

Supporting Information

A method for small molecule discovery based on microscale preparative multidimensional gas chromatography isolation with nuclear magnetic resonance spectroscopy

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Table S1. Samples and acquisition parameters used for 2D NMR experiments at 500 MHz and 800 MHz with cryoprobe (CD₃OD)

2D NMR Experiment	500 MHz	800 MHz + cryoprobe
gCOSY	100 µg/mL std sample ^a 16 h 25 min nt = 308, ni = 128	110.8 µg/ mL prep sample ^b 2 h 45 min nt = 64, ni = 128
	12.3 µg/mL prep sample ^c 14 h 58 min nt = 280, ni = 128	12.3 µg/mL prep sample ^c ≈ 2 h nt = 48, ni = 128
		12.3 µg/mL prep sample ^c ≈ 8 h nt = 230, ni = 128
gHSQC	110.8 µg/mL prep sample ^b 15 h 54 min nt = 144, ni = 128	110.8 µg/mL prep sample ^b 10 h 12 min nt = 256, ni = 128
gHMBC	110.8 µg/mL prep sample ^b 16 h 27 min nt = 244, ni = 200	110.8 µg/mL prep sample ^b 15 h 53 min nt = 300, ni = 128

^a Geraniol standard sample. Geraniol preparative sample isolated from ^b100 injections and ^c10 injections of the essential oil matrix with the prep-MDGC system. 2D NMR results of both data sets are presented in **Table 2** and displayed in **Figure S2** and **Figure S3** for 800 MHz data only.

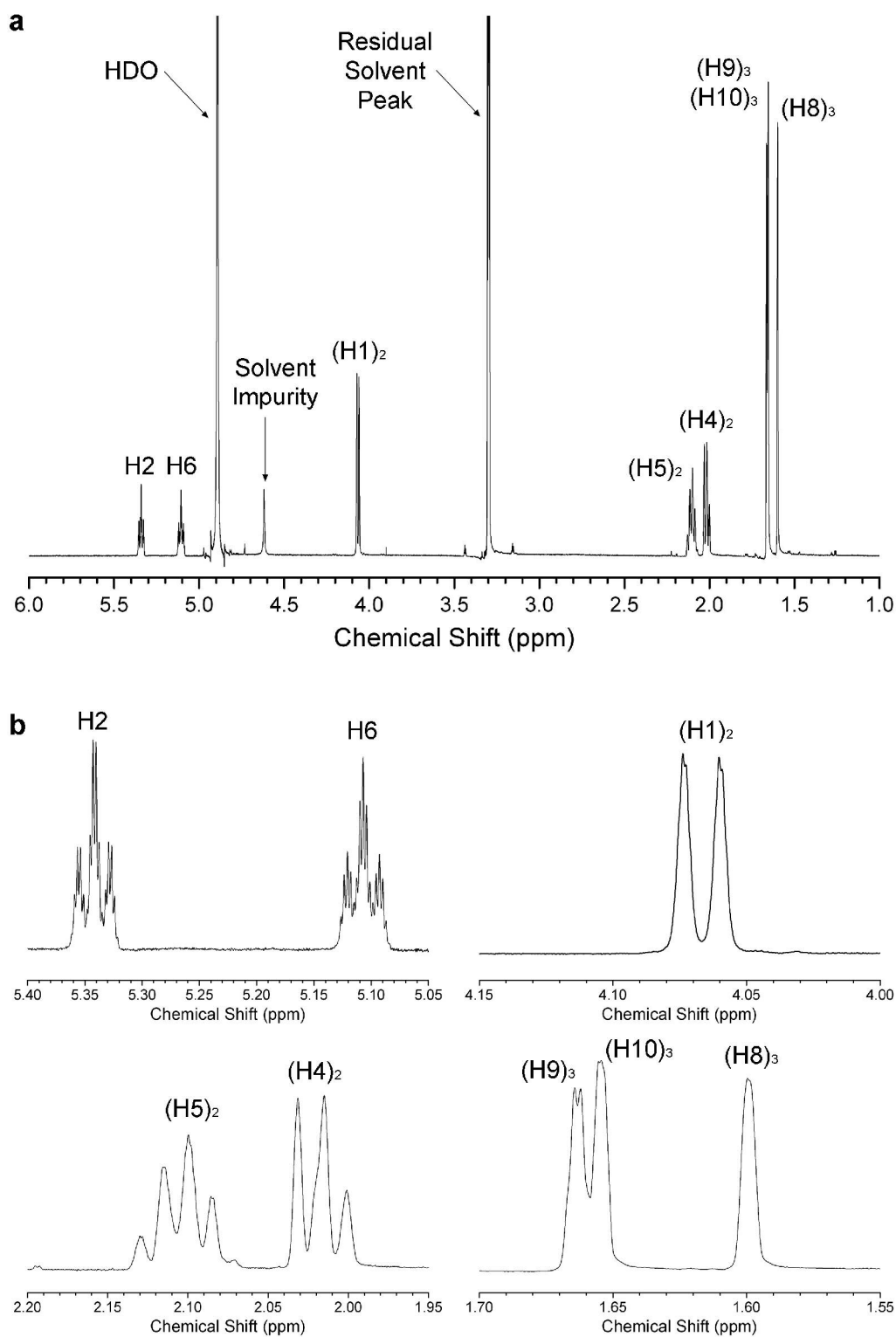


Figure S1. ^1H NMR spectrum of geraniol standard (500 MHz, CD_3OD).

