

Simple Green Purification of Spilanthol from Natural Deep Eutectic Solvent and Ethanolic *Acmella oleracea* (L.) R.K.Jansen Extracts using Solid Phase Extraction

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Supplementary Materials

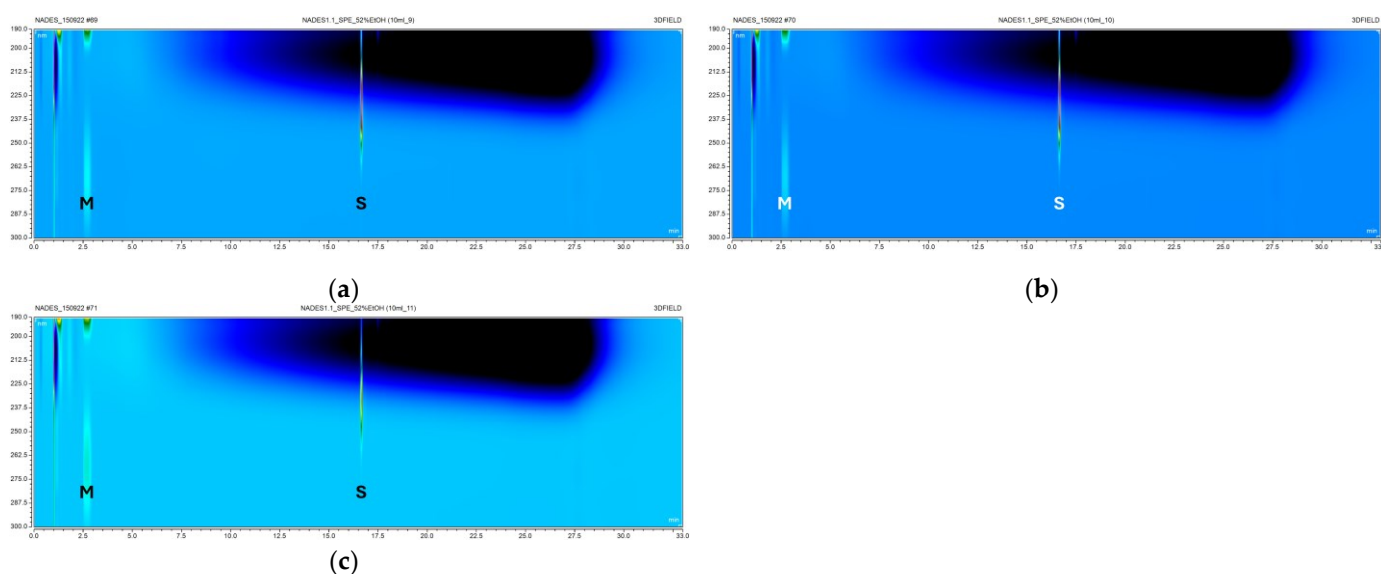


Figure S1. 3D chromatograms (190–300 nm) of extract ChCl/MeU purest SPE fractions, eluted with 52% ethanol. (a) Fraction 9; (b) Fraction 10; (c) Fraction 11; M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthol.

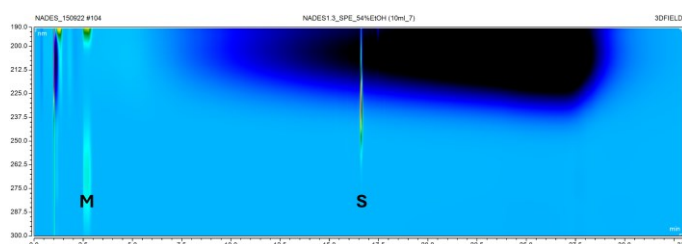


Figure S2. 3D chromatogram (190–300 nm) of extract ChCl/MeU purest SPE fraction 7, eluted with 54% ethanol. M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthol.

