58. Photochemical Reactions

147th Communication[1])

Further Investigation of the Photochemistry of 5,6-Epoxy-5,6-dihydro-β-ionone: Product Formation *via* a Carbonyl-Ylide Intermediate

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On π,π*-excitation of the epoxyenone (*E*)-**1** (λ = 254 nm, MeCN), in addition to the previously isolated compounds **2**–**9**, the new products **10**–**12** derived from the ylide intermediate **c** were isolated. Further evidence for the ylide **c** was obtained by the rapid racemization of the optically active epoxyenone (–)-(*E*)-**1**.

1. Introduction. – Several years ago, we have found that on n,π*-excitation (λ > 347 nm, pentane) of 5,6-dihydro-5,6-epoxy-β-ionone ((*E*)-**1**), the main reaction is cleavage of the C(γ)–O bond of the oxirane ring ((*E*)-**1**→**a**) leading to products **2**, (*E*/*Z*)-**3**, and (*E*/*Z*)-**4** [2]. On the other hand, on π,π*-excitation (λ = 254 nm, pentane) of (*E*)-**1**, compounds **5**–**9** were additionally isolated *(Scheme)*. Whereas compounds **5** and **6** presumably arise from the carbene intermediate **b**, the enol-ethers **7** and **8** may be formed *via* the carbonyl-ylide intermediate **c**. The furan **9**, however, was shown to be formed in an acid-catalyzed or thermal reaction from the epoxyenone (*Z*)-**1**.

The ylide **c** was detected on laser flash photolysis of (*E*)-**1** and its lifetime τ = 22 μs (MeCN) and 11 μs (pentane) was found to be rather long compared to related epoxyenones in the ionone series [3]. On the basis of these findings, it was surprising that on photolysis of (*E*)-**1** (λ = 254 nm, pentane), the ylide products **7** and **8** had been isolated in only small yields of 1 and 7%, respectively [2]. Furthermore, in recent studies of the photolyses of epoxyenones related to (*E*)-**1**, new types of ylide products were isolated which, however, proved to be acid-sensitive and unstable on repeated chromatography [4] [5]. In view of these facts, it was obviously of interest to investigate the photolysis of (*E*)-**1** under following experimental conditions: *a)* in a more polar solvent such as MeCN, *b)* in the presence of the ylide-trapping agents MeOH and H_2O, and *c)* using flash chromatography [6] for the separation of the acid-sensitive photoproducts.

[1]) 146th Communication: see [1].

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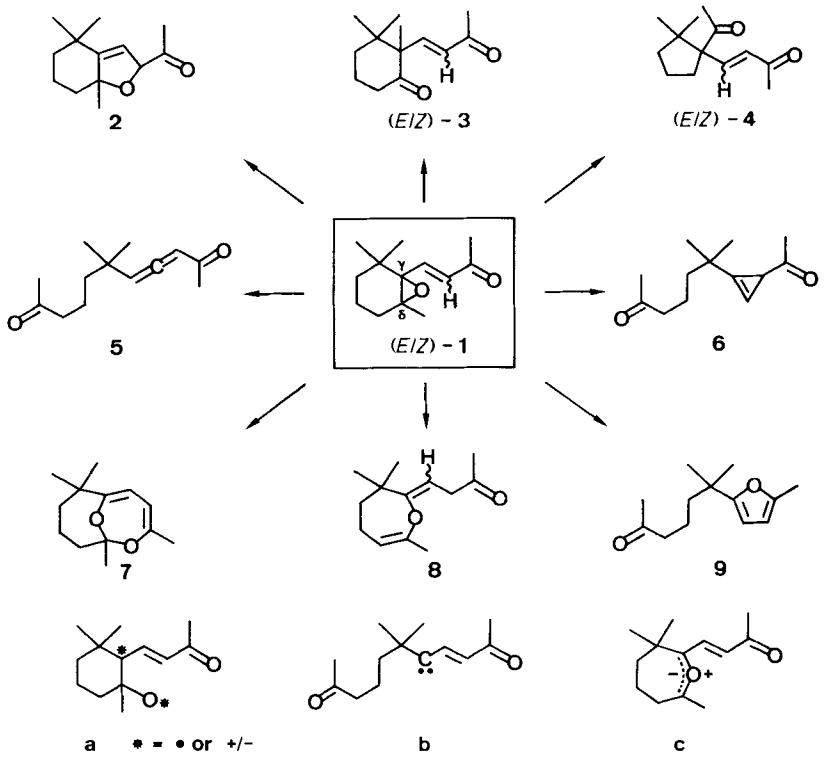
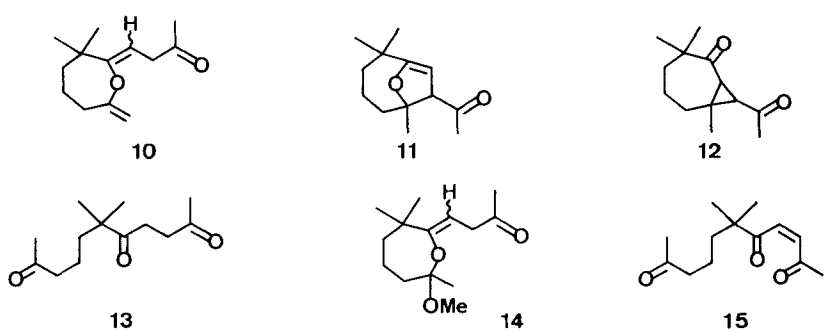
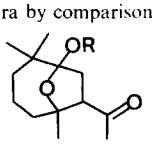


