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# Thiyl Radical Induced Isomerisations of Maleate Esters Provide a Convenient Route to Fumarates and Furanones

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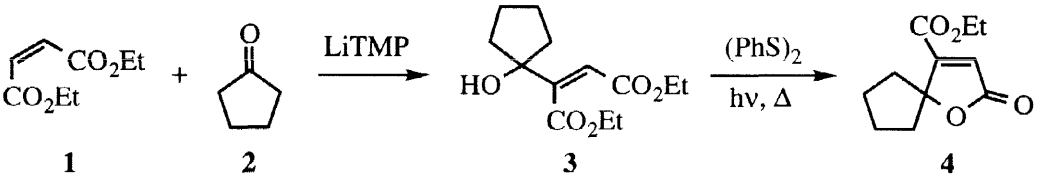
***Abstract:*** Maleate esters can be converted into fumarate esters in near quantitative yield through exposure to thiyl radicals generated in refluxing hexane by photolysis of diphenyl disulfide. When conditions are applied to dialkyl (hydroxyalkyl)maleate esters akin to **3**, 2(5*H*)-furanones are given in good yield. © 1999 Elsevier Science Ltd. All rights reserved.

***Keywords:*** Alkenes; Cyclisation; Isomerisations; Lactones; Radicals and radical reactions; Sulfur Chemistry.

## INTRODUCTION

In a recent paper we described a method of condensing dialkyl maleate esters and ketones using lithium amide bases as mediators, *e.g.* **1** + **2** → **3**.[1] The reaction was notable for its excellent stereoselectivity and as an extension of Baylis-Hillman type condensation reactions to trisubstituted alkenes.[2] We now report a further advance towards furanone synthesis (Scheme 1). A key achievement is the establishment of a mild and high yielding procedure to isomerise maleate esters to fumarate esters that also acts a trigger for the radical - polar crossover sequence outlined in Scheme 2.



**Scheme 1**

## RESULTS AND DISCUSSION

To achieve conversion of **3** into spirolactone **4** first required us to develop an isomerisation protocol for maleate esters that would be tolerant of tertiary allylic alcohol and ester functions.[3] Of those methods commonly employed to transform *cis*-alkenes into *trans*-alkenes, the addition and elimination of thiyl radical intermediates appeared ideal.[4] However, in common with others,[5] we were unable to induce any reaction between maleate ester **3** and a thiyl radical using standard procedures.[4,5]

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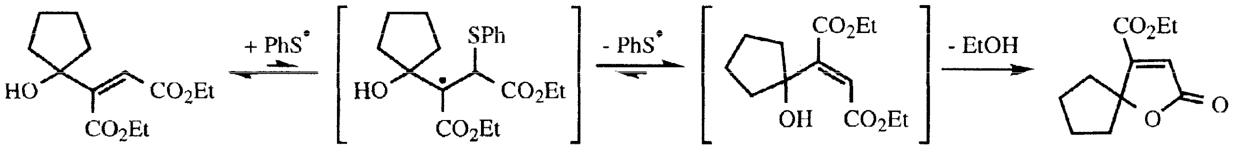
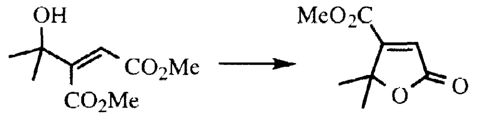
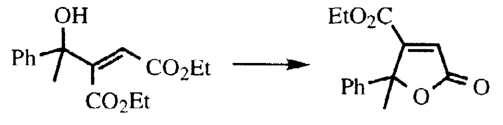
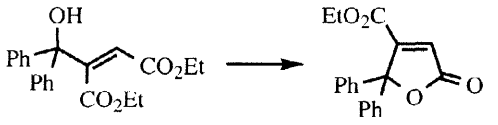
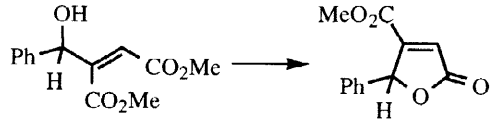
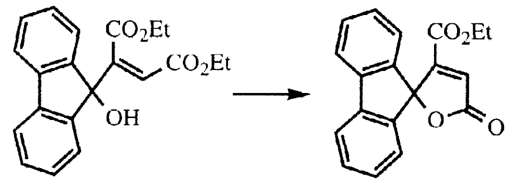
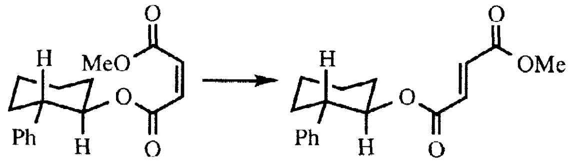
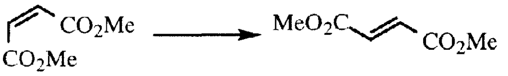
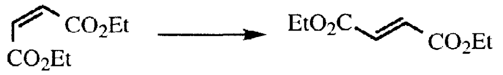