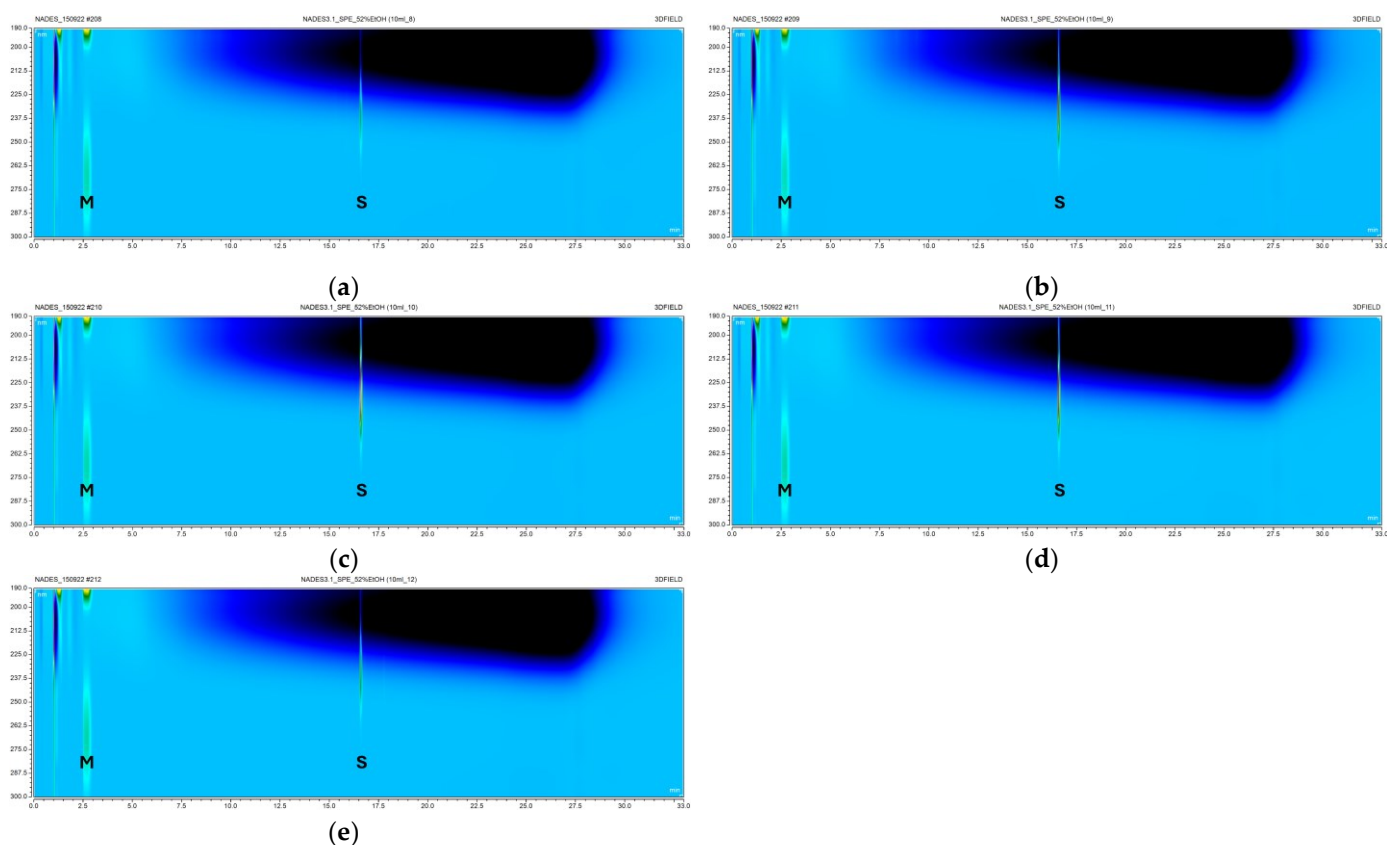


Figure S3. 3D chromatograms (190-300 nm) of extract ChCl/P purest SPE fractions, eluted with 52% ethanol. (a) Fraction 8; (b) Fraction 9; (c) Fraction 10; (d) Fraction 11; (e) Fraction 12; M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthal.

