A Novel Chemoselective Cleavage of (*tert*-Butyl)(dimethyl)silyl (TBS) Ethers Catalyzed by $Ce(SO_4)_2 \cdot 4\,H_2O$

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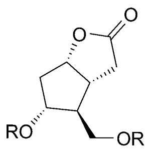
(*tert*-Butyl)(dimethyl)silyl (^{t}BuMe$_2$Si; TBS) phenyl/alkyl ethers were efficiently cleaved to the corresponding parent hydroxy compounds in good yields using catalytic amounts of $Ce(SO_4)_2 \cdot 4\,H_2O$ by microwave-assisted or conventional heating in MeOH. Intramolecular and competitive experiments demonstrated the chemoselective deprotection of TBS ethers in the presence of triisopropylsilyl (^{i}Pr$_3$Si; TIPS) and (*tert*-butyl)(diphenyl)silyl (^{t}BuPh$_2$Si; TBDPS) ethers.

Introduction. – Silyl ethers are among the most frequently used to protect OH groups, because they are easily and efficiently installed and are stable to a variety of reagents and reaction conditions [1]. In this context, the (*tert*-butyl)(dimethyl)silyl (^{t}BuMe$_2$Si; TBS), triisopropylsilyl (^{i}Pr$_3$Si; TIPS), and (*tert*-butyl)(diphenyl)silyl (^{t}BuPh$_2$Si; TBDPS) moieties are among the most important protecting groups and are often used in multistep organic syntheses [2]. Ce-based inorganic reagents for desilylation have been reported, *e.g.*, silica gel-supported ceric ammonium nitrate (CAN-SiO$_2$; stoichiometric amounts) [3], CAN/MeOH (1.2 equiv.) [4], $CeCl_3 \cdot 7\,H_2O$/MeCN (2.0 equiv.) [5], and Ce(OTf)$_4$/MeCN [6]. Unfortunately, none of these reagents lead to selectivity for TBS, TIPS, or TBDPS ethers; moreover, stoichiometric amounts of reagents are particularly disadvantageous for large-scale reactions.

Results and Discussion. – Within our current research, we observed that bis-TBS ether **1a** was cleaved within minutes upon microwave heating in MeOH containing $Ce(SO_4)_2 \cdot 4\,H_2O$. As a consequence, we decided to investigate the $Ce(SO_4)_2 \cdot 4\,H_2O$-catalyzed cleavage of silyl ether **1a** in more detail.

Optimization studies for this cleavage process were carried out on **1a**. Complete removal of the silyl group elimination occurred within 20 min upon microwave heating at 130° in MeOH containing 20 mol-% $Ce(SO_4)_2 \cdot 4\,H_2O$. At room temperature, silyl group loss was slow, but it occurred at an acceptable rate at 60° (conventional heating) and was complete within 12 h.

The optimized conditions, under microwave and conventional heating conditions (12 h), were then used to study the deprotection of a series of silylated alcohols and/or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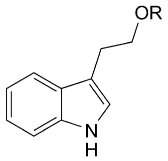
1 R = H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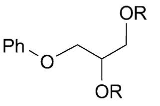
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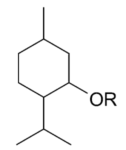
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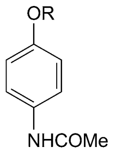
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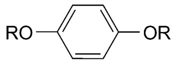
5c R = TBDPS
5 R = H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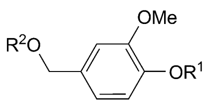
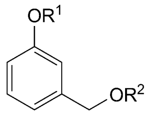
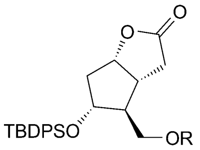
6 R = H


7a R^1 = TIPS, R^2 = TBS
7b R^1 = TIPS, R^2 = H
7c R^1 = TBDPS, R^2 = TBS
7d R^1 = TBDPS, R^2 = H


8a R^1 = TIPS, R^2 = TBS
8b R^1 = TIPS, R^2 = H
8c R^1 = TBDPS, R^2 = TBS
8d R^1 = TBDPS, R^2 = H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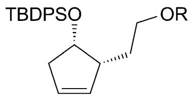
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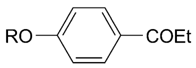
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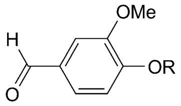
11 R = H


12a R = TBS
12b R = TBDPS
12 R = H

TBS = ^{t}BuMe$_2$Si
TIPS = ^{i}Pr$_3$Si
TBDPS = ^{t}BuPh$_2$Si

phenols (*Table 1*). Both TBS ethers of alcohols (*Entries 1–3* and *5*) and phenols (*Entries 7* and *10*) were very efficiently cleaved in high yields, while TIPS aryl ethers were cleaved only in trace amounts or not at all (*Entries 8* and *11*). TBDPS Alkyl and aryl ethers were essentially inert.

The data in *Table 1* clearly indicate that selective cleavage of TBS alkyl ethers should be possible. Indeed, the results presented in *Table 2* reveal that TBS groups of alkyl ethers are cleanly and efficiently removed in the presence of TIPS phenyl (*Entries 1* and *2*), or TBDPS phenyl (*3* and *4*) or alkyl (*Entries 5* and *6*) ethers within the same molecule. In addition, we have also carried out a series of intermolecular competition experiments (*Table 3*) which led to the conclusion that TBS aryl ethers are very selectively cleaved in the presence of TIPS or TBDPS aryl ethers.

Conclusions. – We reported that catalytic amounts $Ce(SO_4)_2 \cdot 4\,H_2O$ in MeOH selectively cleaves TBS phenyl or alkyl ethers in the presence of TIPS, or TBDPS phenyl or aryl ethers. In addition to the selectivity, this methodology is noteworthy because $Ce(SO_4)_2 \cdot 4\,H_2O$ is mild, easy to handle, and of relatively low toxicity.