2. Results and Discussion. – The results of the photolyses of (*E*)-**1** are given in the *Table*.

On π,π^*-excitation of (*E*)-**1** in MeCN, after flash chromatography, indeed the three new photoproducts **10**, **11**, and **12** could be isolated in addition to the known compounds **2**–**9**, and the hydrolysis product **13** [2] [7][3]).



[3]) The structures of the new compounds **10**–**12** were deduced from their spectra by comparison with that of known analogs [4] [5]. The divinyl ether **10** was previously obtained as the main product (78%) on thermolysis of (*E*)-**1** [8]. The bicyclic dihydrofuran **11** was transformed to the mixed acetal **16** and the hemiacetal **17** by treatment with oxalic acid in MeOH and H_2O, respectively.



16 R = Me
17 R = H

Table. *Results of the Photolyses of* (E)-**1** (λ = 254 nm; r.t.)

Solvent	Conversion	Product Distribution [%][a]														
	[%]	**2**	(*E*)-**3**	(*Z*)-**3**	(*E*)-**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
MeCN	90	4	5	5	2	5	6	0.5	1	9	3	7	3	2	–	–
Pentane	83	7	–	2	2	17	9	–	4	26	2	3	2	9	–	–
MeOH	88	–	3	–	–	–	1	–	–	15	–	–	–	1	58	–
MeCN/H_2O	69	2	7	4	–	3	trace	–	–	4	–	–	–	38	–	2
MeCN[b]	98	2	2	1	–	4	2	–	–	4	–	–	–	2	–	3

[a] Yields were determined after chromatography on SiO_2 by ^{1}H-NMR and GC of the fractions and are based on converted starting material.

[b] Saturated with O_2; −30°.

On photolysis of (*E*)-**1** in pentane, **10**–**12** were also obtained, however, in somewhat smaller yields *(Table)*. On the other hand, photolysis of (*E*)-**1** in MeOH led to the acetal **14** as the main product, whereas the photolysis of (*E*)-**1** in MeCN/H_2O gave the triketone **13** as the main product. The latter is presumably formed by isomerization of the hemiacetal corresponding to **14**. Furthermore, photolysis of (*E*)-**1** in MeCN in the presence of O_2 gave the above photoproducts only in low yields, and in addition to the oxidation product **15** as well as a mixture of acids of unknown structure.

The aforementioned results show that on π,π^*-excitation of the epoxyenone (*E*)-**1**, the main pathway is cleavage of the oxirane to the ylide intermediate **c** *(Scheme)*. In the absence of a trapping agent, **c** rapidly undergoes a ring closure to starting material, or slowly reacts to compounds **7**, **8**, **10**, and **11**[4]). This pathway was also evidenced by the photolysis of optically active epoxyenone (−)-(*E*)-**1** [10] causing rapid racemization[5]).

In the presence of an ylide-trapping agent such as MeOH (or H_2O), the formation of the acetal **14** suppresses the formation of the ylide products **7**, **8**, and **10**–**12**, as well as that of the carbene products **5** and **6**.