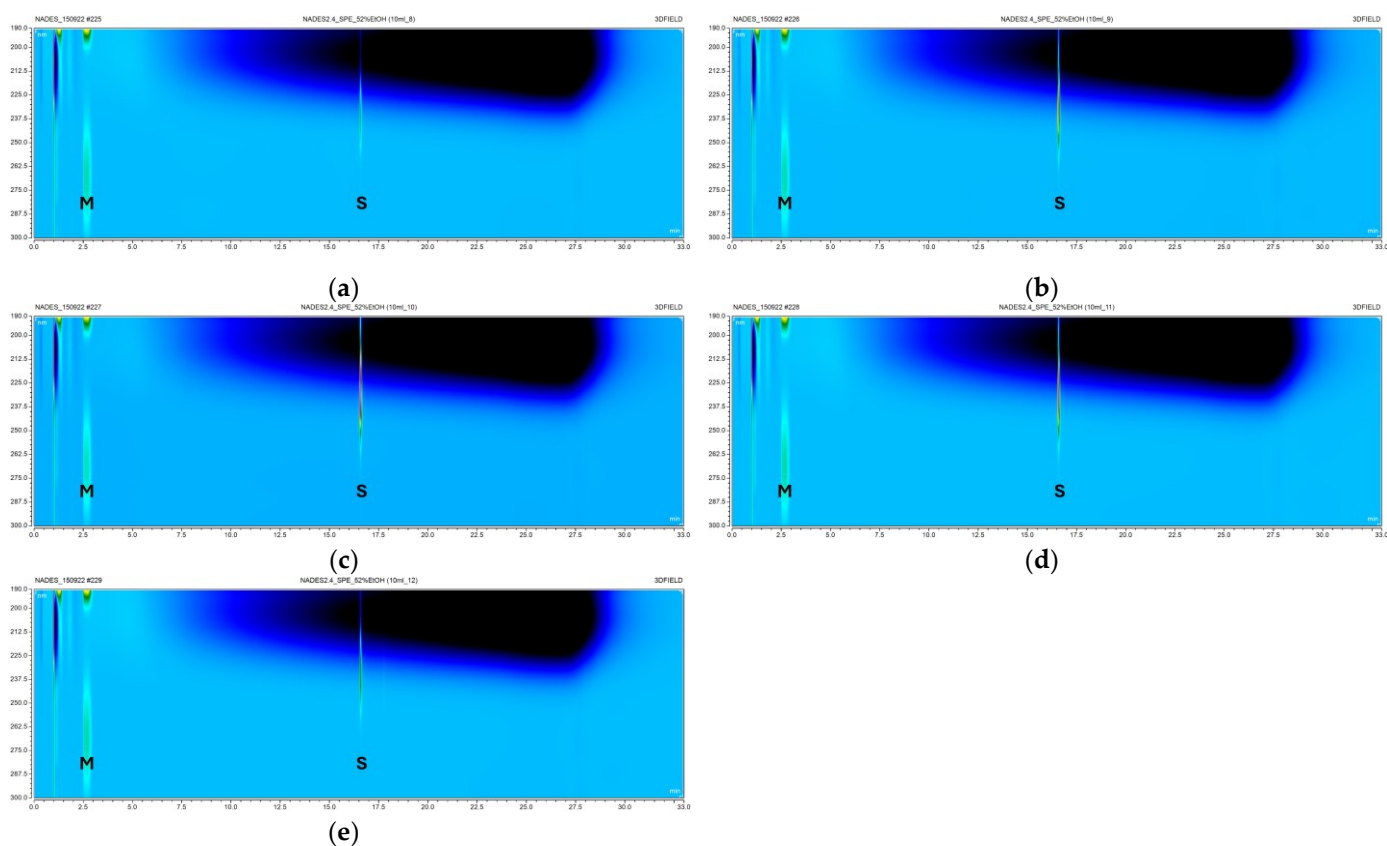


Figure S4. 3D chromatograms (190-300 nm) of extract ChCl/C purest SPE fractions, eluted with 52% ethanol. (a) Fraction 8; (b) Fraction 9; (c) Fraction 10; (d) Fraction 11; (e) Fraction 12; M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthal.

