Differential reactivity of purified bioactive coffee furans, cafestol and kahweol, with acidic nitrite: product characterization and factors controlling nitrosation versus ring opening pathways.

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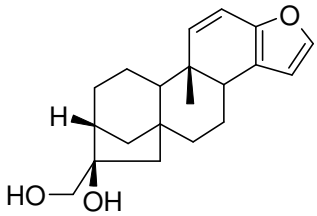
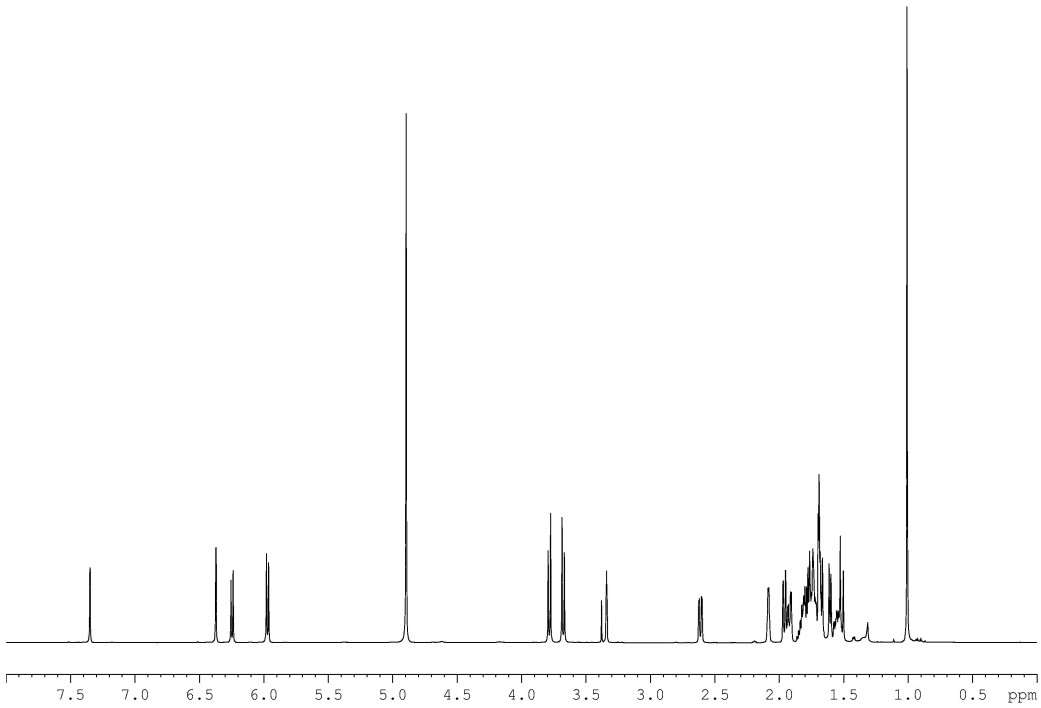
NMR spectra

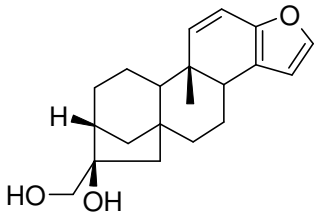
Spectra were acquired using 256 equally spaced evolution-time period t_1 values averaging 16 transients of 2048 points, with 6024 Hz of spectral width. Time-domain data matrices were all zero-filled to 4K in both dimensions, thus yielding a digital resolution of 2.94 Hz/pt. Prior to Fourier transformation, a Lorentz-to-Gauss window with different parameters was applied for both t_1 and t_2 dimensions for all the experiments. ROESY spectra were obtained with 200-ms mixing time, and the recycle time was set to 5 times the longest T_1. TOCSY experiments were recorded with spin-lock periods of 64 ms, achieved with the MLEV-17 pulse sequence. Linear prediction was applied to extend the data to twice their length in t_1. The ^{1}H and ^{13}C natural abundance HSQC and HMBC spectra were recorded at 300 K on the DRX-600 spectrometer, operating at 150.90 MHz for ^{13}C. One hundred twenty eight equally spaced evolution time period t_1 values were acquired, averaging 48 transients of 2048 points and using GARP4 for decoupling. The final data matrix was zero-filled to 4096 in both dimensions, and apodized before Fourier transformation by a shifted cosine window function in t_2 and in t_1. Linear prediction was also applied to extend the data to twice their length in t_1.

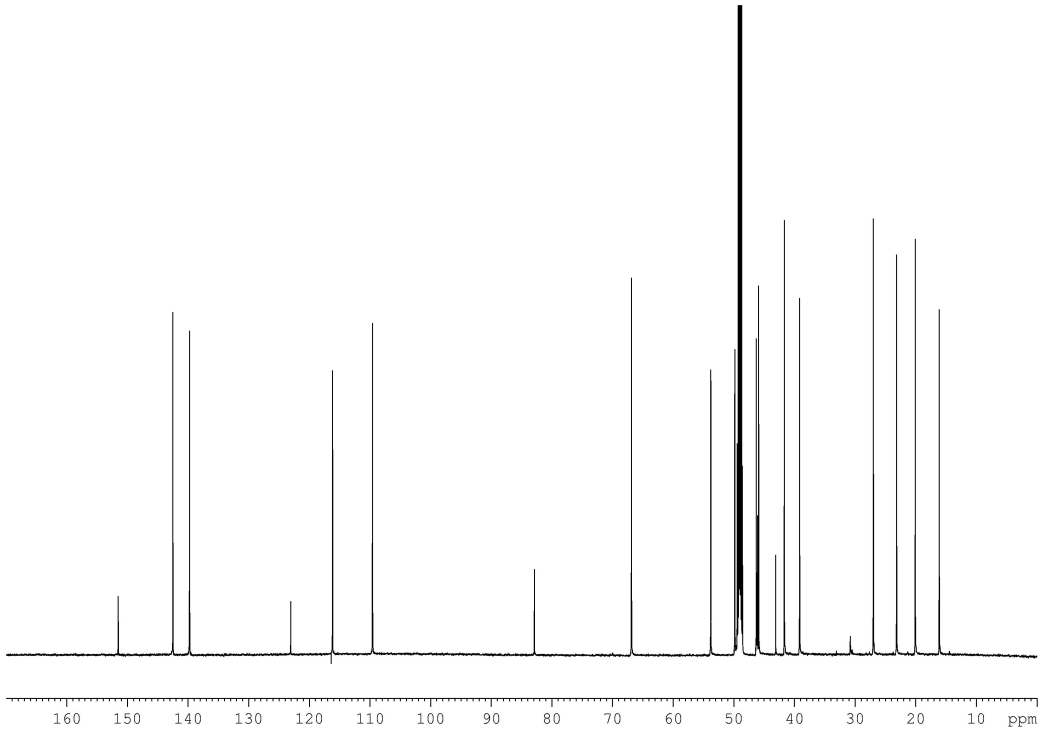
^{1}H NMR spectrum of kahweol in CD_3OD



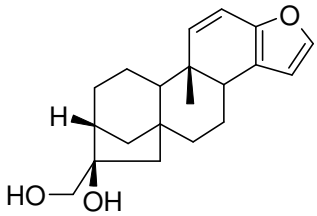


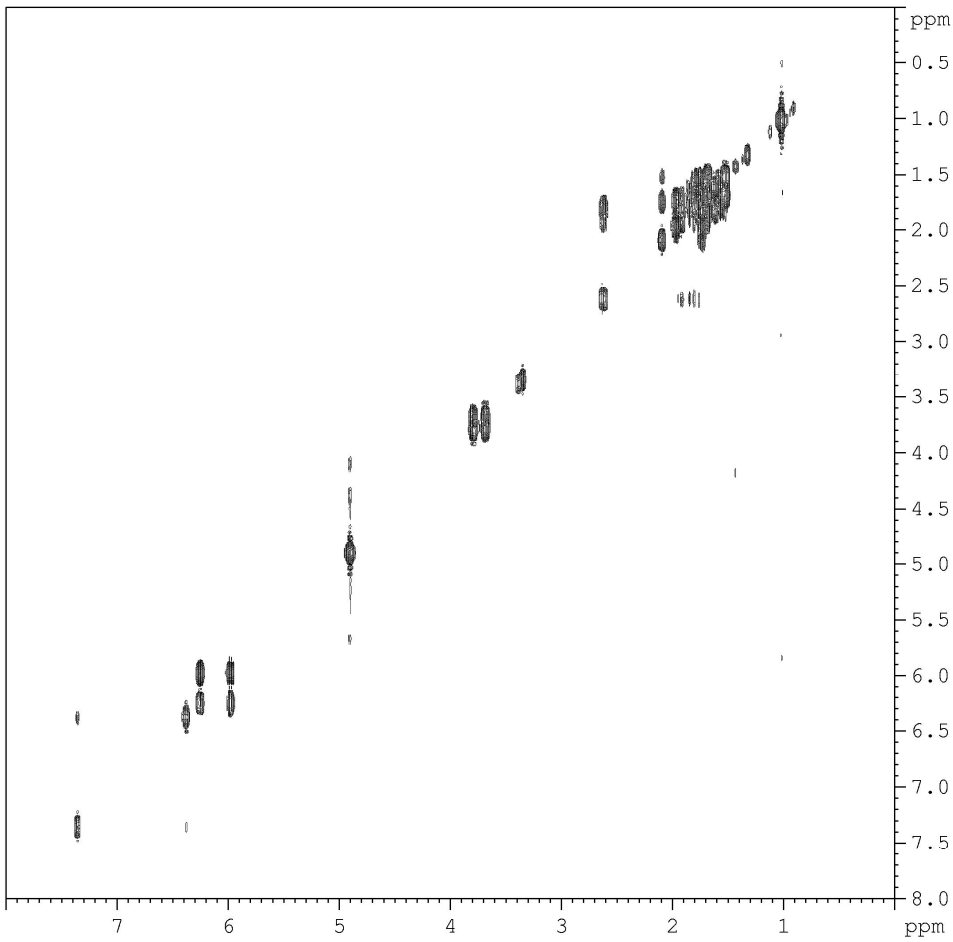
^{13}C NMR spectrum of kahweol in CD_3OD



