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Synthesis and reactivity of cyclic dienol-ethers

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Summary — Several dienes, synthesized from cyclic enol-ethers, were opposed to α-phenyl or α-alkyl sulfinylalkenones in Diels–Alder reactions. α-Phenylsulfinylalkenones gave unstable adducts which spontaneously lost phenylsulfenic acid, while the other dienophiles led to stable adducts in the form of diastereoisomeric mixtures.

Diels–Alder / water / sulfoxide / enol-ether

Résumé — Synthèse et réactivité d'éthers diénoliques cycliques. Plusieurs dièmes, synthétisés à partir d'éthers d'énols cycliques, sont opposés à des α-phényl ou α-alkyl-sulfinyl alcénones dans des réactions de Diels–Alder. Les α-phénylsulfinyl alcénones donnent des adduits instables qui spontanément perdent de l'acide benzène sulfénique, alors que les autres diénophiles conduisent à des adduits stables sous forme d'un mélange de diastéréoisomères.

Diels–Alder / eau / sulfoxyde / éther d'énol

Introduction

Diels–Alder reactions allow the formation of complex cyclic systems from two small molecules and in conjunction with the use of carbohydrate derivatives are a straightforward route to carbocyclic compounds eventually in optically active form. In previous works [1], we have described such reactions between lactonic dienes and ethylenic sulfoxides leading to structures of type I. However, these adducts are not easily transformed as we expected for the synthesis of products such as *dl*-ivangulin [2]. Particularly, selective reduction of the ketone group was not possible without attack on the lactone nucleus. Also we have examined the substitution of this cycle by a furan or a pyran ring (see fig 1).

Some examples of diene derivatives from cyclic enol-ethers are described in the literature. Among these, the first cited was isopropenyldihydropyran **1** [3], and more recently dieno-pyranoside **2** [4, 5] and analogues were synthesized. In the furan series, the first example **3** was reported by Fraser-Reid's group [6]. All of them underwent cycloaddition with maleic anhydride or maleimide, to give bicyclic compounds in high stereoselectivity. These results are obtained in standard conditions (mainly with benzene on reflux) and encouraged us to examine the reactivity of such dienes with ethylenic sulfoxides. In this paper, we describe the synthesis and Diels–Alder reactions of dienes **3–5** (see fig 2).

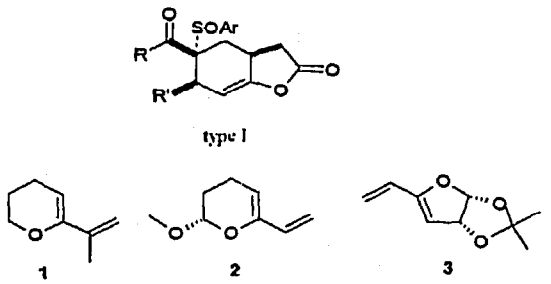
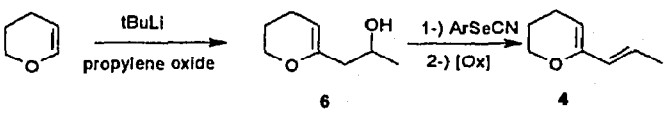


Fig 1

Fig 2

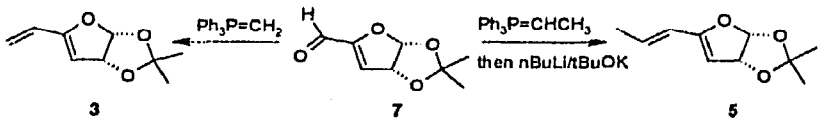
Preparation of dienes

Propenyldihydropyran **4** was prepared as shown in scheme 1, from dihydropyran. The two furans **3** and **5** were obtained from the same aldehyde **7** [6] by



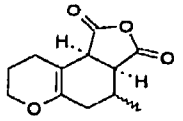
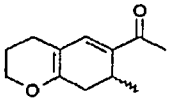
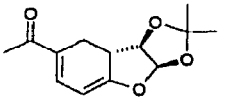
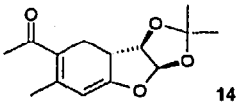
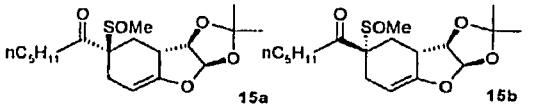
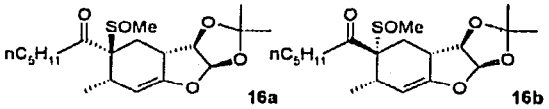
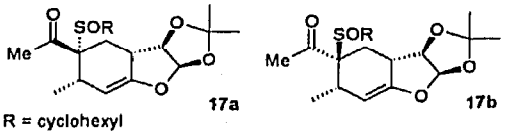
Scheme 1

* Correspondence and reprints



Scheme 2

Table I. Diels–Alder reactions for cyclic dienol-ethers.