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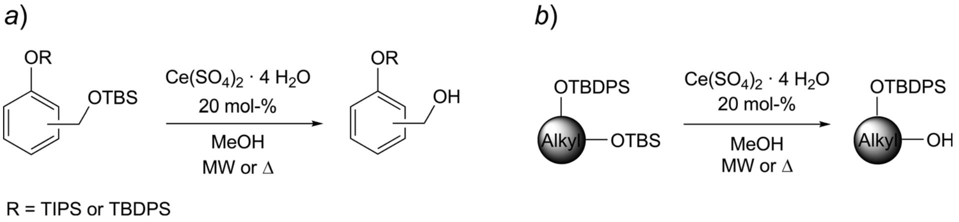
Table 1. *Deprotection of Silyl Ethers Using 20 mol-% $Ce(SO_4)_2 \cdot 4\,H_2O$ by Microwave* (MW) *or Conventional Heating*

Entry	Silyl ether	Alcohol[a]	Yield [%][b][c]	
			MW[d]	Δ[e]
1	**1a**	**1**	94	96
2	**2a**	**2**	90	88
3	**3a**	**3**	93	95
4	**3b**	**3**	NR	NR
5	**4a**	**4**	96	91
6	**4b**	**4**	NR	trace
7	**5a**	**5**	94	94
8	**5b**	**5**	NR	trace
9	**5c**	**5**	NR	NR
10	**6a**	**6**	93	90
11	**6b**	**6**	trace	NR
12	**6c**	**6**	NR	NR

[a] Confirmed by comparison of the, ^{1}H- and ^{13}C-NMR, and MS data with those of the authentic sample. [b] Yields refer to chromatographically isolated pure compounds. [c] In some cases, no reaction (NR) occurred, or product yields were very low, and *ca.* 90–98% of starting material was recovered. [d] At 130°, 20 min. [e] At 60°, 12 h.

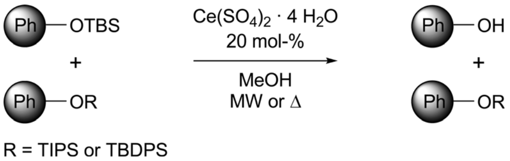
Table 2. *Intramolecular Chemoselective Deprotection of TBS Alkyl Ethers in the Presence of* a) *TIPS or TBDPS Phenyl Ethers* (*Entries 1–4*) *and* b) *TBDPS Alkyl Ethers* (*Entries 5* and *6*)



Entry	Substrate	Product[a]	Yield [%][b]	
			Δ[c]	MW[d]
1	**7a**	**7b**	95	93
2	**8a**	**8b**	96	96
3	**7c**	**7d**	94	92
4	**8c**	**8d**	93	97
5	**9a**	**9**	95	92
6	**10a**	**10**	93	96

[a] Confirmed by comparison of the ^{1}H- and ^{13}C-NMR, and MS data with those of the authentic sample. [b] Yields refer to chromatographically isolated pure compounds. [c] At 60°, 12 h. [d] At 130°, 20 min.

Table 3. *Chemoselective Deprotection of TBS Phenyl Ethers in the Presence of TIPS and TBDPS Phenyl Ethers Evaluated by Competition Experiments*



Substrates	Products[a]) (Yield [%])[b])	
	Δ[c])	MW[d])
11a + **12b**	**11** (90) + **12b** (97)	**11** (92) + **12b** (94)
12a + **11b**	**12** (87) + **11b** (95)	**12** (90) + **11b** (96)

[a]) Confirmed by comparison of the ^{1}H- and ^{13}C-NMR, and MS data with those of the authentic sample. [b]) Yields refer to chromatographically isolated pure compounds. [c]) At 60°, 12 h. [d]) At 130°, 20 min.

Experimental Part

General. The starting materials were purchased from *Aldrich Chemical Co.* and were used without further purification. The microwave-assisted reactions were performed using a focused microwave unit *Anton-Paar Synthos 300*; constant factor of the microwave, 1.214. The temp. was monitored with an IR temp. sensor. In all experiments, the microwave temp. was held constant. Reactions were carried out in 5-ml glass vessels with a magnetic stir bar, which were sealed with a cap septum. The specific reaction time corresponds to the total irradiation time. After completion of the reaction, the mixture was cooled to 25° *via* air-jet cooling. TLC: Silica-gel plates (SiO_2; 0.20-mm thickness); visualization with UV light at 254 nm or by staining with a base soln. of $CoCl_2/H_2SO_4$ ac. (2 g/100 ml H_2SO_4 10%), followed by heating. Flash column chromatography (FC): SiO_2 *60* (230–400 mesh). M.p.: *Fischer–Johns Scientific* melting-point apparatus; uncorrected. ^{1}H- and ^{13}C-NMR spectra: *Bruker Avance 300* MHz, and a *Varian 500* MHz; δ in ppm rel. to Me_4Si as internal standard, J in Hz. MS: *Shimadzu GCMS-QP2010 Plus*; in *m*/*z* (rel. %).

Microwave Irradiation. A 10-ml reaction vessel was charged with a magnetic stir bar, 0.4 mmol of TBS ether, and 20 mol-% $Ce(SO_4)_2 \cdot 4\ H_2O$ in 2 ml of MeOH. A septum cap was affixed, and the vessel was placed in the microwave cavity of an *Anton-Paar* microwave equipment. The stirred mixture was irradiated at 130° for 20 min. After cooling to r.t., TLC indicated the disappearance of starting material. The solid $Ce(SO_4)_2$ was filtered off, and the solvent was removed under reduced pressure. FC afforded the pure alcohol. All products were spectrally identical with authentic alcohols.

Conventional Heating. A 10-ml round-bottom flask was equipped with a magnetic stir bar and a reflux condenser. Then, TBS ether (0.4 mmol) and 20 mol-% $Ce(SO_4)_2 \cdot 4\ H_2O$ were added in MeOH (2.0 ml). The mixture was stirred at 60° for 12 h. After cooling to r.t., TLC indicated the disappearance of starting material. The solid $Ce(SO_4)_2$ was filtered off, and the solvent was removed under reduced pressure. FC afforded the pure alcohol. All products were spectrally identical with authentic alcohols.