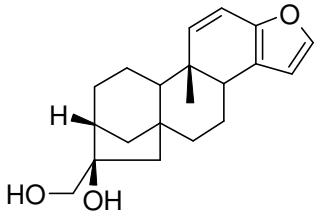


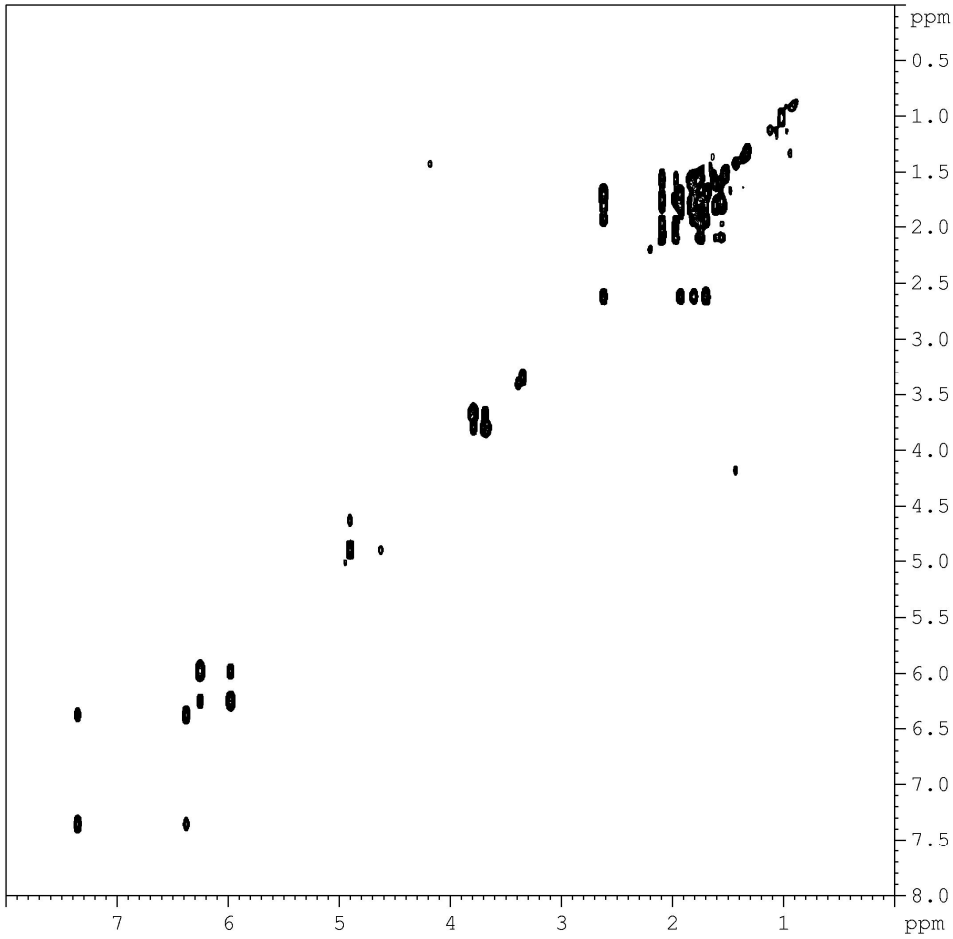
COSY spectrum of kahweol in CD_3OD



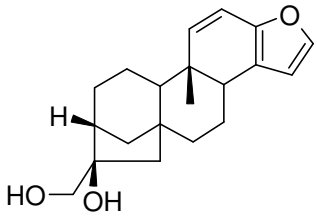


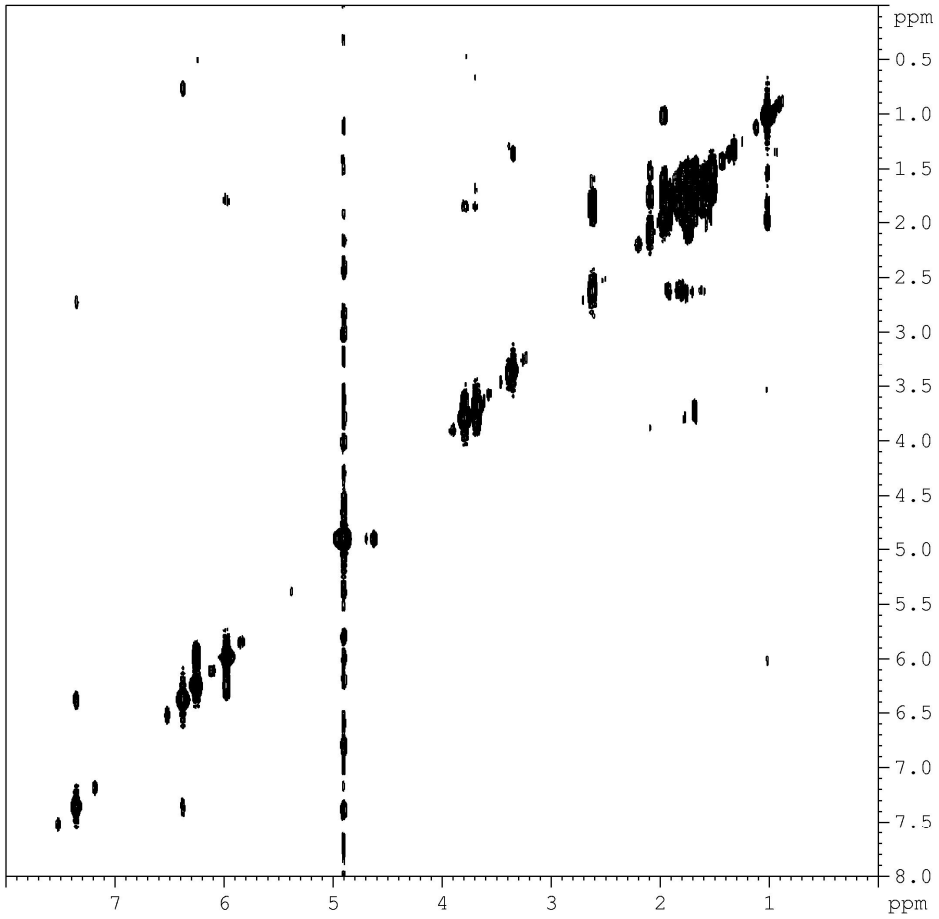
TOCSY spectrum of kahweol in CD_3OD



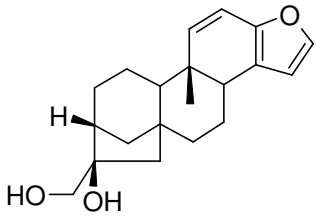


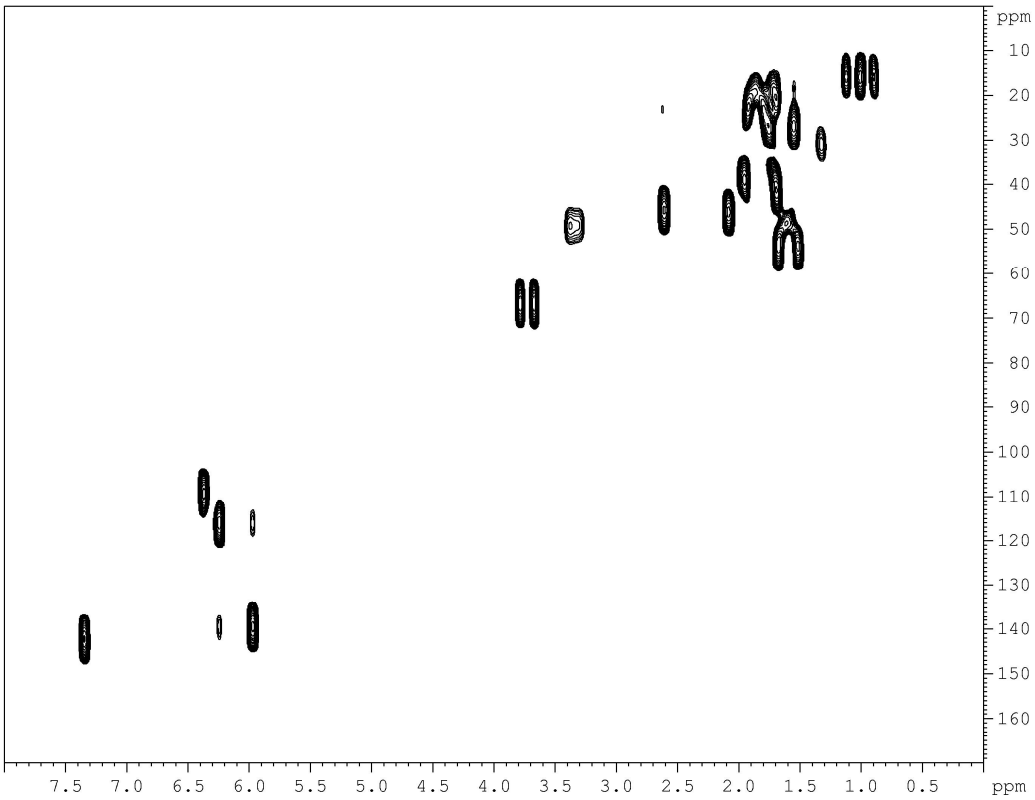
ROESY spectrum of kahweol in CD_3OD