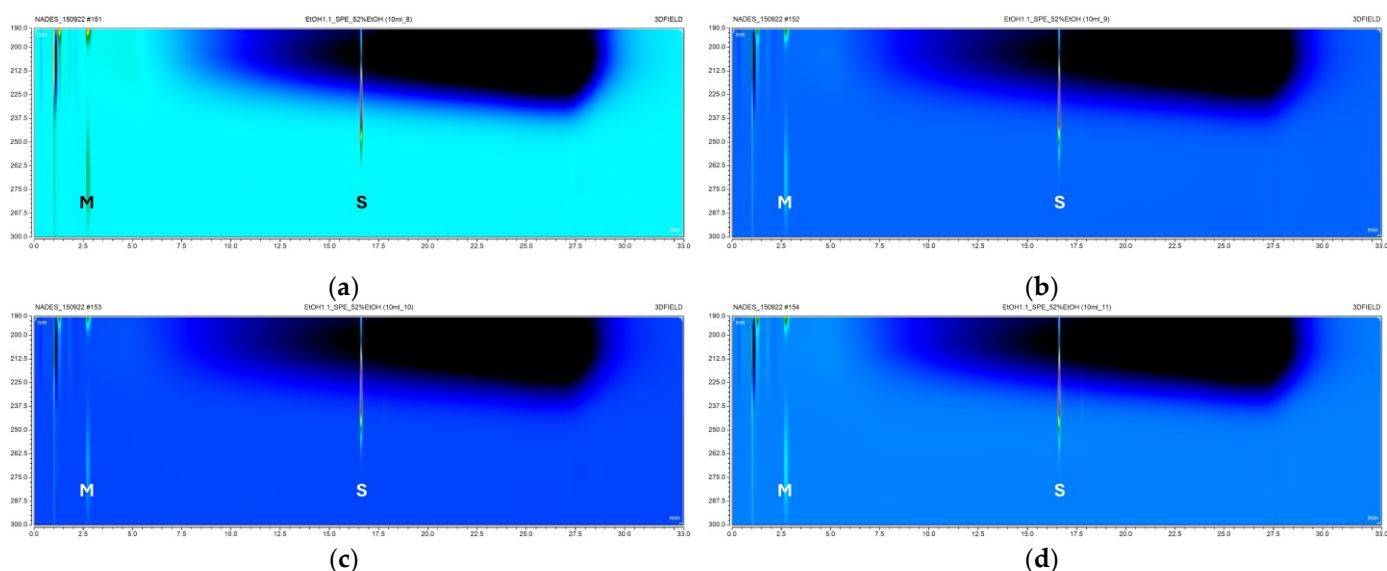


Figure S5. 3D chromatograms (190-300 nm) of extract EtOH 100 mg purest SPE fractions, eluted with 52% ethanol. (a) Fraction 8; (b) Fraction 9; (c) Fraction 10; (d) Fraction 11; M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthol.