*(3a*R*,4*S*,5*R*,6a*S*)-3,3a,4,5,6,6a-Hexahydro-5-hydroxy-4-(hydroxymethyl)-2*H*-cyclopenta[*b*]furan-2-one* (**1**). White solid. M.p. 115–117°. ^{1}H-NMR (300 MHz, $CDCl_3$): 0.9 (*s*, OH); 1.22 (*s*, OH); 1.9–2.2 (*m*, 1 H); 2.41 (*td*, $J = 6.6, 21.5$, 1 H); 2.56 (*d*, $J = 2.4$, 1 H); 2.62 (*td*, $J = 2.4, 5.0$, 2 H); 2.67 (*d*, $J = 2.1$, 1 H); 2.80 (*dd*, $J = 9.6, 18.0$, 2 H); 3.63 (*dd*, $J = 7.2, 10.5$, 1 H); 3.73 (*dd*, $J = 5.7, 10.5$, 1 H); 3.85 (*d*, $J = 6.6$, 1 H); 4.19 (*q*, $J = 6.3$, 1 H); 4.94 (*td*, $J = 2.7, 7.0$, 1 H). ^{13}C-NMR (75 MHz, $CDCl_3$): 35.2 (CH_2); 39.5 (CH); 40.7 (CH_2); 55.1 (CH); 63.6 (CH_2); 75.5 (CH); 83.4 (CH); 176.8 (C=O). EI-MS: 173 ($[M+1]^+$), 154 (12), 137 (15), 126 (30), 95 (18), 82 (40), 67 (23), 54 (100), 41 (30), 28 (19), 4 (15).

*(3a*R*,4*S*,5*R*,6a*S*)-5-{[(*tert*-Butyl)(dimethyl)silyl]oxy}-4-({[(*tert*-butyl)(dimethyl)silyl]oxy}methyl)-3,3a,4,5,6,6a-hexahydro-2*H*-cyclopenta[*b*]furan-2-one* (**1a**). White solid. M.p. 60–63°. ^{1}H-NMR

(500 MHz, $CDCl_3$): 0.045 – 0.036 (*m*, 12 H); 0.86 (*s*, 9 H); 0.88 (*s*, 9 H); 1.99 – 1.95 (*m*, 2 H); 2.25 – 2.2 (*m*, 1 H); 2.53 (*dd*, *J* = 18.0, 3.0, 1 H); 2.68 – 2.63 (*m*, 2 H); 2.77 (*dd*, *J* = 18.0, 10.5, 1 H); 3.47 (*dd*, *J* = 10.5, 5.5, 1 H); 3.54 (*dd*, *J* = 10.5, 5.5, 1 H); 4.15 (*q*, *J* = 5.0, 2 H); 4.92 (*dt*, *J* = 7.0, 2.5, 1 H). ^{13}C-NMR (125 MHz, $CDCl_3$): – 5.5 (4 MeSi); 18.2 (2 C); 25.8 (6 Me); 35.5 (CH_2); 39.1 (CH); 41.0 (CH_2); 56.9 (CH_2); 62.5 (CH); 74.5 (CH); 84.1 (CH); 177.2 (C=O).

*2-(1*H*-Indol-3-yl)ethanol* (**2**). Beige solid. M.p. 56 – 59°. ^{1}H-NMR (500 MHz, $CDCl_3$): 1.9 (*s*, OH); 3.0 (*t*, *J* = 6.5, 2 H); 3.86 (*t*, *J* = 6.5, 2 H); 6.9 (*s*, 1 H); 7.11 (*t*, *J* = 7.5, 1 H); 7.19 (*t*, *J* = 7.5, 1 H); 7.30 (*d*, *J* = 7.5, 1 H); 7.59 (*d*, *J* = 7.5, 1 H); 8.16 (*s*, NH). ^{13}C-NMR (125 MHz, $CDCl_3$): 28.7 (CH_2); 62.5 (CH_2); 111.3 (CH); 112.1 (C); 118.8 (CH); 119.4 (CH); 122.1 (CH); 122.6 (CH); 127.4 (C); 136.4 (C). EI-MS: 161 (M^+), 159 (12), 133 (25), 117 (12), 102 (6), 85 (100), 77 (7).

*3-(2-{[(*tert*-Butyl)(dimethyl)silyl]oxy}ethyl)-1*H*-indole* (**2a**). Amber oil. ^{1}H-NMR (500 MHz, $CDCl_3$): 0.0 (*s*, 6 H); 0.8 (*s*, 9 H); 2.9 (*t*, *J* = 7.5, 2 H); 3.8 (*t*, *J* = 7.5, 2 H); 6.95 – 6.94 (*m*, 1 H); 7.09 – 7.07 (*m*, 1 H); 7.142 – 7.140 (*m*, 1 H); 7.28 – 7.26 (*m*, 1 H); 7.57 – 7.56 (*m*, 1 H); 7.85 (*s*, NH). ^{13}C-NMR (125 MHz, $CDCl_3$): 0.0 (2 MeSi); 23.6 (C); 31.2 (3 Me); 34.2 (CH_2); 69.1 (CH_2); 116.3 (CH); 118.3 (C); 124.1 (CH); 124.4 (CH); 127.1 (CH); 127.3 (CH); 132.9 (C); 141.3 (C). EI-MS: 275 (M^+), 260 (42), 218 (61), 200 (12), 144 (100), 130 (8).

3-Phenoxypropane-1,2-diol (**3**). White solid. M.p. 50 – 53°. ^{1}H-NMR (500 MHz, $CDCl_3$): 3.28 (*s*, 2 H); 3.70 – 3.64 (*m*, 2 H); 3.78 – 3.76 (*m*, 1 H); 3.97 – 3.96 (*m*, 2 H); 4.08 – 4.06 (*m*, 1 H); 6.88 – 6.86 (*m*, 2 H); 6.96 – 6.92 (*m*, 1 H); 7.26 – 7.23 (*m*, 2 H). ^{13}C-NMR (125 MHz, $CDCl_3$): 63.7 (CH_2); 68.9 (CH_2); 70.5 (CH); 114.5 (2 CH); 121.2 (CH); 129.5 (2 CH); 158.4 (C). EI-MS: 168 (M^+), 150 (50), 136 (22), 119 (30), 94 (100), 77 (28), 66 (35).