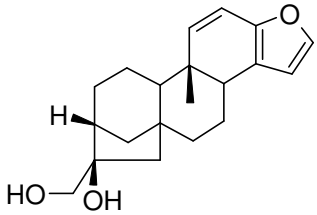


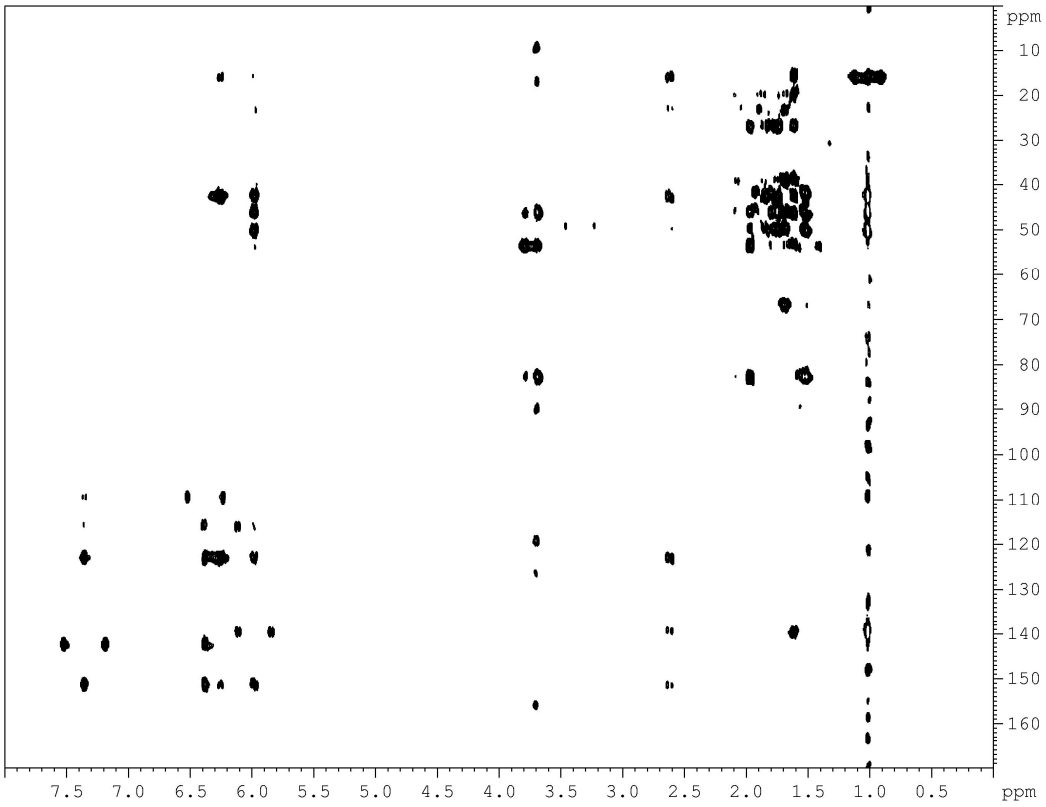
^{1}H, ^{13}C HSQC spectrum of kahweol in CD_3OD



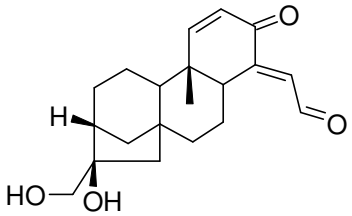


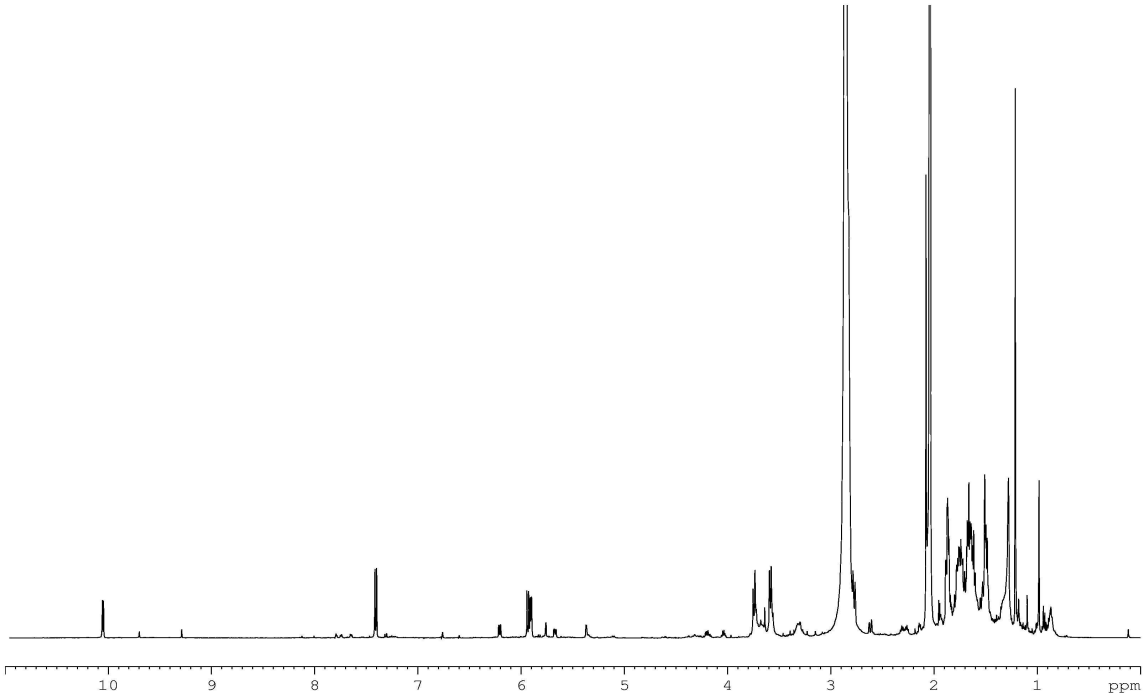
^{1}H, ^{13}C HMBC spectrum of kahweol in CD_3OD



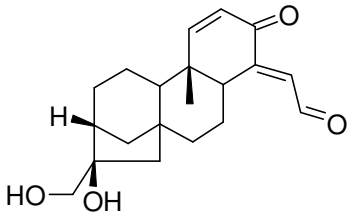


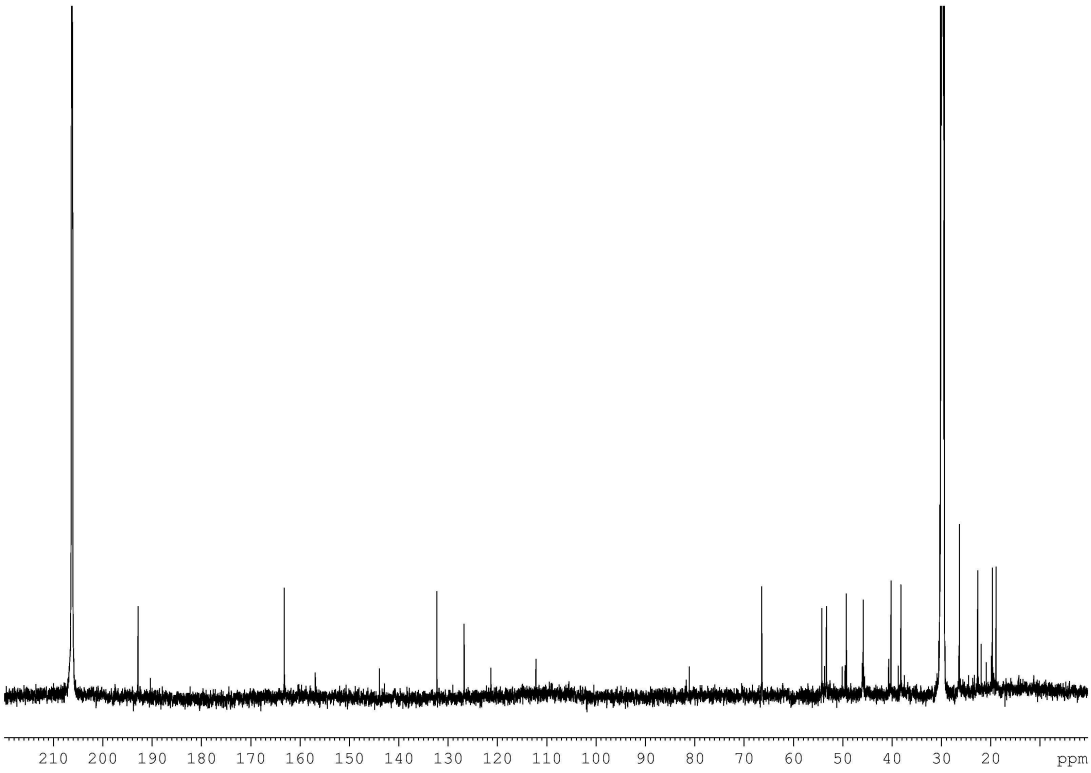
^{1}H NMR spectrum of compound **1** in acetone-d_6



