195. Cyclopropanization of Methyl Carboxylates with *Tebbe*-Type Reagents

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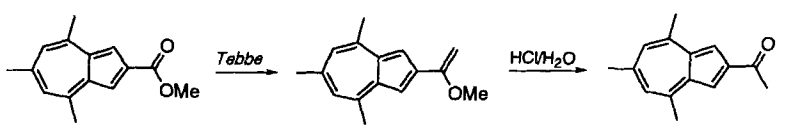
The methylenation reaction of methyl azulene-2-carboxylates (*cf. Schemes 1* and *2*) with *Tebbe*'s or *Takai*'s reagent is described. When the prescribed amount of *Takai*'s reagent is applied in a four-fold excess, the corresponding cyclopropyl methyl ethers are formed instead of the enol ethers (*cf. Schemes 2* and *3*). Similarly, methyl benzoate and methyl 2-naphthoate yield, after treatment with *Takai*'s reagent and hydrolysis, the corresponding cyclopropanols **18** and **19**, respectively (*Scheme 3*). The cyclopropyl methyl ether **4** or cyclopropanol **5** rearrange, on acid catalysis, into the 1-(azulen-2-yl)propan-1-one **20** (*Scheme 4*) whose reduction with Et_3SiH in CF_3COOH yields the 2-propylazulene **21**.

Introduction. – In the course of the investigation of a new synthesis for substituted benz[*a*]azulenes [1], we were interested in a convenient access to methyl 4,6,8-trimethylazulen-2-yl ketone (**1**)[2]). A promising approach seemed to be the methylenation reaction of the corresponding methyl azulene-2-carboxylate **2** with *Tebbe*'s reagent or *Tebbe*-type reagents (*cf.* [4]) to yield the methyl enol ether **3** which, on hydrolysis, leads to **1** (*Scheme 1*).

Scheme 1



2 **3** **1**

The azulene-2-carboxylate **2** is easily accessible by the reaction of methoxycarbonylcyclopentadienide (*cf.* [5]) with 2,4,6-trimethylpyrylium tetrafluoroborate [6] [7][3]). Indeed, when **2** was treated with 1 mol-equiv. of the commercially available *Tebbe* reagent[4]) in THF at 0° and the reaction mixture hydrolyzed, **1** was formed in almost quantitative yield [1].

In later runs, we found that *Takai*'s modification of the methylenation reaction [8] (*cf.* [9]) of carboxylates gave nearly the same results with **2**. The nature of the reactive species in *Takai*'s reagent seems still to be unknown [8]. For a quantitative methylenation

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[2]) Compound **1** has been synthesized together with its 1-acetyl isomer in a yield of 25% by acetylation of 4,6,8-trimethylazulene with hot malonic acid [2] (*cf.* [3]). The thermal rearrangement of methyl 4,6,8-trimethylazulen-1-yl ketone into **1** has also been observed [2].

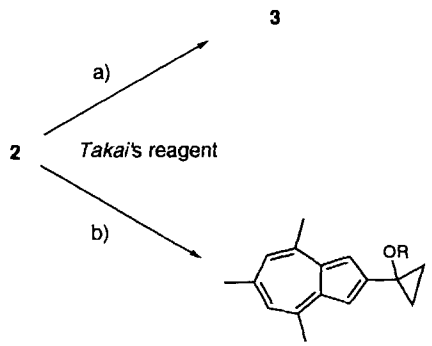
[3]) When the azulene-forming reaction is run in MeOH, **2** separates in microcrystalline form directly from the reaction mixture.

[4]) *Aldrich*®, 0.5M solution in toluene.

of 1 mol-equiv. of a carboxylate, 2.2 mol-equiv. of CH_2Br_2 and 4.0 mol-equiv. of $TiCl_4$ in the presence of 9 mol-equiv. of Zn and 8 mol-equiv. of *N,N,N′,N′*-tetramethylethylenediamine (TMEDA) have to be applied.

We were quite surprised to find that an increase in the molar amount of *Takai*'s reagent per mol of **2** led to the formation of a new product **4** which was formed almost quantitatively, when a four-fold excess of the prescribed amount of the reagent was used (*Scheme 2*)[5]).

Scheme 2



a) Prescribed amount of *Takai*'s reagent (*cf.* [8] and text).
b) Four-fold amount of *Takai*'s reagent.

