

Supporting Information

Miliusanes, A Class of Cytotoxic Agents from *Miliusa sinensis*

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SPECTRA OF MILIUSANE ISOLATES (1-22)

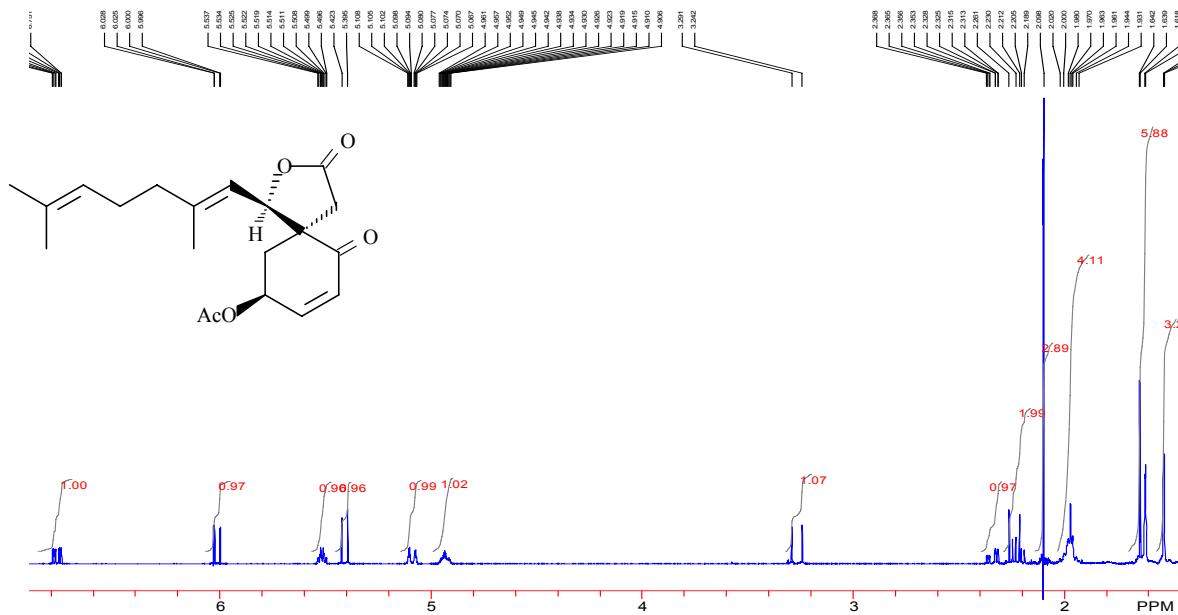


Figure A1. ^1H NMR Spectrum of Miliusate (**1**), CDCl_3

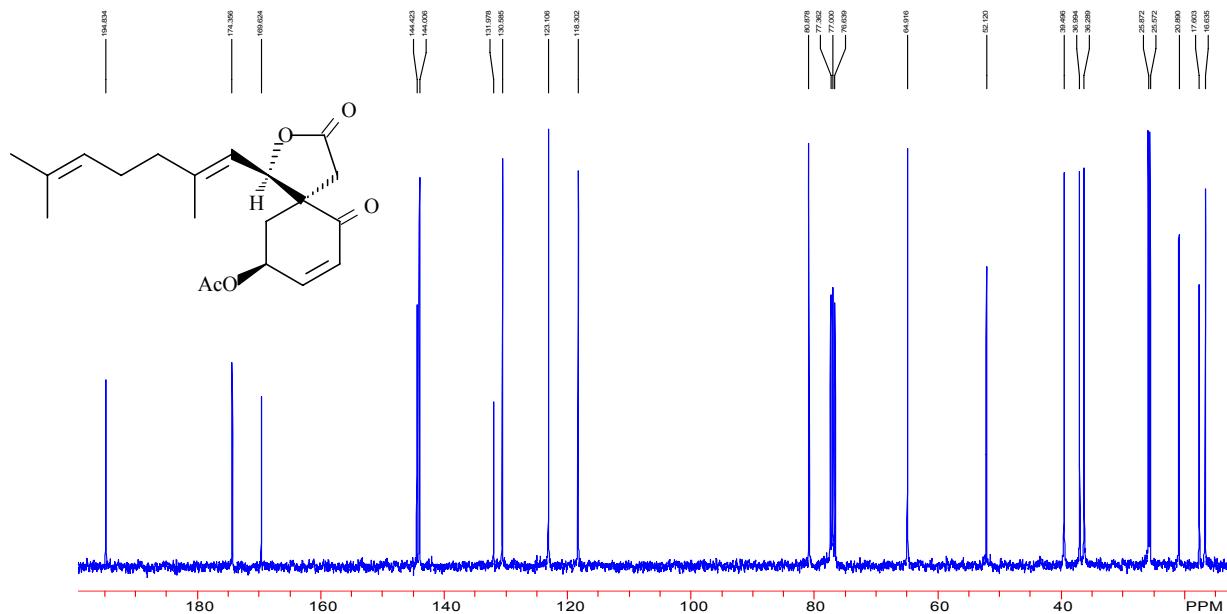


Figure A2. ^{13}C NMR Spectrum of Miliusate (**1**), CDCl_3

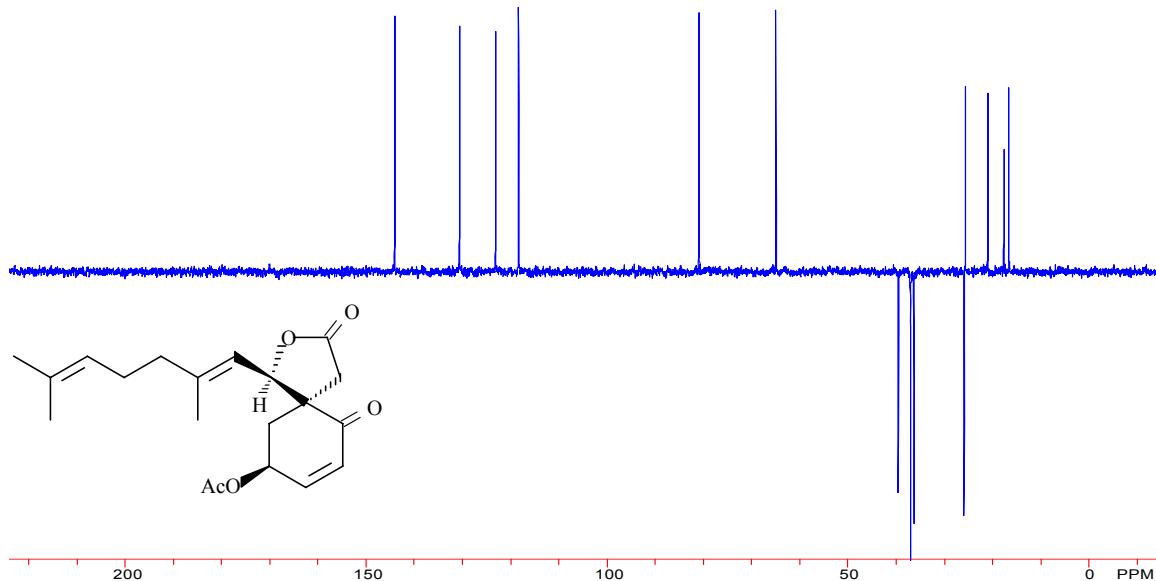


Figure A3. DEPT-135 Spectrum of Miliusate (**1**), CDCl_3

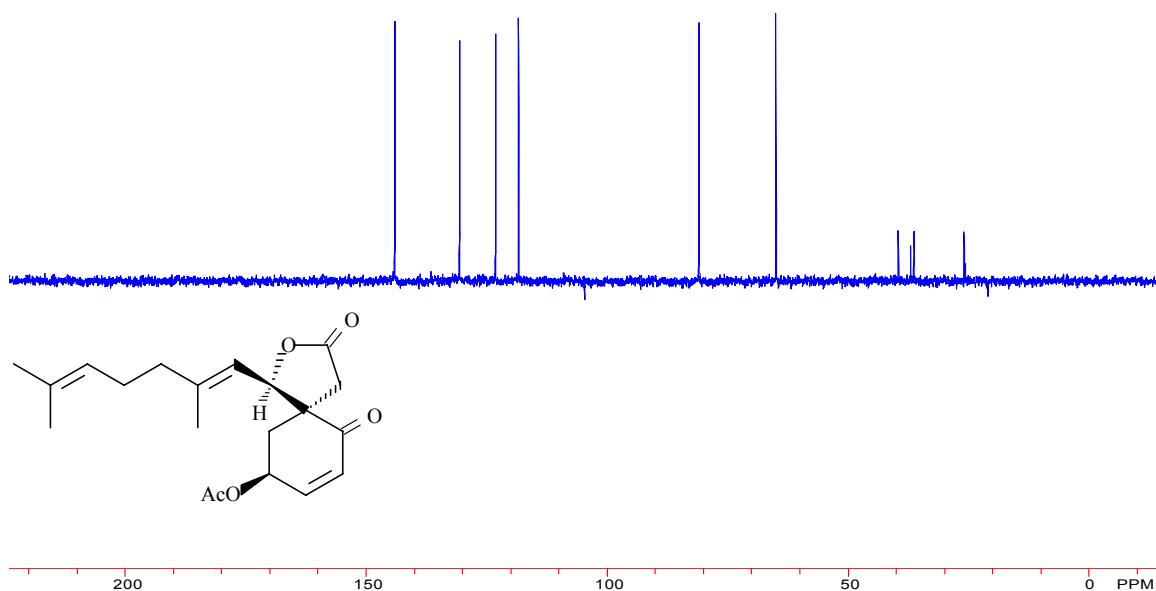