2,2,3,3,8,8,9,9-Octamethyl-5-(phenoxymethyl)-4,7-dioxa-3,8-disiladecane (**3a**). Colorless liquid. ^{1}H-NMR (300 MHz, $CDCl_3$): 0.17 – 0.058 (*m*, 12 H); 0.9 (*s*, 18 H); 3.71 (*d*, *J* = 6.0, 2 H); 3.91 – 3.90 (*m*, 1 H); 4.16 – 4.09 (*m*, 2 H); 6.97 – 6.94 (*m*, 3 H); 7.34 – 7.29 (*m*, 2 H). ^{13}C-NMR (75 MHz, $CDCl_3$): – 4.62 (2 MeSi); – 4.54 (2 MeSi); 18.3 (2 C); 25.9 (6 Me); 65.0 (CH_2); 69.9 (CH_2); 72.1 (CH); 114.4 (2 CH); 120.5 (CH); 129.3 (2 CH); 159.0 (C). EI-MS: 397 (M^+), 303 (70), 277 (10), 249 (12), 207 (11), 171 (52), 133 (75), 89 (12), 73 (16).

2,2,9,9-Tetramethyl-5-(phenoxymethyl)-3,3,8,8-tetraphenyl-4,7-dioxa-3,8-disiladecane (**3b**). Slightly yellow oil. ^{1}H-NMR (500 MHz, $CDCl_3$): 1.09 – 1.02 (*m*, 18 H); 3.76 (*d*, *J* = 2.4, 2 H); 3.98 – 3.96 (*m*, 1 H); 4.16 – 4.12 (*m*, 2 H); 6.75 – 6.73 (*m*, 2 H); 6.93 – 6.91 (*m*, 1 H); 7.25 – 7.21 (*m*, 6 H); 7.34 – 7.31 (*m*, 4 H); 7.42 – 7.40 (*m*, 4 H); 7.63 – 7.57 (*m*, 4 H); 7.70 – 7.67 (*m*, 4 H). ^{13}C-NMR (125 MHz, $CDCl_3$): 19.5 (2 C); 27.0 (12 Me); 64.9 (CH_2); 68.6 (CH); 72.3 (CH_2); 114.6 (CH); 120.6 (CH); 127 (7 CH); 129 (7 CH); 133.6 (5 CH); 134 (4 CH); 136 (4 C); 158.9 (C). EI-MS: 391 (20), 331 (8), 253 (12), 215 (100), 199 (30), 152 (20), 139 (20), 79 (42).

Menthol (=*5-Methyl-2-(1-methylethyl)cyclohexanol*; **4**). White solid. M.p. 41 – 44°. ^{1}H-NMR (500 MHz, $CDCl_3$): 0.62 – 0.61 (*m*, 1 H); 0.83 – 0.81 (*m*, 3 H); 0.92 – 0.92 (*m*, 6 H); 1.05 – 1.03 (*m*, 1 H); 1.42 (*s*, OH); 1.65 – 1.62 (*m*, 1 H); 1.98 – 1.97 (*m*, 1 H); 2.19 – 2.18 (*m*, 1 H); 3.41 – 3.39 (*m*, 1 H). ^{13}C-NMR (125 MHz, $CDCl_3$): 16.0 (Me); 21.0 (Me); 22.2 (Me); 23.1 (CH_2); 25.7 (CH); 31.6 (CH); 34.5 (CH_2); 45.0 (CH_2); 50.1 (CH); 71.4 (CH). EI-MS: 156 (M^+), 138 (100), 119 (20), 106 (55), 95 (30), 86 (10), 57 (40).

*2-{[(*tert*-Butyl)dimethylsilyl]oxy}-4-methyl-1-(1-methylethyl)cyclohexane* (**4a**). Colorless liquid. ^{1}H-NMR (300 MHz, $CDCl_3$): 0.098 (*s*, 6 H); 0.78 – 0.77 (*m*, 6 H); 0.92 (*s*, 9 H); 1.21 – 1.19 (*m*, 1 H); 1.42 – 1.38 (*m*, 1 H); 1.70 – 1.59 (*m*, 2 H); 1.92 – 1.89 (*m*, 1 H); 2.31 – 2.22 (*m*, 1 H); 3.49 – 3.39 (*m*, 1 H). ^{13}C-NMR (75 MHz, $CDCl_3$): – 4.8 (MeSi); – 3.7 (MeSi); 15.7 (C); 18.1 (Me); 21.3 (2 Me); 22.0 (CH_2); 25.0 (CH); 25.0 (3 Me); 31.7 (CH); 34.6 (CH_2); 45.5 (CH_2); 50.3 (CH); 72.4 (CH). EI-MS: 270 (M^+), 255 (30), 216 (65), 155 (10), 143 (81), 131 (42), 128 (100), 115 (8), 85 (53), 51 (20).

*1,1′-{(*tert*-Butyl){[5-methyl-2-(1-methylethyl)cyclohexyl]oxy}silylene}bis(benzene)* (**4b**). White solid. M.p. 61 – 63°. ^{1}H-NMR (500 MHz, $CDCl_3$): 0.51 – 0.49 (*m*, 3 H); 0.71 – 0.68 (*m*, 3 H); 0.79 – 0.76 (*m*, 3 H); 1.1 (*s*, 9 H); 1.27 – 1.24 (*m*, 1 H); 1.54 – 1.50 (*m*, 2 H); 1.73 – 1.69 (*m*, 1 H); 2.28 – 2.25 (*m*, 1 H); 3.42 – 3.40 (*m*, 1 H); 7.30 – 7.28 (*m*, 6 H); 7.59 – 7.55 (*m*, 4 H). ^{13}C-NMR (125 MHz, $CDCl_3$): 15.7 (Me); 19.7 (Me); 21.5 (Me); 22.4 (CH_2); 25.2 (CH); 27.3 (3 Me); 31.7 (CH); 34.6 (CH_2); 45.4 (CH_2); 50.6 (CH); 73.9 (CH); 127 (6 CH); 129 (4 CH); 134.4 (2 C). EI-MS: 337 (30), 217 (20), 199 (80), 139 (100), 137 (28), 95 (21), 81 (57).

