SUPPORTING INFORMATION

Comparison of SEP and SPME enrichment capacity. The effectiveness of the SEP (sample enrichment probe) technique in the analysis of samples containing only trace amounts of volatile constituents had been demonstrated by an exploratory comparison of the results of solid-phase microextration-gas chromatography-mass spectrometry (SPME-GC-MS) and sample enrichment probe-gas chromatography-mass spectrometry (SEP-GC-MS) analyses of the aroma of dry, fermented plant material of *Aspalathus linearis*, known as rooibos tea.[S1] In spite of a long sample enrichment period of 24 h, only some of the peaks visible in the higher retention-time range of the total ion chromatogram (TIC) of the material enriched by SPME gave mass spectra that contained useful diagnostic information. In contrast, sample enrichment for 24 h in the case of a SEP-GC-MS analysis gave more than 140 library searchable or interpretable mass spectra. In the present study, comparison of the results of headspace solid-phase microextraction-gas chromatography-mass spectrometry (HS-SPME-GC-MS) (**Figure S1**) and headspace sample enrichment probe-gas chromatography-mass spectrometry (HS-SEP-GC-MS) (**Figure S2**) analyses confirmed the much higher capacity of the SEP and the latter was therefore selected as the method of choice for the analysis of honeybush volatiles.

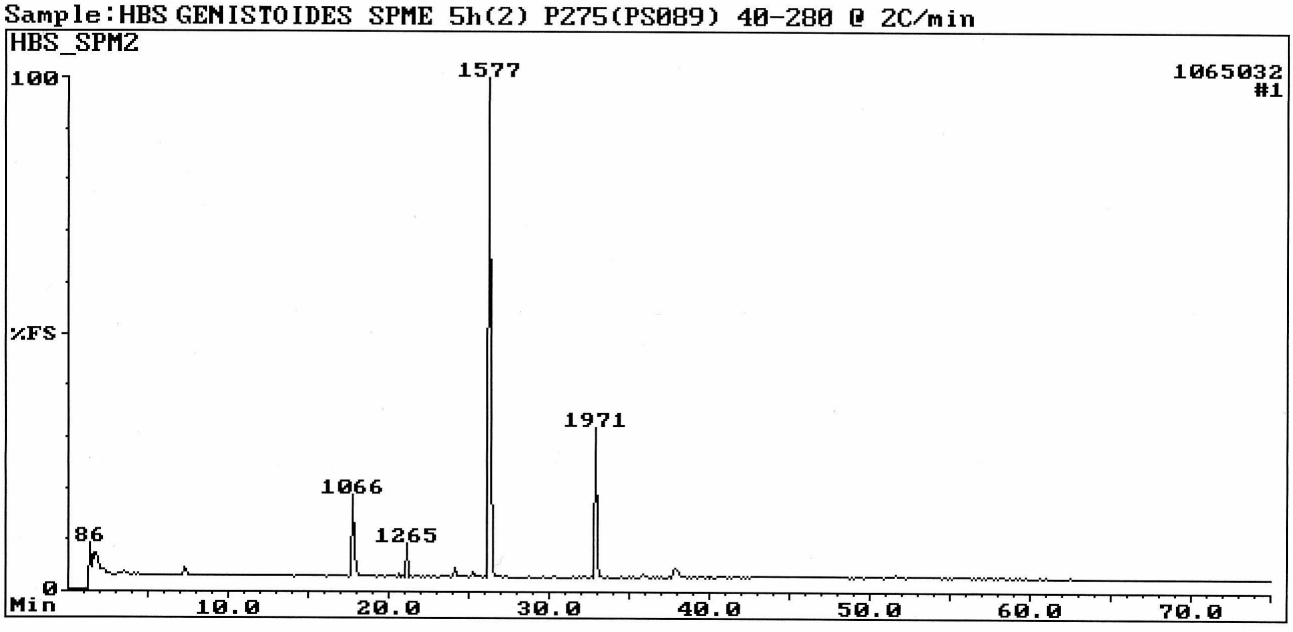


Figure S1. Volatile compounds in dry, fermented honeybush analysed by HS-**SPME**-GC-MS.