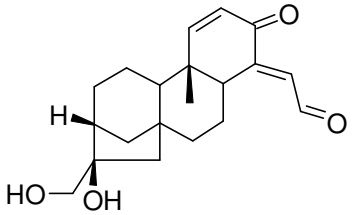


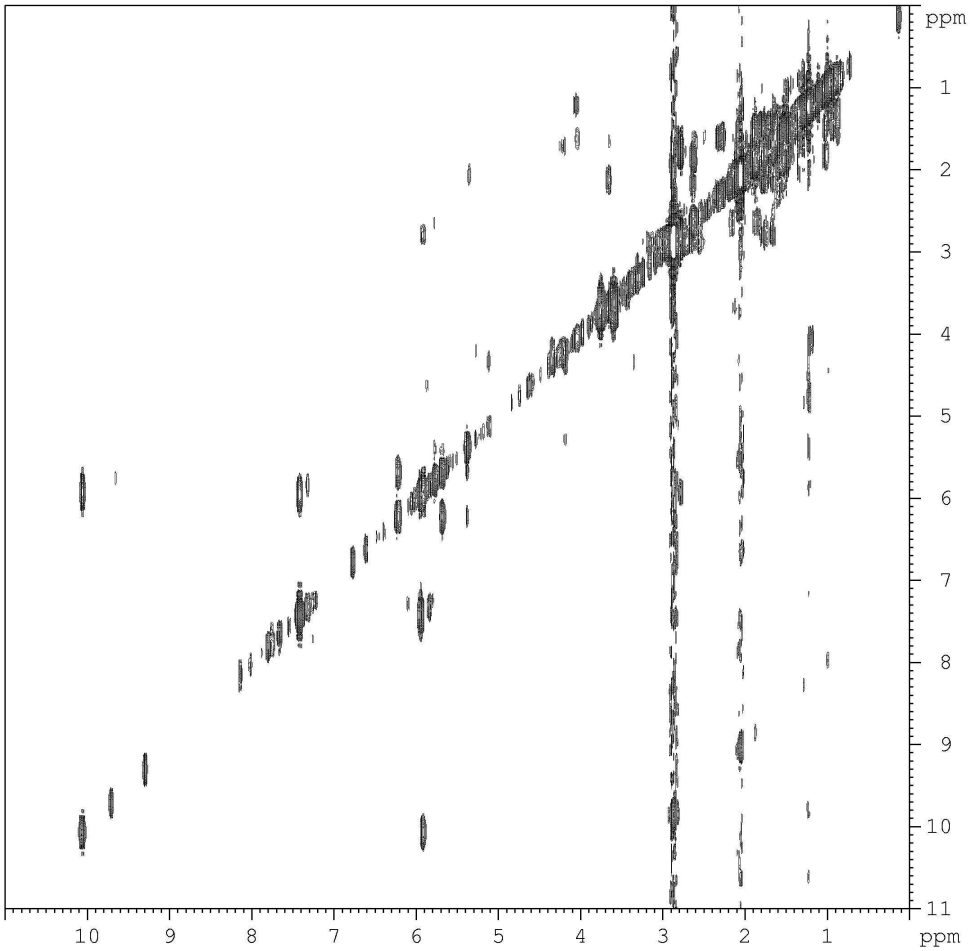
^{13}C NMR spectrum of compound **1** in acetone-d_6



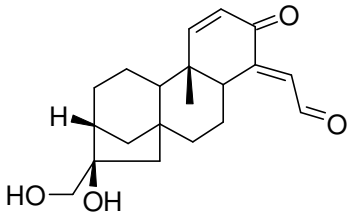


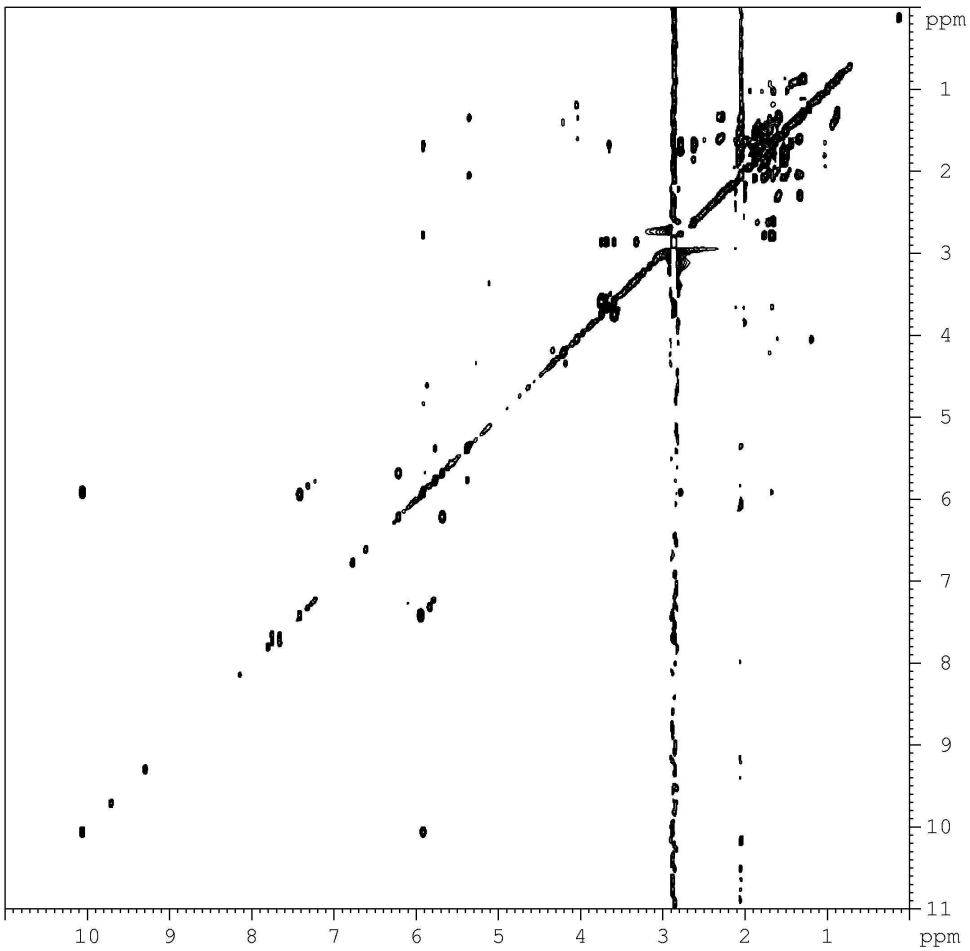
COSY spectrum of compound **1** in acetone-d_6



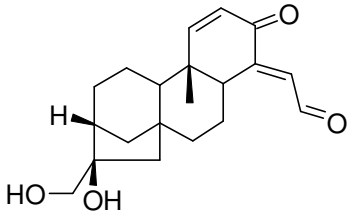


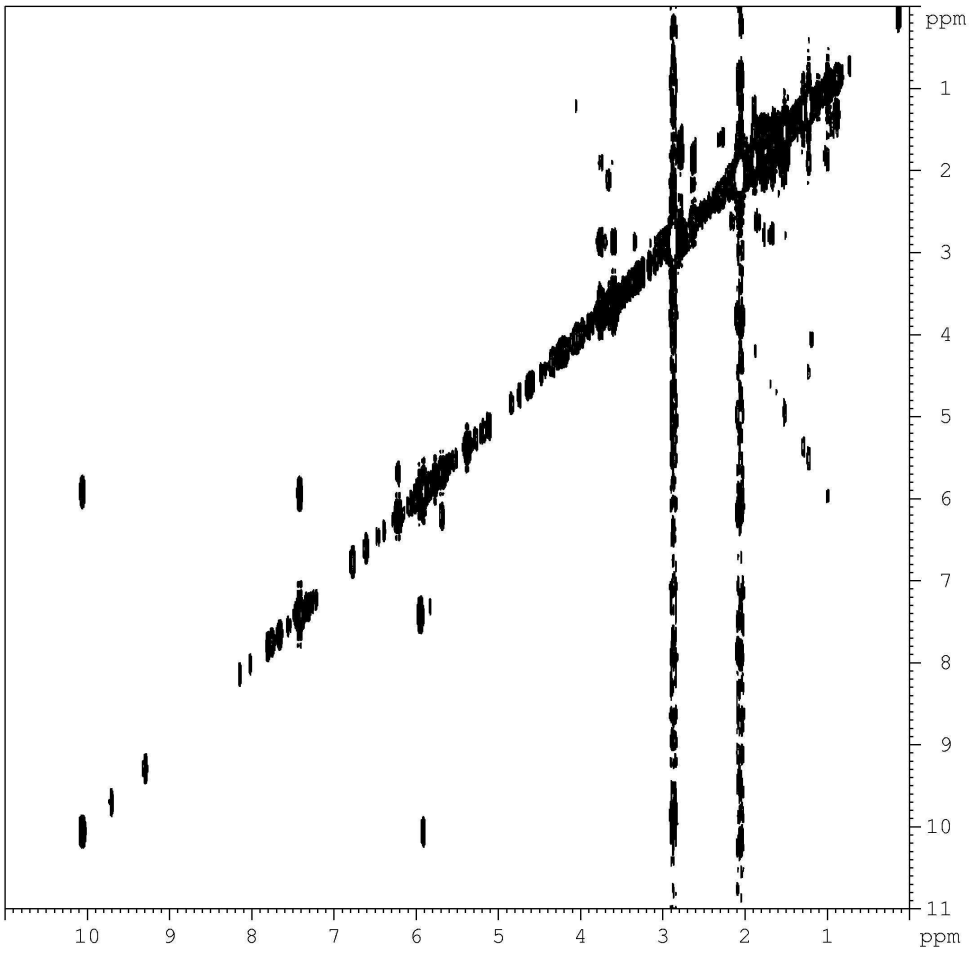
TOCSY spectrum of compound **1** in acetone-d_6