N-*(4-Hydroxyphenyl)acetamide* (**5**). White solid. M.p. 169–171°. ^{1}H-NMR (300 MHz, CDCl$_3$/DMSO): 1.99 (*s*, 3 H); 6.66–6.63 (*m*, 2 H); 7.28–7.23 (*m*, 2 H); 9.12 (*s*, NH). ^{13}C-NMR (75 MHz, CDCl$_3$/DMSO): 23 (Me); 114 (2 CH); 120 (2 CH); 129.9 (C); 152.7 (C); 167.7 (C=O). EI-MS: 151 (M^+), 121 (4), 109 (100), 80 (10), 65 (2), 53 (4), 42 (5), 26 (5).

N-*(4-{[(*tert*-Butyl)(dimethyl)silyl]oxy}phenyl)acetamide* (**5a**). White solid. M.p. 95–97°. ^{1}H-NMR (300 MHz, CDCl$_3$): 0.17 (*s*, 6 H); 0.96 (*s*, 9 H); 2.1 (*s*, 3 H); 6.78–6.75 (*m*, 2 H); 7.35–7.32 (*m*, 2 H); 7.6 (*s*, NH). ^{13}C-NMR (75 MHz, CDCl$_3$): −4.5 (2 Me); 18.1 (C); 24.2 (Me); 25.6 (3 Me); 120.2 (2 CH); 121.6 (2 CH); 131.5 (C); 152.3 (C); 168.4 (C=O). EI-MS: 265 (M^+), 250 (5), 208 (100), 192 (5), 180 (8), 166 (30), 149 (8), 134 (5), 116 (4), 106 (15), 73 (37), 43 (35).

N-*(4-{[Tris(1-methylethyl)silyl]oxy}phenyl)acetamide* (**5b**). White solid. M.p. 64–66°. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.10–1.07 (*m*, 18 H); 1.28–1.21 (*m*, 3 H); 2.0 (*s*, 3 H); 6.80 (*d*, *J* = 9.0, 2 H); 7.34 (*d*, *J* = 9.0, 2 H); 7.9 (*s*, NH). ^{13}C-NMR (75 MHz, CDCl$_3$): 12.5 (3 CH); 17.8 (6 Me); 24.1 (Me); 119.8 (2 CH); 121.5 (2 CH); 131.4 (C); 152.6 (C); 168.5 (C=O). EI-MS: 307 (M^+), 264 (82), 236 (60), 222 (18), 208 (80), 194 (45), 178 (12), 134 (12), 93 (12), 75 (22), 43 (100).

N-*(4-{[(*tert*-Butyl)(diphenyl)silyl]oxy}phenyl)acetamide* (**5c**). Slightly yellow oil. ^{1}H-NMR (500 MHz, CDCl$_3$): 1.0 (*s*, 9 H); 2.0 (*s*, 3 H); 6.69–6.68 (*m*, 2 H); 7.19–7.17 (*m*, 2 H); 7.41–7.33 (*m*, 7 H); 7.73–7.69 (*m*, 3 H); 7.9 (*s*, NH). ^{13}C-NMR (125 MHz, CDCl$_3$): 19.4 (C); 24.2 (Me); 26.6 (3 Me); 115.8 (CH); 119.8 (CH); 121.5 (CH); 122.6 (CH); 127.4 (CH); 129.5 (4 CH); 129.7 (3 CH); 131.2 (C); 132.8 (CH); 134.9 (2 CH); 152.3 (C); 154.1 (C); 162.7 (C); 168.0 (C=O). EI-MS: 389 (M^+), 350 (100), 290 (40), 272 (38), 231 (25), 149 (20), 77 (8).

Hydroquinone (= *Benzene-1,4-diol*; **6**). White solid. M.p. 171°. ^{1}H-NMR (300 MHz, CDCl$_3$): 6.6 (*s*, 4 H); 7.4 (*s*, 2 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 115.8 (4 CH); 149.5 (2 C). EI-MS: 110 (M^+), 81 (33), 63 (8), 53 (32), 39 (21).

*1,4-Bis{[(*tert*-Butyl)(dimethyl)silyl]oxy}benzene* (**6a**). White solid. M.p. 42–44°. ^{1}H-NMR (300 MHz, CDCl$_3$): 0.16 (*s*, 12 H); 0.97 (*s*, 8 H); 6.6 (*s*, 4 H). ^{13}C-NMR (75 MHz, CDCl$_3$): −4.4 (4 Me); 18.1 (2 C); 25 (6 Me); 120.5 (4 CH); 149.7 (2 C). EI-MS: 138 (M^+), 323 (5), 281 (100), 239 (10), 225 (15), 112 (12), 73 (75), 57 (12), 41 (12).

1,4-Bis{[tris(1-methylethyl)silyl]oxy}benzene (**6b**). White solid. M.p. 22°. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.09–1.06 (*m*, 32 H); 1.25–1.17 (*m*, 6 H); 6.7 (*s*, 4 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 12.5 (6 CH); 17.8 (12 Me); 120.3 (4 CH); 150 (2 C). EI-MS: 422 (M^+), 379 (97), 351 (40), 337 (15), 323 (40), 309 (25), 195 (10), 112 (70), 105 (50), 87 (28), 73 (39), 59 (55), 43 (22).

1,4-Bis{[(1,1-dimethylethyl)diphenylsilyl]oxy}benzene (**6c**). White solid. M.p. 121–123°. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.0 (*s*, 18 H); 6.4 (*s*, 4 H); 7.30–7.40 (*m*, 12 H); 7.63–7.60 (*m*, 8 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 19.4 (2 C); 26.5 (6 Me); 120.0 (4 CH); 127.6 (CH); 129.6 (CH); 135.5 (CH); 149.5 (2 C). EI-MS: 586 (M^+), 391 (10), 318 (3), 215 (100), 199 (83), 139 (57), 79 (75), 63 (28).