Figure A4. DEPT-90 Spectrum of Miliusate (**1**), CDCl_3

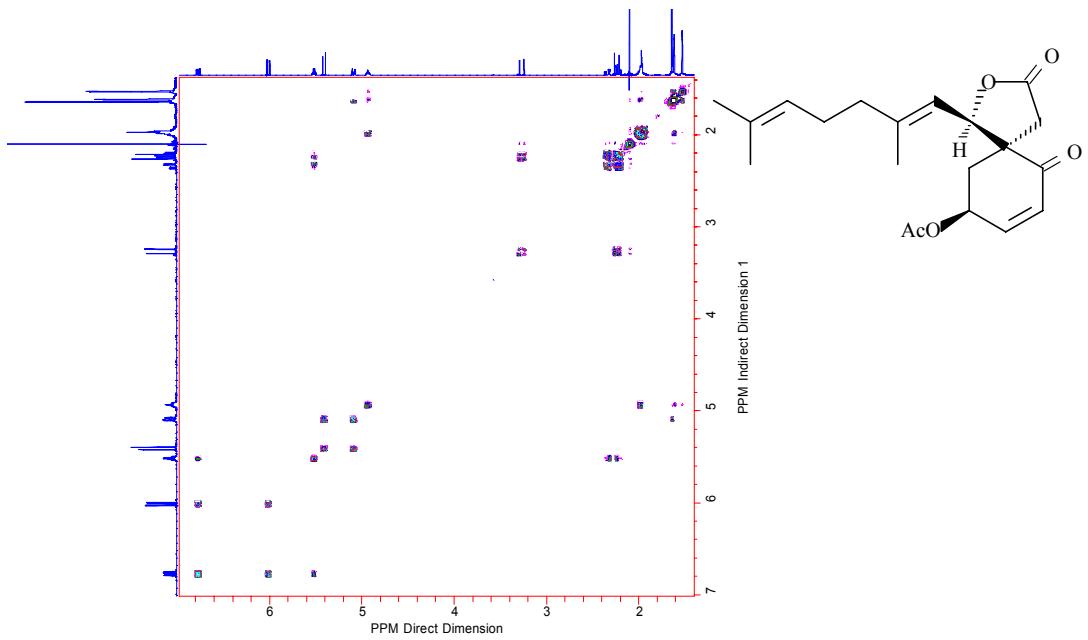


Figure A5. ^1H - ^1H COSY Spectrum of Miliusate (1), CDCl_3

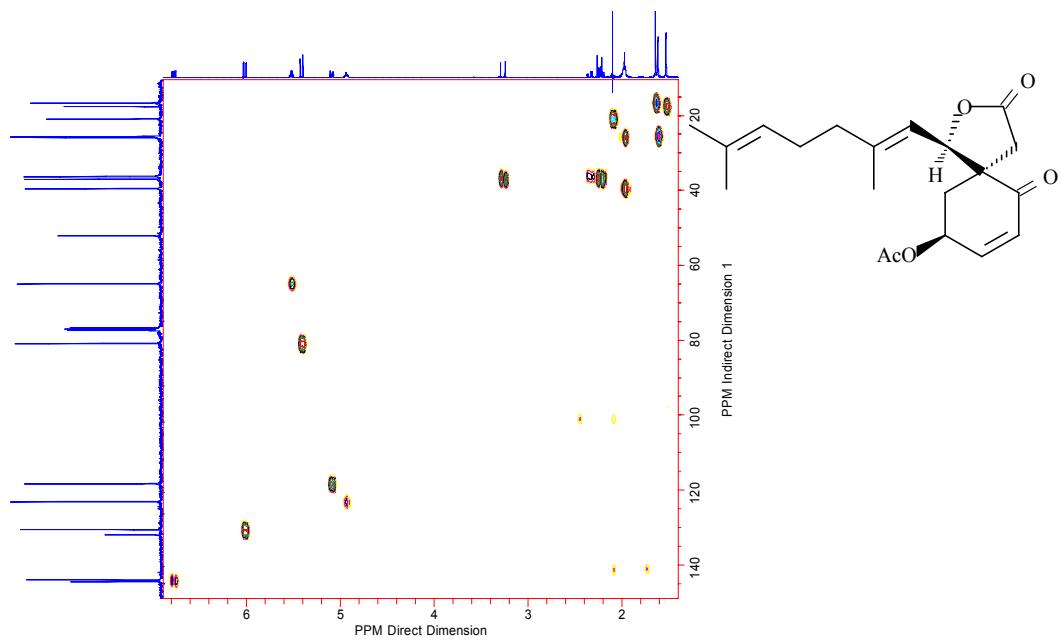


Figure A6. HMQC Spectrum of Miliusate (1), CDCl_3

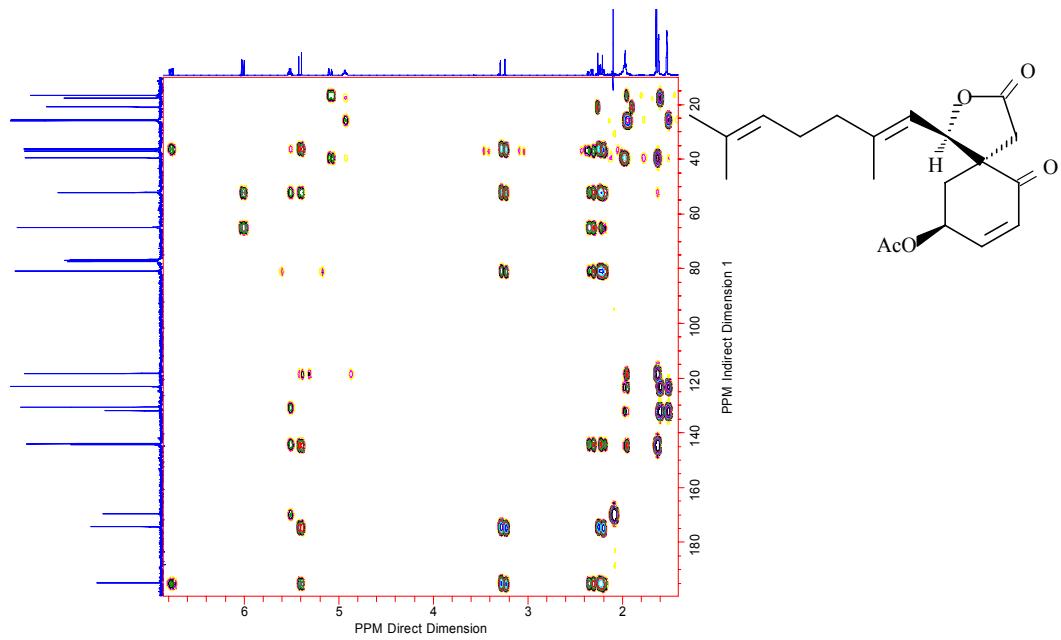


Figure A7. HMBC Spectrum of Miliusate (**1**), CDCl_3

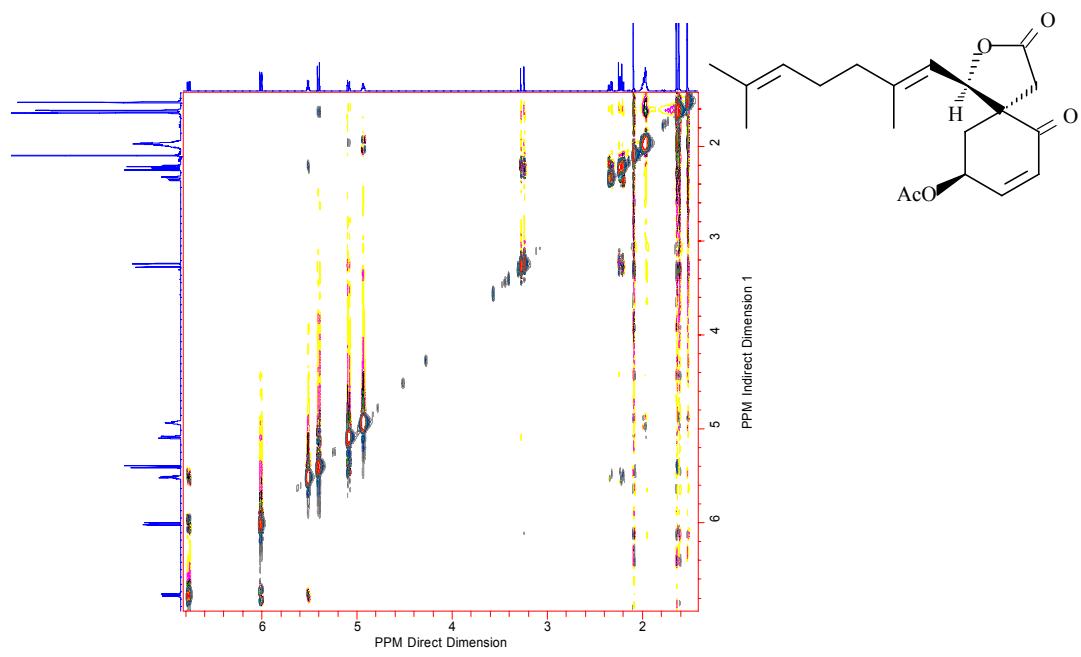


Figure A8. ROESY Spectrum of Miliusate (**1**), CDCl_3

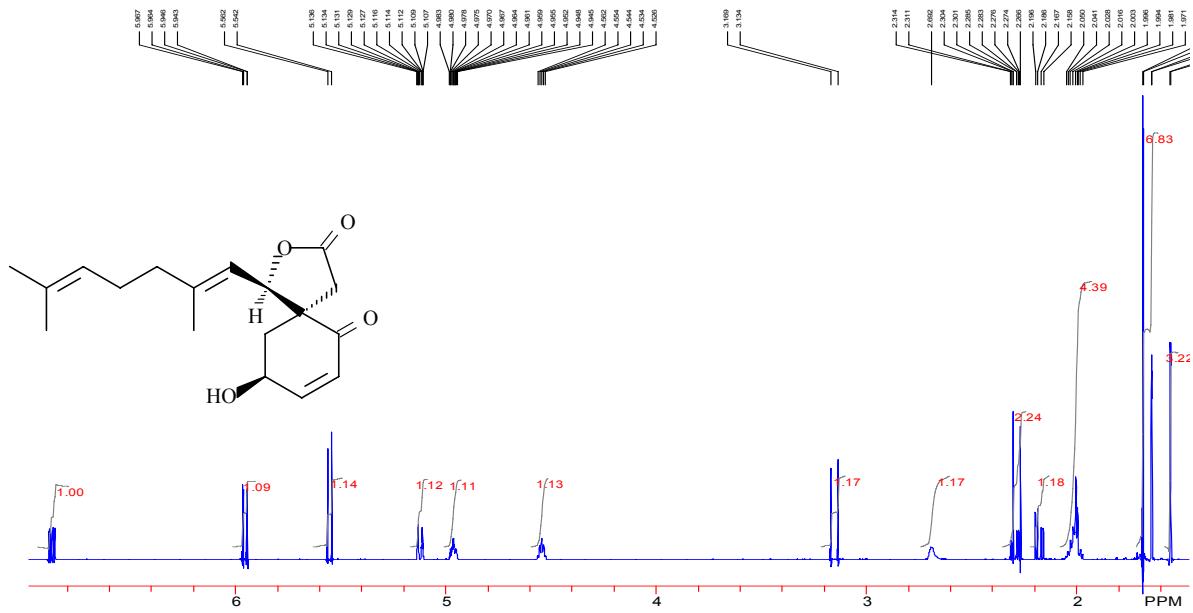


Figure B1. ^1H NMR Spectrum of Miliusol (**2**), CDCl_3