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Experimental Part

General. See [11], except as noted below. Anal. GC was performed using a 25 m × 0.33 mm *Ucon HB-5100* glass capillary. Column chromatographies (CC) were carried out on silica gel (SiO_2) *60 Merck*, 0.040–0.063 mm, 230–400 mesh ASTM. Analytically pure samples were obtained, in general, after repeated CC, in some cases further purification was necessary on HPLC (*Du Pont Instruments Model 830*, UV detector), using a 25 cm × 23.6 mm SiO_2 column, or by prep. GC. ^{1}H-NMR spectra were taken on a Bruker *WP-80/CW* (80 MHz) instrument in $CDCl_3$ solns. Yields reported are based on converted starting material.

1. Photolysis Experiments. – 1.1. π,π^*-*Excitation of* (E)-**1**. 1.1.1. *In MeCN.* A soln. of (*E*)-**1** (725 mg, 3.49 mmol) and hexadecane (50 µl) in MeCN (200 ml) was irradiated with a Hg low-pressure lamp for 100 min (quartz,

[4]) The bicyclic diketone **12** is presumably a secondary photoproduct of the bicyclic dihydrofuran **11** as was previously proven for a compound related to **12** [9].

[5]) Photolysis of (−)-(*E*)-**1** [10] ($[\alpha]_D = -93°$) up to 22 and 58% conversion gave recovered (−)-(*E*)-**1** with $[\alpha]_D = -78°$ and −33°, respectively (see *Exper. Part*).

90% conversion). The mixture was chromatographed on SiO_2 (75 g) with Et_2O to yield fractions containing (*Z*)-**3** (35 mg, 5%), **6** (40 mg, 6%), **13** (10 mg, 2%), and a nonpolar mixture which was further chromatographed on SiO_2 (75 g) with hexane (20 ml), hexane/AcOEt (4:1, 500 ml), and hexane/AcOEt (3:2, 500 ml) to yield fractions containing: (*E*)-**1** (69 mg), **2** (25 mg, 4%), (*E*)-**3** (30 mg, 5%), (*E*)-**4** (10 mg, 2%), **5** (35 mg, 5%), **7** (3 mg, 0.5%), **8** (7 mg, 1%), **9** (60 mg, 9%), **10** (18 mg, 3%), **11** (45 mg, 7%), and **12** (20 mg, 3%). An anal. sample of **12** was obtained by CC on SiO_2 (acetone/CH_2Cl_2 1:99).

(2,2,6-Trimethyl-9-oxabicyclo[4.2.1]non-8-en-7-yl) Methyl Ketone (**11**). ^{1}H-NMR: 4.01 (*d*, *J* = 2); 5.13 (2*d*, *J* = 2, H–C(7), H–C(8)).

8-Acetyl-3,3,7-trimethylbicyclo[5.1.0]octan-2-one (**12**). B.p. 120°/0.03 Torr. UV (3.55 mg in 2 ml pentane): 285 (156). IR: 2960*s*, 2925*s*, 2860*m*, 1715*s*, 1692*s*, 1455*m*, 1420*m*, 1380*s*, 1358*s*, 1325*m*, 1298*w*, 1275*w*, 1046*w*, 1210*w*, 1188*w*, 1168*s*, 1148*w*, 1079*m*, 1063*m*, 1038*w*, 1018*w*, 966*w*, 953*m*, 920*w*, 900*w*, 852*w*. ^{1}H-NMR: 1.04, 1.08, 1.13 (3*s*, 2 CH_3–C(3), CH_3–C(7)); 1.0–2.2 (*m*, 2 H–C(4), 2 H–C(5), 2 H–C(6)); 2.28 (*s*, CH_3CO); 2.89 (*AB*-system, *J* = 8, δ_A = 2.72, δ_B = 3.05, H–C(1), H–C(8)). ^{13}C-NMR: 20.6, 22.4, 28.8, 31.6 (4*q*, 2 CH_3–C(3), CH_3–C(7), CH_3CO); 23.5, 30.0, 40.3 (3*t*, C(4), C(5), C(6)); 42.4, 42.7 (2*d*, C(1), C(8)); 33.4 (*s*, C(7)); 48.1 (*s*, C(3)); 202.2, 205.2 (2*s*, C(2), CH_3CO). MS: 208 (9, M^+, $C_{13}H_{20}O_2$), 165 (11), 138 (12), 123 (17), 122 (29), 111 (11), 110 (16), 109 (18), 107 (10), 96 (12), 95 (26), 93 (10), 81 (32), 69 (62), 67 (17), 55 (17), 53 (12), *43* (100), 41 (43). Anal. calc. for $C_{13}H_{20}O_2$ (208.29): C 74.96, H 9.68; found: C 74.69, H 9.50.

