Journal of Organometallic Chemistry, 271 (1984) 281–288
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

THE RELATIVE STEREOCHEMISTRY OF SOME α-METHYL-β-SILYLCARBONYL COMPOUNDS PRODUCED BY THE DIASTEREOSELECTIVE ALKYLATION OF β-SILYLENOLATES *,**

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(Received February 14th, 1984)

Summary

Conjugate addition of lithium bis(dimethylphenylsilyl)cuprate to methyl cinnamate (**1**), to 4-phenylbut-3-en-2-one (**4**), and to dec-3-en-2-one (**15**), followed by methylation of the intermediate enolate, gives largely one diastereoisomer of the α-methyl-β-silylcarbonyl compound (the β-silyl ester (**3**), 3-methyl-4-dimethyl(phenyl)silyl-4-phenylbutan-2-one (**5**), and 4-dimethyl(phenyl)silyl-3-methyldecan-3-one (**16**), respectively). The relative configurations of the two chiral centres in these products are proved to be (*RR*,*SS*), (*RR*,*SS*), and (*RS*,*SR*), respectively, by conversion of the ester **3** into the ketone **5**, and by Baeyer–Villiger oxidation of the ketone **5**, its diastereoisomer (*RS*,*SR*)-3-methyl-4-dimethyl(phenyl)silyl-4-phenylbutan-2-one (**8**), and the ketone **16** to the corresponding acetates ((*RR*,*SS*)-1-phenyl-1-dimethyl(phenyl)silylprop-2-yl acetate (**9**), (*RS*,*SR*)-1-phenyl-1-dimethyl(phenyl)silylprop-2-yl acetate (**12**), and (*RR*,*SS*)-3-dimethyl(phenyl)silylnon-2-yl acetate (**17**)). Fluoride ion-catalysed elimination of the silyl and acetate groups is not stereospecific when the silyl group is benzylic (**9** and **12**), but is stereospecifically *anti* for the saturated acetate (**17**). Reduction of the acetates **9**, **12** and **17** followed by *syn*-Peterson elimination gives the alkenes *E*-phenylpropene (**11**), *Z*-phenylprop-1-ene (**14**) and *E*-non-2-ene (**19**).

Introduction

In a preliminary communication [1] we reported that the enolate **2** reacted with methyl iodide to give very largely (97/3) one diastereoisomer of the β-silyl ester **3**. In this paper, we give full details of the method by which we proved the relative

* Dedicated to Professor M. Kumada in honour of his important contributions to organosilicon chemistry.

** No reprints available.

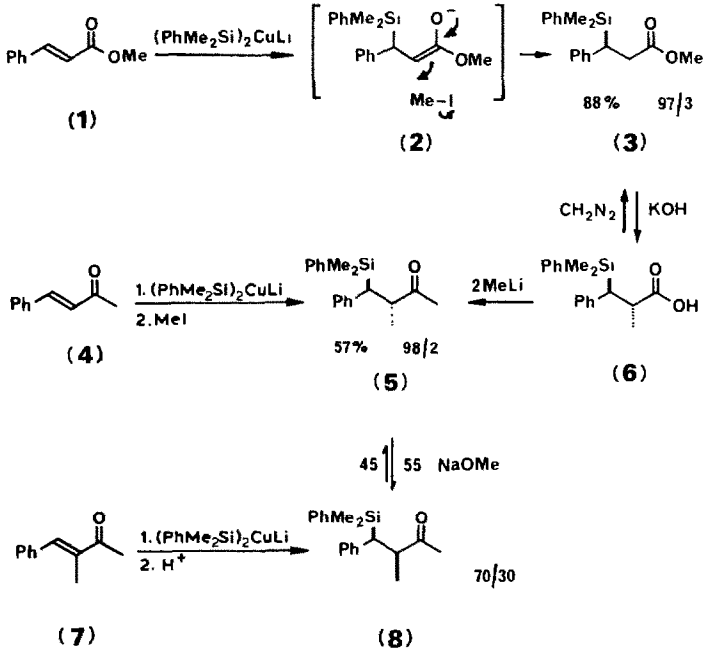
0022-328X/84/$03.00

stereochemistry of the two chiral centres in this product. In the course of our work, we have found that the fluoride ion-catalysed β-elimination of silyl and acetate groups is not stereospecific when the α-carbon carries a phenyl group (**9** or **12** → **11**), but that it is *anti*-stereospecific when the α-carbon carries an alkyl group (**17** → **18**).