Entry	*Diene*	*Dienophile*	*Adduct*	*Yields (ratio)*
1	4	Maleic anhydride	 11	63%
2	4	8	 12	60%
3	3	8	 13	59%
4	5	8	 14	51%
7	3	9	 15a 15b	52% (66:34)
5	5	9	 16a 16b	66% (64:36)
6	5	10	 17a 17b R = cyclohexyl	40% (95:5)

Wittig's reaction using standard conditions for **3** [6], but in adopting Schlosser's method [7] to obtain **5** with a good E/Z ratio (95:5) (see scheme 2).

Cycloadditions

Diels–Alder reactions were attempted with several dienophiles in water and at 6 °C (table I). The choice of water as a solvent was suggested by previous works on lactonic dienes. Dienophiles used in these reactions were maleic anhydride and phenyl- (or alkyl-) sulfinylbutenone (**8–10**) prepared by standard methods (see fig 3) [8]. Results of the Diels–Alder reactions are summarized in table I.

For diene **4**, good yields were obtained either with maleic anhydride or α-phenylsulfinylbutenone **8** (entries 1, 2). Only one adduct was isolated in each experiment. In the first case, the product resulted from *endo* mode addition. The double bond, initially formed, migrated into the 4,5 position as observed with similar reactions [5] and for entry 2 we isolated the diene **12** issued from elimination of sulfenic acid. Dienes **3** and **5**, recorded in entries 3 and 4, treated with α-(phenylsulfinyl)butenone gave also a single adduct in 59% and

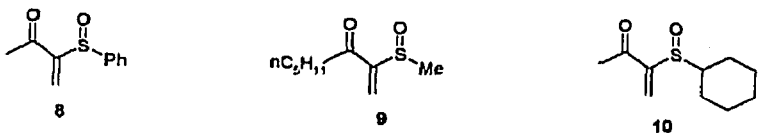


Fig 3

51% yield respectively. The structures of these compounds were assigned on the basis of their 400 MHz ^{1}H NMR spectra.

The configuration of the new stereocenter generated at C_4 in the adducts corresponded with a cycloaddition to the convex face of the diene. These results show the great tendency for elimination of the aromatic sulfinyl group in these series, in contrast with the lactonic series, and compromise the possibility of a selective reduction of ketone in the side chain. To overcome this problem two other dienophiles were tested, **9** and **10**, in which an alkyl group replaced the aromatic substituent on sulfur. We thought this route would minimize the elimination since alkylsulfinyl groups are more stable than their aromatic counterparts. Their syntheses were effected by standard methods, first using addition of alkyl sulfenic acid on acetylenic alcohol and then oxidation of the resulting product. Dienes **3** and **5** opposed to these dienophiles under the same conditions as above, led effectively to more stable adducts. A mixture of two stereoisomers was isolated in all cases. Coupling constants in ^{1}H NMR spectra of hydrogen atoms on C_4 and C_5 along with NOE effect measurements show that addition proceeded also on the convex face of the dienes. Oxidation with 3-chloroperbenzoic acid of adducts **15a** and **15b** led to two different sulfones, demonstrating that the configuration of carbon C-5 was not the same in the two adducts.

In summary, we observed that cyclic dienol-ethers were good dienes in Diels–Alder reactions with α-phenyl- or α-alkylsulfinylalkenones. Alkyl substituents at sulfoxide gave stable cycloadducts and allowed some modifications on the side chain. In particular we hope that selective reduction of the carbonyl group proceeds with good selectivity in order to introduce an asymmetric centre at this position.

Experimental section

General methods

^{1}H and ^{13}C NMR were recorded with a Bruker AC400 (400 MHz) spectrometer. Chemical shifts were reported in ppm (δ) relative to tetramethylsilane as internal standard, for solutions in $CDCl_3$; coupling constants (J) are given in Hz with the following abbreviations for splitting patterns: s = singlet, ps = pseudo-singlet, d = doublet, t = triplet, q = quartet, m = multiplet. High-resolution mass measurements were performed at the CRMPO (Rennes) with a Varian Mat 311 spectrometer. Melting points were measured using a Reichert apparatus and are uncorrected. IR spectra were recorded with a Genesis Matteson infrared spectrophotometer. Flash chromatography was performed on 230–400 mesh Merck Silica gel 60.