3 5 6 4

**Scheme 2**

Reasoning that the equilibrium established between an electrophilic thiyl radical and an electron deficient alkene was likely to favour reactants (Scheme 2), we decided to bias the system. The approach soon paid dividends as simply irradiating a refluxing hexane solution of **3** containing 5 equivalents of diphenyl disulfide provided lactone **4** in 71% yield after 12 hours and recovered starting material accounted for most of the remaining mass balance (20%). A series of maleate derivatives akin to **3** were then subjected to the aforementioned reaction conditions and in each case γ-lactones were formed in moderate to good yield (Table entries A - E). Likewise, three unsubstituted maleate esters were transformed into the corresponding fumarate esters in near quantitative yield using this protocol (Table entries F - H).

**Table:** Conversion of Maleate Esters to Fumarates Esters and 5*H*-Furanones[†]

| | | | |
|---|---|---|---|
| **A** |  | **B** |  |
| | $(PhS)_2$, hv, reflux, hexane, 24h, 66% | | $(PhS)_2$, hv, reflux, hexane, 23h, 77% (21%) |
| **C** |  | **D** |  |
| | $(PhS)_2$, hv, reflux, hexane, 4h, 44% (53%) | | $(PhS)_2$, hv, reflux, hexane, 24h, 46% |
| **E** |  | **F** |  |
| | $(PhS)_2$, hv, reflux, hexanc, 72h, 75% (19%) | | $(PhS)_2$, hv, reflux, hexane, 12h, 97% |
| **G** |  | **H** |  |
| | $(PhS)_2$, hv, reflux, hexane, 24h, 93% | | $(PhS)_2$, hv, reflux, hexane, 24h, 99% |

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[†] Figures in parentheses refer to % of recovered starting material

## CONCLUSION

To conclude, we have developed a versatile two step route to 2-furanones from maleate esters involving a new radical - polar crossover sequence. In addition we have established mild reaction conditions to effect the isomerisation of dialkyl maleate esters to dialkyl fumarate esters in near quantitative yield.

## ACKNOWLEDGEMENTS

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## EXPERIMENTAL SECTION

### GENERAL REMARKS

Melting points were obtained using a Mel-Temp (II) apparatus and are uncorrected. UV spectra were recorded on a Pye Unicam SP800 spectrometer. IR spectra were recorded on a Perkin Elmer 1600 series Fourier transform infrared spectrometer using NaCl cells except **4** which was recorded using a Bio-Rad FTS 135 Fourier transform infrared spectrometer equipped with a Golden Gate Single Reflection Diamond ATR. NMR spectra were recorded on a Bruker AC300 (operating at 300 MHz for $^1$H and at 75 MHz for $^{13}$C). Chemical shifts ($\delta_H$) are reported as values in parts per million relative to tetramethylsilane ($\delta_H$ 0.00, $\delta_C$ 0.00) or residual $CHCl_3$ ($\delta_H$ 7.27, $\delta_C$ 77.2). Mass spectra were recorded on a variety of instruments either in house or at the EPSRC mass spectrometry centre, Swansea.