Results and discussion

Hydrolysis of the ester **3** gave a crystalline acid **6** (see Scheme 1). The chiral centre α to the carbonyl group had not suffered epimerisation in this step, since treatment of this acid with diazomethane gave back the original ester **3**. Treatment of the acid **6** with methyllithium gave the ketone **5**. We also found that this ketone was the major product (98/2) when we treated the enone **4** successively with our silylcuprate reagent [2] and methyl iodide. On the other hand, the diastereoisomeric ketone **8** was the major product (70/30) when we treated the enone **7** successively with the silylcuprate reagent and acid (Scheme 1). Equilibration of the two ketones,

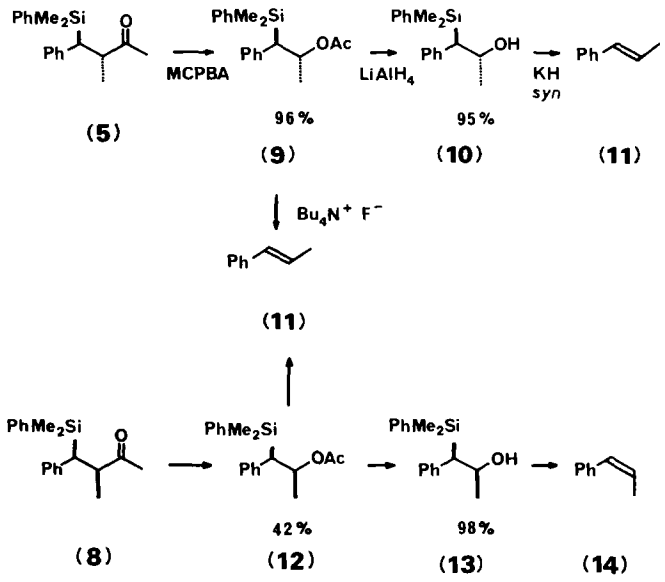
SCHEME 1



using sodium methoxide in methanol, gave a ratio **5**/**8** of 45/55. The two ketones, like the ester **3** and its diastereoisomer, are easily distinguished by their ^{1}H NMR spectra. We also separated the ketones by conventional column chromatography, thus enabling us to study each isomer independently.

Baeyer–Villiger oxidation [3] of each ketone (**5** and **8**) cleanly gave the acetates **9** and **12**, respectively (Scheme 2). Lithium aluminium hydride converted the acetates into the corresponding alcohols **10** and **13**, and Peterson elimination then gave the *E*- and *Z*-styrenes (**11** and **14**, respectively). Since the Peterson reaction is a reliably *syn* process [4], and since the Baeyer–Villiger reaction is known to take place with

