 °C. At temperatures of −40, −10, 0 and 17 °C the reactions also gave almost quantitative recovery of unchanged **10**; traces of **8** and **6** were detected by NMR from the ambient temperature reaction.

With aqueous KOH (20 mg, 0.36 mol, in 1 cm^3 water) at 22 °C, disilane **10** (99 mg, 0.35 mmol) gave a pale yellow solid (61 mg) that contained recovered **10**, **8** and **6** in a ratio of *ca.* 11 : 3 : 19 representing approximately 30, 8 and 51% yields, respectively.

C. Alkylidenecyclopropa[*b*]naphthalenes were provided from disilane **10** (103 mg, 0.36 mmol) in THF (5 cm^3) that was added to a suspension of KOH (20 mg, 0.36 mmol) in THF (10 cm^3) containing the carbonyl compound (0.36 mmol) at 0 °C (except as specified) and the mixture stirred for 24 h while attaining RT. Reactions employing 18-crown-6 were performed using one molar equivalent (132 mg, 0.36 mmol) of the compound.

1-(Diphenylmethylidene)-1*H*-cyclopropa[*b*]naphthalene 11

At −70 °C with benzophenone, chromatography afforded white needles of cyclopropanaphthalene **6** (1.2 mg, 3%) as the most mobile component from the crude product mixture. Further elution (light petroleum) provided a bright yellow oil (103 mg) which on trituration (diethyl ether) gave bright yellow needles of *compound* **11** (98 mg, 90%), mp 109–111 °C (lit.,[12] 95%, mp 110–111 °C). Repetitions of the reaction at 0 °C provided **11** in yields of 85–95%.

1-[(4-Fluorophenyl)phenylmethylidene]-1*H*-cyclopropa[*b*]-naphthalene 12

With 4-fluorobenzophenone and 18-crown-6 *compound* **12** was obtained in 65% yield; mp 105–106 °C and identical to the sample recorded above.

1-Fluoren-9′-ylidene-1*H*-cyclopropa[*b*]naphthalene 17

From fluoren-9-one the first chromatographic fraction contained unchanged **10** and hydrocarbon **6** (*ca.* 15% each). The second fraction contained *compound* **17** (70%); mp 258–259 °C (lit.,[20] 92%, mp 259–260 °C).

1-[4-(Dimethylamino)phenyl]methylidene-1*H*-cyclopropa[*b*]-naphthalene 18

From 4-(dimethylamino)benzaldehyde *compound* **18** was obtained in 84% yield; mp 142–144 °C (lit.,[6] 94%, mp 141–142 °C).

1-(Phenylmethylidene)-1*H*-cyclopropa[*b*]naphthalene 19

From benzaldehyde and 18-crown-6 *compound* **18** was obtained in 37% yield; mp 153–155 °C (lit.,[12] 68%, mp 114–117 °C). Spectroscopic data matched those reported previously.[12]

Acknowledgements

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