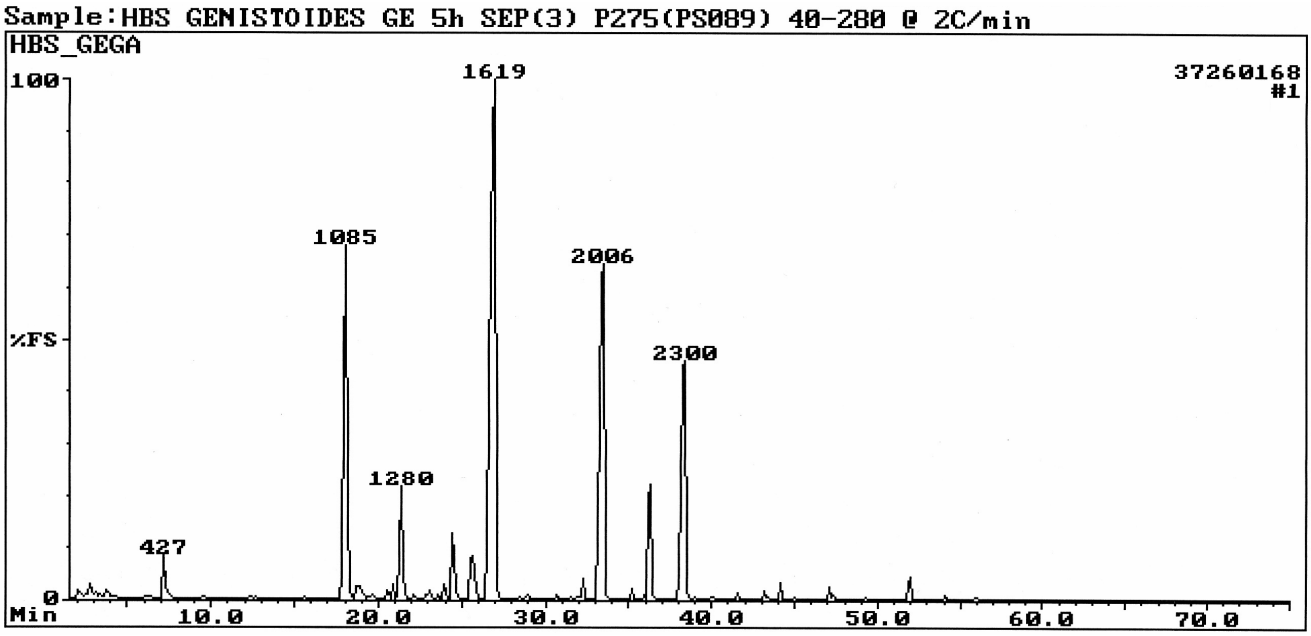


Figure S2. Volatile compounds in dry, fermented honeybush analysed by HS-**SEP**-GC-MS.

Nuclear magnetic resonance spectroscopy. ^{1}H and ^{13}C NMR spectra of synthesized compounds were recorded in $CDCl_3$ on Varian VnmrS 300, Unity *INOVA* 400 and UNITY *INOVA* 600 NMR instruments (Varian, Palo Alto, USA). Chemical shifts are given in parts per million (δ) relative to chloroform (7.26 and 77.04 ppm for ^{1}H and ^{13}C NMR, respectively).

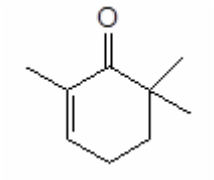
Syntheses.

2,6,6-Trimethylcyclohex-2-enone:

2-Bromo-2,6,6-trimethylcyclohexanone: Following the protocol of Tietze and Eicher[S2] for the α-bromination of an α-substituted ketone, a solution of bromine (0.57 g; 3.57 mmol; 0.18 ml) in CCl_4 (5 ml) was added dropwise over a period of 15 min to a magnetically stirred solution of 2,2,6-trimethylcyclohexanone (0.5 g; 3.57 mmol) in CCl_4 (10 ml) in a round-bottom flask fitted with a cooler and dropping funnel. After addition of the bromine, ether (10 ml) was added and the reaction mixture was washed once with water (10 ml) and dried on anhydrous $MgSO_4$. The drying agent was filtered off and the solvent removed on a rotary evaporator to yield 2-bromo-2,6,6-trimethylcyclohexanone (0.72 g; 92%) in a purity of 98% (GC-MS).