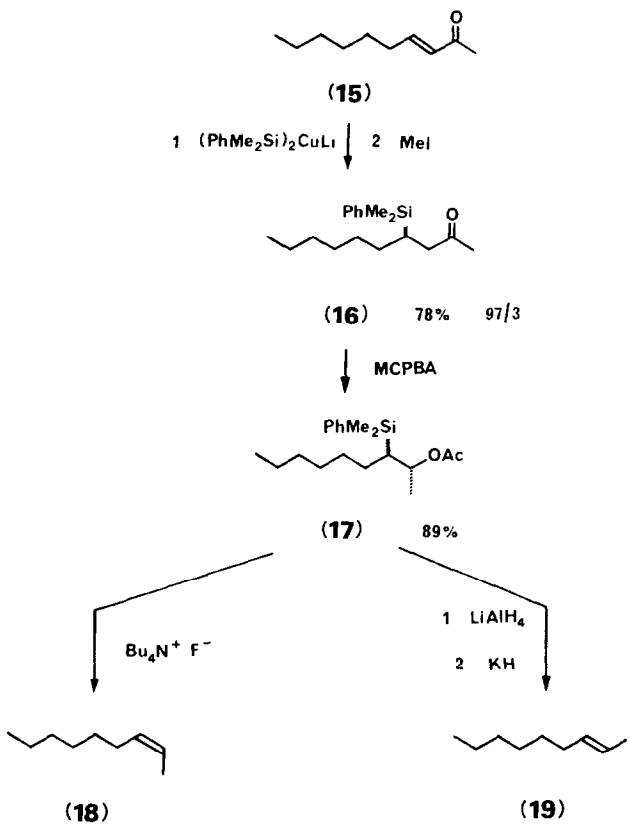
SCHEME 2



retention of configuration in the migrating group [5], the relative configurations of all our compounds are those shown.

The fluoride ion-catalysed β-elimination of a silyl and a carboxylate ion has been used to produce alkenes [6,7], and has even been shown to be stereospecific [7]. However, it had not strictly been proved to be *anti*, although this has naturally been assumed. We thought that we could prove the *anti*-stereospecificity by treating each of the acetates **9** and **12** with fluoride ion. In the event, both acetates gave the same styrene **11**, even though the *Z*-styrene **14** was configurationally stable to the reaction conditions. We reasoned that the presence of the phenyl group might have interfered with the stereospecificity of this reaction and turned, therefore, to the corresponding series of reactions in the alkyl series **15**–**19** (Scheme 3). Again, we got essentially a single diastereoisomer (**16**) in the silyl-cupration-alkylation reaction. Baeyer–Villiger reaction (**16** → **17**), treatment with lithium aluminium hydride, and Peterson elimination gave the *E*-alkene (**19**), showing that the alkylation step giving the ketone **16** was diastereoselective in the same sense as in the reactions described above. However, in this series, the acetate **17** cleanly gave the *Z*-alkene **18** on treatment with fluoride ion, showing that the problem in the reactions **9** and **12** → **11** did indeed stem from the presence of the phenyl group. Presumably, the phenyl group stabilises an intermediate benzyl anion, configurational inversion of which, followed by *anti* elimination of acetate ion, allows the acetate **9** to give the product **11** of what amounts overall to a *syn* elimination. Easy configurational inversion of a "naked" benzyl anion is not proved by these results, but its intervention is the most economical explanation of our observations. The known configurational stability of some alkyllithiums [8] points to the possibility of a difference between alkyllithiums and "naked" anions, but this point will have to be settled by more definitive experiments than those described here.

SCHEME 3



In the alkyl series **15**–**19**, we did not carry the diastereoisomer through the same sequence, because it was not easy to separate it. The formation of the less-stable *Z*-alkene in the fluoride ion-catalysed reaction (**17** → **18**) proved that this type of reaction was *anti* in nature. Since we and others have already shown that the reaction is stereospecific [6] (in the absence of phenyl substituents), we are confident that this useful alkene-forming reaction can now be described as *anti*-stereospecific.

Experimental

Light petroleum refers to the fraction, b.p. 40–60 °C unless otherwise stated.

Correlation of the ester 3 with the ketone 5

The ester **3** [1,11] (190 mg, 0.61 mmol) in ethanol (3 ml) and potassium hydroxide (300 mg) in water (3 ml) were heated for 100 min at 90 °C. An aqueous work up gave (*RR*,*SS*)-2-methyl-3-dimethyl(phenyl)silyl-3-phenylpropanoic acid (**6**) (145 mg, 80%) m.p. 99–101 °C (from ether/hexane). ν_{max}(film) 1680 cm^{-1} (C=O), δ (90 MHz, CCl_4) 9.38 (1H, br s, OH), 7.8–6.9 (10H, m, Ph), 3.0 (1H, m, C(2)-H), 2.65 (1H, d, *J* 12 Hz, C(3)-H), 1.14 (3H, d *J* 7 Hz, *Me*CH), 0.33 (6H, s, $SiMe_2$) (Found: *M* − Me,

283.113 7. $C_{17}H_{19}O_2Si$ calcd.: M − Me, 283.115 4), m/z 284 (8%, M + 1 − Me), 283 (30, M − Me), 221 (34, M − Ph), 220 (43, M − benzene), 205 (60), 161 (9), 137 (40), 135 (100, $PhMe_2Si^+$), 118 (39), 91 (21). Methyllithium (1.1 mmol in ether) was added at room temperature to the acid **6** (145 mg, 0.48 mmol) in ether (10 ml) and kept for 30 min. Aqueous work-up gave (*RR,SS*)-3-methyl-4-dimethyl(phenyl)silyl-4-phenylbutan-2-one (**5**) (115 mg, 77%). This product was identical (^{1}H NMR, MS, IR) with the ketone described below.