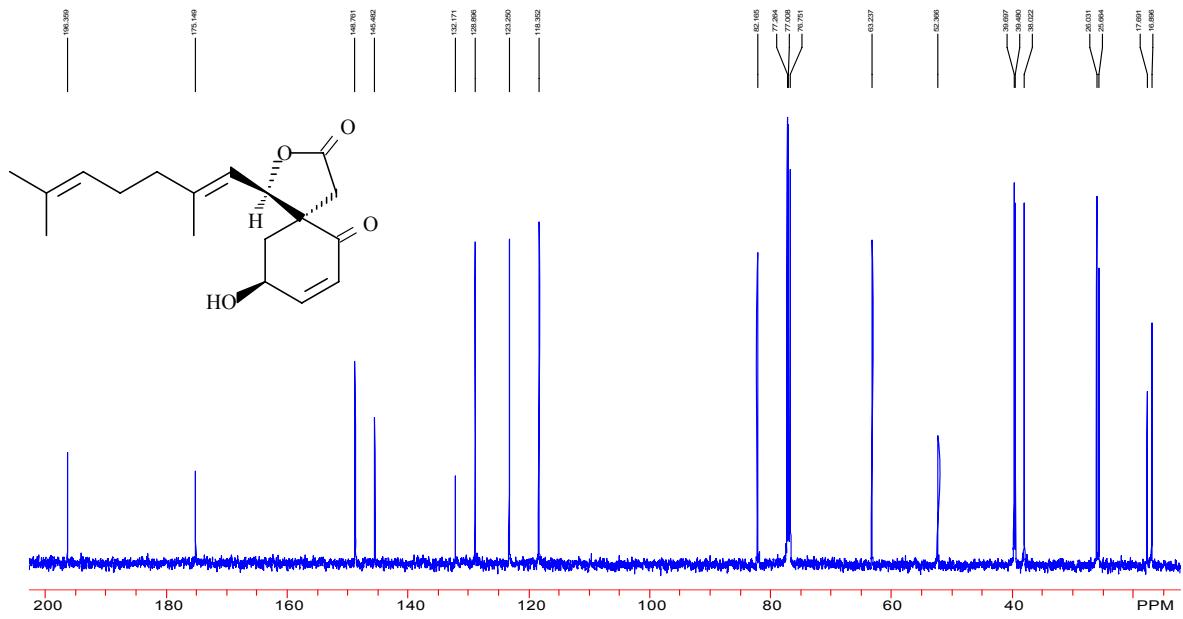


Figure B2. ^{13}C NMR Spectrum of Miliusol (**2**), CDCl_3

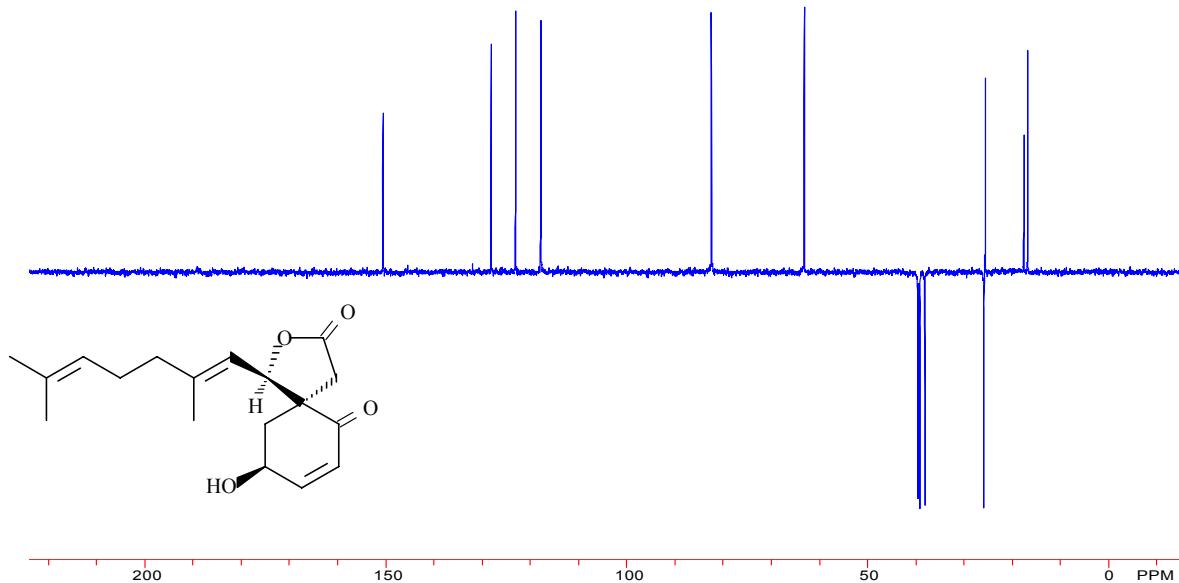


Figure B3. DEPT-135 Spectrum of Miliusol (**2**), CDCl_3

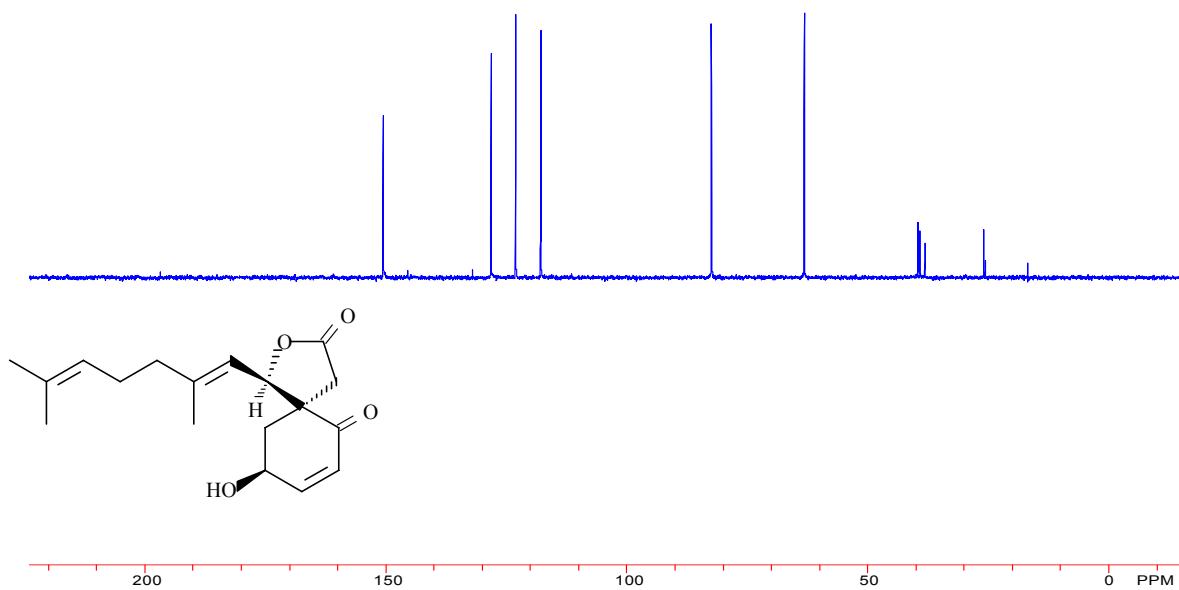


Figure B4. DEPT-90 Spectrum of Miliusol (**2**), CDCl_3

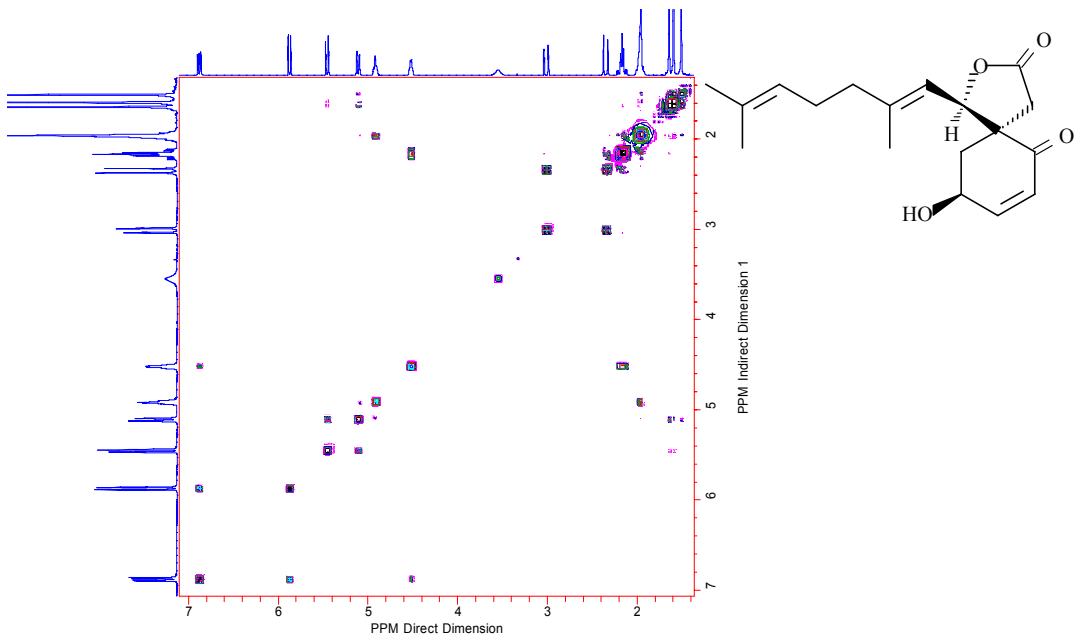


Figure B5. ^1H - ^1H COSY Spectrum of Miliusol (2), CDCl_3

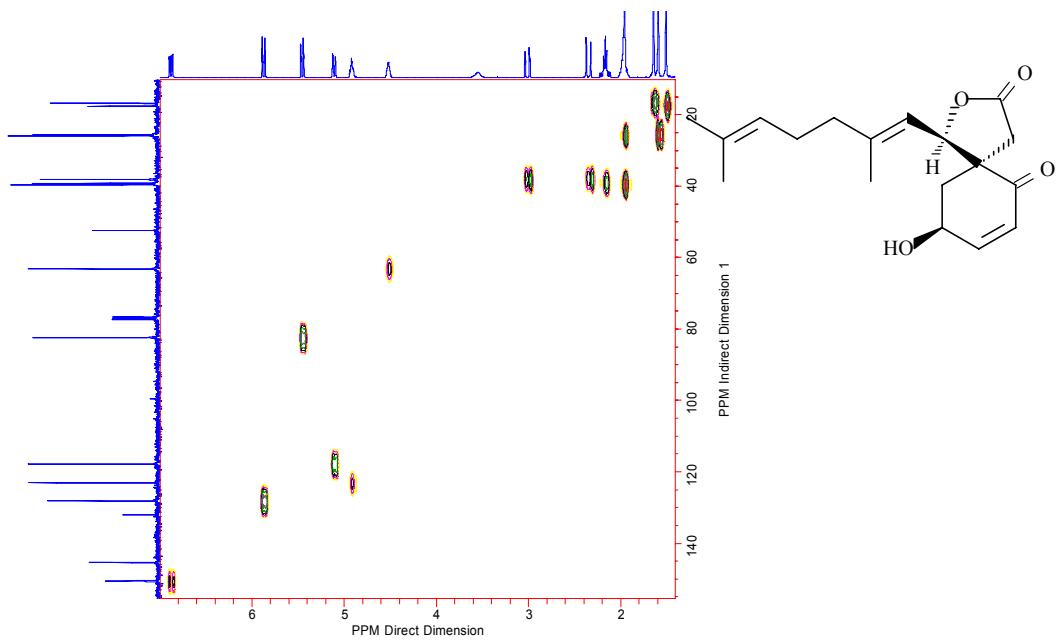


Figure B6. HMQC Spectrum of Miliusol (2), CDCl_3