• *1-(3,4-Dihydro-2H-pyran-6-yl)propan-2-ol* **6**

A solution of *n*-BuLi 1.6 M in hexane (41 mL, 65.6 mmol) was added to 3,4-dihydro-2*H*-pyran (5 mL, 54 mmol) in THF at 0 °C. The solution was heated 2 h at 50 °C before cooling at 0 °C to introduce propylene oxide (17 mL, 240 mmol). The mixture was stirred 2.5 h at 50 °C. After cooling, water (30 mL) was added and the aqueous layer was extracted with diethyl ether, dried ($MgSO_4$) and evaporated under reduced pressure at 0 °C. The crude product was purified by chromatography on silica gel to give **6** (4.9 g, 34.5 mmol) as a colourless liquid. Yield 64%.

IR (film): 3 399, 1 675, 1 062 cm^{-1}.

^{1}H NMR δ (ppm): 1.13 (d, 3H, CH_3, J = 6.2 Hz); 1.76 (m, 2H, CH_2); 1.96 (m, 2H, CH_2); 2.04 et 2.14 (dm, 2H, CH_2, J^2 = 14.2 Hz); 2.39 (s, 1H, OH); 3.90 (m, 1H, CH); 3.95 (m, 2H, CH_2); 4.51 (t, 1H, CH, J = 3.6 Hz).

^{13}C NMR δ (ppm): 20.1, 22.2, 22.6, 43.9, 66.2, 66.2, 98.1, 152.0.

• *6-Prop-1-enyl-3,4-dihydro-2H-pyran* **4**

Under argon, at room temperature, tributylphosphine (1.3 mL) was added slowly to a mixture of alcohol **6** (0.6 g, 4.2 mmol) and (2-nitrophenyl) selenocyanate (1.2 g, 5.3 mmol) in 16 mL of THF. After 2 h at room temperature, the solution was concentrated under reduced pressure and chromatographed on silica gel (1:20; cyclohexane/ether: 9:1) to give 1.3 g (4 mmol) of seleno-intermediate. This product in methanol (11 mL) was added, at room temperature, to a mixture of $NaHCO_3$ (60 mg, 0.07 mmol) and $NaIO_4$ (300 mg, 1.4 mmol) in water (2 mL). After 1 h the mixture was poured in a saturated solution of $NaHCO_3$ (40 mL). The aqueous phase was extracted with pentane/ether (3:1) and the organic layer was washed with water and NaCl saturated before being dried ($MgSO_4$) and evaporated under reduced pressure at 0 °C. The crude product was purified on alumina (1:20; petroleum ether/ether: 83:17) to give diene **4** (156 mg, 1.3 mmol). Yield 30%.

IR (film): 1 648, 1 608 cm^{-1}.

^{1}H NMR δ (ppm): 1.71 (dd, 3H, J = 1.0 and 7.1 Hz); 1.80 (m, 2H); 2.06 (m, 2H); 4.01 (t, 2H, J = 5.1 Hz); 4.62 (t, 1H, J = 4.0 Hz); 5.72 (qd, 1H, J = 1.8 and 15.4 Hz); 5.88 (qd, 1H, J = 7.1 and 15.4 Hz).

^{13}C NMR δ (ppm): 17.6, 20.9, 22.3, 66.0, 100.2, 123.4, 127.1, 152.0.

• *3,5,6-Trideoxy-1,2-O-isopropylidene-α-D-glycerohepta-3,5-dienofuranose* **5**

Under argon atmosphere, a solution of *n*-BuLi 1.6 M in hexane (13 mL, 21 mmol) was added to a suspension of ethyltriphenylphosphonium iodide (8.3 g, 21 mmol) in diethyl ether (12 mL). The mixture was stirred 40 min, then cooled at −70 °C to add aldehyde **7** (2.2 g, 13 mmol) in diethyl ether (8 mL). After 1 h at this temperature a solution of *n*-BuLi 1.6 M in hexane (13 mL, 21 mmol) was added at −40 °C. Stirring was continued for one hour at −30 °C and potassium *tert*-butoxide (2.3 g, 21 mmol) was added, the resulting mixture was left one night at room temperature. After filtration on celite the solution was evaporated under reduced pressure and the residue chromatographed on silica gel (1:20; ether) to give diene **5** (1.48 g, 8.5 mmol) as a colourless liquid. Yield 64%.