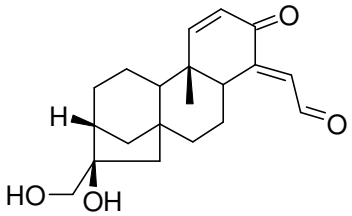


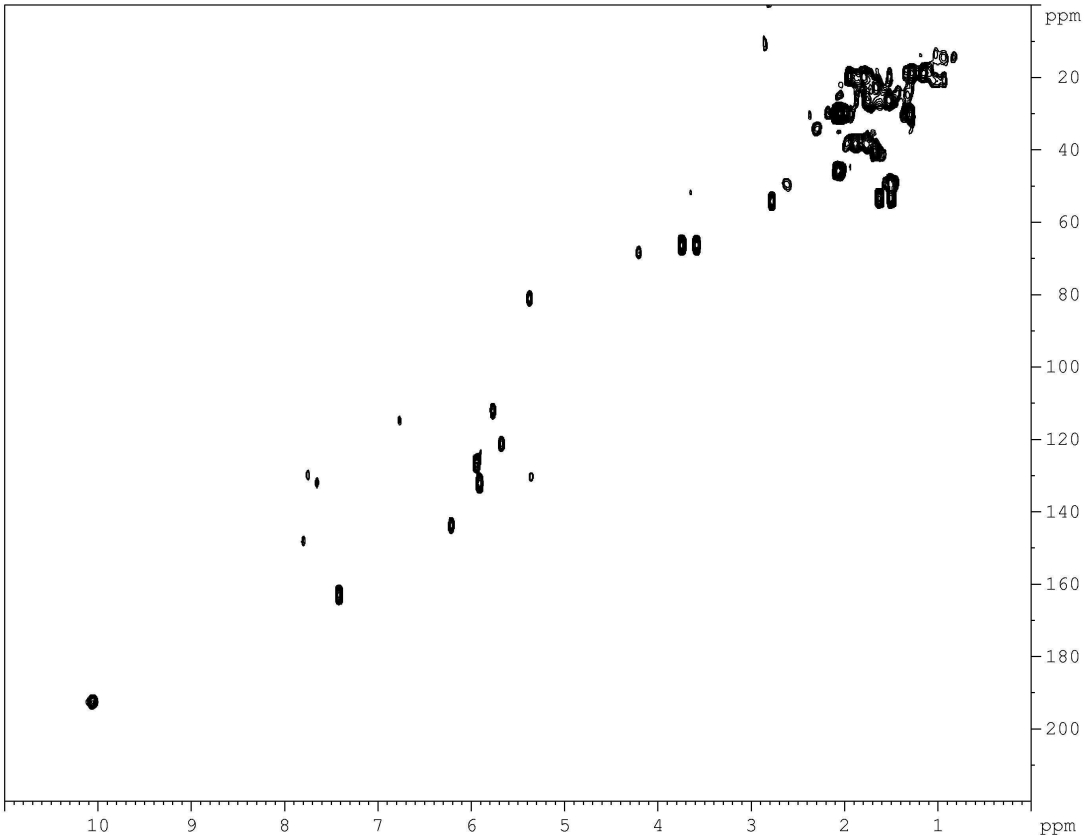
ROESY spectrum of compound **1** in acetone-d_6



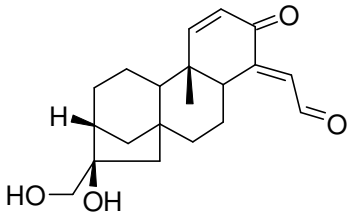


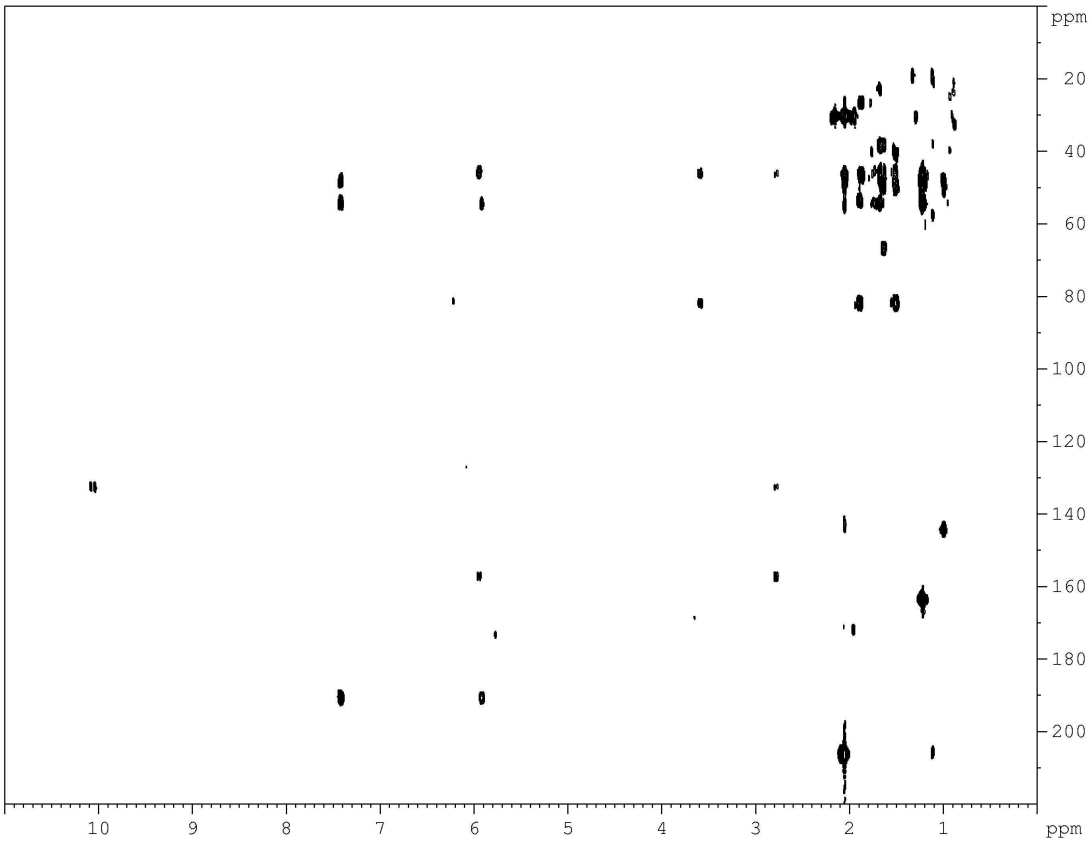
^{1}H, ^{13}C HSQC spectrum of compound **1** in acetone-d_6