The silylation reaction followed by methylation

A solution of dimethyl(phenyl)silyllithium (18.5 mmol) [10] in dry THF was added to copper(I) cyanide (0.85 g, 9.5 mmol) under nitrogen at 0 °C. The α,β-unsaturated ketone (**4** or **15**) (8.75 mmol) in THF (7 ml) was added dropwise to the cuprate reagent at −23 °C, and the mixture stirred for 5 h. Methyl iodide (9 mmol) was added dropwise at −23 °C, and after 1 h the mixture was allowed to warm to room temperature. Ammonium chloride solution (36 ml) was added, then light petroleum (50 ml) and the organic phase washed with ammonium chloride solution. The organic layer was dried and evaporated in vacuo, and the products purified by column chromatography (silica gel, light petroleum/ether, 7/3, v/v). The following compounds were prepared by this method: (*RR,SS*)-3-methyl-4-dimethyl(phenyl)silyl-4-phenylbutan-2-one (**5**) (56%), R_F (light petroleum/ether 7/3) 0.66, ν_{max} (CCl_4) 1710 (C=O) and 1600 cm^{-1} (Ph), δ (250 MHz, $CDCl_3$) 7.41–6.92 (10H, m, Ph), 3.1–2.85 (1H, dq, J 10.8 and 6.9 Hz, C(3)-H), 2.66 (1H, d, J 10.8 Hz, C(4)-H), 1.85 (3H, s, MeC=O), 0.92 (3H, d, J 6.9 Hz, C(3)-Me), 0.22 and 0.14 (6H, 2s, $SiMe_2$) (Found: M^+ 296.1602. $C_{19}H_{24}OSi$ calcd.: M, 296.1596), m/z 296 (16% M^+), 282 (10), 281 (38, M − Me), 137 (21), 136 (13), 135 (100, Me_2PhSi^+), and (*RS,SR*)-4-dimethyl(phenyl)silyl-3-methyldecan-3-one (**16**) (78%), R_F (light petroleum/ether, 7/3) 0.76, ν_{max} (film) 1695 cm^{-1} (C=O), δ (250 MHz, $CDCl_3$) 7.54–7.25 (5H, m, Ph), 2.52 (1H, dq, J 11.9 and 6.96 Hz, C(3)-H), 2.04 (3H, s, MeC=O), 1.6–1.0 (11H, m with peak at 1.15), 0.94 (3 H, d, J 6.96 Hz, C(3)-Me) (the doublet of the (*RR,SS*)-isomer appears at 1.03 ppm, J 6.9 Hz; this was established by partial epimerisation of **16**; the diastereoisomers in the crude mixture before epimerisation appeared to be present in the ratio ca. 97/3), 0.83 (3H, t, J 6.7 Hz, CH_3CH_2), and 0.35 and 0.34 (6H, 2s, Me_2Si), δ (62.897 MHz, $CDCl_3$) 177.24 (C=O), 133.87, 133.80, 128.95 and 127.80 (Ph), 47.18 (C(4)), 31.58, 30.17, 29.56, 27.84, 27.76, 26.22, 22.56, 13.98, 12.53, −2.66, −3.15. (Found: M^+, 304.2215. $C_{19}H_{32}OSi$ calcd.: M 304.222 2), m/z 304 (18% M^+), 289 (14, M − Me), 261 (8, M − Me − C=O), 219 (17, M − n-hexyl), 137 (12), 136 (12), 135 (100, $SiMe_2Ph^+$).