IR (film): 3 056, 2 967, 1 604, 1 438, 1 249, 1 180, 1 120 cm^{-1}.

^{1}H NMR δ (ppm): 1.42 and 1.43 (2s, 6H); 1.81 (dd, 3H, J = 1.5 and 6.8 Hz); 4.99 (d, 1H, J = 2.3 Hz); 5.31 (dd, 1H, J = 2.3 and 5.3 Hz); 5.89 (dq, 1H, J = 1.5 and 15.4 Hz); 6.06 (d, 1H, J = 5.3 Hz); 6.23 (dq, 1H, J = 6.8 and 15.4 Hz).

HRMS calc for $C_{10}H_{14}O_3$ $(M)^+$ 182.09429, found 182.0947.

m/z (%): 182(28.57); 167(20.42); 154(10.20); 153(77.27); 125(42.11); 111(11.02); 97(45.45); 96(27.01); 95(72.27); 81(19.83); 69(49.76); 67(16.93); 58(16.49); 43(100); 41(42.12); 39(25.15); 28(25.25).

General procedure for Diels–Alder reactions

A mixture of diene (1 mmol) and dienophile (1 mmol) was added into 10 mL of water; the resulting suspension was stirred during one night at 6 °C.

• *4-Methyl-3a,5,7,8,9,9b-hexahydro-3H-furo[3,4-f][1]benzopyran-1,3(4H)-dione* **11**

Yield 63%. Purification on silica gel (1:30; cyclohexane/AcOEt: 9:1).

IR (film): 2 928, 1 775, 1 610, 1 475, 1 345, 1 201, 1 152 cm^{-1}.

^{1}H NMR δ (ppm): 1.34 (d, 3H, J = 6.9 Hz); 1.85–2.15 (m, 6H); 2.38 (m, 2H, J = 2.9 and 6.9 Hz); 3.28 (ddd, 1H, J = 1.0, 4.4 and 8.4 Hz); 3.48 (dd, 1H, J = 1.0 and 8.4 Hz); 3.35 (ddd, 1H, J = 3.9, 8.8 and 10.8 Hz); 4.02 (m, 1H, J = 1.5, 3.9 and 10.8 Hz).

^{13}C NMR δ (ppm): 17.4, 22.2, 22.5, 31.9, 28.0, 45.3, 46.4, 65.9, 96.8, 150.6, 170.3, 171.4.

• *1-(7-Methyl-3,4,7,8-tetrahydro-2H-1-benzopyran-6-yl)ethanone* **12**

Yield 60%. Purification on silica gel (1:30; cyclohexane/AcOEt: 9:1).

IR (film): 2 928, 1 708, 1 610, 1 428, 1 335, 1 211, 1 162 cm^{-1}.

^{1}H NMR δ (ppm): 0.93 (d, 3H, J = 6.9 Hz); 1.93 (m, 3H, J = 17.0 Hz); 2.16 (m, 2H); 2.27 (s, 3H); 2.61 (ddt, 1H, J = 3.1, 8.6 and 17.0 Hz); 3.02 (ddq, 1H, J = 1.4, 6.9 and 8.6 Hz); 4.10 (m, 2H, J = 7.0 and 11.3 Hz); 6.75 (s, 1H).

^{13}C NMR δ (ppm): 18.1, 22.5, 23.0, 24.9, 26.1, 34.1, 67.0, 102.9, 133.5, 139.3, 157.2, 196.0.

HRMS calc for $C_{12}H_{16}O_2$ $(M)^+$: 192.11502, found 192.1147.

m/z (%): 192(9.21); 177(15.76); 175(9.34); 149(19.33); 140(13.19); 97(10.21); 87(13.28); 77(12.09); 71(30.21); 69(57.75); 55(11.20); 43(100); 41(21.79); 39(15.97); 28(26.68); 18(46.13).

• *1-(2,2-Dimethyl-3a,8,8a,8b-tetrahydro-1,3-dioxolo[4,5-b]benzofuran-7-yl)ethanone* **13**

Yield 59%. Purification on silica gel (1:30; cyclohexane/AcOEt: 8:2).

$[\alpha]_D$ +148 (*c* 0.71, propanone).

IR (film): 3 020, 2 929, 1 650, 1 524, 1 460, 1 375, 1 241, 1 152, 1 075, 1 025 cm^{-1}.