2,6,6-Trimethylcyclohex-2-enone: 2-Bromo-2,6,6-trimethylcyclohexanone (0.35 g; 1.6 mmol) was heated to 170 °C in 2,4,6-trimethylpyridine (collidine) (1.4 ml) for 70 min, after which the reaction mixture was cooled to 0 °C. Ether (2.8 ml) was added to the reaction mixture, which was left to stand for 1 h. The collidinium hydrobromide was filtered off, washed with ether (3 x 2 ml) and the combined

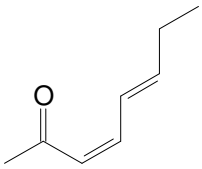
filtrate and ether washings washed consecutively with 4 M HCl solution (2 ml), water (2 ml) and 5% NaOH solution (2 ml), and dried on anhydrous $MgSO_4$. Because GC-MS analysis of the product mixture indicated that dehydrohalogenation was not yet complete, a further volume of collidine (0.5 ml) was added and the mixture heated for another hour and worked up as before to yield 2,6,6-trimethylcyclohex-2-enone (0.156 g; 71%) in a purity of 96% (GC-MS).



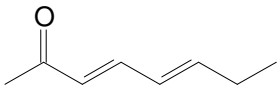
MS (70 eV): *m/z* (%) 138 (M^+, 9), 123 (1), 110 (5), 95 (5), 82 (100), 67 (6), 54 (56), 39 (36).
^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.096 (s, 6H, 6-CH_3s), 1.75 (dd, 3H, J = 2.0 Hz, J = 3.4 Hz, 2-CH_3), 1.81 (t, 2H, J = 6.1 Hz, 5-CH_2), 2.28-2.36 (m, 2H, 4-CH_2), 6.624 (br s, 1H, 3-CH).
^{13}C NMR ($CDCl_3$, 300 MHz): δ = 16.46 (q, C-2a), 23.05 (s, C-6), 24.30 (q, C-6a & C-6b), 36.65 (t, C-4), 41.21 (t, C-5), 133.74 (d, C-3), 143.50 (s, C-2), 204.74 (s, C-1).

(*E,E*)- and (*Z,E*)-3,5-Octadien-2-one:

Following an adapted version of the protocol of Heydanek and McGorrin,[S3] a solution of (*E*)-2-pentenal (1.013 g; 12 mmol) in THF (5 ml) was added dropwise to a solution of sodium hydride (122 mg; 3.1 mmol; 60% NaH) and acetone (3.017 g; 52 mmol) in THF (30 ml) over a period of 2.5 h, while cooling the reaction mixture to between 10–15 °C. After completion of the addition the solution was stirred for 1 h at room temperature, quenched with 20% HCl (1 ml) and the solvent removed on a rotary evaporator. Ether (40 ml) was added to the oily yellow residue. The ether solution was then washed with water (3 x 10 ml) and dried on anhydrous Na_2SO_4. Evaporation of the solvent yielded a yellow oil (1.31 g; 79%). The 90% pure (GC-MS) target product contained (*E,E*)- and (*Z,E*)-3,5-octadien-2-one in a ratio of 99:1.



(*Z,E*)-3,5-Octadien-2-one

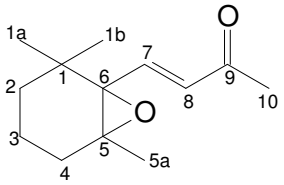


(*E,E*)-3,5-Octadien-2-one

MS (70 eV): *m/z* (%): 124 (M^+, 29), 109 (12), 96 (7), 95 (100), 81 (44), 79 (26), 77 (8), 65 (8), 53 (24), 43 (35), 41(13), 39 (15).
^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.03 (t, 3H, J = 7.4 Hz, 8-CH_3), 2.15-2.26 (m, 2H, 7-CH_2), 2.23 (s, 3H, 1-CH_3), 6.03 (d, 1H, J = 15.8 Hz, 3-CH), 6.12-6.25 (m, 2H, 5- and 6-CH), 7.08 (dd, 1H, J = 9.6 Hz, J = 15.6 Hz, 4-CH).
^{13}C ($CDCl_3$, 400 MHz): δ = 13.06 (q, C-8), 26.37 (t, C-7), 27.30 (q, C-1), 128.07 (d, C-3), 128.97 (d, C-5), 144.41 (d, C-6), 147.33 (d, C-4), 199.14 (s, C-2).