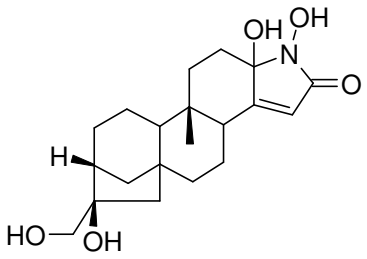


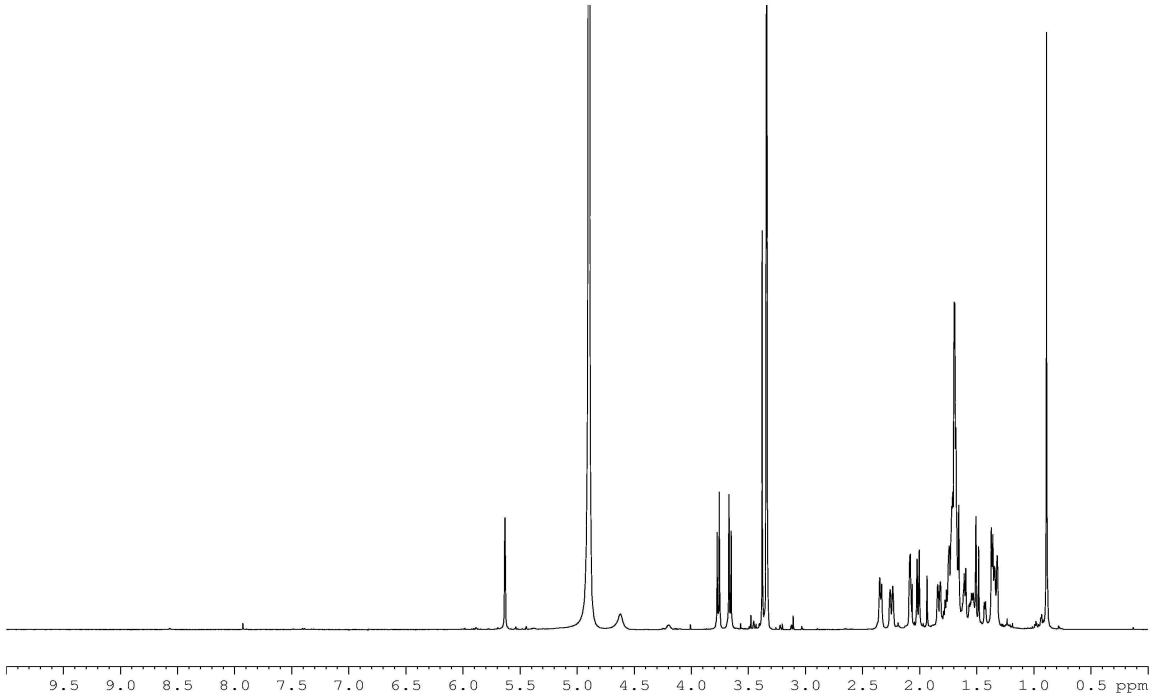
^{1}H, ^{13}C HMBC spectrum of compound **1** in acetone-d_6



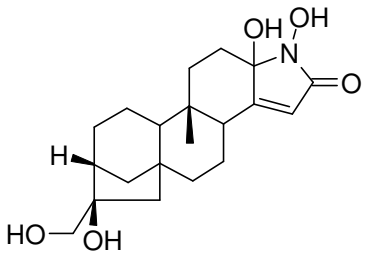


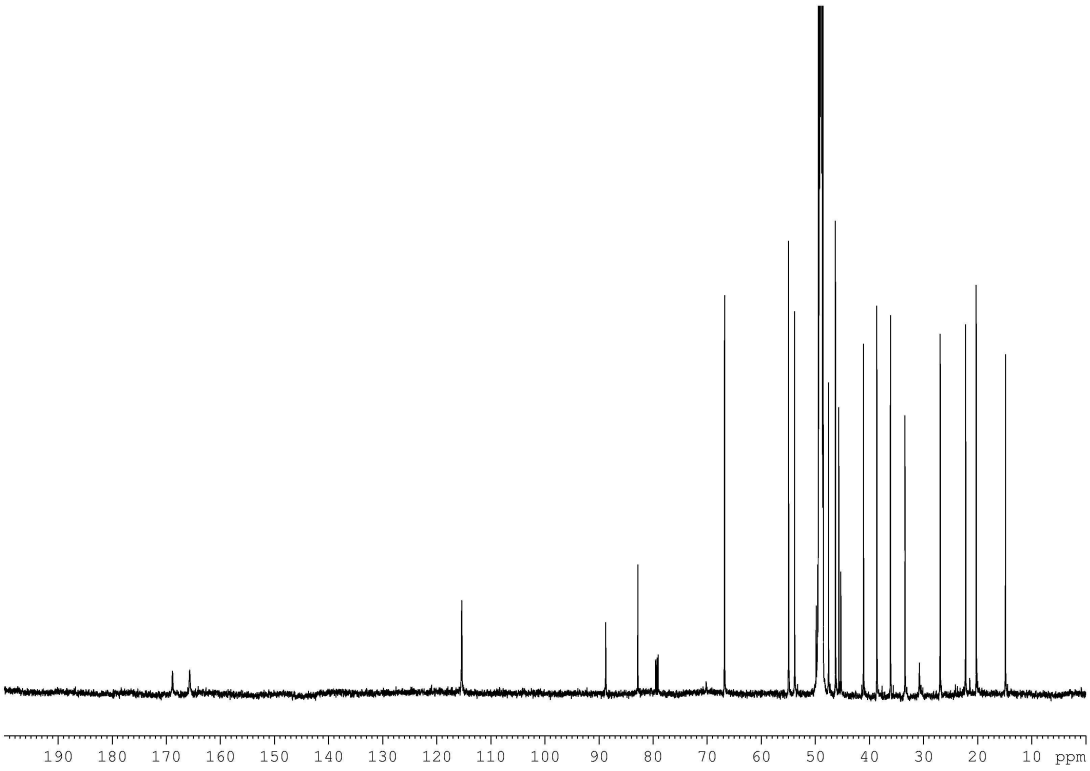
^{1}H NMR spectrum of compound **2** in CD_3OD



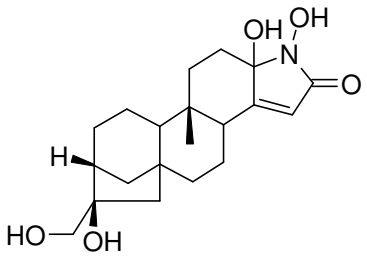


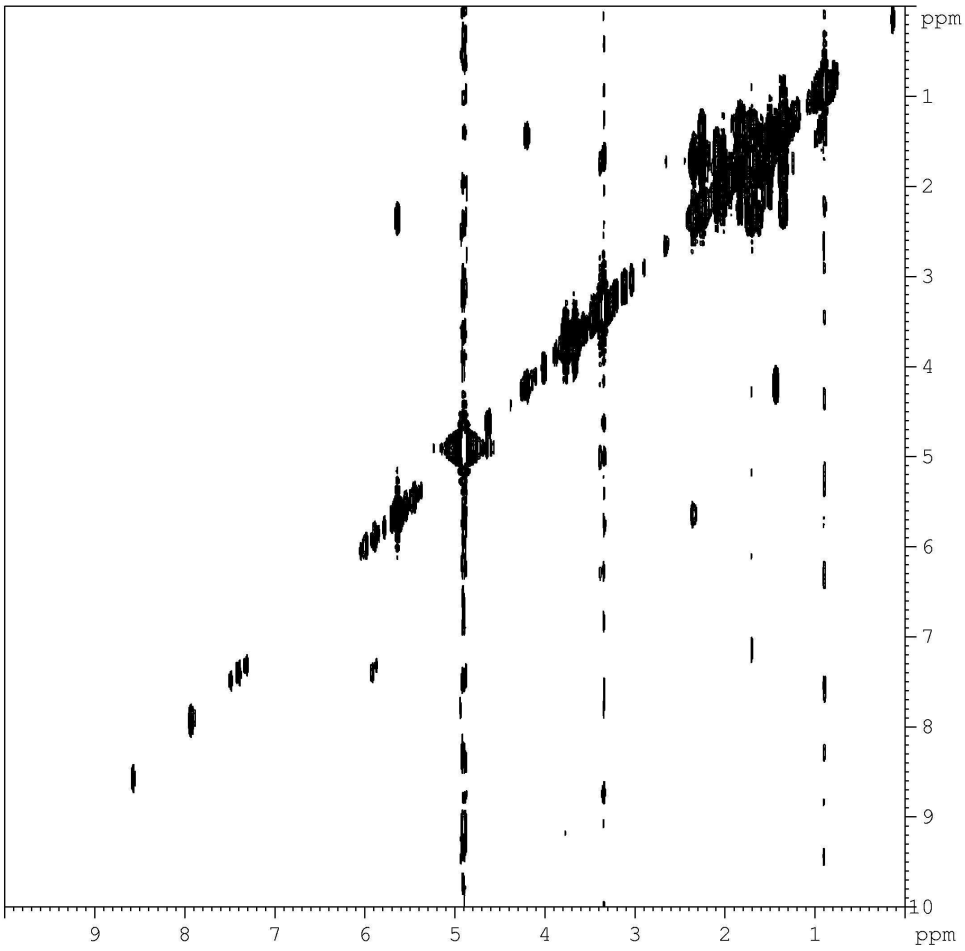
^{13}C NMR spectrum of compound **2** in CD_3OD