All reactions were magnetically stirred under a nitrogen atmosphere and performed in a Quartz reaction vessel with irradiation from a 125W medium pressure mercury lamp (no additional heating source being required). Reactions were monitored by thin layer chromatography using Macherey-Nagel Alugram Sil G/UV$_{254}$ precoated aluminium foil plates of layer thickness 0.25mm. Compounds were visualised firstly by UV irradiation, then by exposure to iodine vapour and finally by heating plates exposed to solutions of phosphomolybdic acid in ethanol. Column chromatography was performed on Sorbsil 60 silica (230-400 mesh), slurry packed and run under low pressure. Hexane was dried over calcium hydride and degassed using ultrasound irradiation prior to use. Diphenyl disulfide, dimethyl maleate and diethyl maleate were used as supplied. The dialkyl (hydroxyalkyl)maleate esters were prepared by the procedure of Harrowven and Poon.[1] Methyl (1*R*, 2*S*)-2-phenylcyclohexyl maleate was prepared by the procedure of Maruoka *et al.*[6]

### GENERAL PROCEDURE FOR EFFECTING THE ISOMERISATION OF MALEATES TO FUMARATES

A solution of the maleate derivative (1.0 mmol) and diphenyl disulfide (5.0 mmol) in hexane (20 mL) was refluxed under irradiation from a 125W medium pressure mercury lamp for the stated time. The resulting mixture was concentrated *in vacuo* and purified by column chromatography on silica using the stated solvent as eluant to give firstly the furanone (or fumarate) and then recovered maleate derivative.

### 4'-Carboethoxyspiro[cyclopentane-1,5'-furan-2-one] 4

Prepared using the general procedure with maleate derivative (0.256 g, 1.0 mmol), diphenyl disulfide (1.092 g, 5.0 mmol) and hexane (20 mL). Irradiation at reflux for 12 h. Purification by column chromatography (silica, 10% ether in petrol) and recrystallisation from petrol gave the product (0.149 g, 0.71 mmol, 71% yield) as a pale yellow solid and then recovered starting material (0.051 g, 20%). **MP** (petrol) 36-38°C; **CHN** Calcd for

$C_{11}H_{14}O_4$: C, 62.85; H, 6.71. Found: C, 62.89; H, 6.51; **FT-IR** (solid) $\nu_{max}$, 3090 m, 2990 m, 2945 m, 1745 s, 1715 vs, 1625 m, 1480 m, 1380 m, 1230 s, 1155 s, 1115 m, 1015 m, 960 m and 945 m cm$^{-1}$; **$^1$H NMR** (300 MHz, $CDCl_3$) $\delta_H$, 6.60 (1H, s, =C*H*), 4.34 (2H, q, *J* = 7.2 Hz, OC*H*$_2$), 2.40-2.28 (2H, m), 2.06-1.80 (6H, m) and 1.37 (3H, t, *J* = 7.2 Hz, $OCH_2CH_3$) ppm; **$^{13}$C NMR** (75 MHz, $CDCl_3$) $\delta_C$, 170.4 (s), 161.1 (s), 158.8 (s), 126.8 (d), 97.3 (s), 62.1 (t), 37.2 (2 x t), 25.4 (2 x t) and 14.2 (q) ppm; **LRMS** (CI), 252 (100%, [MH+MeCN]$^+$) and 211 (20%, MH$^+$); **HRMS** (CI), Calcd. for $C_{11}H_{15}O_4$: 211.0970; Found: MH$^+$ 211.0979.

**4-Carboethoxy-5,5-dimethyl-2-furanone** (Table entry A)

Prepared using the general procedure with maleate derivative (0.230 g, 1.0 mmol), diphenyl disulfide (1.092 g, 5.0 mmol) and hexane (20 mL). Irradiation at reflux for 24 h. Purification by column chromatography (silica, 10-30 % ether in petrol) gave the product (0.122 g, 0.7 mmol, 66%) as a colourless oil; **FT-IR** (thin film) $\nu_{max}$, 3110 w, 2985 s, 2940 m, 2875 w, 1820 m, 1770 s, 1730 s, 1635 m, 1460 m, 1370 m, 1315 m, 1270 s, 1225 s, 1200 m, 1130 s, 1050 m, 975 m and 940 m cm$^{-1}$; **$^1$H NMR** (300 MHz, $CDCl_3$) $\delta_H$, 6.59 (1H, s, =C*H*), 4.35 (2H, q, *J* = 7.1 Hz, OC*H*$_2$), 1.65 (6H, s, 2 x CC*H*$_3$) and 1.38 (3H, t, *J* = 7.1 Hz, $OCH_2CH_3$) ppm; **$^{13}$C NMR** (75 MHz, $CDCl_3$) $\delta_C$, 170.1 (s), 161.5 (s), 161.0 (s), 126.2 (d), 87.4 (s), 62.2 (t), 25.3 (2 x q) and 14.2 (q) ppm; **LRMS** (APCI), 226 (100%, [MH+MeCN]$^+$); **HRMS** (EI), Calcd. for $C_9H_{13}O_4$: 185.0814; Found: MH$^+$ 185.0813.