The structure of the new product turned out to be that of the corresponding cyclopropyl methyl ether **4** which could easily be hydrolyzed to cyclopropanol **5**. The structure of this compound was established by an X-ray crystal-structure analysis (see *Exper. Part*). One can speculate that **4** is a follow-up product of **3**. However, treatment of **3** with *Takai*'s reagent did not lead in our hands to the formation of **4**. Furthermore, we checked that the reaction of **1** with an excess of *Takai*'s or *Tebbe*'s reagent did not yield a corresponding cyclopropane derivative, but stopped at the stage of 2-isopropenyl-4,6,8-trimethylazulene (*cf.* [7]). These observations may be considered as an indication that the cyclopropanization reaction of **2** occurs in two consecutive methylenation steps of intermediates linked with their O-atoms to an appropriate Ti-ylide complex.

The cyclopropanization reaction of methyl carboxylates with *Takai*'s reagent seems to be quite general as is demonstrated by the examples delineated in *Scheme 3*.

The reaction of the azulene-2-carboxylates **6** and **7** with the appropriate amount of *Takai*'s reagent yielded the enol ethers **8** and **9**[6]), or the corresponding cyclopropyl methyl ethers **12** and **13**, respectively. In the case of the azulene-1,2-dicarboxylate **7**, only the MeOCO group at C(2) underwent the methylenation reactions[7]). Further examples are the cyclopropanization reaction of methyl benzoate (**14**) and methyl 2-naphthoate (**15**)

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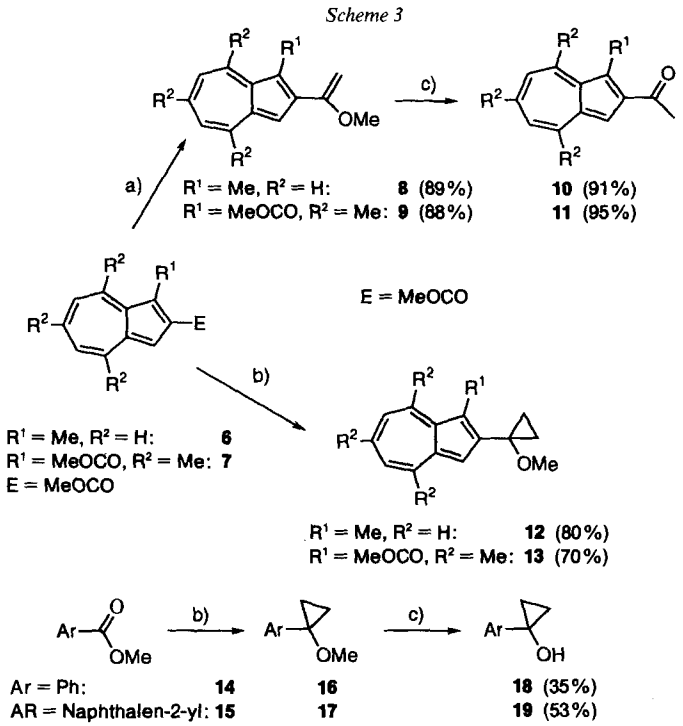
[5]) The same result was achieved, when 2 mol-equiv. of *Tebbe*'s reagent from *Aldrich*® was employed, provided that the reagent was taken from a freshly opened bottle.

[6]) Compounds **8** and **9** lead, on hydrolysis, to **10** and **11**, respectively.

[7]) Ethyl 2,4,6,8-tetramethylazulene-1-carboxylate reacts also with the four-fold amount of *Takai*'s reagent in THF at 0°. However, no cyclopropane derivative could be isolated from the reaction mixture.

Scheme 3

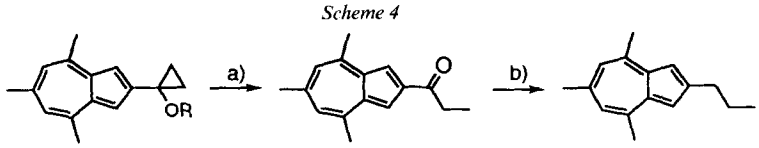


a), b) See *Scheme 2*. c) 2N HCl.

with the four-fold amount of *Takai*'s reagent. However, no cyclopropane derivative could be obtained from the reaction of methyl 2-phenylacetate with *Takai*'s reagent[8]).

The formation of cyclopropyl methyl ethers in the methylenation reaction of methyl carboxylates with Ti-based methanides allows the synthesis of further products. For example, the acid-catalyzed rearrangement of cyclopropyl compounds **4** and **5** leads to the formation of 1-(4,6,8-trimethylazulen-2-yl)propan-1-one (**20**) which, on reduction with Et_3SiH in CF_3COOH (TFA) (*cf.* also [10]) yields the corresponding 2-propylazulene **21** (*Scheme 4*). Both steps can be combined, *i.e.*, treatment of **4** or **5** with Et_3SiH in CF_3COOH leads directly to **21** with **20** as intermediate.