^{1}H NMR δ (ppm): 1.44 and 1.49 (2s, 6H); 2.03 (ddd, 1H, J = 2.7, 16.1 and 19.0 Hz); 2.29 (s, 3H); 3.04 (m, 1H); 3.24 (dd, 1H, J = 8.4 and 16.1 Hz); 4.61 (dd, 1H, J = 4.4 and 4.9 Hz); 5.37 (dd, 1H, J = 2.6 and 6.1 Hz); 6.16 (d, 1H, J = 4.9 Hz); 7.03 (dd, 1H, J = 2.7 and 6.1 Hz).

^{13}C NMR δ (ppm); 24.9, 25.6, 27.8, 28.1, 46.4, 82.9, 94.2, 110.0, 115.0, 130.3, 138.4, 165.4, 196.8.

HRMS calc for $C_{13}H_{16}O_4$ $(M)^+$: 236.10485, found 236.1044.

m/z (%): 236(18.86); 205(10.08); 178(30.00); 163(14.81); 148(14.91); 145(15.75); 121(19.84); 107(25.78); 77(18.57); 58(18.18); 43(100); 28(11.72).

• *1-(2,2,6-Trimethyl-3a,8,8a,8b-tetrahydro-1,3-dioxolo[4,5-b]benzofuran-7-yl)ethanone* **14**

Yield 51%. Purification on silica gel (1:30; cyclohexane/AcOEt: 8:2).

$[\alpha]_D$ +11 (*c* 0.6, propanone).

Mp = 94–95 °C (diethyl ether/petroleum ether: 8:2).

IR (nujol): 2 929, 1 654, 1 525, 1 463, 1 376, 1 243, 1 157, 1 078, 1 039 cm^{-1}.

^{1}H NMR δ (ppm): 1.42 and 1.48 (2s, 6H); 2.12 (d, 3H, J = 2.3 Hz); 2.25 (s, 3H); 2.30 (m, 1H, J = 15.0 Hz); 2.95 (dd, 1H, J = 7.6 and 15.0 Hz); 3.05 (m, 1H); 4.58 (dd, 1H, J = 4.5 and 4.5 Hz); 5.24 (d, 1H, J = 2.3 Hz); 6.13 (d, 1H, J = 4.5 Hz).

^{13}C NMR δ (ppm): 22.0, 28.2, 28.5, 30.1, 30.6, 46.4, 83.1, 101.3, 109.9, 115.1, 124.4, 146.0, 163.0, 199.8.

HRMS for $C_{14}H_{18}O_4$ $(M)^+$: 250.12050, found 250.1208.

m/z (%): 250(4.96); 235(1.76); 219(2.41); 192(7.47); 177(4.46); 175(2.62); 164(6.27); 150(4.63); 145(5.67); 135(9.55); 121(10.85); 109(7.96); 96(5.89); 91(8.51); 77(6.98); 59(5.40); 43(100); 28(15.77).

• *1-[2,2-Dimethyl-7-(methylsulfinyl)-3a,6,7,8,8a,8b-hexahydro-1,3-dioxolo[4,5-b]benzofuran-7-yl]hexan-1-one* **15a, 15b**

Yield 52%. Purification on silica gel (1:30; cyclohexane/AcOEt: 8:2).

IR (film): 2 925, 2 855, 1 683, 1 457, 1 372, 1 160, 1 068, 1 044 cm^{-1}.

^{1}H NMR δ (ppm): (*: minor diastereomer) 0.88* and 0.89 (t, 3H, CH_3, J = 6.8 Hz); 1.25 (m, 4H); 1.43 (s, 3H, CH_3); 1.45* and 1.46 (2s, 3H); 1.58 (m, 2H); 1.78–1.84 and 1.92–1.98 (2m, 2H); 2.34* and 2.38 (2s, 3H); 2.41–2.74 (m, 3H); 2.96–3.10 (m, 2H); 3.19* (dd, 1H, J* = 4.9 and 12.8 Hz); 4.58 and 4.85* (dd, 1H, CH, J* = 5.0 and 4.9 Hz); 4.79 and 4.89* (t, 1H, J = 4.9 Hz); 6.02 (d, 1H, J = 4.9 Hz).

• *1-[2,2,6-Trimethyl-7-(methylsulfinyl)-3a,6,7,8,8a,8b-hexahydro-1,3-dioxolo[4,5-b]benzofuran-7-yl]hexan-1-one* **16a, 16b**

Yield 66%. Separation of diastereomers ($64_{(a)}$:$36_{(b)}$) by purification on silica gel (1:30; cyclohexane/AcOEt: 8:2).