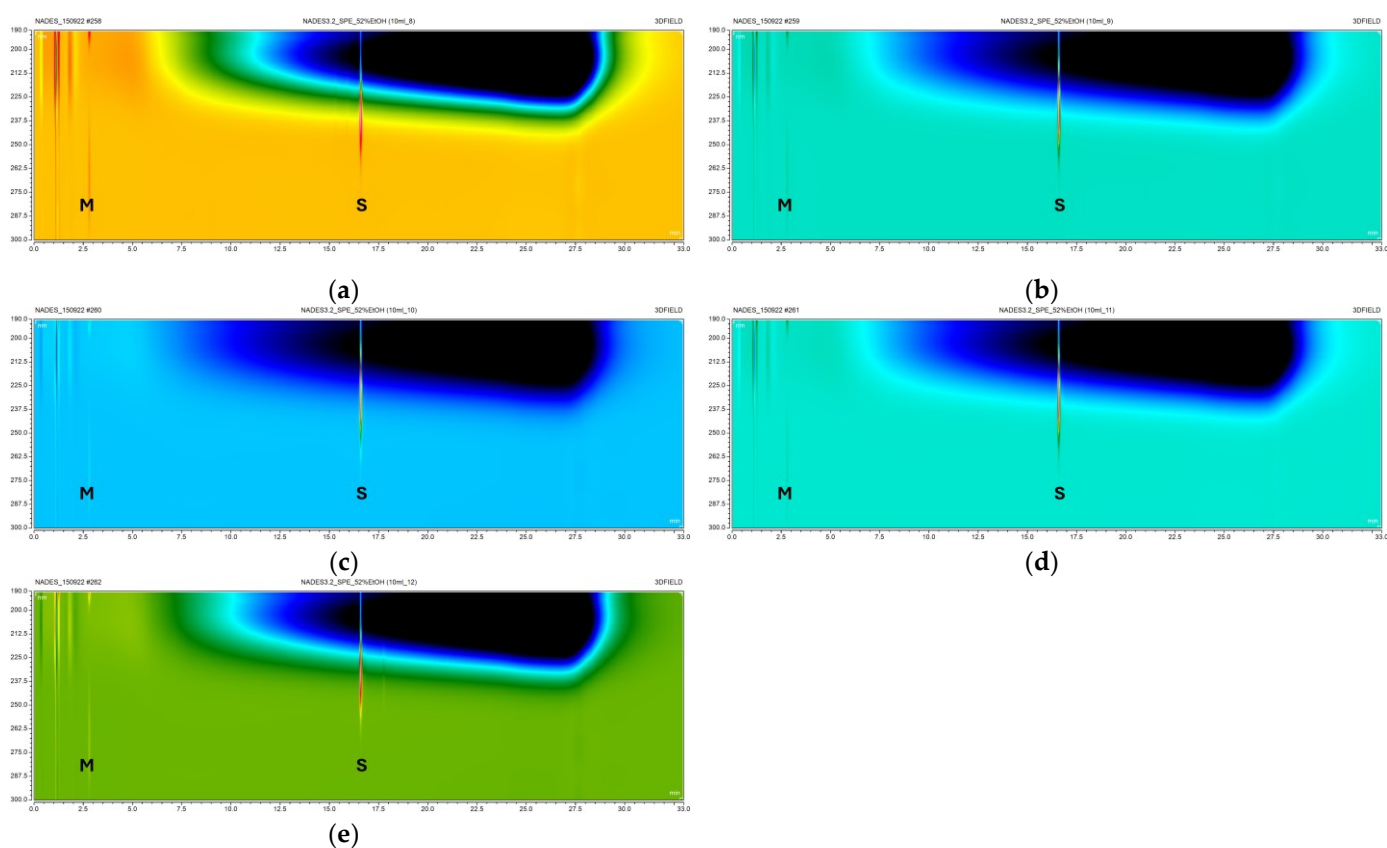


Figure S6. 3D chromatograms (190-300 nm) of extract ChCl/P (100 mL) purest SPE fractions, eluted with 52% ethanol. (a) Fraction 8; (b) Fraction 9; (c) Fraction 10; (d) Fraction 11; (e) Fraction 12; M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthol.