Scheme 4



a) TFA, 25°. b) Et_3SiH/TFA, 60°.

[8]) We cannot exclude at the moment that our workup procedure destroys the expected 1-benzylcyclopropyl methyl ether.

We thank Dr. *A. Linden* for the X-ray crystal-structure analysis of **5**, Prof. *M. Hesse* and his coworkers for mass spectra, Prof. *W. von Philipsborn* and his coworkers for NMR support and ^{1}H-NOE measurements, and *H. Frohofer* for elemental analysis. The financial support of this work by the *Swiss National Science Foundation* is gratefully acknowledged.

Experimental Part

General. See [11]. TLC on silica gel on aluminium foils (silica gel *60 F 254, Merck*; layer thickness 0.2 mm); eluant: hexane/Et_2O 1:1. UV Spectra: on a *Perkin-Elmer* spectrophotometer (model *Lambda 9*). Unless otherwise stated, all ^{1}H-NMR spectra were measured on a *Bruker* instrument (model *AC 300*) in $CDCl_3$ as solvent.

General Procedure for the Methylenation of Methyl Carboxylates Leading to Ethenyl Methyl or Cyclopropyl Methyl Ethers. The reagent for the methylenation was prepared as described by *Takai* and coworkers [8] (see also [9]). For the formation of the ethenyl methyl ethers, 2.2 mmol of CH_2Br_2 (*Fluka, puriss.*) were applied per mmol of the methyl carboxylates. The synthesis of the cyclopropyl methyl ethers was realized with 8.8 mmol CH_2Br_2 per mmol of the methyl carboxylates. The reaction mixtures were stirred for 3 h at 25° and then poured on ice in 2N NaOH. The resulting mixtures were filtered over *Celite* and the filter cakes thoroughly washed with CH_2Cl_2. The CH_2Cl_2 phases were separated and dried ($MgSO_4$). The solvent was distilled off and the residue subjected to CC (silica gel; hexane/Et_2O 1:1). For the separation of ethenyl methyl esters, silica gel was pretreated with Et_3N. Crystalline compounds were recrystallized from hexane/Et_2O. Liquid compounds were further purified by bulb-to-bulb distillation *in vacuo*.

1. Ethenyl Methyl Ethers. – 1.1. *2-(1-Methoxyethenyl)-4,6,8-trimethylazulene* (**3**). From 0.88 mmol of *methyl 4,6,8-trimethylazulene-2-carboxylate* (**2**) [7], 0.85 mmol (97%) of **3** were obtained. Blue crystals from hexane/Et_2O. M.p. 124–125°. R_f 0.35. ^{1}H-NMR[9]): 7.46 (*s*, H–C(1,3)); 7.03 (*s*, H–C(5,7)); 5.01 (*d*, ^2J = 2.4, H_A–C(2′)); 4.39 (*d*, ^2J = 2.4, H_B–C(2′)); 3.83 (*s*, MeO); 2.85 (*s*, Me–C(4,8)); 2.60 (*s*, Me–C(6)).

The hydrolysis of **3** with 2N HCl in Et_2O yielded pure *4,6,8-trimethylazulen-2-yl methyl ketone* (**1**) (*cf.* [1]).

1.2. *2-(1-Methoxyethenyl)-1-methylazulene* (**8**). From 0.76 mmol of *methyl 1-methylazulene-2-carboxylate* (**6**) [12], 0.68 mmol (89%) of **8** were obtained. Blue oil. R_f 0.36. ^{1}H-NMR: 8.24 (*d*, *J*(8,7) = 9.9, H–C(8)); 8.18 (*d*, *J*(4,5) = 8.9, H–C(4)); 7.48 (*t*, *J*(6,5) ≈ *J*(6,7) = 9.8, H–C(6)); 7.48 (*s*, H–C(3)); 7.08 (*t*, *J*(7,6) ≈ *J*(7,8) = 9.9, H–C(7)); 7.04 (*t*, *J*(5,6) ≈ *J*(5,4) = 9.9, H–C(5)); 4.85 (*d*, ^2J = 2.6, H_A–C(2′)); 4.67 (*d*, ^2J = 2.6, H_B–C(2′)); 3.82 (*s*, MeO); 2.73 (*s*, Me).