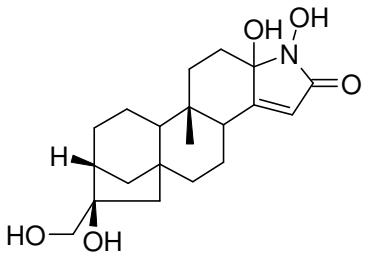


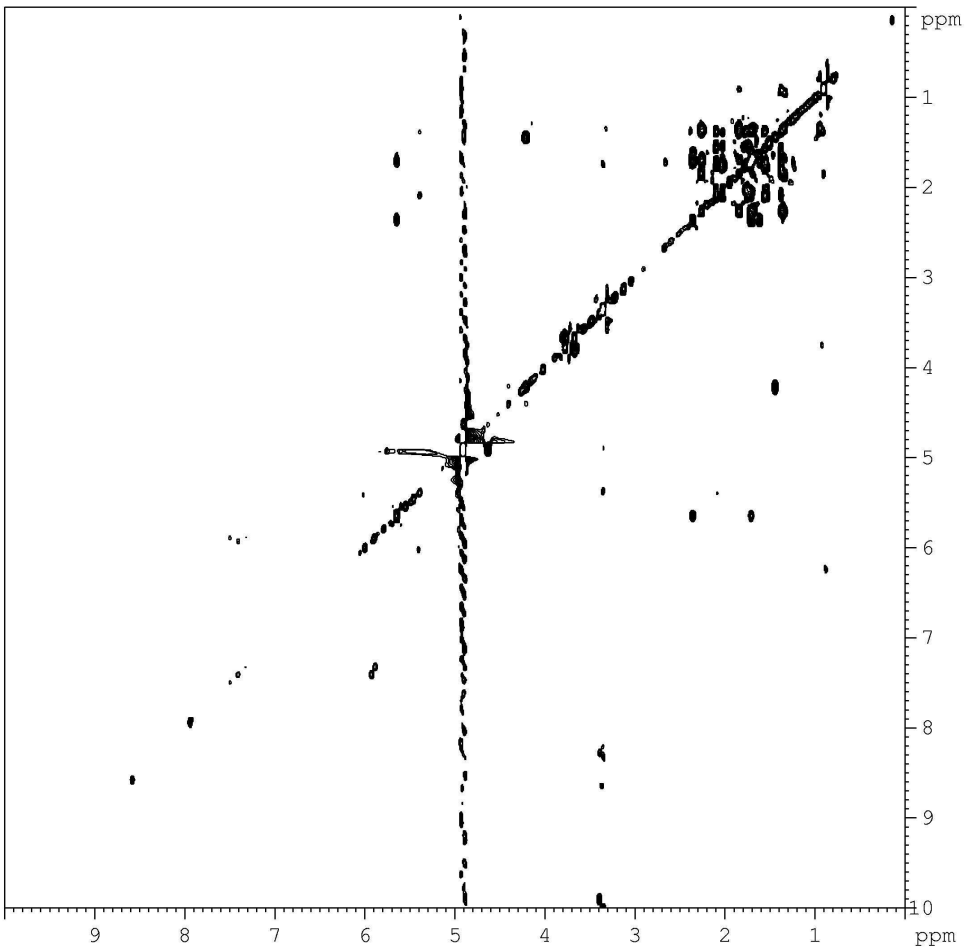
COSY spectrum of compound **2** in CD_3OD



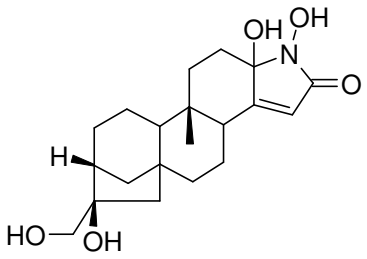


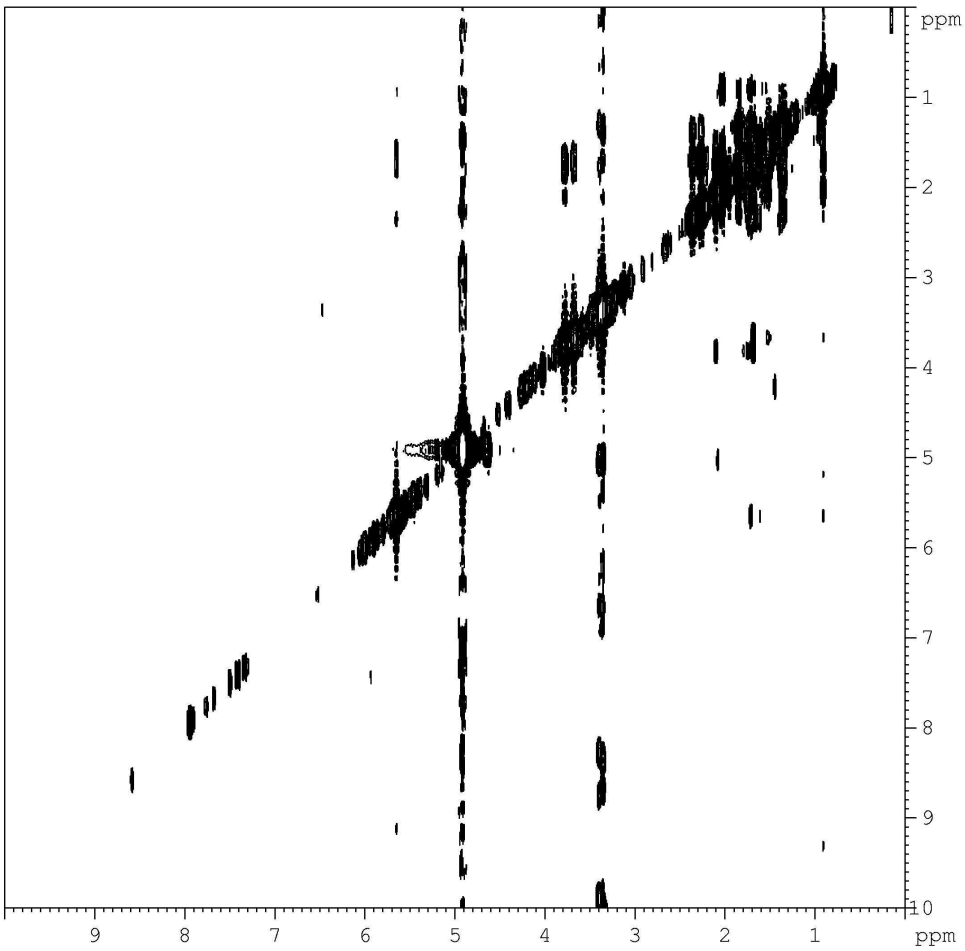
TOCSY spectrum of compound **2** in CD_3OD



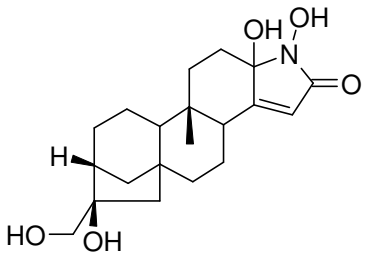


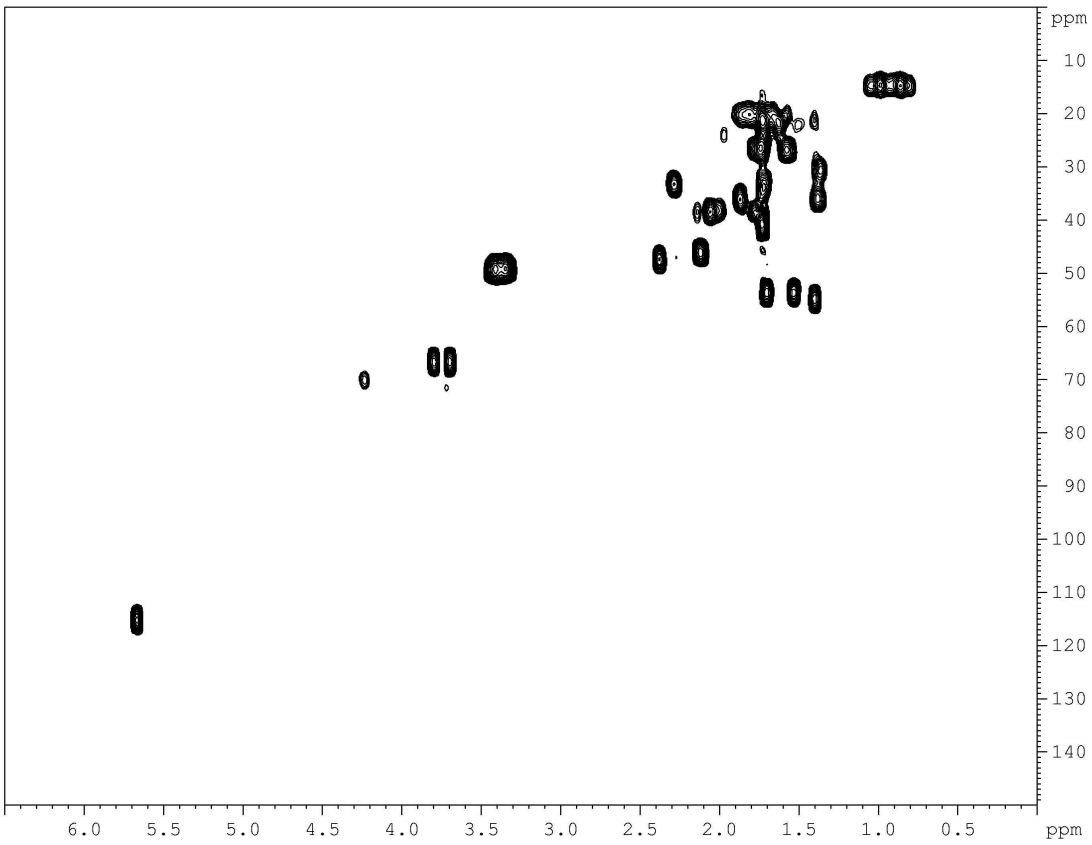
ROESY spectrum of compound **2** in CD_3OD