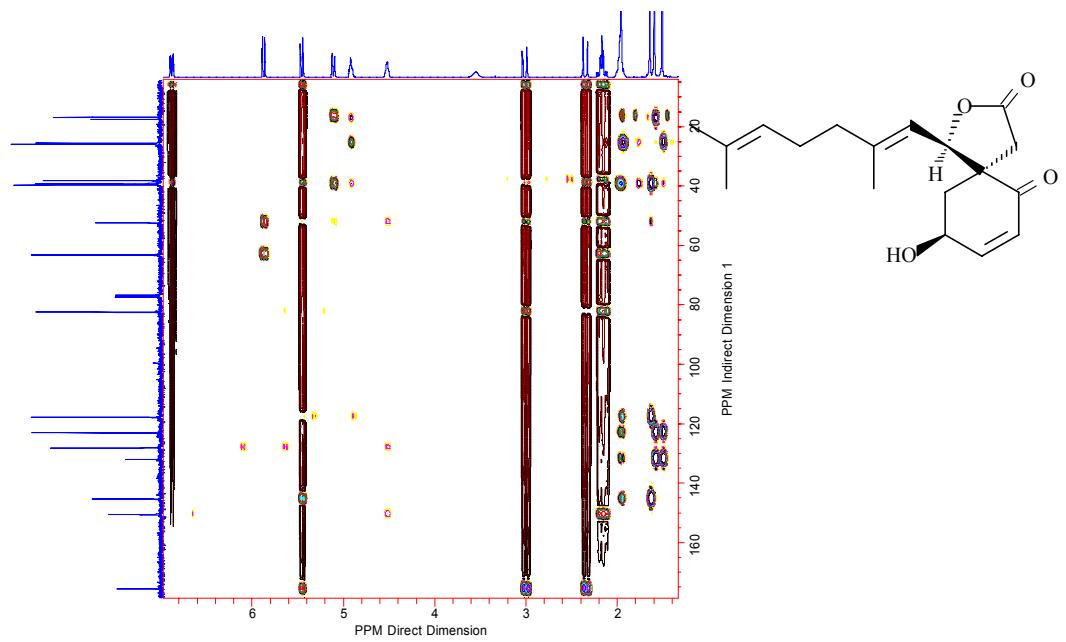


Figure B7. HMBC Spectrum of Miliusol (**2**), CDCl_3

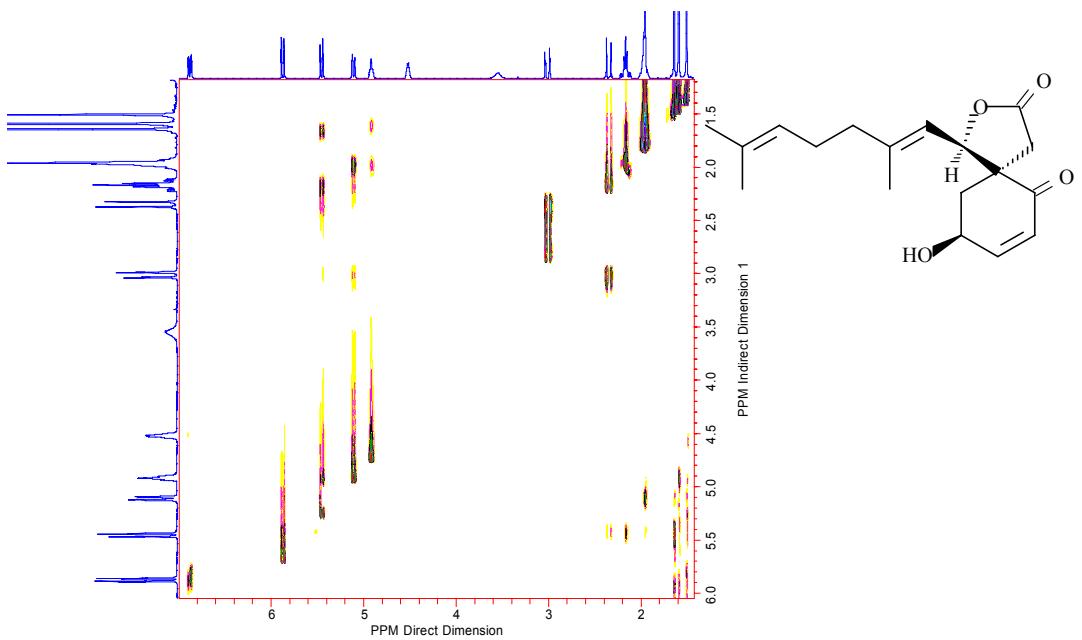


Figure B8. ROESY Spectrum of Miliusol (**2**), CDCl_3

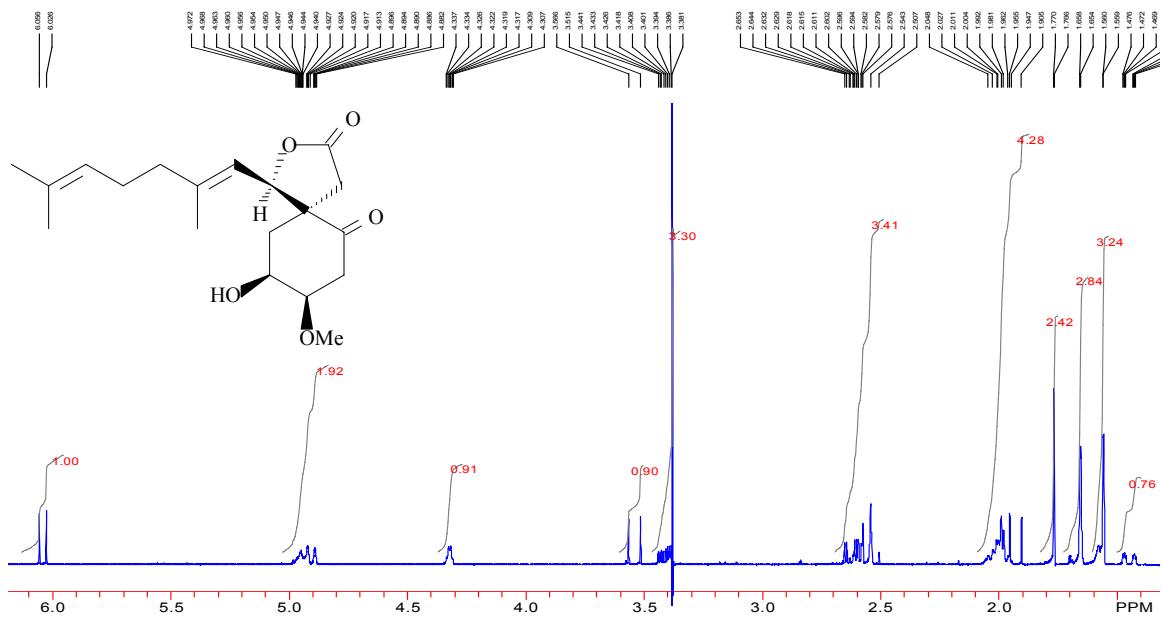


Figure C1. ^1H NMR Spectrum of Miliusane I (**3**), CDCl_3

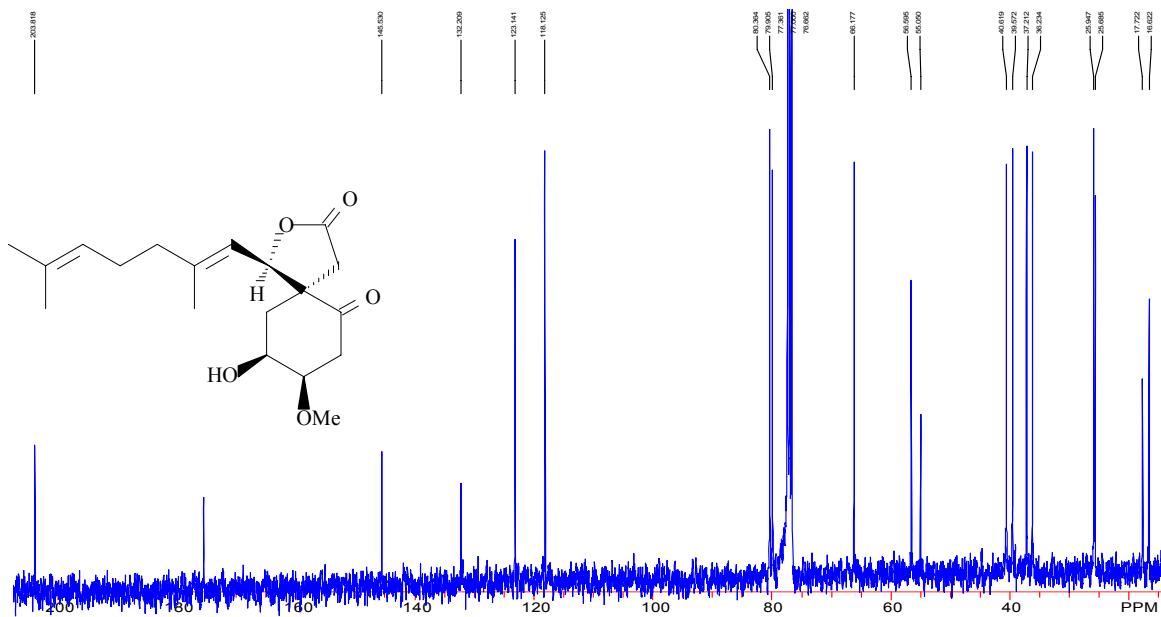


Figure C2. ^{13}C NMR Spectrum of Miliusane I (**3**), CDCl_3

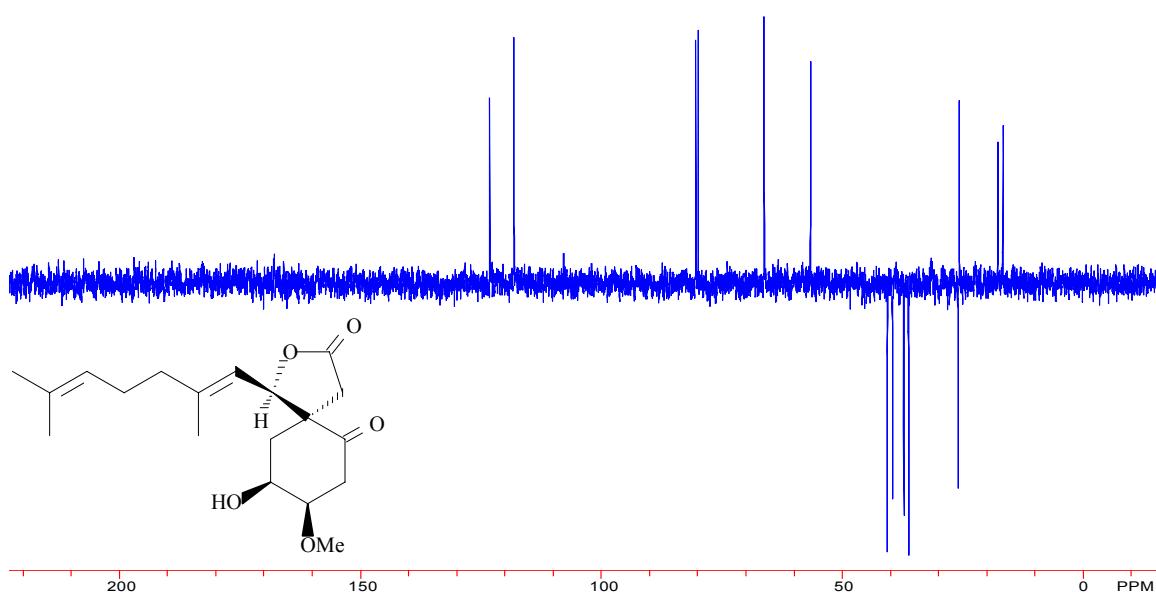


Figure C3. DEPT-135 Spectrum of Miliusane I (**3**), CDCl_3

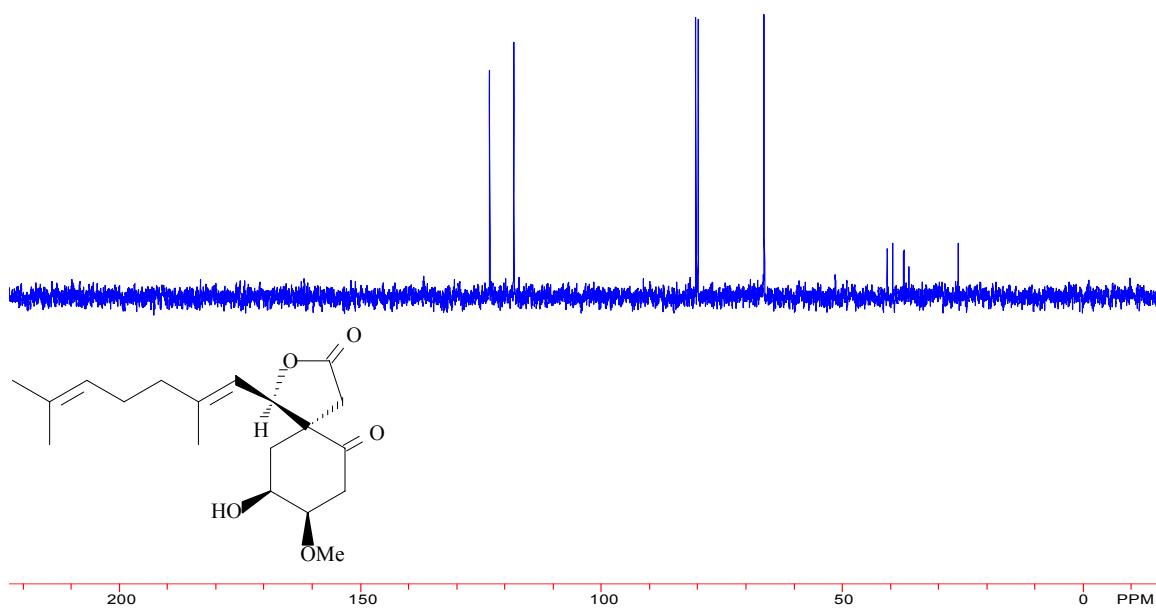