1.2.1. *Methyl 1-Methylazulen-2-yl Ketone* (**10**). In 2N HCl (20 ml), **8** (0.10 g, 0.50 mmol) was dissolved and extracted twice with Et_2O (30 ml). The extracts were dried ($MgSO_4$), and the solvent distilled off. After purification by CC, 0.085 g (91%) of **10** was obtained. Green oil. R_f 0.50. UV (hexane): λ_{max} 367 (3.75), 354 (4.06), 339 (4.04), 286 (4.98), 241 (4.54); λ_{min} 344 (4.00), 331 (4.01), 252 (4.44), 226 (4.37). IR (KBr): 2922*m*, 1663*s*, 1574*m*, 1420*m*, 1376*m*, 1355*m*, 1280*m*, 1233*s*, 1203*s*, 1102*m*, 930*m*, 797*s*, 740*s*. ^{1}H-NMR (C_6D_6): 7.97 (*d*, H–C(8), *J*(8,7) = 9.8); 7.88 (*d*, *J*(4,5) = 9.5, H–C(4)); 7.34 (*s*, H–C(3)); 7.08 (*t*, *J*(6,5) ≈ *J*(6,7) = 9.8, H–C(6)); 6.60 (*t*, *J*(7,6) ≈ *J*(7,8) = 9.9, H–C(7)); 6.59 (*t*, *J*(5,6) ≈ *J*(5,4) = 9.9, H–C(5)); 2.89 (*s*, Me–C(1)); 2.41 (*s*, MeCO).

1.3. *Methyl 2-(1-Methoxyethenyl)-4,6,8-trimethylazulene-1-carboxylate* (**9**). From 1.17 mmol of *dimethyl 4,6,8-trimethylazulene-1,2-dicarboxylate* (**7**), 1.03 mmol (88%) of **9** were obtained. Red-violet crystals from hexane/Et_2O. M.p. 95–96°. R_f 0.36. ^{1}H-NMR: 7.36 (*s*, H–C(3)); 7.10 (*s*, H–C(5)); 7.08 (*s*, H–C(7)); 4.80 (*d*, ^2J = 2.6, H_A–C(2′)); 4.39 (*d*, ^2J = 2.6, H_B–C(2′)); 3.92 (*s*, MeOCO); 3.75 (*s*, MeO–C(1′)); 2.83 (*s*, Me–C(4,8)); 2.60 (*s*, Me–C(6)).

1.3.1. *Methyl 2-Acetyl-4,6,8-trimethylazulene-1-carboxylate* (**11**). In 2N HCl (20 ml) **9** (0.10 g, 0.35 mmol) was dissolved and extracted twice with Et_2O (40 ml). The extracts were dried ($MgSO_4$) and the solvent distilled off. After purification by CC, 0.090 g (95%) of **11** was obtained. Blue-violet crystals from hexane/Et_2O. M.p. 133–134°. R_f 0.15. ^{1}H-NMR: 7.61 (*s*, H–C(3)); 7.16 (*s*, H–C(5)); 7.13 (*s*, H–C(7)); 4.00 (*s*, MeOCO); 2.91 (*s*, Me–C(8)); 2.86 (*s*, Me–C(4)); 2.68 (*s*, MeCO–C(2)); 2.63 (*s*, Me–C(6)). Anal. calc. for $C_{17}H_{18}O_3$ (270.33): C 75.53, H 6.71; found: C 75.41, H 6.58.

2. Cyclopropyl Methyl Ethers and Cyclopropanols. – 2.1. *2-(1-Methoxycyclopropyl)-4,6,8-trimethylazulene* (**4**). From 1.75 mmol of **2**, 1.67 mmol (95%) of **4** were obtained. Red liquid. R_f 0.35. ^{1}H-NMR: 7.13 (*s*, H–C(1,3)); 7.06 (*s*, H–C(5,7)); 3.48 (*s*, MeO); 2.83 (*s*, Me–C(4,8)); 2.62 (*s*, Me–C(6)); 1.40 (*AA′* of *AA′BB′*, H_A–C(2′,3′)); 1.20 (*BB′* of *AA′BB′*, H_B–C(2′,3′)).

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[9]) For clarity, the locants of the substituent(s) are primed.