1.1.2. *In Pentane.* A soln. of (*E*)-**1** (727 mg, 3.50 mmol) and hexadecane (250 mg) in pentane (180 ml) was irradiated as described in *Sect. 1.1.1* (83% conversion). The solvent was evaporated and the mixture chromatographed on SiO_2 (75 g) with hexane (100 ml), hexane/AcOEt (9:1, 250 ml), (4:1, 250 ml) and (3:2, 250 ml) to produce fractions containing: (*E*)-**1** (125 mg), **2** (40 mg, 7%), (*Z*)-**3** (15 mg, 2%), (*E*)-**4** (10 mg, 2%), **5** (105 mg, 17%), **6** (55 mg, 9%), **8** (23 mg, 4%), **9** (161 mg, 26%), **10** (12 mg, 2%), **11** (17 mg, 3%), **12** (10 mg, 2%), and **13** (55 mg, 9%).

1.1.3. *In MeOH.* A soln. of (*E*)-**1** (448 mg, 2.15 mmol) and K_2CO_3 (50 mg, 0.362 mmol) in MeOH (180 ml) was irradiated as described in *Sect. 1.1.1* for 45 min (88% conversion). The solvent was then evaporated, azeotroped with benzene, filtered through SiO_2 (Et_2O) and chromatographed on SiO_2 (75 g), eluting with hexane (20 ml), hexane/AcOEt (9:1, 250 ml), (4:1, 250 ml), (3:2, 500 ml) to yield fractions containing: (*E*)-**1** (52 mg), (*E*)-**3** (10 mg, 3%), **6** (5 mg, 1%), **9** (60 mg, 15%), **13** (3 mg, 1%), and **14** (263 mg, 58%).

4-(3'-Methoxy-3',7',7'-trimethyl-2'-oxa-1'-cycloheptylidene)-2-butanone (**14**). UV (5.907 mg in 2 ml pentane): 279 (92). IR: 2960*s*, 2905*m* (sh), 2880*m* (sh), 2830*w*, 1720*s*, 1705*s*, 1662*w*, 1460*m*, 1448*m*, 1395*w*, 1385*m*, 1376*m*, 1353*m*, 1302*w*, 1277*w*, 1210*m* (br.), 1182*m*, 1158*m*, 1123*w*, 1095*m*, 1082*m*, 1050*m* (sh), 1040*m*, 1015*w*, 987*w*, 955*w*, 922*w*, 908*w*. ^{1}H-NMR: 1.08, 1.14 (2*s*, 2 CH_3–C(7')); 1.48 (*s*, CH_3–C(3')); 0.9–1.7 (*m*, 2 H–C(4'), 2 H–C(5'), 2 H–C(6')); 2.15 (*s*, 3 H–C(1)); 3.26 (*d*, *J* = 7, 2 H–C(3)); 3.43 (*s*, CH_3O); 5.20 (*t*, *J* = 7, H–C(4)). ^{13}C-NMR: 22.1, 28.1, 28.5, 29.2 (4*q*, C(1), CH_3–C(3'), 2 CH_3–C(7')); 49.1 (*q*, CH_3O); 19.2, 36.5, 41.6 (3*t*, C(4'), C(5'), C(6')); 106.4 (*d*, C(4)); 39.1 (*s*, C(7')); 104.9 (*s*, C(3')); 160.7 (*s*, C(1')); 206.4 (*s*, C(2)). MS: 240 (< 1, M^+, $C_{14}H_{26}O_3$), 142 (88), 123 (13), 109 (21), 99 (77), 98 (84), 85 (70), 84 (13), 83 (10), 72 (45), 71 (27), 69 (73), 67 (14), 55 (37), *43* (100), 41 (39).

1.1.4. *In MeCN/H_2O.* A soln. of (*E*)-**1** (310 mg, 1.49 mmol) in MeCN (50 ml) and H_2O (50 ml) was irradiated as described in *Sect. 1.1.1* for 25 min (69% conversion). The solvent was evaporated and the aq. layer treated with NaCl (aq. sat., 30 ml) and extracted with Et_2O to yield 260 mg of a mixture, which was chromatographed on SiO_2 (75 g) with hexane (10 ml); hexane/AcOEt (3:2, 500 ml); (4:1, 500 ml) to yield fractions containing: (*E*)-**1** (97 mg), (*Z*)-**1** (13 mg, 6%; decomposed to **9**), **2** (4 mg, 2%), (*E*)-**3** (14 mg, 7%), (*Z*)-**3** (9 mg, 4%), **5** (6 mg, 3%), **6** (trace), **9** (9 mg, 4%), **13** (87 mg, 38%), and **15** (4 mg, 2%).