Data acquired on a standard solution of 1 mg/mL (nt = 16). (a) Full spectrum. (b) Zoomed regions of the spectrum. Chemical shifts, multiplicity and coupling constants of the corresponding proton resonances are provided in **Table 2**.

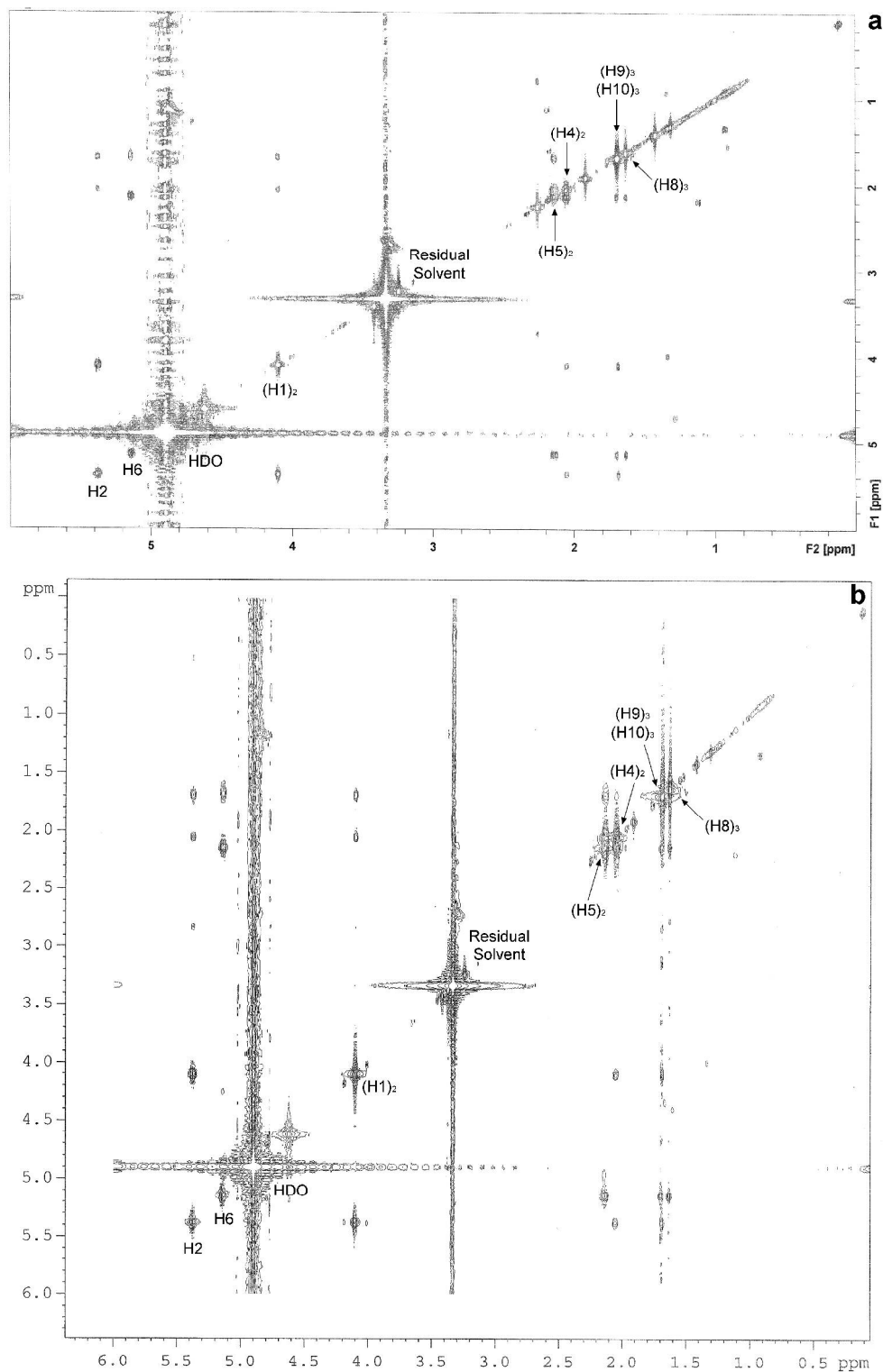


Figure S2. 2D homonuclear NMR spectra of geraniol isolated by preparative MDGC (800 MHz with cryoprobe, CD₃OD).