5,6-Epoxy-β-ionone:

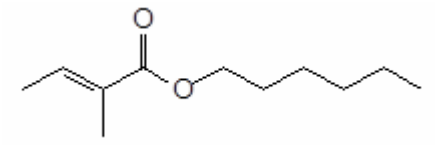
m-Chloroperbenzoic acid (*m*CPBA; 70%) (9.1 mmol) was slowly added in small portions, to a magnetically stirred solution of β-ionone (1.93 g; 10 mmol) in a biphasic solvent system of DCM (100 ml) and 0.05 M $NaHCO_3$ (30 ml).[S4] After addition of the *m*CPBA the solution was stirred at room temperature for a further 2 h. The two phases were separated and the organic phase was washed consecutively with 1 M NaOH (30 ml) and water (30 ml), and dried on anhydrous Na_2SO_4. GC-MS analysis of the product mixture at this stage indicated the presence of approximately 80% 5,6-epoxy-β-ionone and 20% unepoxidised β-ionone. The synthesis was repeated with half of the product mixture, using the amount of *m*CPBA necessary to epoxidise the remaining β-ionone, but no further epoxidation took place. The second half of the crude product (1 g) was chromatographed on a 25-cm column of Kieselgel 60 (230–400 mesh), and the pure (GC-MS) 5,6-epoxy-β-ionone (0.46 g, 22%) was eluted with ether:petroleum ether (25:75).



MS (70 eV): *m/z* (%) 208 (M^+, <0.1), 193 (3), 165 (3), 135 (33), 124 (25), 123 (100), 43 (83), 41 (32).
^{1}H NMR ($CDCl_3$, 400 MHz): δ = 0.895 (s, 3H, 5-CH_3), 1.11 (s, 6H, 1-CH_3s), 1.35–1.91 (m, 6H, 2,3,4-CH_2), 2.24 (s, 3H, 10-CH_3), 6.25 (d, 1H, J = 15.6 Hz, 8-CH), 6.98 (d, 1H, J = 15.6 Hz, 7-CH).
^{13}C ($CDCl_3$, 300 MHz): δ = 16.86 (t, C-3), 20.81 (q, C-5a), 25.81 and 25.87 (q, C-1a and C-1b), 28.23 (q, C-10), 29.74 (t, C-4), 33.52 (s, C-1), 35.46 (t, C-2), 65.84 (s, C-5), 70.57 (s, C-6), 132.45 (d, C-8), 142.63 (d, C-7), 197.49 (s, C-9).

Hexyl tiglate:

A solution of tiglic acid (5 g; 0.05 mol) and 1-hexanol (1.7 g; 0.0167 mol) in benzene (30 ml) was refluxed with Amberlyst 15 ion-exchange catalyst granules (1 g), using a Dean-Stark water separator. The reaction mixture was washed with NaOH solution (1.332 g in 10 ml water) and dried on anhydrous $MgSO_4$ The drying agent was filtered off and the solvent evaporated to yield hexyl tiglate (2.34 g; 73%) in a purity of 96% (GC-MS).

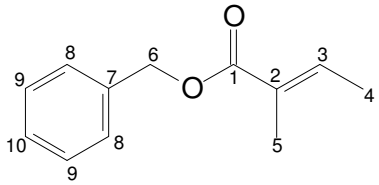


MS (70 eV): *m/z* (%) 181 (M^+, 0), 111 (4), 101 (100), 100 (15), 83 (45), 69 (9), 56 (24), 55 (81), 43 (42), 41 (36), 39 (25).

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 0.89 (t, 3H, J = 6.9 Hz, 11-CH_3), 1.27–1.43 (m, 6H, 8,9,10-CH_2), 1.62–1.70 (m, 2H, 7-CH_2), 1.77–1.80 (m, 3H, 4-CH_3), 1.82–1.85 (m, 3H, 5-CH_3), 4.12 (t, 2H, J = 6.7 Hz, 6-CH_2), 6.82–6.89 (qq, 1H, J = 1.4 Hz, J = 7.0 Hz, 3-CH).
^{13}C NMR ($CDCl_3$, 300 MHz): δ = 11.99 (q, C-5), 13.97 (q, C-11), 14.27 (q, C-4), 22.53 (t, C-10), 25.69 (t, C-8), 28.66 (t, C-9), 31.45 (t, C-7), 64.56 (t, C-6), 128.78 (s, C-2), 136.76 (d, C-3), 168.21 (s, C-1).