**4-Carboethoxy-5-methyl-5-phenyl-2-furanone** (Table entry B)

Prepared using the general procedure with maleate derivative (0.292 g, 1.0 mmol), diphenyl disulfide (1.092 g, 5.0 mmol) and hexane (20 mL). Irradiation at reflux for 23 h. Purification by column chromatography (silica, 10-40 % ether in petrol) gave firstly the product (0.189 g, 0.8 mmol, 77%) as a pale yellow oil and then recovered starting material (0.061 g, 21%). **FT-IR** (thin film) $\nu_{max}$, 3110 w, 2985 m, 2940 w, 1770 s, 1730 s, 1635 m, 1495 m, 1445 m, 1220 s, 1150 m, 1100 m, 1040 m, 950 m, 895 m and 765 m cm$^{-1}$; **UV** (MeOH) $\lambda_{max}$ ($\varepsilon$), 234 (6010) nm; **$^1$H NMR** (300 MHz, $CDCl_3$) $\delta_H$, 7.44-7.32 (5H, m, 5x Ar*H*), 6.69 (1H, s, =C*H*), 4.26 (1H, dq, *J* = 16.2, 7.2 Hz, OC*H*H), 4.22 (1H, dq, *J* = 16.2, 7.2 Hz, OCH*H*), 2.11 (3H, s, CC*H*$_3$) and 1.27 (3H, t, *J* = 7.2 Hz, $OCH_2CH_3$) ppm; **$^{13}$C NMR** (75 MHz, $CDCl_3$) $\delta_C$, 170.6 (s), 161.4 (s), 160.8 (s), 137.3 (s), 128.9 (d), 128.8 (2 x d), 125.8 (2 x d), 125.6 (d), 89.1 (s), 62.2 (t), 24.3 (q) and 14.1 (q) ppm; **LRMS** (APCI), 288 (60%, [MH+MeCN]$^+$), 247 (80%, MH$^+$), 201 (100%, [M-OEt]$^+$) and 173 (50%, [M-$CO_2Et$]$^+$); **HRMS** (CI), Calcd. for $C_{14}H_{15}O_4$: 247.0970; Found: MH$^+$ 247.0970.

**4-Carboethoxy-5,5-diphenyl-2-furanone** (Table entry C)

Prepared using the general procedure with maleate derivative (0.354 g, 1.0 mmol), diphenyl disulfide (1.092 g, 5.0 mmol) and hexane (20 mL). Irradiation at reflux for 4 h. Purification by column chromatography (silica, 30% $CH_2Cl_2$ in petrol) gave firstly the product (0.136 g, 0.4 mmol, 44% yield) as a colourless oil and then recovered starting material (0.188 g, 0.5 mmol, 53% yield). **FT-IR** (thin film) $\nu_{max}$, 3110 w, 3060 w, 2985 w, 1770 s, 1730 s, 1630 w, 1495 m, 1450 m, 1335 m, 1220 s, 1000 m, 935 m, 760 m and 700 s cm$^{-1}$; **UV** (EtOH) $\lambda_{max}$ ($\varepsilon$), 270 inf (2300), 258 inf (3000) and 232 (5800) nm; **$^1$H NMR** (300 MHz, $CDCl_3$) $\delta_H$, 7.40-7.33 (10H, m, 10 x Ar*H*), 6.88 (1H, s, =C*H*), 4.25 (2H, q, *J* = 7.1 Hz, OC*H*$_2$) and 1.25 (3H, t, *J* = 7.1 Hz, $OCH_2CH_3$) ppm; **$^{13}$C NMR** (75 MHz, $CDCl_3$) $\delta_C$, 170.1 (s), 161.1 (s), 160.1 (s), 138.0 (2 x s), 129.2 (2 x d), 128.4 (4 x d), 128.2 (4 x d), 127.3 (d), 95.5 (s), 62.4 (t) and 14.0 (q) ppm; **LRMS** (CI), 326 (100%, [M+NH$_4$]$^+$); **HRMS** (CI), Calcd. for $C_{19}H_{20}NO_4$: 326.1391; Found: [M+NH$_4$]$^+$ 326.1392.