(RS,SR)-3-Methyl-4-dimethyl(phenyl)silyl-4-phenylbutan-2-one (8)

The ketone **5** (670 mg, 2.26 mmol) in methanol (15 ml) and sodium methoxide in methanol (from 670 mg of sodium in 21 ml) were kept together under nitrogen for 96 h at room temperature. An aqueous work-up gave a yellow oil (670 mg). The ratio **5**/**8** was 45/55 as determined by integration of the doublets at 0.92 (5) and 1.05 (8). Column chromatography (silica gel, light petroleum/ether 7/3 v/v) gave the ketone **5** (18%) a mixed fraction (15%) and the ketone **8** (270 mg, 40%), R_F (light petroleum/ether, 7/3) 0.575, ν_{max} (CCl_4) 1715 and 1705 (C=O), and 1595 cm^{-1} (Ph), δ (250 MHz, $CDCl_3$) 7.47–6.94 (10H, m, Ph), 3.13 (1H, m, C(3)-H), 2.58 (1H, d, J 11.7 Hz, C(4)-H), 1.79 (3H, s, MeC=O), 1.05 (3H, d, J 6.97 Hz, C(3)-Me) and

0.29 and 0.12 (6H, 2s, $SiMe_2$) (Found: M^+, 296.159 2. $C_{19}H_{24}OSi$ calcd.: M, 296.159 6), m/z 296 (9%, M^+), 281 (15, M − Me), 162 (17, $M - C_8H_{10}Si$), 147 (11), 137 (14), 136 (13), 135 (100, $SiMe_2Ph^+$). The same ketone was the major product (70/30 by ^1H NMR, integrating the well-separated CH*Me* doublets) from the reaction of the silylcuprate reagent (2.6 mmol) with the ketone **7** [12] (2.5 mmol) in THF at −23°C for 4.5 h, followed by injection of this mixture into trifluoroacetic acid (7.8 mmol) in THF (4 ml) at −77°C.

The Baeyer–Villiger oxidations

Anhydrous disodium hydrogenphosphate (360 mg) and *m*-chloroperbenzoic acid (MCPBA) (310 mg, 1.8 mmol) were added to the ketone (1 mmol) in dichloromethane (4 ml) and the reaction mixture stirred at room temperature for 24 h. The mixture was filtered, the filtrate washed with sodium hydrogencarbonate solution (10%, 250 ml), dried ($MgSO_4$) and evaporated in vacuo at room temperature to give the acetates. The following compounds were prepared using this method: (*RR,SS*)-1-phenyl-1-dimethyl(phenyl)silylprop-2-yl acetate (**9**) (96%) R_F (light petroleum/ether, 7/3) 0.74, $\nu_{max}(CCl_4)$ 1730s (C=O) and 1590 cm^{-1} (Ph), δ (250 MHz, $CDCl_3$) 7.43–6.94 (10H, m, Ph), 5.35 (1H, dq, J 10.03 and 6.02 Hz, C(2)-H), 2.58 (1H, d, J 10.03 Hz, C(1)-H), 1.79 (3H, s, MeC=O), 1.10 (3H, d, J 6.02 Hz, CH*Me*), and 0.293 and 0.130 (6H, 2s, Me_2Si), m/z 314 (6%), 313 (46, M − 1), 281 (7, $M + 1$ − MeOH), 277 (16), 275 (45), 271 (11), 269 (48), 254 (11), 252 (28, M − MeCOOH), 237 (38), 215 (2), 213 (13, $Ph_2SiMe_2H^+$), 197 (20), 193 (9), 179 (3), 137 (8), 135 (29, Me_2PhSi^+), 128 (8), 119 (11), 118 (100), 117 (19); (*RS,SR*)-1-phenyl-1-dimethyl(phenyl)silylprop-2-yl acetate (**12**) (95%) R_F (light petroleum/ether, 7/3) 0.66, ν_{max} (CCl_4) 1730s (C=O) and 1600 cm^{-1} (Ph), δ (250 MHz, $CDCl_3$) 7.39–7.1 (10H, m, Ph), 5.35 (1H, quintet J 6.5 Hz, C(2)-H), 2.46 (1H, d, J 6.50 Hz, C(1)-H), 1.86 (3H, s, MeC=O), 1.10 (3H, d, J 6.23 Hz, CH*Me*) and 0.306 and 0.178 (6H, 2s, Me_2Si), m/z 313 (6%, $M + 1$), 282 (20), 281 (89, $M + 1$ − MeOH), 277 (22), 275 (60), 269 (10), 254 (3), 252 (27, M − MeCOOH), 237 (31), 213 (56), 197 (33), 179 (10), 137 (29), 135 (72 Me_2PhSi^+), 128 (11), 119 (29), 118 (100), 117 (47), 116 (17), 115 (6); and (*RR,SS*)-3-dimethyl(phenyl)silylnon-2-yl acetate (**17**) (42%) R_F (light petroleum/ether, 7/3) 0.79, ν_{max} (CCl_4) 1710 cm^{-1} (C=O), δ (250 MHz, $CDCl_3$) 7.53–7.25 (5H, m, Ph), 5.09 (1H, dq, C(2)-H), 1.93 (3H, s, MeCO), 1.56–1.0 (11H, m, with peak at 1.18 ppm), 1.13 (3H, d, J 6.51 Hz, CH*Me*) (the doublet of the diastereoisomer appears at 1.04 ppm, J 7.11 Hz), 0.84 (3H, t, J 6.6 Hz, CH_3CH_2), and 0.339 and 0.325 (6H, 2s, Me_2Si) δ (62.897 MHz, $CDCl_3$) 170.51 (C=O), 133.85, 133.76, 128.86 and 127.70 (SiPh), 73.33 (C(2)), 32.31, 31.64, 29.95, 29.37, 26.50, 22.60, 21.41, 19.25, 14.02, −2.54 and −3.12 ($SiMe_2$).