Benzyl tiglate:

Tiglic acid (2 g; 0.02 mol) was added to thionyl chloride (3.57 g, 0.03 mol) in a round-bottom flask at room temperature. The apparatus was set up as if for a distillation procedure with a pear flask as collecting vessel. The pear flask was fitted with a $CaCl_2$ drying tube that was connected by rubber tubing to a funnel of which the rim was positioned just below the surface of water in a beaker in order to make provision for the absorbtion of the SO_2(g) and HCl(g) formed in the reaction. The reaction mixture was stirred for 2 h at room temperature and then heated to 110 °C to distill off any excess thionyl chloride in a fume hood. The mixture was left to cool to room temperature, benzyl alcohol (1.5 g; 0.014 mol) was added and the mixture was stirred at room temperature overnight. The mixture was then heated for an hour at 80 °C, allowed to cool, and diluted with ether (20 ml). The ether solution was washed with water (8 x 20 ml) and dried on anhydrous Na_2SO_4. Evaporation of the solvent yielded benzyl tiglate (1.97 g; 75%) in a purity of 84% (GC-MS).

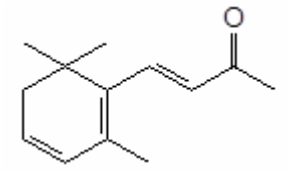


MS (70 eV): *m/z* (%) 190 (M^+, 3), 172 (23), 145 (28), 129 (5), 107 (7), 91 (100), 83 (61), 65 (20), 55 (42), 39 (17).
^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.79–1.81 (m, 3H, 4-CH_3), 1.86–1.88 (m, 3H, 5-CH_3), 5.19 (s, 2H, 6-CH_2), 6.89–6.96 (qq, 1H, J = 1.4 Hz, J = 7.1 Hz, 3-CH), 7.28–7.39 (m, 5H, 8,9,10-CH).
^{13}C ($CDCl_3$, 300 MHz): δ = 12.30 (q, C-5), 14.59 (q, C-4), 66.39 (t, C-6), 128.19 (d, C-8), 128.24 (d, C-10), 128.72 (d, C-9), 128.73 (s, C-2), 137.87 (d, C-3), 140.04 (s, C-7), 168.12 (s, C-1).

3,4-Dehydro-β-ionone:

$NaHCO_3$ (3.28 g; 0.039 mol), CaO (2.62 g; 0.047 mol) and *N*-bromosuccinimide (NBS) (7 g; 0.039 mol) were added to β-ionone (6 g; 0.031 mol) in CCl_4 (40 ml) in a round-bottom flask equipped with a condenser, magnetic follower and thermometer, and the reaction mixture was heated to boiling point.[S5] The external heat source was removed and the mixture was allowed to boil vigorously for at least 10 min, after which the exothermic reaction began to subside. The reaction mixture was then cooled down to 40 °C and dimethyl aniline (9 ml) was added. The succinimide formed in the reaction was filtered off and washed with CCl_4. After evaporation of the solvent from the combined filtrate and

washings and heating the residual product mixture in a nitrogen atmosphere for 2 h, pyridine (3 ml) was added and heating continued for 1 h. After the mixture had cooled the product was poured into cold water (40 ml) and extracted with petroleum ether (3 x 40 ml). The combined extracts were washed consecutively with 2% H_2SO_4 (50 ml), water (50 ml) and a 2% $NaHCO_3$ (50 ml), and then dried on Na_2SO_4. Evaporation of the solvent and vacuum distillation yielded 3,4-dehydro-β-ionone (3.69 g; 62%) in a purity of 88% (GC-MS); bp 90–95 °C (0.3 mmHg).



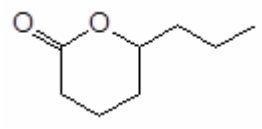
MS (70 eV): *m/z* (%) 190 (M^+, 14), 175 (47), 157 (8), 147 (15), 131 (19), 115 (19), 105 (17), 91 (21), 77 (10), 43 (100).