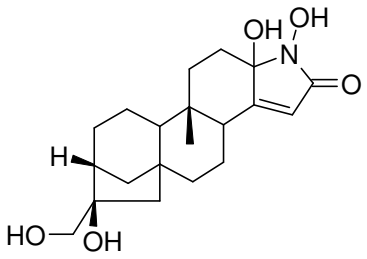


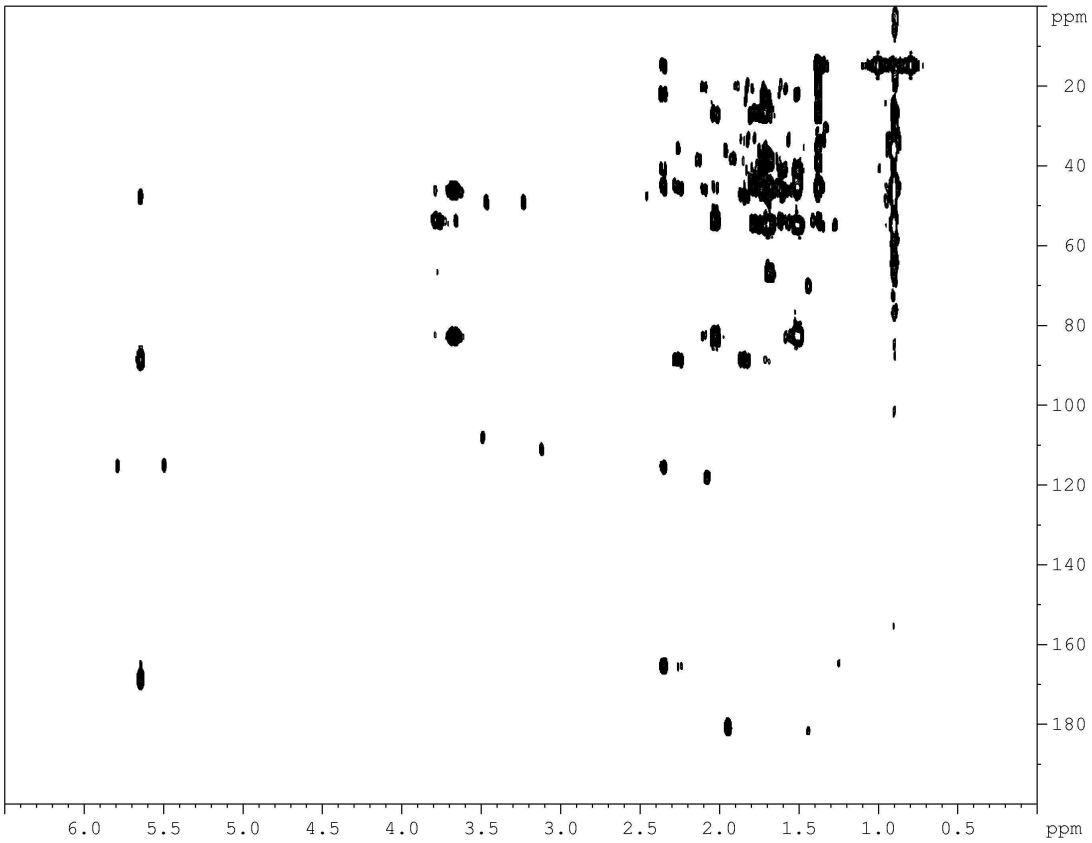
^{1}H, ^{13}C HSQC spectrum of compound **2** in CD_3OD





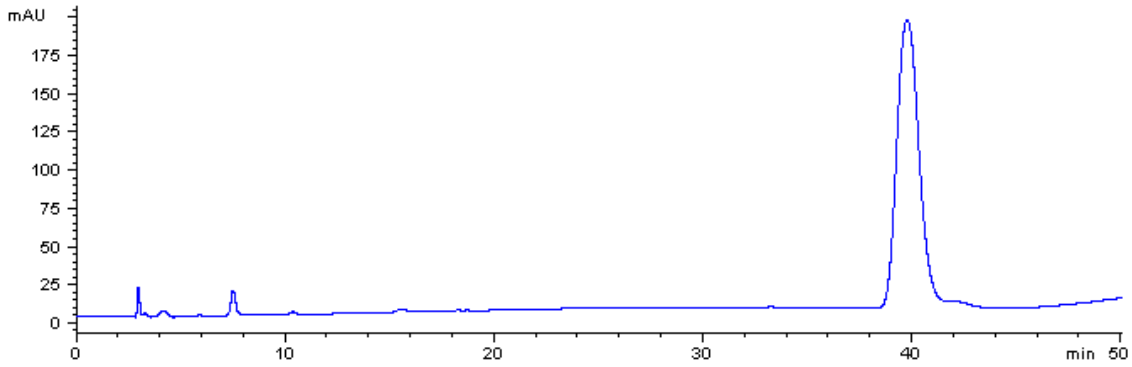
^{1}H, ^{13}C HMBC spectrum of compound **2** in CD_3OD

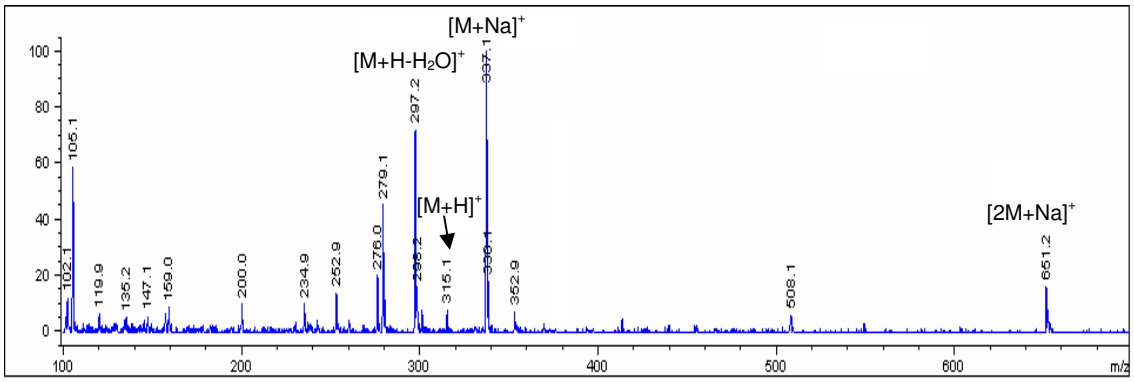




HPLC elution profile and ESI+/MS spectrum of kahweol isolated from green coffee beans. Elution conditions are described in the Experimental Procedures.

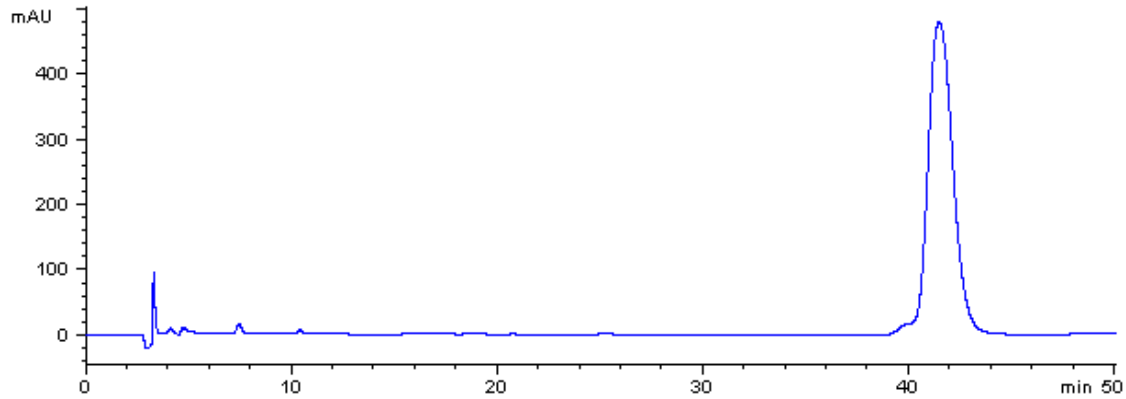
Kahweol

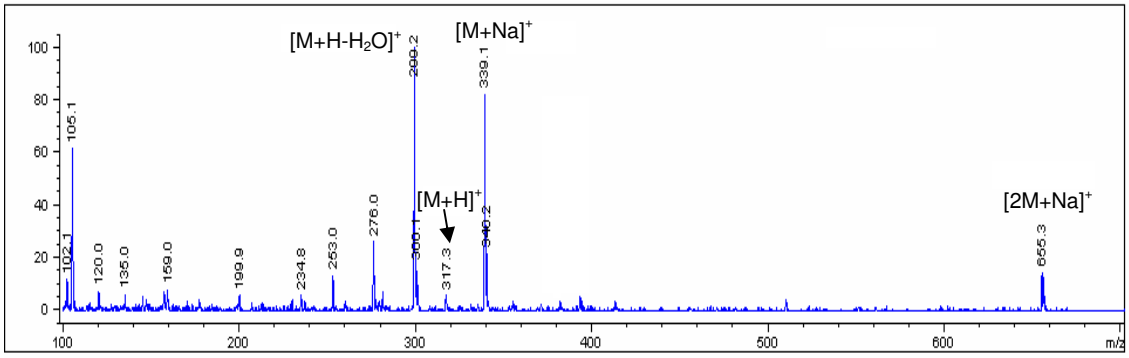




HPLC elution profile and ESI+/MS spectrum of cafestol obtained from green coffee beans. Elution conditions are described in the Experimental Procedures.

Cafestol





HPLC elution profiles of the ethyl acetate extract from the reaction mixture of coffee with nitrite ions (2 mM) at pH 3 (blue trace: 0 min; red trace: 2 h). Elution conditions are described in the Experimental Procedures.