(Z)-6,6-Dimethyl-3-undecen-2,5,10-trione (**15**). B.p. 130°/0.1 Torr. IR: 2960*m*, 2930*m*, 1710*s* (br.), 1688*s*, 1605*w*, 1467*w*, 1455*w*, 1406*w*, 1385*m*, 1355*m*, 1175*m*, 1070*w*, 976*w*. ^{1}H-NMR: 1.17 (*s*, 2 CH_3–C(6)); 1.1–1.7 (*m*, 2 H–C(7), 2 H–C(8)); 2.12, 2.29 (2*s*, 3 H–C(1), 3 H–C(11)); 2.2–2.6 (*m*, 2 H–C(9)); 6.46 (*AB*-system, *J* = 12, δ_A = 6.38, δ_B = 6.54, H–C(3), H–C(4)).

1.1.5. *In MeCN under O_2 at −30°.* A soln. of (*E*)-**1** (1.0 g, 4.81 mmol) in MeCN (200 ml) was irradiated with a Hg low-pressure lamp through a quartz finger, which was cooled with a N_2 stream, while the soln. was cooled to −30° and saturated with O_2. After 4 h of irradiation, the solvent was evaporated and the residue dissolved in Et_2O and extracted with $NaHCO_3$ (5% aq.) to give a neutral fraction (702 mg). The aq. soln. was acidified and reextracted into Et_2O to give a mixture of carboxylic acids (127 mg). The neutral fraction was chromatographed on SiO_2 (75 g) with Et_2O to give two fractions which were separately chromatographed with hexane/AcOEt gradients on SiO_2 to give fractions containing: (*E*)-**1** (15 mg), **2** (15 mg, 2%), (*E*)-**3** (20 mg, 2%), **5** (40 mg, 4%), **6** (20 mg, 2%), **9** (35 mg, 4%), **13** (20 mg, 2%), and **15** (30 mg, 3%). An anal. sample of **15** was obtained by CC on SiO_2 first with DME/hexane (1:4), then again with an acetone/CH_2Cl_2 gradient (up to 1:24).

1.2. *π,π*-Excitation of (−)-(E)-***1**. *a)* A soln. of (−)-(*E*)-**1** [10] ($[\alpha]_D = -93°$, $c = 1.0$, $CHCl_3$, 32 mg) and hexadecane (2 mg) in MeCN (5 ml) was irradiated as described in *Sect. 1.1.1* for 30 min. GC showed 42% (−)-(*E*)-**1**. CC on SiO_2 (12 g) with hexane/AcOEt 4:1 yielded (−)-(*E*)-**1** (12 mg, 95% pure by GC, $[\alpha]_D = -33°$, $c = 1.2$, $CHCl_3$).

b) A soln. of (−)-(*E*)-**1** (32 mg) and undecanenitrile (15 mg) in MeCN (10 ml) was irradiated as described in *Sect. 1.1.1* for 18 min. GC showed 77% (−)-(*E*)-**1**. CC as above led to (−)-(*E*)-**1** (20 mg, $[\alpha]_D = -78°$, $c = 1.0$, $CHCl_3$).

c) A soln. of (−)-(*E*)-**1** (32 mg) and undecanenitrile (15 mg) was irradiated as described in *Sect. 1.1.1* for 5 min. GC indicated 96% (−)-(*E*)-**1**. CC as above yielded (−)-(*E*)-**1** (30 mg, $[\alpha]_D = -95°$, $c = 1.0$, $CHCl_3$).

2. Additional Experiments. 2.1. *Methanolysis of* **11**. A soln. of (*E*)-**1** (500 mg, 2.40 mmol) in MeCN was irradiated as described above. After 90 min, the solvent was evaporated and the residue azeotroped with toluene before treatment with oxalic acid (15 mg) in dry MeOH (15 ml). After 25 h, GC indicated complete disappearance of **11**, so the mixture was worked up between $NaHCO_3$ (5%, aq.) and Et_2O. The org. layer was washed with H_2O, dried ($MgSO_4$), evaporated and azeotroped with EtOH and toluene to remove traces of H_2O. CC on SiO_2 (75 g) with AcOEt/hexane (1:4, 500 ml) and (2:3, 1000 ml). One fraction (186 mg) containing both (*E*)-**1** and **16** was further chromatographed on SiO_2 (20 g) with hexane (5 ml), and Et_2O/hexane (1:4, 250 ml) to yield pure **16** (65 mg, 11%).