(a) gCOSY (8 h) of 10 injection sample (12.3 μg/mL), (b) gCOSY of 100 injection sample (110.8 μg/mL), with correlations listed in **Table 2** and acquisition parameters given in **Table S1**.

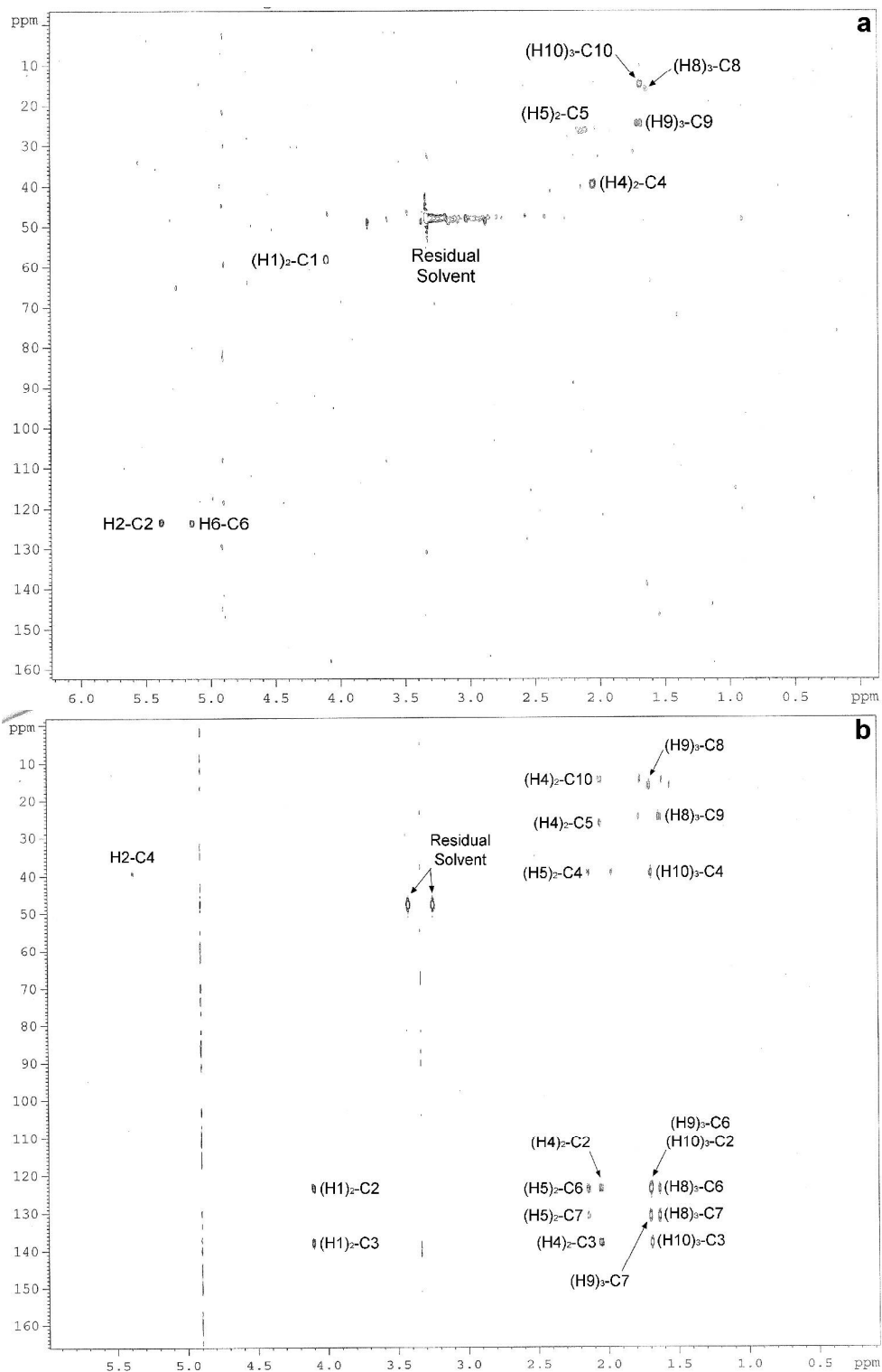


Figure S3. 2D heteronuclear NMR spectra of geraniol isolated by preparative MDGC (800 MHz with cryoprobe, CD_3OD).

(a) gHSQC of 100 injection sample (110.8 $\mu\text{g/mL}$), (b) gHMBC of 100 injection sample (110.8 $\mu\text{g/mL}$), with correlations listed in **Table 2** and acquisition parameters given in **Table S1**.