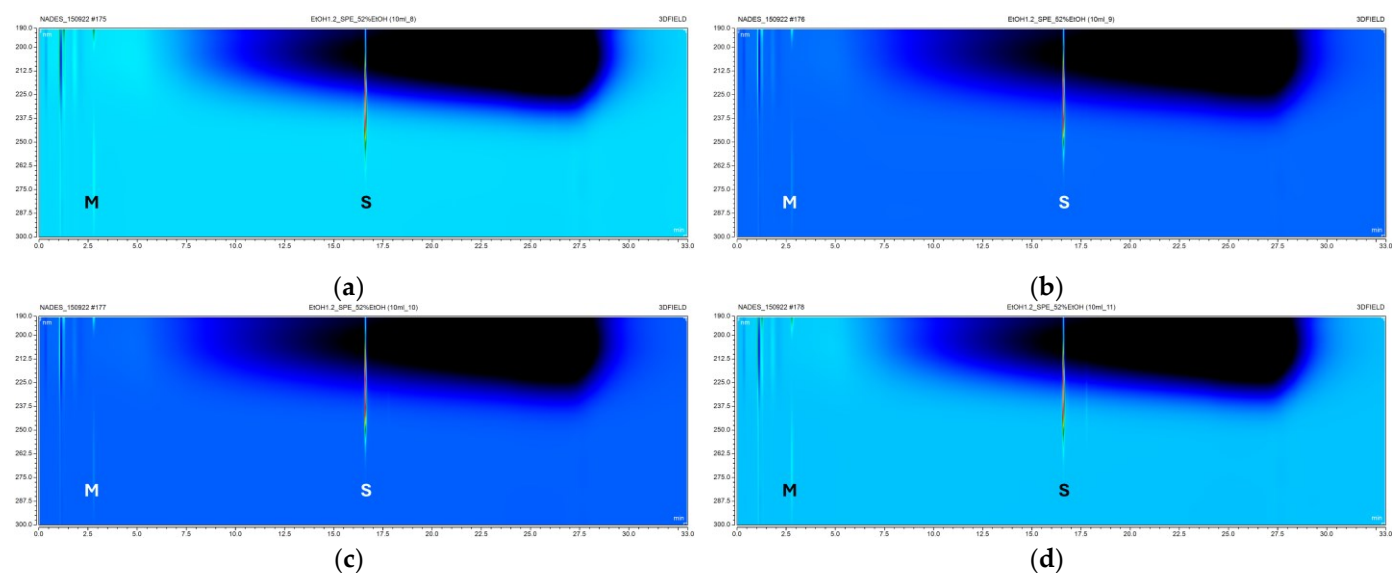


Figure S7. 3D chromatograms (190-300 nm) of extract EtOH 500 mg purest SPE fractions, eluted with 52% ethanol. (a) Fraction 8; (b) Fraction 9; (c) Fraction 10; (d) Fraction 11; M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthol.