Figure C4. DEPT-90 Spectrum of Miliusane I (**3**), CDCl_3

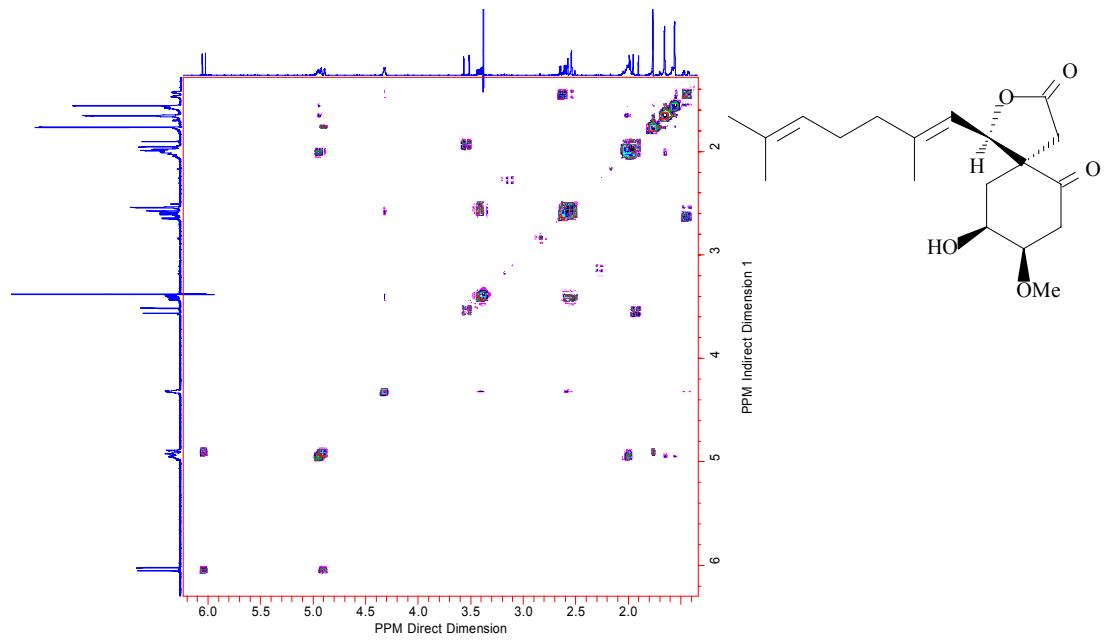


Figure C5. ^1H - ^1H COSY Spectrum of Miliusane I (**3**), CDCl_3

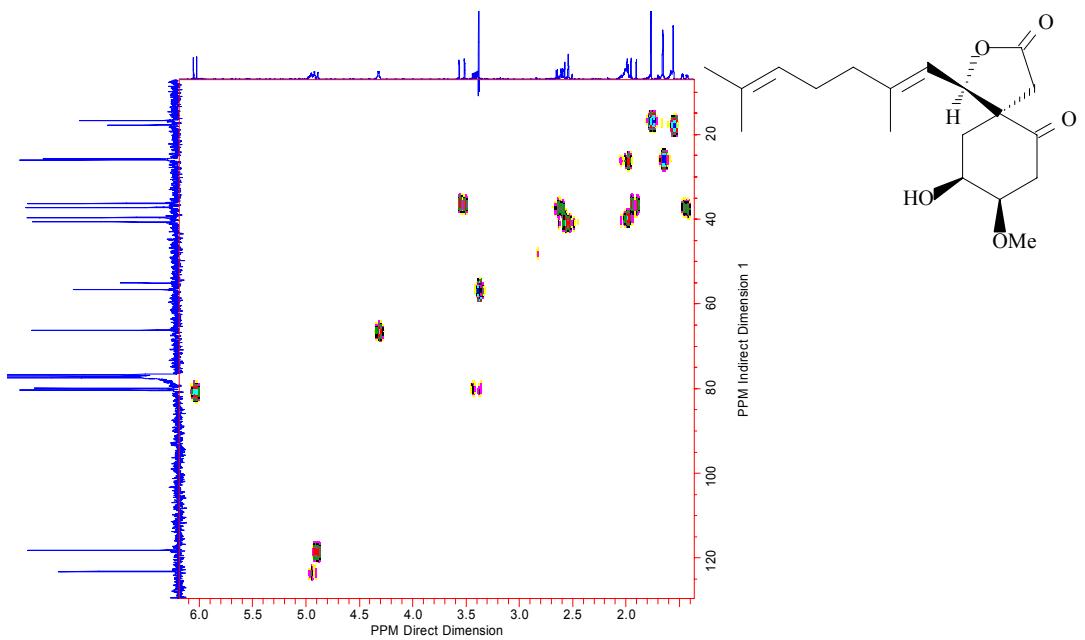


Figure C6. HMQC Spectrum of Miliusane I (**3**), CDCl_3

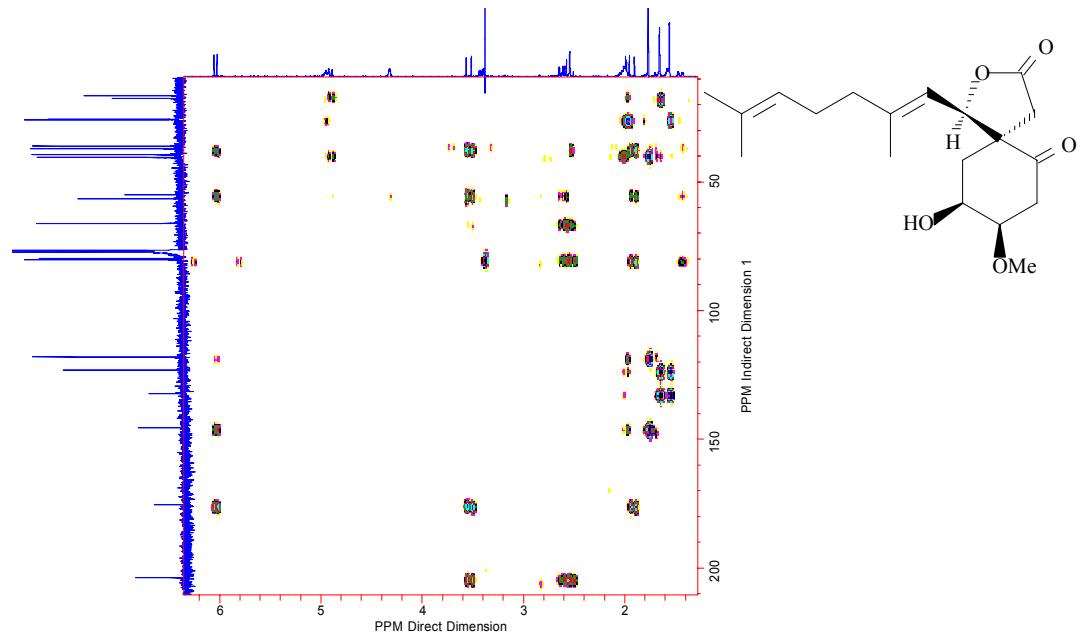


Figure C7. HMBC Spectrum of Miliusane I (**3**), CDCl_3

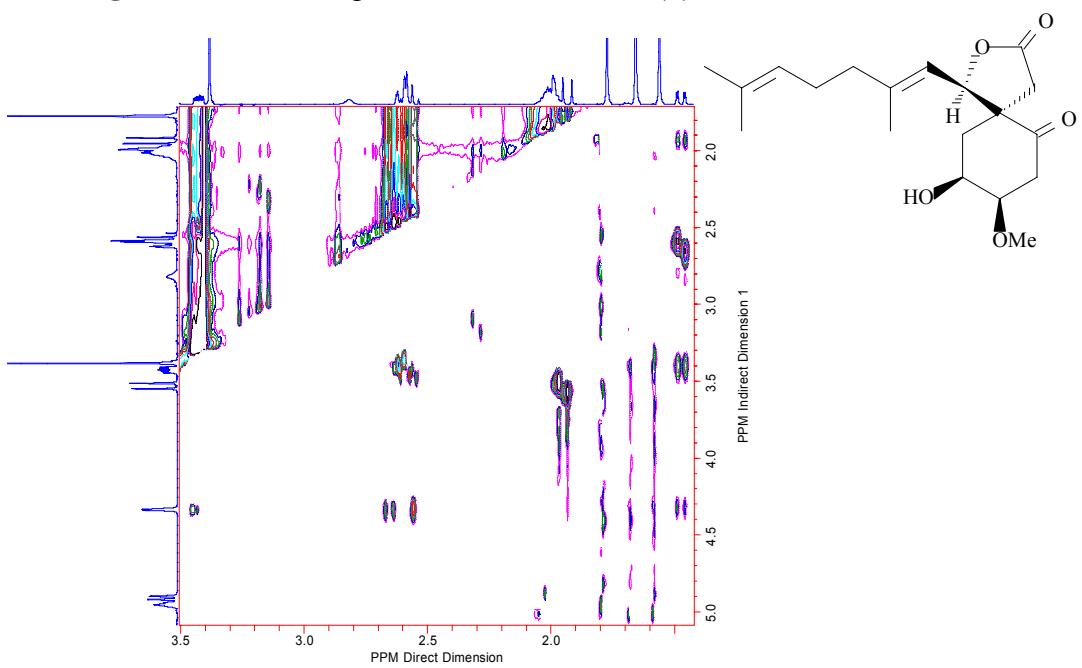


Figure C8. ROESY Spectrum of Miliusane I (**3**), CDCl_3

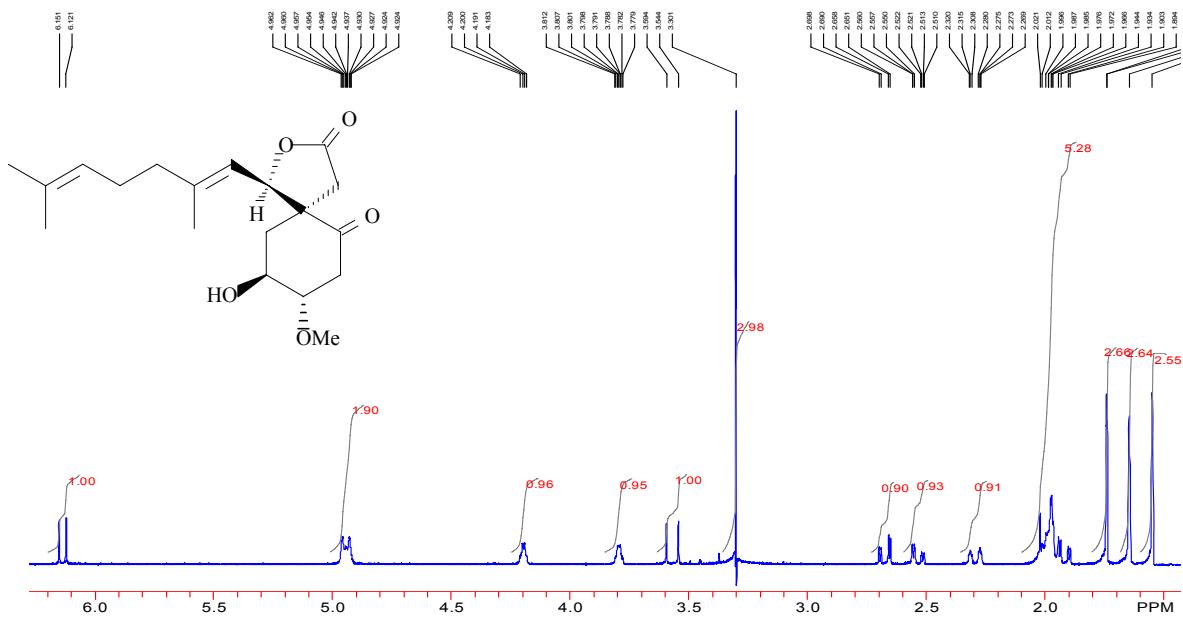