2.2. *1-(4,6,8-Trimethylazulen-2-yl)cyclopropanol* (**5**). From 1.67 mmol of **4**, after hydrolysis in 2N HCl, 1.67 mmol (99%) of **5** were obtained. Pink needles from hexane/Et_2O. M.p. 130°. R_f 0.18. UV (hexane): λ_{max} 375 (3.15), 358 (3.20), 343 (3.14), 315 (sh, 3.33), 300 (4.23), 289 (sh, 4.17), 249 (3.85); λ_{min} 369 (2.90), 351 (3.09), 372 (3.03), 261 (3.13), 208 (3.51). IR (CH_2Cl_2): 3684*s*, 3585*m*, 2978*s*, 2872*s*, 1605*m*, 1383*m*, 1215*m*, 1113*s*, 1065*m*, 896*s*. ^{1}H-NMR: 7.10 (*s*, H–C(1′,3′)), 7.06 (*s*, H–C(5′,7′)); 2.83 (*s*, Me–C(4′,8′)); 2.61 (*s*, Me–C(6′)); 1.45 (*AA′* of *AA′BB′*, H_A–C(2,3)); 1.24 (*BB′* of *AA′BB′*, H_B–C(2,3)). EI-MS: 227 (29, $[M + 1]^+$), 226 (100, $M^{+\cdot}$), 211 (63), 197 (34), 183 (19), 170 (10), 169 (25), 153 (15), 152 (11), 115 (13), 77 (11), 63 (11), 57 (14), 55 (60), 51 (18).

X-Ray Structure Analysis of **5**. The structure of **5** has been solved without unusual features. There are two symmetry-independent molecules in the asymmetric unit. However, there are no significant geometrical differences between the molecules. The independent molecules are linked to each other by intermolecular H-bonds. These interactions form infinite one-dimensional chains run parallel to *x*-axis.

2.3. *2-(1-Methoxycyclopropyl)-1-methylazulene* (**12**). From 1.01 mmol of **7**, 0.81 mmol (80%) of **12** were obtained. Blue liquid. R_f 0.32. UV (hexane): λ_{max} 350 (3.43), 337 (3.38), 286 (4.56), 242 (4.07), 213 (sh, 3.97); λ_{min} 344 (3.34), 322 (3.29), 254 (3.98), 224 (3.91). IR (CH_2Cl_2): 3056*s*, 2989*s*, 2933s, 1575*m*, 1447*s*, 1421*m*, 1395*m*, 1351*m*, 1292*m*, 1229*s*, 1064*s*, 774*m*. ^{1}H-NMR: 8.24 (*d*, J(8,7) = 9.9, H–C(8)); 8.15 (*d*, J(4,5) = 8.9, H–C(4)); 7.50 (*t*, J(6,7) ≈ J(6,5) = 9.8); 7.15 (*s*, H–C(3)); 7.08 (*t*, J(7,6) ≈ J(7,8) = 9.9, H–C(7)); 7.04 (*t*, J(5,6) ≈ J(5,4) = 9.9, H–C(5)); 3.19 (*s*, MeO); 2.75 (*s*, Me); 1.25 (*AA′* of *AA′BB′*, H_A–C(2′,3′)); 1.07 (*BB′* of *AA′BB′*, H_B–C(2′,3′)). EI-MS: 213 (4, $[M + 1]^+$), 212 (9, $M^{+\cdot}$), 197 (15), 169 (13), 167 (9), 165 (18), 155 (100), 153 (13), 152 (10), 141 (32), 139 (21), 128 (12), 115 (49), 71 (11), 63 (17), 57 (15), 55 (19), 51 (11).

2.4. *Methyl 2-(1-Methoxycyclopropyl)-4,6,8-trimethylazulene-1-carboxylate* (**13**). From 1.06 mmol of **6**, 0.74 mmol (70%) of **13** were obtained. Red crystals from hexane/Et_2O. M.p. 111–112°. R_f 0.25. UV (hexane): λ_{max} 374 (sh, 3.37), 358 (3.66), 302 (4.60), 249 (4.30), 230 (4.21); λ_{min} 336 (3.60), 264 (3.75), 234 (4.20), 208 (4.09). IR (CH_2Cl_2): 2949*m*, 1714*s*, 1581*m*, 1500*w*, 1436*s*, 1389*m*, 1375*m*, 1348*w*, 1216*s*, 1164*m*, 1140*m*, 1062*m*, 1029*m*. ^{1}H-NMR: 7.12 (*s*, H–C(5,7)); 6.97 (*s*, H–C(3)); 3.95 (*s*, MeOCO); 3.20 (*s*, MeO); 2.86 (*s*, Me–C(8)); 2.83 (*s*, Me–C(4)); 2.61 (*s*, Me–C(6)); 1.23 (*AA′* of *AA′BB′*, H_A–C(2′,3′)); 1.15 (*BB′* of *AA′BB′*, H_B–C(2′,3′)). EI-MS: 299 (9, $[M + 1]^+$), 298 (52, $M^{+\cdot}$), 283 (46), 268 (36), 266 (18), 255 (19), 251 (32), 240 (20), 239 (100), 237 (20), 223 (48), 209 (47), 197 (27), 196 (19), 195 (25), 193 (40), 181 (27), 180 (24), 179 (56), 178 (51), 167 (27), 166 (28), 165 (91), 153 (45), 152 (63), 141 (26), 115 (28), 59 (26), 55 (31).