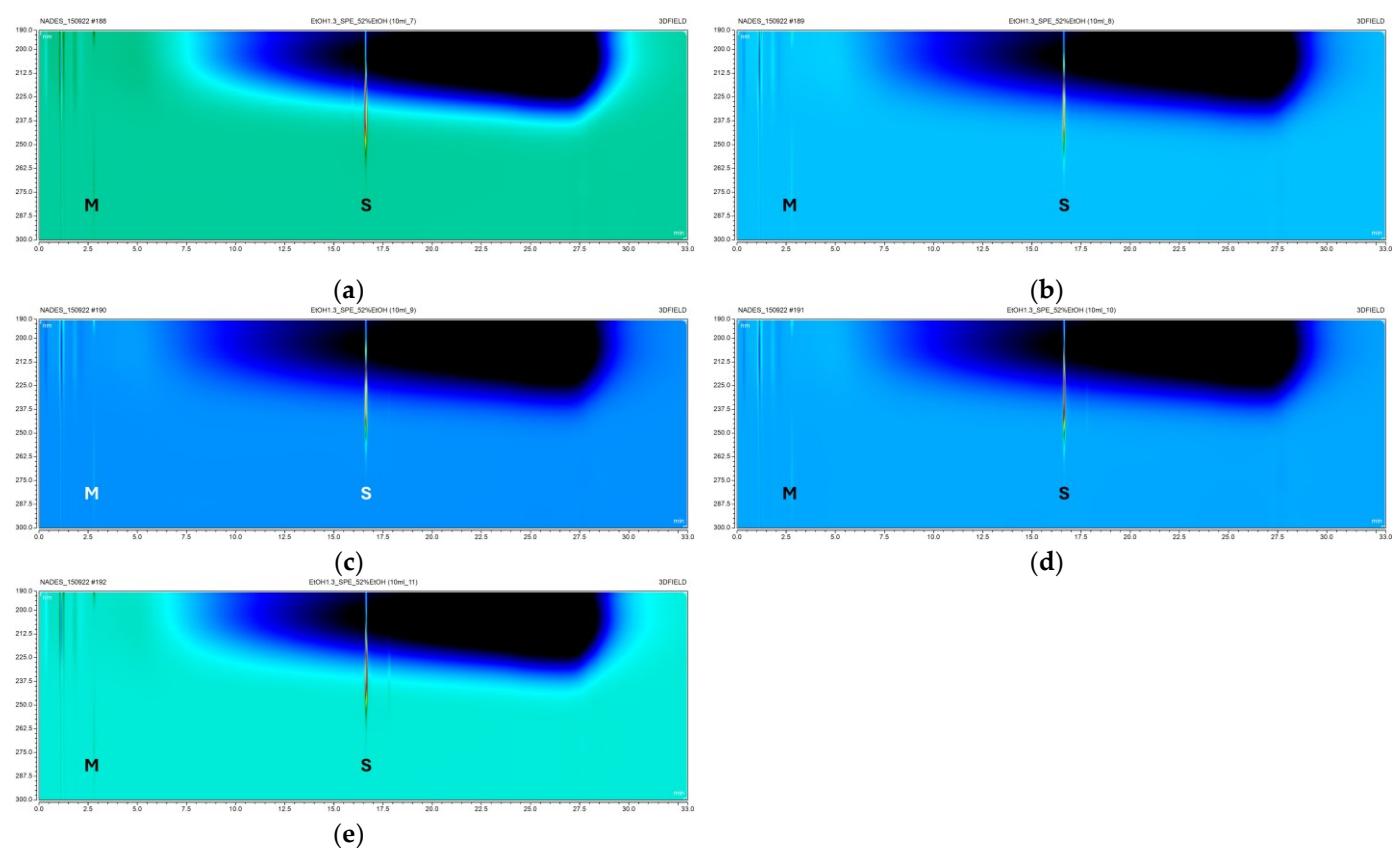


Figure S8. 3D chromatograms (190-300 nm) of extract EtOH 1000 mg (1) purest SPE fractions, eluted with 52% ethanol. (a) Fraction 7; (b) Fraction 8; (c) Fraction 9; (d) Fraction 10; (e) Fraction 11; M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthol.