^{1}H NMR ($CDCl_3$, 300 MHz): δ = 1.08 (s, 6H, 1,1-CH_3), 1.91 (s, 3H, 5-CH_3), 2.12 (d, 2H, J = 2.2 Hz, 2-CH_2), 2.31 (s, 3H, 10-CH_3), 5.89 (s, 2H, 3,4-CH), 6.21 (d, 1H, J = 16.4 Hz, 8-CH), 7.28 (d, 1H, J = 16.4 Hz, 7-CH).

^{13}C ($CDCl_3$, 300 MHz): δ = 20.34 (q, C-5a), 26.56 (q, C1a, C1b), 27.36 (q, C-10), 34.04 (s, C-1), 39.98 (t, C-2), 128.25 (d), 129.61 (d), 130.36 (s), 132.75 (d, C-8), 135.94 (s), 141.76 (d, C-7), 198.40 (s, C-9).

Octan-5-olide:

Mercury(II) acetate (9.1 g; 25.56 mmol) and 1-pentene (2g; 28.57 mmol) were added to a mixture of THF (70 ml) and water (30 ml).[S6] After stirring the mixture for 1 h, acrylonitrile (4.54 g; 85.71 mmol), 2 M NaOH (30 ml) and a solution of $NaBH_4$ (2.16 g; 57.14 mmol) dissolved in 2 M NaOH (110 ml) were added, and the reaction mixture stirred for another 30 min. The product was extracted with DCM, dried on anhydrous Na_2SO_4, and the solvent evaporated to give 5-hydroxyoctanenitrile. NaOH (2 M; 28 ml) was added to the nitrile and the mixture extracted with ether (6 x 5 ml) to remove any unsaponified material. The water layer was acidified with H_2SO_4 (50% solution), the organic product was extracted with ether (6 x 5 ml), and the combined extracts were washed with water and dried on $MgSO_4$. Evaporation of the solvent yielded octan-5-olide (0.80 g; 30%) in a purity of 86% (GC-MS) as a slightly yellow liquid with a distinctly pleasant smell.



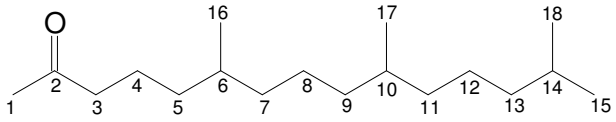
MS (70 eV): *m/z* (%) 142 (M^+, <0.7), 124 (3), 114 (9), 99 (100), 71 (60), 70 (39), 55 (45), 42 (69).

^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.93 (t, 3H, J = 7.2 Hz, 8-CH_3), 1.37–1.46 (m, 1H), 1.46–1.6 (m, 3H), 1.66–1.74 (m, 1H), 1.84–1.94 (m, 3H), 2.42 (ddd, 1H, J = 1.6 Hz, J = 8.7 Hz, J = 17.3 Hz, 2-CH), 2.56–2.63 (m, 1H, 2-CH), 4.24–4.34 (m, 1H, 5-CH).

^{13}C ($CDCl_3$, 300 MHz): δ = 13.81 (q, C-8), 18.15 (t, C-7), 18.44 (t, C-3), 27.75 (t, C-4), 29.43 (t, C-2), 37.85 (t, C-6), 80.41 (d, C-5), 172.28 (s, C-1).

Hexahydrofarnesylacetone:

A solution of farnesylacetone (0.53 g; 0.2 mmol) in absolute ethanol (15 ml) was hydrogenated in a conventional hydrogenation apparatus[S7] using platinum on activated charcoal (0.04 g; 10% Pt/C) as catalyst. The hydrogenation was carried out at approximately atmospheric pressure and the consumption of hydrogen was monitored. The reaction was allowed to run to completion. A total volume of 138.6 ml (6.2 mmol) hydrogen was taken up. Most of the ethanol was then evaporated using a slow stream of nitrogen. Petroleum ether (20 ml) was added to the residue consisting of the product and catalyst, and the catalyst was filtered off through a layer of magnesium sulphate. The petroleum ether was evaporated to afford hexahydrofarnesylactone (0.42 g; 79%) in a purity of 90% (GC-MS).