4-({[(1,1-Dimethylethyl)dimethylsilyl]oxy}methyl)-2-methoxy-1-{[tris(1-methylethyl)silyl]oxy}benzene (**7a**). Yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 0.03 (*s*, 3 H); 0.08 (*s*, 3 H); 0.94 (*s*, 9 H); 1.10 (*d*, *J* = 7.2, 18 H); 1.3–1.2 (*m*, 3 H); 3.8 (*s*, 3 H); 4.67 (*s*, 2 H); 6.72 (*dd*, *J* = 8.0, 1.5, 1 H); 6.82 (*d*, *J* = 8.0, 1 H); 6.87 (*d*, *J* = 1.5, 1 H). ^{13}C-NMR (75 MHz, CDCl$_3$): −5.1 (MeSi); −2.9 (MeSi); 12.9 (3 CH); 17.9 (6 Me); 18.4 (C); 25.7 (3 Me); 55.4 (MeO); 65.0 (CH$_2$); 110.5 (CH); 118.3 (CH); 120.0 (CH); 134.6 (C); 144.4 (C); 150.7 (C).

3-Methoxy-4-{[tris(1-methylethyl)silyl]oxy}benzenemethanol (**7b**). Yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.09 (*d*, *J* = 7.2, 18 H); 1.32–1.18 (*m*, 3 H); 3.8 (*s*, 3 H); 4.58 (*s*, 2 H); 6.72 (*dd*, *J* = 8.0, 1.8, 1 H); 6.82 (*d*, *J* = 8.0, 1 H); 6.86 (*d*, *J* = 1.8, 1 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 12.9 (3 CH); 17.9 (6 Me); 55.4 (MeO); 65.4 (CH$_2$); 111.3 (CH); 119.4 (CH); 120.2 (CH); 134.1 (C); 145.1 (C); 151.0 (C).

(*tert*-*Butyl)[4-({[(*tert*-butyl)(dimethyl)silyl]oxy}methyl)-2-methoxyphenoxy]diphenylsilane* (**7c**). Yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): −0.31 (*s*, 3 H); −0.14 (*s*, 3 H); 0.73–0.72 (*m*, 9 H); 0.95–0.97 (*m*, 9 H); 4.44–4.30 (*m*, 3 H); 5.28 (*s*, 2 H); 6.55–7.06 (*m*, 3 H); 7.20–7.35 (*m*, 6 H); 7.58–7.65 (*m*, 4 H). ^{13}C-NMR (75 MHz, CDCl$_3$): −5.0 (MeSi); −4.6 (MeSi); 10.2 (C); 19.4 (C); 26.5 (3 Me); 31.6 (3 Me); 55.1 (Me); 64.8 (CH$_2$); 115.5 (2 CH); 119.5 (2 CH); 121.6 (CH); 126.9 (2 CH); 127.7 (2 CH); 129.8 (2 CH); 132.9 (CH); 134.8 (CH); 135.5 (2 C); 136.9 (C); 138.3 (C); 154.9 (C).

*(4-{[(*tert*-Butyl)(diphenyl)silyl]oxy}-3-methoxyphenyl)methanol* (**7d**). Yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.12–1.01 (*m*, 9 H); 2.97 (*s*, OH); 3.55 (*s*, 3 H); 4.49 (*s*, 2 H); 6.9–6.5 (*m*, 3 H);

7.5–7.2 (*m*, 6 H); 7.7 (*d*, *J* = 7.8, 4 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 19.0 (C); 26.6 (3 Me); 55.4 (Me); 65.3 (CH$_2$); 111.5 (CH); 119.3 (CH); 120.0 (CH); 127.3 (CH); 127.5 (CH); 127.7 (CH); 128.1 (CH); 129.3 (CH); 129.6 (CH); 129.7 (CH); 133.6 (CH); 134.1 (CH); 134.4 (CH); 138.8 (2 C); 135.3 (C); 144.6 (C); 150.6 (C).

*1-({[(*tert*-Butyl)(dimethyl)silyl]oxy}methyl)-3-{[tris(1-methylethyl)silyl]oxy}benzene* (**8a**). Yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 0.19 (*s*, 6 H); 1.05 (*s*, 9 H); 1.20 (*d*, *J* = 7.0, 18 H); 1.45–1.29 (*m*, 3 H); 4.79 (*s*, 2 H); 6.85 (*dd*, *J* = 7.8, 2.1, 1 H); 7.04–6.98 (*m*, 2 H); 7.22–7.34 (*m*, 1 H). ^{13}C-NMR (75 MHz, CDCl$_3$): –5.3 (2 MeSi); 12.7 (3 CH); 17.9 (6 Me); 18.0 (C); 25.9 (3 Me); 64.7 (CH$_2$); 117.5 (CH); 118.4 (CH); 118.5 (CH); 129.0 (CH); 143.0 (C); 156.1 (C).

3-{[Tris(1-methylethyl)silyl]oxy}benzenemethanol (**8b**). Yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.10 (*d*, *J* = 7.2, 18 H); 1.33–1.18 (*m*, 3 H); 1.77 (*s*, OH); 4.61 (*s*, 2 H); 6.80 (*d*, *J* = 8.0, 1 H); 6.90 (*d*, *J* = 8.0, 2 H); 7.19 (*t*, *J* = 8.0, 1 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 12.7 (3 CH); 17.9 (6 Me); 65.2 (CH$_2$); 118.4 (CH); 119.0 (CH); 119.4 (CH); 129.5 (CH); 142.5 (C); 156.3 (C).

*(*tert*-Butyl)[3-({[(*tert*-butyl)(dimethyl)silyl]oxy}methyl)phenoxy]diphenylsilane* (**8c**). Yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 0.0 (*s*, 6 H); 0.87 (*s*, 9 H); 1.1 (*s*, 9 H); 4.57 (*s*, 2 H); 6.62 (*dd*, *J* = 8.0, 2.0, 1 H); 6.80 (*d*, *J* = 2.0, 1 H); 6.83 (*s*, 1 H); 7.03 (*t*, *J* = 8.0, 1 H); 7.43–7.32 (*m*, 6 H); 7.72 (*d*, *J* = 8.0, 4 H). ^{13}C-NMR (75 MHz, CDCl$_3$): –5.3 (2 Me); 18.4 (C); 19.5 (C); 25.9 (3 Me); 26.6 (3 Me); 64.7 (CH$_2$); 117.5 (CH); 118.2 (CH); 118.7 (CH); 127.7 (CH); 128.9 (2 CH); 129.8 (4 CH); 133.1 (2 C); 135.5 (4 CH); 142.9 (C); 155.6 (C).