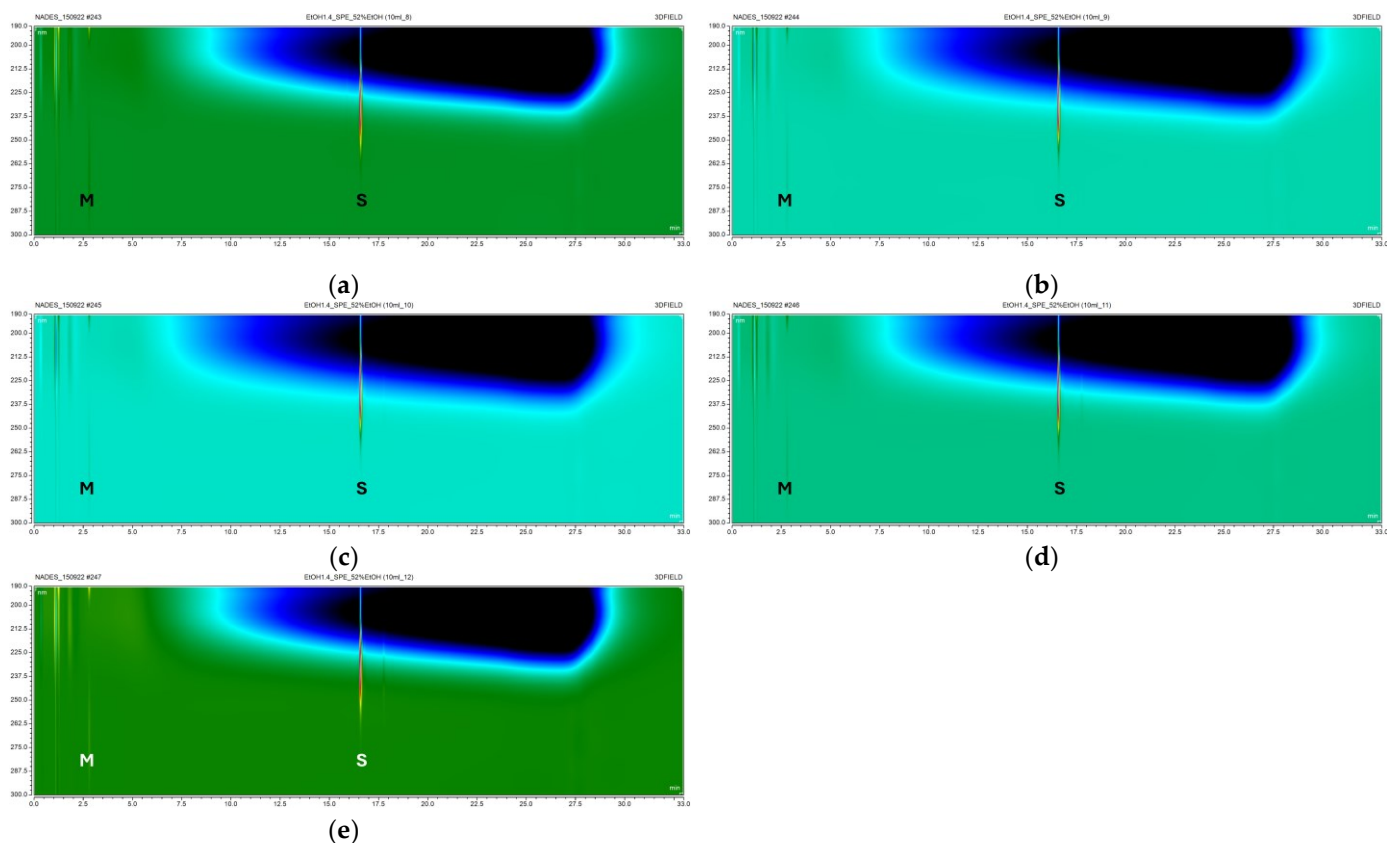


Figure S9. 3D chromatograms (190-300 nm) of extract EtOH 1000 mg (2) purest SPE fractions, eluted with 52% ethanol. (a) Fraction 8; (b) Fraction 9; (c) Fraction 10; (d) Fraction 11; (e) Fraction 12; M: methyl ethyl ketone from partly denatured ethanol in samples; S: spilanthol.

Table S1. Proton (100 MHz, CDCl_3) and carbon NMR data of spilanthol. The experimental temperature was 25 °C, and TMS was used as the internal standard.

Position	δ ^1H [ppm], Mult. ¹	δ ^{13}C [ppm]
1	-	166.0
2	5.81, brd (J = 15.4 Hz)	124.2
3	6.83, dt (J = 15.3 Hz, J = 6.7 Hz)	143.5
4	2.26, m	32.1
5	2.33, m	26.4
6	5.26, dt (J = 10.7 Hz, J = 6.9 Hz)	127.7
7	5.98, t (J = 10.8 Hz)	129.4
8	6.29, dd (J = 13.0 Hz, J = 11.0 Hz)	126.7
9	5.69, m	130.0
10	1.78, d (J = 6.5 Hz)	18.3
1'	3.14, t (J = 6.6 Hz)	46.9
2'	1.80, m	28.6
3'	0.92, d (J = 6.7 Hz)	20.1
4'	0.92, d (J = 6.7 Hz)	20.1
-NH	5.68, m	-

¹ d: doublet, brd: broad doublet, dd: doublet of doublets, t: triplet, dt: doublet of triplets, m: multiplet, multiplicities are given only for not overlapping resonances.