2.5. *1-Phenylcyclopropanol* (**18**; *cf.* [13]). From 1.49 mmol of *methyl benzoate* (**14**), 0.52 mmol (35%) of **18** were obtained. Colorless liquid. R_f 0.42. ^{1}H-NMR: 7.3–7.1 (*m*, arom. H); 2.68 (br. *s*, OH); 1.27 (*AA′* of *AA′BB′*, H_A–C(2,3)); 1.07 (*BB′* of *AA′BB′*, H_B–C(2,3)).

2.6. *1-(Naphthalen-2-yl)cyclopropanol* (**19**). From 2.15 mmol of *methyl 2-naphthoate* (**15**), 1.14 mmol (53%) of **19** were obtained. Colorless crystals from hexane/Et_2O. M.p. 86–87°. R_f 0.32. UV (hexane): λ_{max} 285 (sh, 2.63), 276 (2.77), 226 (3.86); λ_{min} 251 (2.56). ^{1}H-NMR: 7.9–7.7 (*m*, 4 arom. H); 7.55–7.40 (*m*, 2 arom. H); 7.31 (*dd*, ^3J = 8.6, ^4J = 1.7, 1 arom. H); 2.52 (br. *s*, OH); 1.35 (*AA′* of *AA′BB′*, H_A–C(2,3)); 1.17 (*BB′* of *AA′BB′*, H_B–C(2,3)). EI-MS: 185 (25, $[M + 1]^+$), 184 (100, $M^{+\cdot}$), 167 (13), 155 (25), 144 (10), 128 (16).

3. Reduction of Cyclopropanol 5. – Compound **5** (0.05 g, 0.22 mmol) was dissolved in TFA (50%, 2 ml) and stirred during 2 h at r.t. After neutralization with 2N NaOH, the mixture was extracted with Et_2O. After usual workup, *1-(4,6,8-trimethylazulen-2-yl)propan-1-one* (**20**) (0.04 g, 80%) was purified by CC. Blue crystals from hexane/Et_2O. M.p. 130–131°. R_f 0.29. ^{1}H-NMR: 7.73 (*s*, H–C(1′,3′)); 7.08 (*s*, H–C(5′,7′)); 3.16 (*q*, J = 7.7); 2.89 (*s*, Me–C(4,8)); 2.63 (*s*, Me–C(6)); 1.03 (*t*, J = 7.7).

3.1. *4,6,8-Trimethyl-2-propylazulene* (**21**). Compound **5** (0.05 g, 0.22 mmol) was dissolved in TFA (4 ml), and Et_3SiH (2 ml) was added. The mixture was stirred during 16 h at 60°. The mixture was poured on chopped ice and neutralized with powdered K_2CO_3. After extraction with Et_2O and usual workup, **20** (0.010 g, 20%) and **21** (0.25 g, 51%) were separated by CC. *Data of* **21**: red liquid. R_f 0.50. ^{1}H-NMR: 7.14 (*s*, H–C(1,3)); 7.02 (*s*, H–C(5,7)); 2.83 (*s*, Me–C(4,8)); 2.60 (*s*, Me–C(6)); 1.83 (*t*, J = 7.7, Pr–C(6)); 1.41 (*sext.*, J = 7.7, Pr–C(6)); 1.03 (*t*, J = 7.7, Pr–C(6)). EI-MS: 212 (15, $M^{+\cdot}$), 198 (11), 185 (11), 184 (63), 183 (32), 169 (32), 167 (15), 165 (25), 160 (13), 155 (11), 153 (26), 152 (23), 141 (15), 129 (14), 128 (22), 115 (23), 97 (15), 95 (12), 92 (14), 91 (100), 83 (23), 81 (16), 71 (21), 69 (31), 65 (219), 57 (42), 55 (43).

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