*(3-{[(*tert*-Butyl)(diphenyl)silyl]oxy}phenyl)methanol* (**8d**). Yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.1 (*s*, 9 H); 1.58 (*s*, OH); 4.48 (*s*, 2 H); 6.62 (*dd*, *J* = 8.0, 2.0, 1 H); 6.81 (*t*, *J* = 7.5, 2 H); 7.02 (*t*, *J* = 7.8, 1 H); 7.43–7.31 (*m*, 6 H); 7.70 (*d*, *J* = 7.5, 4 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 19.5 (C); 26.6 (3 Me); 65.1 (CH$_2$); 118.3 (CH); 118.9 (CH); 119.6 (CH); 127.8 (CH); 129.3 (2 CH); 129.9 (4 CH); 132.9 (2 C); 135.5 (4 CH); 132.9 (2 C); 135.5 (4 CH); 142.3 (C); 155.8 (C).

*(3a*R*,4*S*,5*R*,6a*S*)-5-{[(*tert*-Butyl)(diphenyl)silyl]oxy}-3,3a,4,5,6,6a-hexahydro-4-(hydroxymethyl)-2*H*-cyclopenta[*b*]furan-2-one* (**9**). White solid. M.p. 141–144°. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.06 (*s*, 9 H); 1.61 (*s*, OH); 2.04 (*q*, *J* = 5.0, 3 H); 2.55 (*d*, *J* = 2.4, 1 H); 2.59 (*d*, *J* = 2.4, 1 H); 2.70–2.64 (*m*, 1 H); 2.80 (*dd*, *J* = 17.4, 10.0, 1 H); 3.38 (*t*, *J* = 6.3, 2 H); 4.10 (*q*, *J* = 5.1, 1 H); 7.41–7.39 (*m*, 7 H); 7.68–7.66 (*m*, 5 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 19.0 (C); 26.9 (3 Me); 35.7 (CH$_2$); 39.0 (CH$_2$); 40.8 (CH); 56.8 (CH); 62.1 (CH$_2$); 75.6 (CH); 84.2 (CH); 127.7 (2 CH); 129.9 (4 CH); 133.4 (2 CH); 135.9 (2 CH); 177.5 (C=O).

*(3a*R*,4*S*,5*R*,6a*S*)-4-({[(*tert*-Butyl)(dimethyl)silyl]oxy}methyl)-5-{[(*tert*-butyl)(diphenyl)silyl]oxy}-3,3a,5,5,6,6a-hexahydro-2*H*-cyclopenta[*b*]furan-2-one* (**9a**). Colorless oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 0.01 (*s*, 6 H); 0.87 (*s*, 9 H); 1.12 (*s*, 9 H); 2.08–2.06 (*m*, 3 H); 2.75–2.41 (*m*, 2 H); 2.88 (*dd*, *J* = 18.6, 11.0, 1 H); 3.33 (*dd*, *J* = 10.0, 6.0, 1 H); 3.48 (*dd*, *J* = 10.0, 6.0, 1 H); 4.15 (*q*, *J* = 5.0, 1 H); 7.51–7.41 (*m*, 7 H); 7.72 (*d*, *J* = 7.5, 4 H). ^{13}C-NMR (75 MHz, CDCl$_3$): –5.6 (Me); –5.6 (Me); 18.1 (C); 19.0 (C); 25.8 (3 Me); 26.9 (3 Me); 35.9 (CH$_2$); 39.9 (CH$_2$); 41.1 (CH); 57.2 (CH); 63.32 (CH$_2$); 76.3 (CH); 84.7 (CH); 127.7 (2 CH); 129.7 (4 CH); 133.6 (2 C); 135.8 (4 CH); 177.4 (C=O).

*2-[(1*R*,5*S*)-5-{[(*tert*-Butyl)(diphenyl)silyl]oxy}cyclopent-2-en-1-yl]ethanol* (**10**). Colorless oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 1.1 (*s*, 9 H); 1.83–1.71 (*m*, 1 H); 2.06–1.93 (*m*, 2 H); 2.19 (*dd*, *J* = 17.0, 7.5, 1 H); 2.29–2.26 (*m*, 1 H); 2.34–2.31 (*m*, 1 H); 2.63–2.59 (*m*, 1 H); 3.78–3.64 (*m*, 1 H); 4.54 (*q*, *J* = 7.2, 1 H); 5.59–5.56 (*m*, 1 H); 5.66–5.63 (*m*, 1 H); 7.43–7.35 (*m*, 6 H); 7.70–7.66 (*m*, 4 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 19.3 (C); 27.0 (3 Me); 31.7 (CH$_2$); 39.8 (CH$_2$); 45.6 (CH); 61.4 (CH$_2$); 75.2 (CH); 127.6 (2 CH); 127.7 (2 CH); 128.4 (CH); 129.7 (CH); 129.7 (CH); 133.0 (CH); 133.7 (C); 134.2 (C); 135.7 (2 CH); 135.8 (2 CH).