Figure D1. ^1H NMR Spectrum of Miliusane II (**4**), CDCl_3

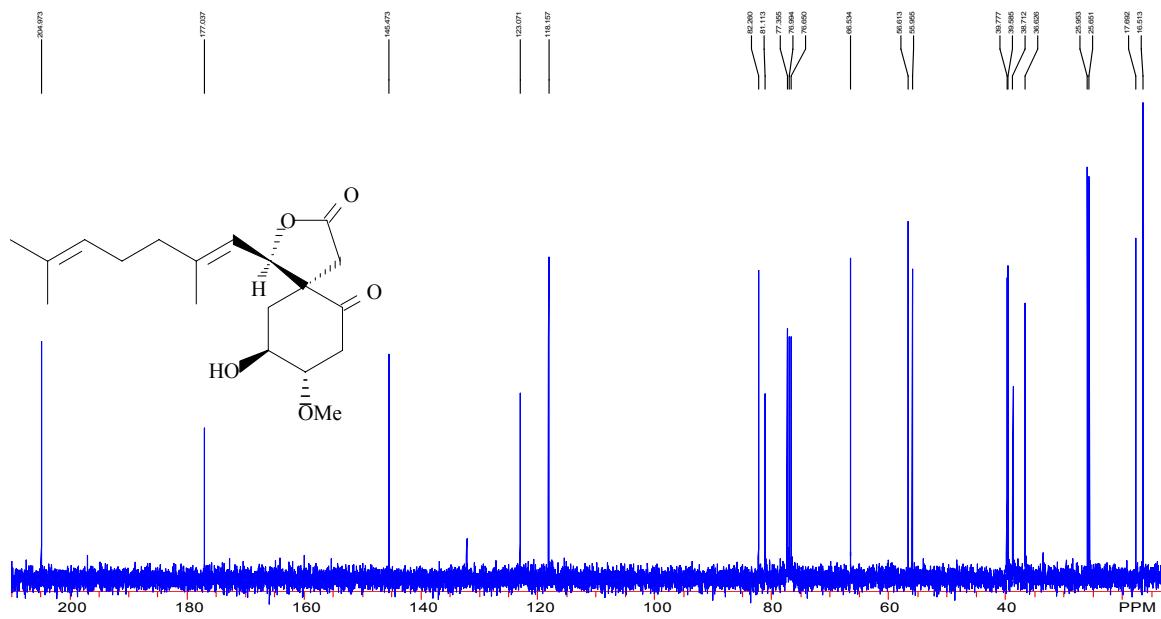


Figure D2. ^{13}C NMR Spectrum of Miliusane II (**4**), CDCl_3

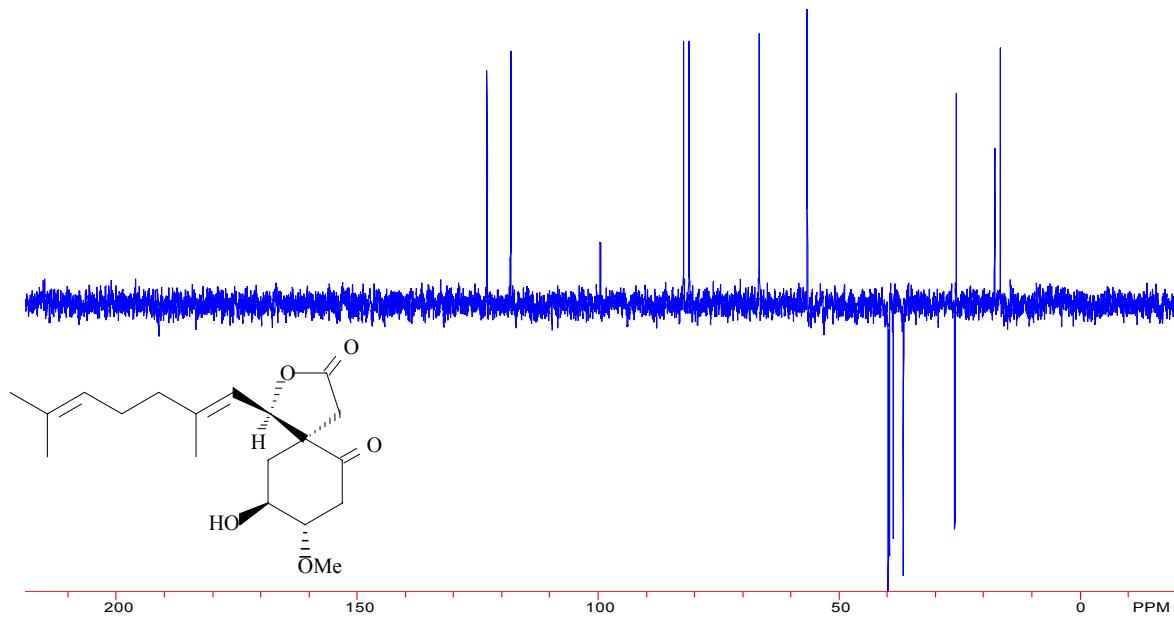


Figure D3. DEPT-135 Spectrum of Miliusane II (**4**), CDCl_3

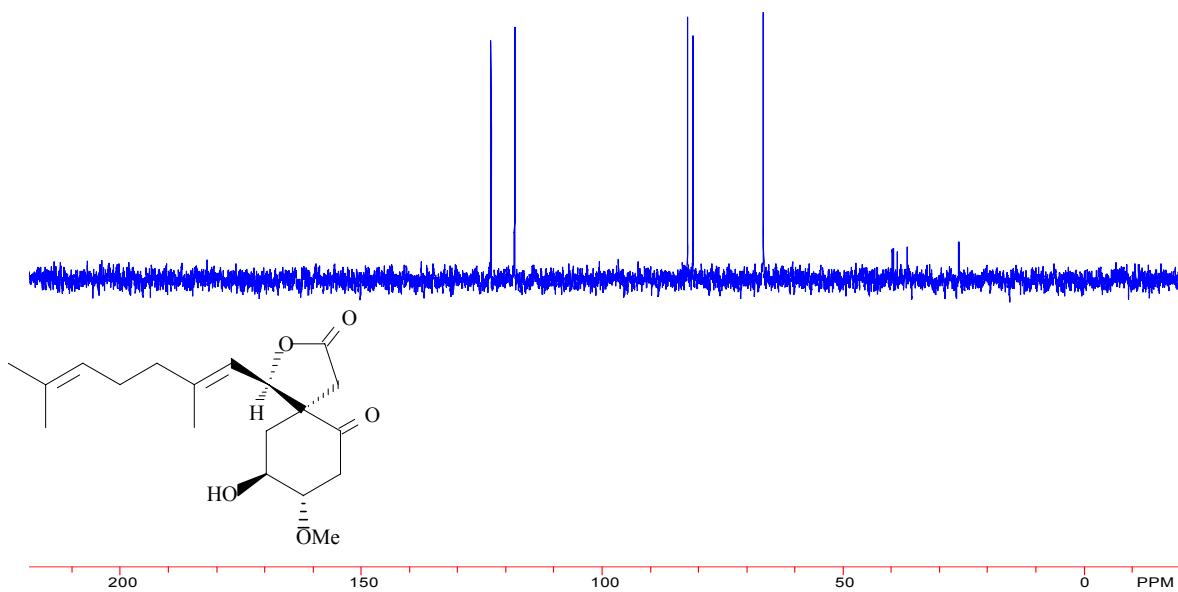


Figure D4. DEPT-90 Spectrum of Miliusane II (**4**), CDCl_3

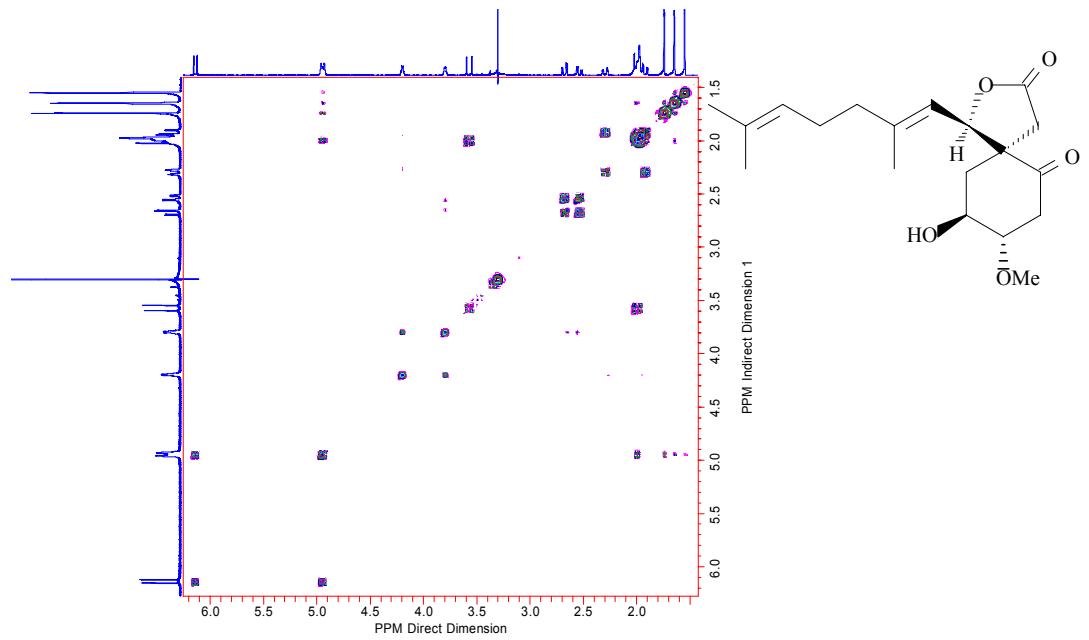


Figure D5. ¹H-¹H COSY Spectrum of Miliusane II (**4**), CDCl₃

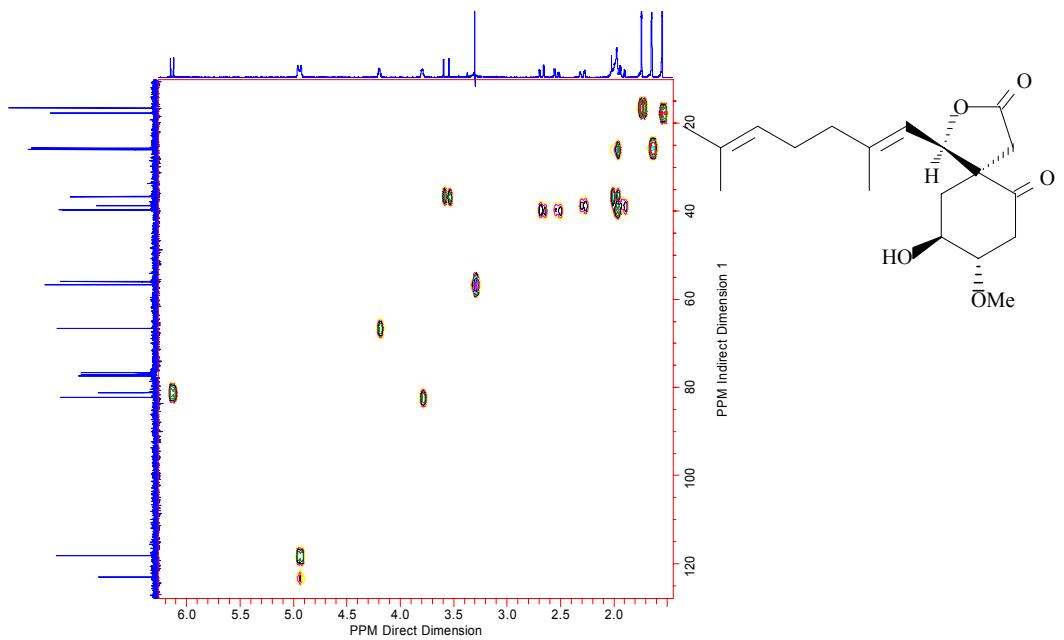


Figure D6. HMQC Spectrum of Miliusane II (**4**), CDCl₃