**4-Carbomethoxy-5-diphenyl-2(5*H*)-furanone** (Table entry D)

Prepared using the general procedure with maleate derivative (0.250 g, 1.0 mmol), diphenyl disulfide (1.092 g, 5.0 mmol) and hexane (20 mL). Irradiation at reflux for 24 h. Purification by column chromatography (silica, 20% ether in petrol) and recrystallisation from petrol gave the product (0.101 g, 0.46 mmol, 46%) as a white solid; **MP** (petrol) 108-110°C; **CHN** Calcd for $C_{12}H_{10}O_4$: C, 66.05; H, 4.62. Found: C, 65.82; H, 4.51; **FT-IR** ($CHCl_3$) $\nu_{max}$, 2925 s, 2855 s, 1765 m, 1725 m, 1640 w, 1455 m, 1370 w, 1300 w, 1225 m, 1160 m, 985 m, 910 w, 845 w and 705 m $cm^{-1}$; **UV** (EtOH) $\lambda_{max}$ ($\varepsilon$), 282 (1100) and 233 (5100) nm; $^1$**H NMR** (300 MHz, $CDCl_3$) $\delta_H$, 7.43-7.36 (3H, m, 3 x Ar*H*), 7.34-7.26 (2H, m, 2 x Ar*H*), 6.80 (1H, d, *J* = 2.0 Hz, PhC*H*), 6.16 (1H, d, *J* = 2.0 Hz, =C*H*) and 3.77 (3H, s, OC*H₃*) ppm; $^{13}$**C NMR** (75 MHz, $CDCl_3$) $\delta_C$, 171.1 (s), 161.1 (s), 157.1 (s), 133.3 (s), 129.9 (d), 129.1 (2 x d), 127.3 (2 x d), 126.2 (d), 83.7 (d) and 53.0 (q) ppm; **LRMS** (APCI), 218 (8%, $M^+$), 214 (20%, $[M-CO_2H+MeCN]^+$), 200 (20%, $M-H_2O]^+$), 187 (20%, $[M-OMe]^+$), 173 (100%, $[M-CO_2H]^+$) and 159 (75%, $[M-CO_2Me]^+$); **HRMS** (CI), Calcd. for $C_{12}H_{10}O_4$: 218.0579; Found: $M^+$ 218.0579.

**4'-Carboethoxyspiro[fluorene-9,5'-furan-2-one]** (Table entry E)

Prepared using the general procedure with maleate derivative (0.352 g, 1.0 mmol), diphenyl disulfide (1.092 g, 5.0 mmol) and hexane (20 mL). Irradiation at reflux for 72 h. Purification by column chromatography (silica, 50% $CH_2Cl_2$ in petrol) gave recovered starting material (0.067 g, 19%) and the product (0.230 g, 0.8 mmol, 75% after recrystallisation from aqueous methanol) as a white solid; **MP** ($MeOH/H_2O$) 124-125°C (Lit.[7] 124-125°C); **FT-IR** (nujol mull) $\nu_{max}$, 2985 w, 1770 s, 1730 s, 1635 w, 1450 m, 1370 w, 1300 m, 1255 m, 1215 s, 1115 m, 1010 m, 920 m, 775 m and 735 m $cm^{-1}$; **UV** (EtOH) $\lambda_{max}$ ($\varepsilon$), 284 (14000), 275 (17000), 269 inf (16000), 237 (21000) and 230 inf (18000) nm; $^1$**H NMR** (300 MHz, $CDCl_3$) $\delta_H$, 7.72 (2H, d, *J* = 7.5 Hz, 2 x Ar*H*), 7.47 (2H, td, *J* = 7.5, 0.9 Hz, 2 x Ar*H*), 7.30 (2H, td, *J* = 7.5, 0.9 Hz, 2 x Ar*H*), 7.22 (2H, d, *J* = 7.5 Hz, 2 x Ar*H*), 7.05 (1H, s, =C*H*), 3.94 (2H, q, *J* = 7.1 Hz, OC*H₂*) and 0.92 (3H, t, *J* = 7.1 Hz, OCH₂C*H₃*) ppm; $^{13}$**C NMR** (75 MHz, $CDCl_3$) $\delta_C$, 170.8 (s), 159.5 (s), 157.6 (s), 141.5 (2 x s), 139.7 (2 x s), 130.7 (2 x d), 128.2 (2 x d), 127.1 (d), 123.5 (2 x d), 120.7 (2 x d), 92.7 (s), 61.8 (t) and 13.4 (q) ppm; **LRMS** (APCI), 306 (100%, $M^+$), 233 (5%, $[M-CO_2Et]^+$); **HRMS** (EI), Calcd. for $C_{19}H_{14}O_4$: 306.0892; Found: $M^+$ 306.0869.