(1-Methoxy-2,2,6-trimethyl-9-oxabicyclo[4.2.1]non-7-yl) Methyl Ketone (**16**). B.p. 100°/0.08 Torr. UV (2.045 mg in 10 ml pentane): 224 (sh, 600). UV (2.352 mg in 2 ml pentane): 287 (40). IR: 2970*s*, 2950*s*, 2930*s*, 2910*s* (sh), 2870*m*, 2850*m*, 2820*w*, 1708*s*, 1470*m*, 1450*m*, 1438*m*, 1382*m*, 1375*m*, 1369*s*, 1315*w*, 1299*w*, 1260*m*, 1235*m*, 1180*m*, 1162*m*, 1153*m*, 1138*s*, 1127*m*, 1098*s*, 1070*m*, 1060*m*, 1042*m*, 1028*m*, 1008*m*, 950*m*, 940*m*. ^{1}H-NMR: 0.90, 1.05 (2*s*, $2\,CH_3$–C(2)); 0.9–1.7 (*m*, 2 H–C(3), 2 H–C(4), 2 H–C(5)); 1.65 (*s*, CH_3–C(6)); 1.95 (*dd*, $J = 14, 8$, H–C(8)); 2.23 (*s*, CH_3CO); 2.57 (*dd*, $J = 14, 12$, H–C(8)); 3.06 (*dd*, $J = 12, 8$, H–C(7)); 3.26 (*s*, CH_3O). ^{13}C-NMR: 21.7, 25.0, 28.9, 31.0 (4*q*, 2 CH_3–C(2), CH_3–C(6), CH_3CO); 48.5 (*q*, CH_3O); 20.4, 30.9, 37.1, 41.7 (4*t*, C(3), C(4), C(5), C(8)); 63.4 (*d*, C(7)); 41.0 (*s*, C(2)); 81.2 (*s*, C(6)); 111.2 (*s*, C(1)); 206.0 (*s*, CH_3CO). MS: 240 (4, M^+, $C_{14}H_{24}O_3$), 197 (13), 140 (11), 123 (27), 113 (12), 111 (11), 110 (11), 109 (18), 97 (10), 95 (17), 81 (21), 69 (35), 67 (13), 55 (19), *43* (100), 41 (30). Anal. calc. for $C_{14}H_{20}O_3$ (240.30): C 69.96, H 10.07; found: C 69.90, H 9.96.

2.2. *Hydrolysis of* **16**. A soln. of **16** (12 mg, 0.05 mmol) and oxalic acid (20 mg) in MeOH (5 ml) and H_2O (5 ml) was heated at 60° for 12 h. The mixture was then separated between Et_2O and $NaHCO_3$ (5%, aq.), the Et_2O layer washed with H_2O, evaporated and chromatographed on SiO_2 (12 g, hexane/AcOEt 3:2) to yield recovered **16** (3 mg, 25%) and **17** (8 mg, 70%).

(1-Hydroxy-2,2,6-trimethyl-9-oxabicyclo[4.2.1]non-7-yl) Methyl Ketone (**17**). IR: 3610*w*, 2950*m* (sh), 2925*s*, 1707*s*, 1465*w*, 1440*w*, 1385*w*, 1376*m*, 1356*m*, 1262*w*, 1180*w*, 1160*w*, 1122*w*, 1065*m*, 1005*w*, 848*w*. ^{1}H-NMR: 0.98, 1.07 (2*s*, 2 CH_3–C(2)); 0.9–1.6 (*m*, 2 H–C(3), 2 H–C(4), 2 H–C(5)); 1.92 (*dd*, $J = 14, 8$, H–C(8)); 2.22 (*s*, CH_3CO); 2.63 (*dd*, $J = 14, 12$, H–C(8)); 3.20 (*dd*, $J = 12, 8$, H–C(7)).

2.3. *Catalytic Hydrogenation of* **15**. A soln. of (**15**, 10 mg) in EtOH (3 ml) was stirred with 10% Pd/C (20 mg) under H_2 for 12 h. It was filtered through *Celite* and evaporated to yield **13** [2] [7] (10 mg, 100%).

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