Reductive cleavage of acetates to alcohols

The alcohol (1 mmol) in ether (10 ml) was stirred with a suspension of lithium aluminium hydride (260 mg) in ether (10 ml) at room temperature for 2 h, and worked-up in the usual way. The following compounds were prepared by this method: (*RR,SS*)-1-dimethyl(phenyl)silyl-1-phenylpropan-2-ol (**10**) (95%), R_F (light petroleum/ether, 7/3) 0.39, ν_{max} (CCl_4) 3590 and 3570 (OH) and 1595 cm^{-1} (Ph), δ (250 MHz, $CDCl_3$) 7.55–6.92 (10H, m, Ph), 4.23 (1H, m, C(2)-H), 2.32 (1H, d, J 9.50 Hz, C(1)-H), 1.57 (1H, br s, OH, exchanges with D_2O), 1.08 (3H, d, J 6.05 Hz, CH*Me*), and 0.269 and 0.224 (6H, 2s, Me_2Si) (Found: $M^+ - H_2O$: 252.1331.

$C_{17}H_{20}Si$ calcd.: M, 252.133 4) m/z 270 (16%, M), 252 (9, $M - H_2O$), 237 (13), 223 (5), 210 (14), 209 (10), 197 (27), 195 (13), 193 (11), 277 (10), 167 (11), 165 (22), 137 (66, $PhMe_2SiH_2^+$), 135 (49, $PhMe_2Si^+$), 119 (28), 118 (100, $M - PhMe_2SiOH$), 117 (60), 116 (11), 115 (10); (*RS,SR*)-1-dimethyl(phenyl)silyl-1-phenylpropan-2-ol (**13**) (98%), R_F (light petroleum/ether 7/3) 0.42, ν_{max} (CCl_4) 3630 and 3590 (OH), and 1595 cm^{-1} (Ph), δ (250 MHz, $CDCl_3$) 7.47–7.08 (10H, m, Ph), 4.28 (1H, quintet, J 6 Hz, C(2)-H), 2.36 (1H, d, J 8.10 Hz, C(1)-H), 1.56 (1H, s, OH), 1.13 (3H, d, J 6.17 Hz, Me), 0.314 and 0.149 (6H, 2s, Me_2Si) (Found: $M^+ - H_2O$, 252.1331. $C_{17}H_{20}Si$ requires $M - H_2O$, 252.1334) m/z 252 (3%), 146 (9), 137 (47, $PhMe_2SiH_2^+$), 136 (14), 135 (73), 120 (3), 119 (32), 118 (100), 117 (51), 116 (8); and (*RR,SS*)-3-dimethyl(phenyl)silylnonan-2-ol (82%), R_F (light petroleum/ether 7/3) 0.45, ν_{max} (film) 3350 br (OH) cm^{-1} δ (80 MHz, $CDCl_3$) 7.60–7.24 (5H, m, Ph), 3.97 (1H, dq, C(2)-H), 1.65–1.0 (15 H, m with peaks at 1.18 and 1.09), 0.85 (3H, t, $MeCH_2$), 0.33 (6H, s, Me_2Si) (Found: $M^+ - H_2O$, 260.1943. $C_{17}H_{28}Si$ calcd.: $M - H_2O$, 260.1960) m/z 260 (8%, $M - H_2O$), 137 (100, $H_2SiMe_2Ph^+$), 135 (55, $SiMe_2Ph^+$).