*(*tert*-Butyl){[(1*S*,2*R*)-2-(2-{[(*tert*-butyl)(dimethyl)silyl]oxy}ethyl)cyclopent-3-en-1-yl]oxy}diphenylsilane* (**10a**). Thick yellow oil. ^{1}H-NMR (300 MHz, CDCl$_3$): 0.02 (*s*, 6 H); 0.85 (*s*, 9 H); 1.01 (*s*, 9 H); 1.61–1.51 (*m*, 1 H); 2.22–1.94 (*m*, 3 H); 2.57–2.47 (*m*, 1 H); 3.71–3.57 (*m*, 2 H); 4.44 (*q*, *J* = 7.0, 1 H); 5.50–5.45 (*m*, 1 H); 5.67–5.62 (*m*, 1 H); 7.39–7.25 (*m*, 7 H); 7.65–7.58 (*m*, 5 H). ^{13}C-NMR (75 MHz, CDCl$_3$): 5.1 (2 Me); 18.4 (C); 19.4 (C); 26.0 (3 Me); 27.0 (3 Me); 31.9 (CH$_2$); 39.9 (CH$_2$); 45.3 (CH); 62.2 (CH$_2$); 75.3 (CH); 127.5 (2 CH); 127.6 (2 CH); 127.8 (CH); 129.5 (CH); 129.6 (CH); 133.8 (CH); 134.1 (2 C); 134.8 (CH); 135.6 (CH); 135.78 (CH); 135.84 (CH).

1-(4-Hydroxyphenyl)propan-1-one (**11**). White solid. M.p. 147–149°. ^{1}H-NMR (300 MHz, $CDCl_3$/DMSO): 1.07 (*t*, *J* = 7.0, 3 H); 2.82 (*q*, *J* = 7.0, 2 H); 6.77 (*d*, *J* = 9.0, 2 H); 7.74 (*d*, *J* = 9.0, 2 H). ^{13}C-NMR (75 MHz, $CDCl_3$/DMSO): 7.5 (Me); 30.0 (CH_2); 114.3 (2 CH); 127.5 (C); 129.2 (2 CH); 161.0 (C); 198.0 (C=O). EI-MS: 150 (M^+), 120 (2), 93 (20), 65 (10), 63 (3), 38 (2), 26 (2).

*1-(4-{[*tert*-Butyl(dimethyl)silyl]oxy}phenyl)propan-1-one* (**11a**). White solid. M.p. 20°. ^{1}H-NMR (300 MHz, $CDCl_3$): 0.2 (*s*, 6 H); 0.9 (*s*, 9 H); 1.19 (*t*, *J* = 7.2, 3 H); 2.93 (*q*, *J* = 7.3, 2 H); 6.86 (*d*, *J* = 8.9, 2 H); 7.89 (*d*, *J* = 8.9, 2 H). ^{13}C-NMR (75 MHz, $CDCl_3$): −4.4 (2 MeSi); 8.4 (Me); 18.2 (C); 25.5 (3 Me); 31.3 (CH_2); 119.8 (2 CH); 130.1 (2 CH); 130.5 (C); 160.0 (C); 199.5 (C=O). EI-MS: 264 (M^+), 235 (40), 207 (100), 179 (17), 151 (15), 135 (10), 121 (5), 105 (2), 89 (5), 73 (7), 57 (11).

1-(4-{[Tris(1-methylethyl)silyl]oxy}phenyl)propan-1-one (**11b**). Thick yellow liquid. ^{1}H-NMR (300 MHz, $CDCl_3$): 1.12–1.05 (*m*, 18 H); 1.31–1.23 (*m*, 3 H); 2.98–2.91 (*m*, 2 H); 6.90 (*d*, *J* = 9.0, 2 H); 7.90–7.87 (*m*, 2 H). ^{13}C-NMR (75 MHz, $CDCl_3$): 8.4 (Me); 13.0 (3 CH); 17.0 (6 Me); 31.3 (CH_2); 119.6 (2 CH); 130.1 (2 CH); 130.3 (C); 160.4 (C); 199.5 (C=O). EI-MS: 306 (M^+), 263 (38), 235 (22), 207 (33), 151 (6), 135 (6), 121 (6), 89 (25), 57 (100), 43 (20).

Vanillin (=*4-Hydroxy-3-methoxybenzaldehyde*; **12**). Thick yellow solid. M.p. 80–82°. ^{1}H-NMR (300 MHz, $CDCl_3$): 3.93 (*s*, 3 H); 6.45 (*s*, OH); 7.02 (*d*, *J* = 8.4, 1 H); 7.42–7.39 (*m*, 2 H); 9.8 (*s*, 1 H). ^{13}C-NMR (75 MHz, $CDCl_3$): 56.0 (Me); 108.8 (CH); 114.4 (CH); 127.4 (CH); 129.7 (C); 147.1 (C); 151.7 (C); 190.9 (C=O). EI-MS: 152 (M^+), 151 (100), 137 (5), 123 (20), 109 (22), 93 (4), 81 (40), 65 (12), 53 (22), 39 (12).

*4-{[(*tert*-Butyl)(dimethyl)silyl]oxy}-3-methoxybenzaldehyde* (**12a**). Brown liquid. ^{1}H-NMR (300 MHz, $CDCl_3$): 0.1 (*s*, 6 H); 0.9 (*s*, 9 H); 3.8 (*s*, 3 H); 6.95 (*d*, *J* = 8.0, 1 H); 7.39–7.35 (*m*, 2 H); 9.8 (*s*, H). ^{13}C-NMR (75 MHz, $CDCl_3$): −4.5 (2 Me); 18.4 (C); 25 (3 Me); 55.4 (Me); 110.1 (CH); 120.6 (CH); 126.1 (CH); 130.9 (C); 151.3 (C); 151.6 (C); 190.9 (C=O). EI-MS: 267 ($[M+1]^+$), 251 (3), 225 (10), 209 (85), 194 (100), 179 (6), 165 (5), 73 (4).

*4-{[(*tert*-Butyl)(diphenyl)silyl]oxy}-3-methoxybenzaldehyde* (**12b**). Yellow oil. ^{1}H-NMR (300 MHz, $CDCl_3$): 1.0 (*s*, 9 H); 3.6 (*s*, 3 H); 7.39–7.35 (*m*, 9 H); 7.73–7.67 (*m*, 5 H); 9.7 (*s*, 1 H). ^{13}C-NMR (75 MHz, $CDCl_3$): 19.0 (C); 26.5 (3 Me); 109–135 (13 CH); 151.1 (2 C); 151.3 (2 C); 191.0 (C=O). EI-MS: 391 ($[M+1]^+$), 333 (70), 318 (70), 261 (10), 217 (55), 199 (100), 152 (30), 139 (60), 79 (70), 63 (11).

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