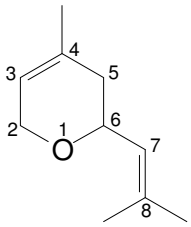


MS (70 eV): *m/z* (%) 268 (M^+, <0.5), 250 (6), 210 (3), 179 (4), 165 (5), 137 (5), 124 (9), 109 (17), 95 (17), 85 (20), 71 (39), 58 (74), 43 (100), 41 (31).
^1H NMR ($CDCl_3$, 400 MHz): δ = 0.827–0.880 (m, 12 H), 1.04–1.17 (m, 6H), 1.22–1.31 (m, 7H), 1.32–1.44 (m, 3H), 1.45–1.64 (m, 3H), 2.13 (s, 3H, 1-CH_3), 2.40 (t, 2H, J = 7.5 Hz, 3-CH_2).
^{13}C ($CDCl_3$, 300 MHz): δ = 19.51 (q, C-17), 19.67 (q, C-16), 21.44 (t, C-4), 22.62 (q, C-18/15), 22.72 (q, C18/15), 24.42 (t, C-12), 24.81 (t, C-8), 27.97 (d, C-14), 29.83 (q, C-1), 32.65 (d, C-6), 32.76 (d, C-10), 36.49 (t, C-7), 36.58 (t, C-11), 37.23 (t, C-5), 37.38 (t, C-13), 39.37 (t, C-9), 44.13 (t, C-3), 209.32 (s, C-2).

Nerol oxide:

Nerol oxide was prepared according to the procedure of Gupta *et al.*[S8] by adding nerol (15 g; 0.0974 mol) to anhydrous methanol (50 ml) in a round-bottom flask fitted with a magnetic follower, thermometer and gas inlet. 1,3-Dibromo-5,5-dimethylhydantoin (DDH) (15 g; 0.0525 mol) was added in small portions to the nerol solution in a nitrogen atmosphere, while keeping the temperature at 5–10 °C. The mixture was then poured into cold water (25 ml) and extracted with ethyl acetate (3 x 25 ml). The combined organic layers were washed consecutively with 5% Na_2CO_3 (3 x 15 ml) and water (3 x 15 ml), dried on anhydrous Na_2SO_4, and the solvent evaporated. The crude product (22.22 g) was dissolved in methanol (60 ml), containing 10.5 g of KOH, and refluxed for 9 h. Most of the solvent was then evaporated under reduced pressure, the product was poured into cold water (20 ml), extracted with $CHCl_3$ (4 x 25 ml), and the combined extracts were washed with water (3 x 15 ml) and dried on $CaCl_2$. The drying agent was filtered off and the solvent removed under reduced

pressure. GC-MS analysis of the crude product indicated that nerol oxide was present as the main product (50% purity, GC-MS). It was distilled to give nerol oxide (0.96 g; 23%) in a purity of 87% (GC-MS); bp 88–92°C (20 mmHg).



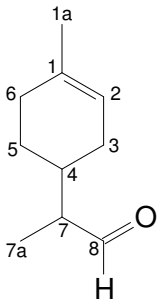
MS (70 eV): *m/z* (%) 152 (M^+, 11), 137 (2), 109 (11), 96 (17), 85 (28), 83 (77), 68 (100), 67 (94), 55 (32), 53 (52), 41 (79), 39 (61).

^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.70, 1.703 (2s, 6H, 8-CH_3s), 1.74 (s, 3H, 4-CH_3), 2.04 (m, 2H, 5-CH_2), 4.15–4.24 (m, 3H, 2-CH_2; 6-CH), 5.22 (tdd, 1H, J = 1.4 Hz, J = 2.8 Hz, J = 8.1 Hz, 7-CH), 5.41 (br. s, 1H, 3-CH).

^{13}C ($CDCl_3$, 300 MHz): δ = 18.34 (q), 22.94 (q), 25.7 (q), 35.95 (t, C-5), 65.52 (t, C-2), 70.68 (d, C-6), 119.66 (d, C-3), 125.68 (d, C-7), 131.82 (s, C-8), 136.01 (s, C-4).