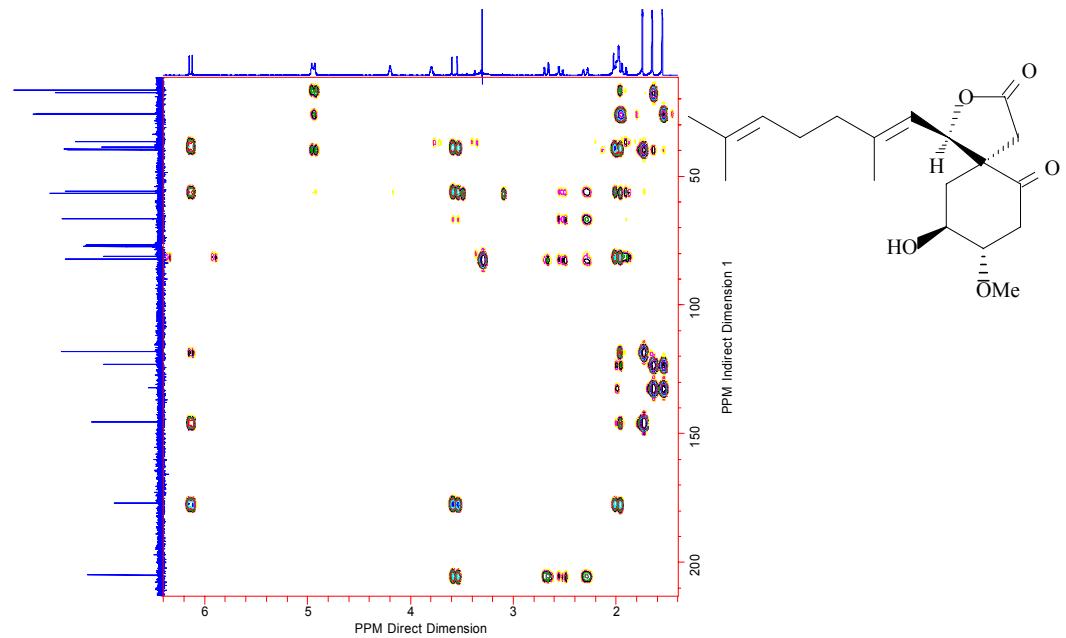


Figure D7. HMBC Spectrum of Miliusane II (**4**), CDCl_3

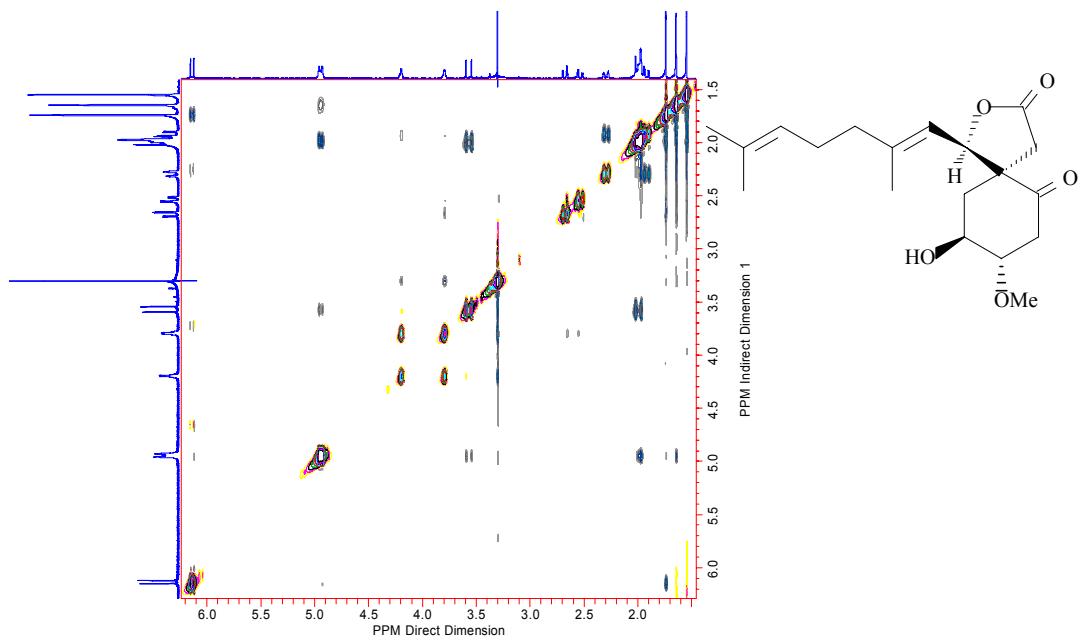


Figure D8. ROESY Spectrum of Miliusane II (**4**), CDCl_3

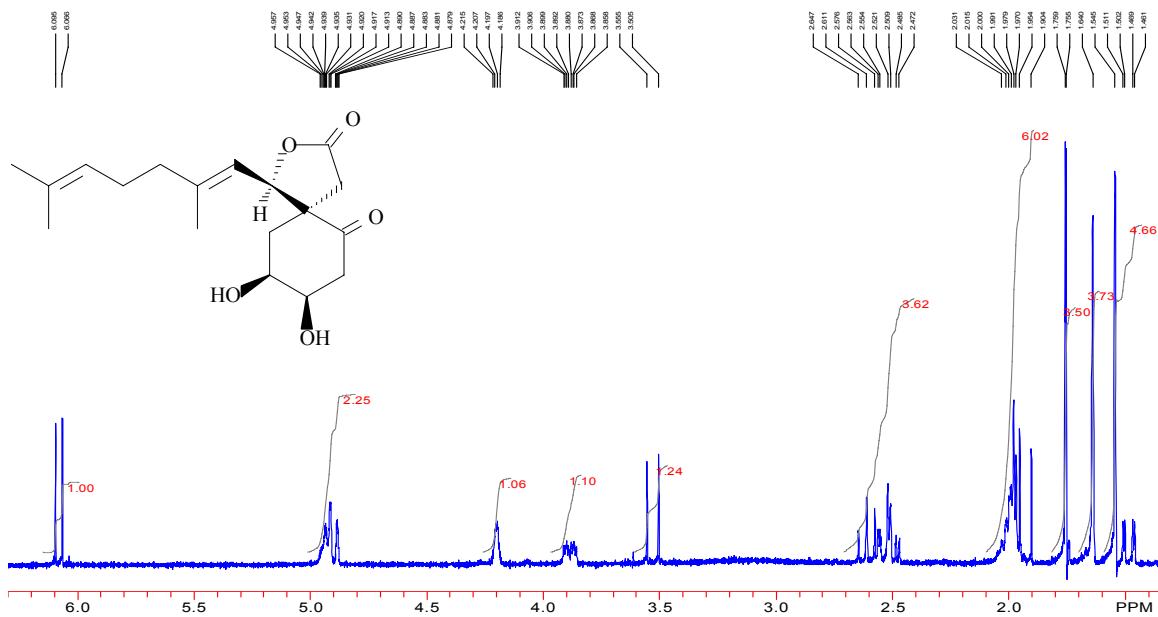


Figure E1. ^1H NMR Spectrum of Miliusane III (**5**), CDCl_3

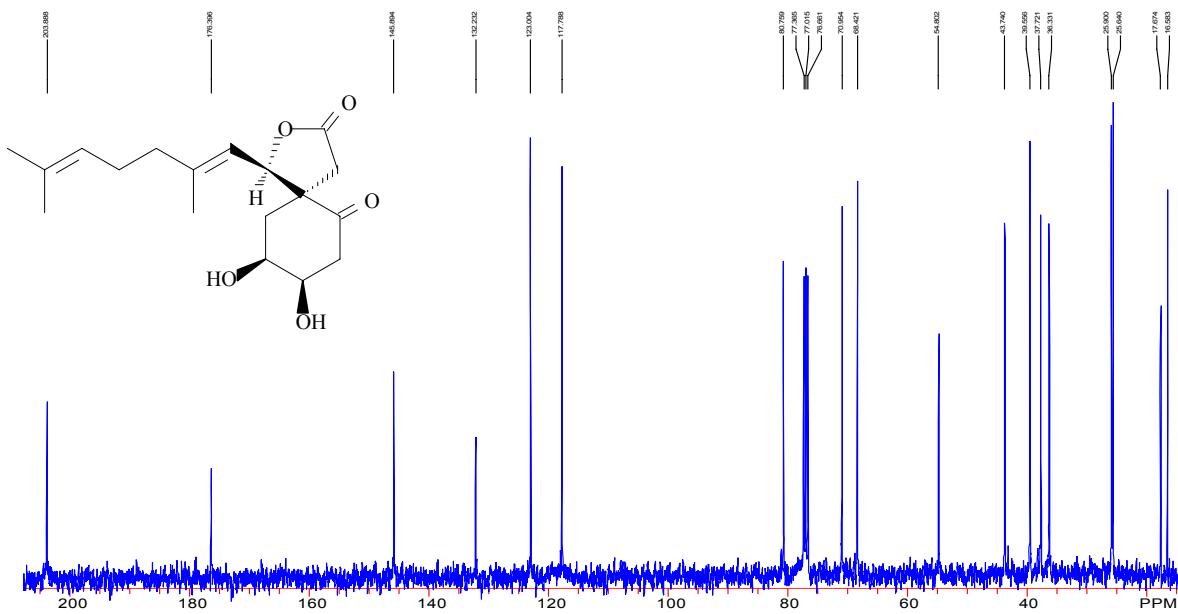


Figure E2. ^{13}C NMR Spectrum of Miliusane III (**5**), CDCl_3

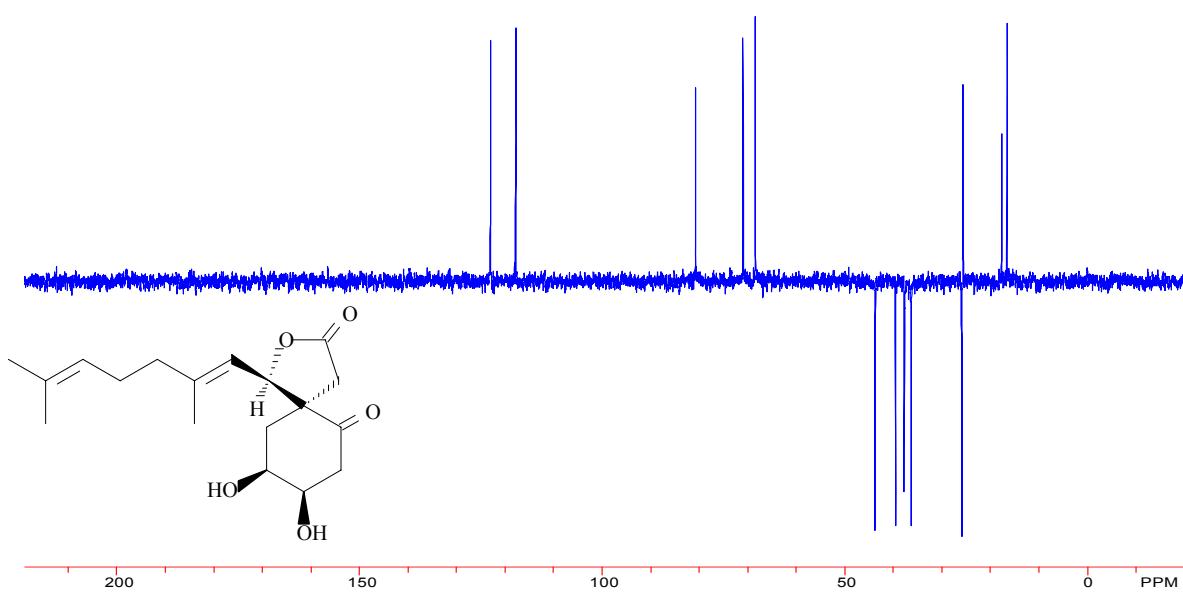


Figure E3. DEPT-135 Spectrum of Miliusane III (**5**), CDCl_3