Fluoride-induced elimination of the acetates

Tetra-n-butylammonium fluoride (5 mmol) in THF (5 ml) was added to the acetate (1 mmol) in THF (3 ml) and the mixture stirred for 1 h at room temperature. The olefin was extracted with light petroleum (b.p. 30–40 °C), the extract washed with water and dried ($MgSO_4$). Cautious evaporation in vacuo and distillation (Kugelrohr 60–100 °C, 20 mmHg) gave the olefins almost quantitatively. The acetates **9** and **12** gave under these conditions *E*-phenylpropene ν_{max} (CCl_4) [13] 965 and 968 cm^{-1}. The 60 MHz ^1H NMR spectrum was identical with that in the Aldrich NMR catalogue (4/19A). The acetate **17** gave *Z*-non-2-ene (**18**), ν_{max} (film) 3500, 3000, 2950, 2925 and 2850 (C–H stretch), 1715 and 1708 (C=C), 1460, 1445 and 1420 cm^{-1}, δ (250 MHz, $CDCl_3$) 5.40 (2H, m, CH=CH), 2.02 (2H, m, which becomes a t, J 5.6 on irradiation at 5.4, CH_2CH=CH), 1.60 (3H, d, J 6 Hz, which becomes a s on irradiation at 5.4 Hz, $MeCH$=CH), 1.29 (8H, brs, CH_2) and 0.89 (3H, t, J 6, $MeCH_2$) (Found: M^+, 126.1409. C_9H_{18} calcd.: M, 126.1408).

Hydride induced elimination of the alcohols

Potassium hydride (ca. 350 mg washed with hexane (5 ml)), THF (5 ml) and the alcohol (0.5 mmol) were stirred for 30 min at room temperature. Ammonium chloride solution (5 ml) was added at 0 °C and the products extracted with light petroleum (b.p. 30–40 °C). The organic phase was washed with ammonium chloride solution (200 ml) and dried ($MgSO_4$). Evaporation and distillation (Kugelrohr 60–80 °C/20 mmHg) gave the olefins in essentially quantitative yield. The alcohol **10** gave *E*-phenylpropene (**11**) identical to the sample described above. The alcohol (**13**) gave *Z*-phenylprop-1-ene (**14**), ν_{max} (film) 770 cm^{-1}, δ (80 MHz, $CDCl_3$) 7.7–7.2 (5H, m, Ph), 6.45 (1H, dq, J 11.2 and 1.7 Hz, C(1)-H), 5.99–5.57 (1H, m, C(2)-H), 1.91 (3H, dd, J 7.2 and 1.7 Hz, Me) Found: M^+, 118.0781. C_9H_{10} calcd.: M, 118.0782). The alcohol derived from the acetate **17** gave *E*-non-2-ene (**19**) ν_{max} (film) 3050, 3000, 2950, 2920, 2850, 1460, 1445, 1430 and 970 cm^{-1}, δ (400 MHz, $CDCl_3$) 5.43 (2H, m, C(2)-H and C(3)-H), 1.97 (2H, m, CH_2CH=CH), 1.65 (3H, dd, J 4.0 and 1 Hz, CH_2CH=CH), 1.28 (8H, br s, CH_2 s), 0.90 (3H, t, J 6 Hz, $MeCH_2$) (Found: M, 126.1410. C_9H_{18} calcd.: M, 126.1408).

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