(+)-*p*-Menth-1-en-9-al:

Following the procedure of Meinwald and Jones,[S9] (+)-*p*-menth-1-en-9-ol (3 g; 19.5 mmol) in DCM (20 ml) was added to a vigorously stirred solution of pyridinium chlorochromate (PCC) (6.3 g; 0.0292 mol) in DCM (60 ml). After stirring the reaction mixture for 2 h the reaction mixture was diluted with ether (60 ml) and stirred for another hour. The reaction mixture was then filtered through Florosil (Merck Chemical Co.), the solvent evaporated under reduced pressure, and the resultant residue distilled to give two diastereomers of (+)-*p*-menth-1-en-9-al (0.38 g, 13%); bp 82–85 °C (8 mmHg) in a purity of 93% (GC-MS).



MS (70 eV): *m/z* (%) 152 (M^+, <1), 105 (3), 95 (16), 94 (100), 93 (10), 79 (46), 67 (14), 55 (13), 53 (12), 41 (18), 39 (17).

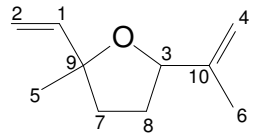
^{1}H NMR ($CDCl_3$, 600 MHz): δ = 1.07 (dd, 3H, J = 7.0 Hz, J = 7.8 Hz, 7-CH_3), 1.60–2.32 (m, 11H), 5.36 (br. s, 1H, 2-CH), 9.67 (dd, 1H, J = 2.5 Hz, J = 7.8 Hz, 8-CHO).

^{13}C ($CDCl_3$, 600 MHz): δ = 10.54 and 10.45 (q, C-7a, C-7a'), 23.60 (q, C-1a, C-1a'), 25.65 (t, C-5), 27.51 (t, C-3), 28.27 (t, C-5'), 29.87 (t, C-3'), 30.35 and 30.19 (t, C-6, C-6'), 34.55 and 34.48 (d, C-4,

C-4'), 51.18 and 50.87 (d, C-7, C-7'), 120.15 and 120.08 (d, C-2, C-2'), 134.27 and 134.23 (s, C-1, C-1'), 205.72 and 205.64 (s, C-8, C-8').

***cis*- and *trans*-Dehydroxylinalool oxide:**

Linalool oxide (1.018 g; 5.99 mmol) was dissolved in hexamethylphosphoric triamide (HMPT) (30 g; 0.168 mol)[S10] and refluxed for 1 h. The reaction mixture was allowed to cool, pentane (25 ml) was added, the solution was washed with brine (3 x 60 ml), dried on anhydrous Na_2SO_4, and the solvent removed to yield a product (0.23 g, 25%) consisting of 42% *cis*-and *trans*-dehydroxylinalool oxide ~~(~~in a ratio of 66:34~~)~~ and 58% of an unknown compound (GC-MS). The *cis*- and *trans*-dehydroxylinalool oxide was further purified by dissolving a quarter of the crude product (0.2 ml) in an equal volume of DCM. This solution was then chromatographed on a 20 × 20 cm silica (2 mm thickness) glass plate (Kieselgel 60 F_{254}, Merck) using ether:petroleum ether (3:1) as mobile phase. One of the bands was scraped from the plate, extracted with deuterochloroform (5 ml) in a 25-ml flask, and filtered. The purity of the dehydroxy isomers relative to the unknown compound had improved to 75:25, according to GC-MS analysis. The identity of the compound was confirmed by comparison of published ^{1}H NMR (Ohloff *et al.*, 1964) and MS (Adams, 2004) data with the experimental data.



MS (70 eV): *m/z* (%) 152 (M^+, 4,), 137 (14), 119 (10), 110 (22), 109 (18), 105 (9), 96 (28), 91 (19), 82 (27), 81 (20), 79 (16), 68 (59), 67 (100), 55 (47), 53 (15), 43 (56), 41 (47), 39 (37).
^{13}C ($CDCl_3$, 600 MHz): δ = 18.08 (*cis*) and 18.31 (*trans*) (q, C-6), 26.84 and 26.46 (q, C-5), 30.65 and 31.0 (t, C-8), 36.97 and 37.33 (t, C-7), 81.97 and 82.16 (d, C-3), 83.24 and 82.84 (s, C-9), 110.23 and 110.39 (t, C-4), 111.46 and 111.44 (t, C-2), 143.85 and 144.50 (d, C-1), 146.09 and 145.62 (s, C-10).

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