HRMS calc for $C_{16}H_{24}O_4S$ (M – MeCOMe)$^+$: 312.13952, found 312.1402.

Analytical data for the major adduct **16a** are as follows:

$[\alpha]_D$ +79 (*c* 0.30; propanone).

IR (nujol): 2 923, 2 854, 1 683, 1 455, 1 370, 1 159, 1 070, 1 045 cm^{-1}.

^{1}H NMR δ (ppm): 0.89 (t, 3H, J = 6.8 Hz); 0.99 (d, 3H, J = 8.6 Hz); 1.27 (m, 4H); 1.43 and 1.55 (2s, 6H); 1.54 (m, 2H); 1.90 (dd, 1H, J = 9.8 and 12.3 Hz); 2.48 (s, 3H); 2.38 (dd, 1H, J = 7.4 and 14.2 Hz); 2.48 (dd, 1H, J = 7.4 and 14.2 Hz); 2.56 (m, 1H); 3.16 (m, 1H, J = 12.3 Hz); 3.20 (m, 1H); 4.61 (dd, 1H, J = 4.6 and 4.6 Hz); 4.79 (dd, 1H, J = 4.5 and 4.0 Hz); 5.85 (d, 1H, J = 4.6 Hz).

^{13}C NMR δ (ppm): 13.9, 19.2, 22.5, 25.6, 26.9, 27.9, 28.3, 31.2, 31.4, 33.9, 42.1, 43.9, 74.8, 83.7, 100.1, 106.2, 114.8, 154.1, 207.0.

m/z (%): 312(3.24); 306(15.93); 249(20.61); 248(27.70); 192(28.45); 177(56.75); 164(11.29); 163(13.25); 151(12.38);

150(30.75); 149(67.17); 135(17.28); 121(32.56); 107(19.82); 99(43.77); 93(16.29); 91(20.91); 79(13.78); 77(23.46); 71(46.46); 69(16.09); 55(13.93); 43(100).

Analytical data for the minor adduct **16b** are as follows:

$[\alpha]_D$ +384 (c 0.52, propanone).

Mp: 86–87 °C (diethyl ether/petroleum ether: 1:1).

^{1}H NMR δ (ppm): 0.88 (t, 3H, J = 6.8 Hz); 0.97 (d, 3H, J = 7.0 Hz); 1.30 (m, 4H); 1.42 and 1.49 (2s, 6H); 1.61 (m, 2H); 1.83 (t, 1H, J = 12.7 and 12.7 Hz); 2.33 (s, 3H); 2.52–2.58 (m, 1H); 2.54–2.62 (dt, 1H, J = 19.7 and 7.3 Hz); 2.78 (dt, 1H, J = 7.0 and 19.7 Hz); 3.06 (dd, 1H, J = 5.2 and 12.7 Hz); 3.13 (m, 1H); 4.53 (t, 1H, J = 5.0 and 5.0 Hz); 4.84 (dd, 1H, J = 4.2 and 2.7 Hz); 5.85 (d, 1H, J = 5.0 Hz).

^{13}C NMR δ (ppm): 13.9, 17.6, 22.6, 23.0, 25.0, 28.0, 28.3, 29.1, 31.4, 33.5, 42.0, 42.3, 72.3, 83.1, 103.3, 105.7, 114.8, 152.3, 204.3.

- *1-[7-(Cyclohexylsulfinyl)-2,2,6-trimethyl-3a,6,7,8,8a,8b-hexahydro-1,3-dioxolo[4,5-b]benzofuran-7-yl]hexan-1-one* **17a**

Yield 40%. Purification on silica gel (1:30; cyclohexane/AcOEt: 8:2).

Analytical data for the major adduct **17a** are as follows:

IR (film): 2 929, 2 853, 1 682, 1 454, 1 372, 1 160, 1 072, 1 046 cm^{-1}.

^{1}H NMR δ (ppm): 1.02 (d, 3H, J = 6.6 Hz); 1.02–1.50 and 1.65–1.83 (2m, 10H); 1.41 and 1.55 (2s, 6H); 1.90 (dd, 1H, J = 10.0 and 13.4 Hz); 2.22 (s, 3H); 2.55 (m, 1H, CH); 2.92 (m, 1H); 3.06 (dd, 1H, J = 6.7 and 13.4 Hz); 3.24 (m, 1H); 4.58 (t, 1H, J = 4.9 Hz); 4.78 (dd, 1H, J = 2.7 and 4.2 Hz); 5.81 (d, 1H, J = 4.9 Hz).

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