**Methyl (1*R**,2*S**)-2-phenylcyclohexyl fumarate** (Table entry F)

Prepared using the general procedure with maleate derivative (0.287 g, 1.0 mmol), diphenyl disulfide (1.092 g, 5.0 mmol) and hexane (20 mL). Irradiation at reflux for 12 h. Purification by column chromatography (silica, 5 % ether in petrol) gave the product (0.279 g, 9.7 mmol, 97% yield) as a pale yellow oil; **FT-IR** (thin film) $\nu_{max}$, 3065 w, 3030 w, 2935 s, 2860 m, 1715 s, 1645 w, 1602 w, 1495 w, 1450 m, 1435 m, 1350 w, 1295 s, 1260 s, 1225 m, 1155 s, 1020 m, 980 m and 760 m $cm^{-1}$; **UV** (MeOH) $\lambda_{max}$ ($\varepsilon$), 231 (4400) nm; $^1$**H NMR** (300 MHz, $CDCl_3$) $\delta_H$, 7.30-7.14 (5H, m, 5 x Ar*H*), 6.62 (2H, s, 2 x =C*H*), 5.07 (1H, td, *J* = 10.6, 4.6 Hz, OC*H*), 3.76 (3H, s, OC*H₃*), 2.73 (1H, ddd, *J* = 12.1, 10.6, 3.9 Hz, PhC*H*), 2.22-2.14 (1H, m), 2.03-1.76 (3H, m) and 1.69-1.34 (4H, m) ppm; $^{13}$**C NMR** (75 MHz, $CDCl_3$) $\delta_C$, 165.6 (s), 164.3 (s), 142.8 (s), 134.2 (d), 132.8 (d), 128.5 (2 x d), 127.6 (2 x d), 126.7 (d), 77.4 (d), 52.4 (q), 49.8 (d), 33.9 (t), 32.3 (t), 25.9 (t) and 24.8 (t) ppm; **LRMS** (CI), 306 (100%, $[M+NH_4]^+$), 176 (20%, $[PhC_6H_{10}OH]^+$) and 158 (30%, $[PhC_6H_{10}]^+$); **HRMS** (CI), Calcd. for $C_{17}H_{24}NO_4$: 306.1705; Found: $[M+NH_4]^+$ 306.1678.

**Dimethyl Fumarate** (Table entry G)
Prepared using the general procedure with dimethyl maleate (0.288 g, 2.0 mmol), diphenyl disulfide (2.18 g, 10.0 mmol) and hexane (40 mL). Irradiation at reflux for 24 h. Purification by column chromatography (silica, 5 % ether in petrol) gave diethyl fumarate (0.268 g, 1.86 mmol, 93% yield) as a colourless solid identical in all respects with an authentic sample;[8] **MP** 102-103°C (Lit.[8] 103-104°C)).

**Diethyl Fumarate** (Table entry H)
Prepared using the general procedure with diethyl maleate (0.344 g, 2.0 mmol), diphenyl disulfide (2.18 g, 10.0 mmol) and hexane (40 mL). Irradiation at reflux for 24 h. Purification by column chromatography (silica, 5 % ether in petrol) gave diethyl fumarate (0.341 g, 1.99 mmol, 99% yield) as a colourless oil and identical in all respects with an authentic sample.[8]

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