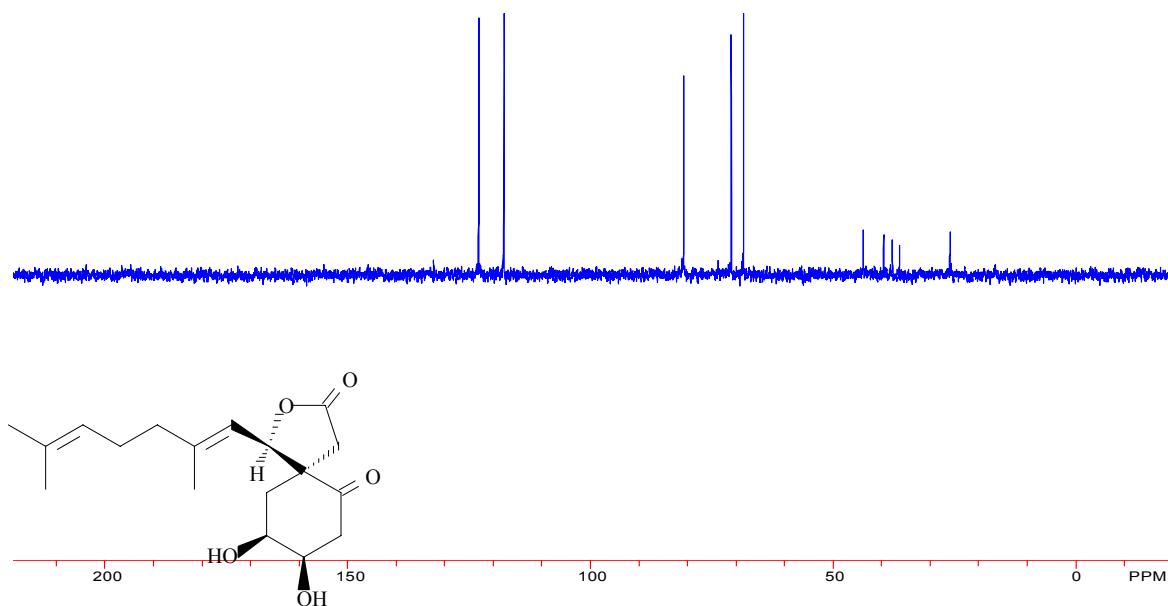


Figure E4. DEPT-90 Spectrum of Miliusane III (**5**), CDCl_3

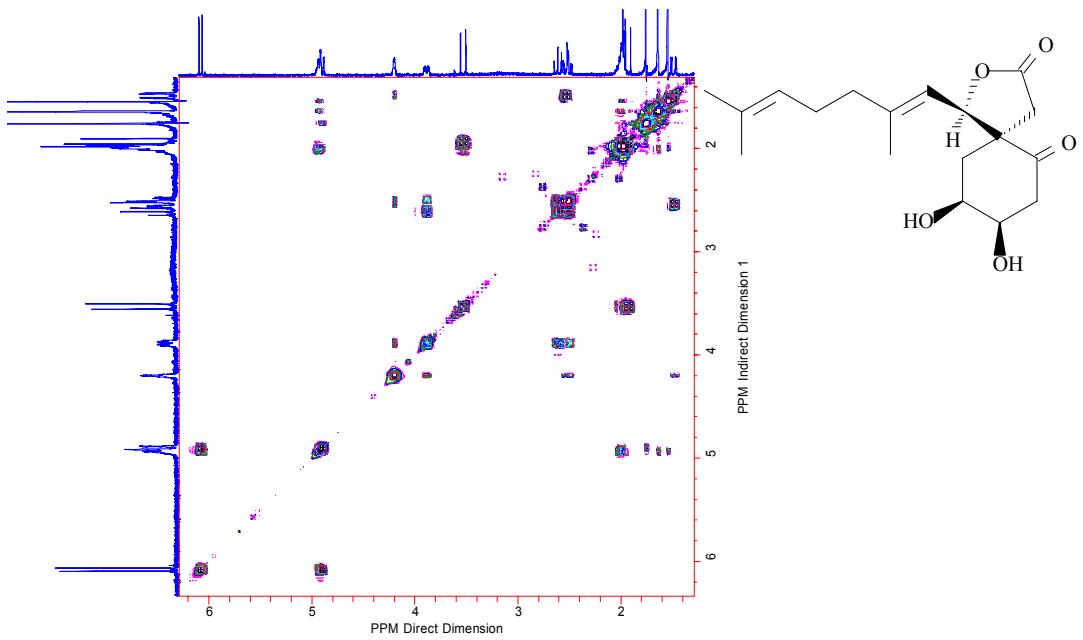


Figure E5. ^1H - ^1H COSY Spectrum of Miliusane III (**5**), CDCl_3

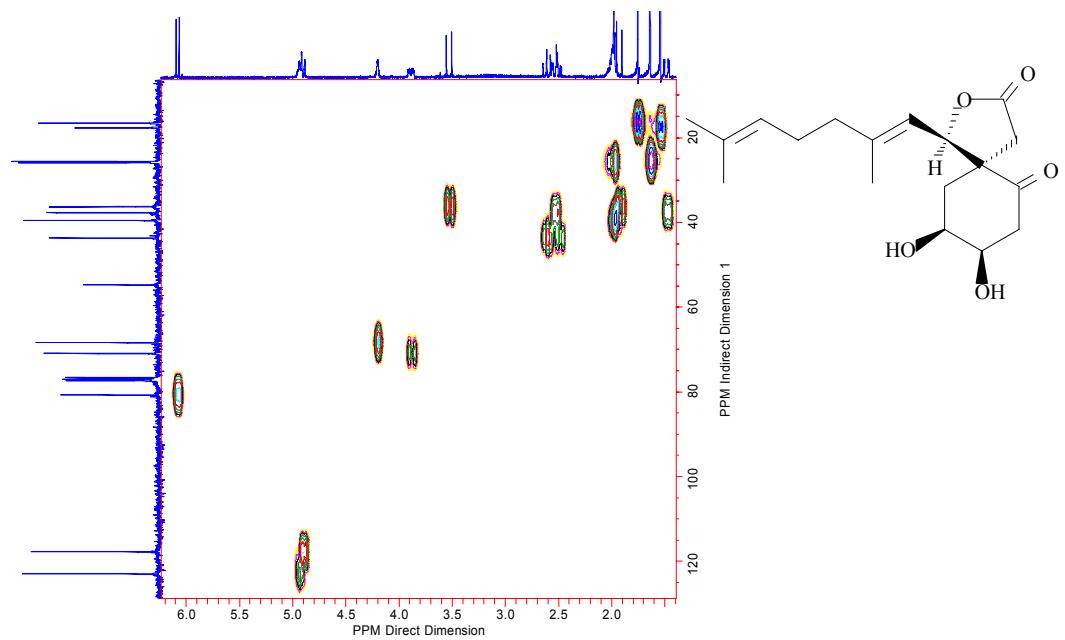


Figure E6. HMQC Spectrum of Miliusane III (**5**), CDCl_3

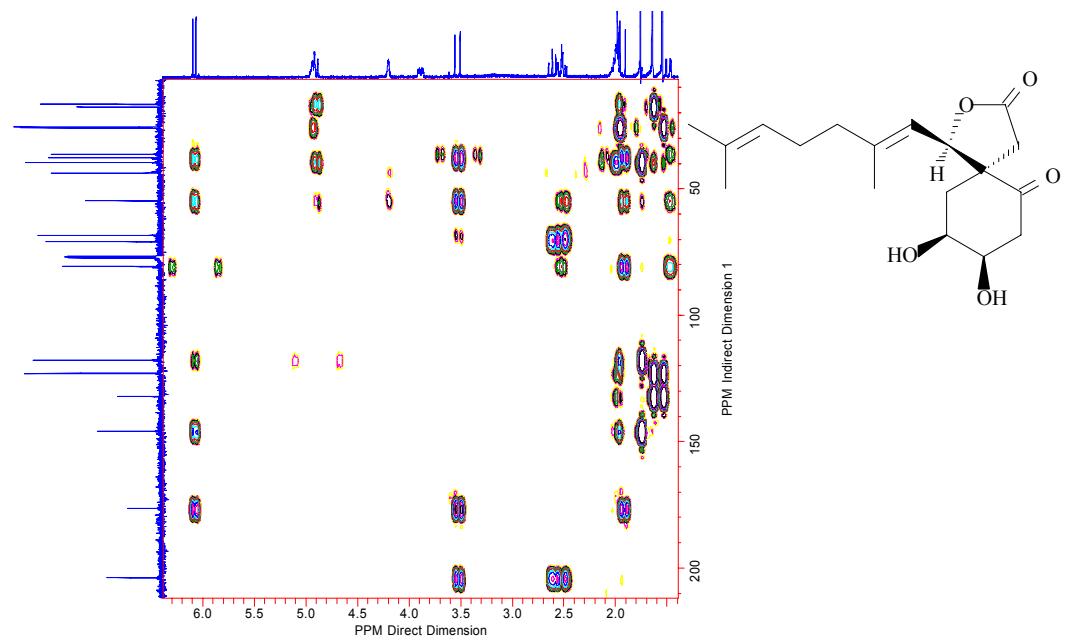


Figure E7. HMBC Spectrum of Miliusane III (**5**), CDCl_3

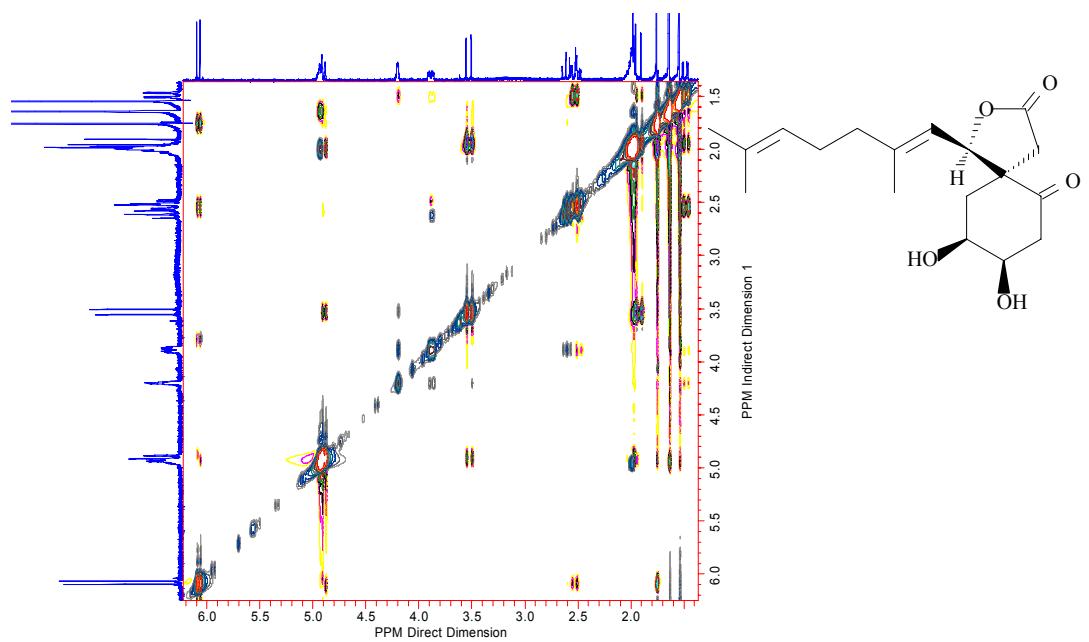


Figure E8. ROESY Spectrum of Miliusane III (**5**), CDCl_3

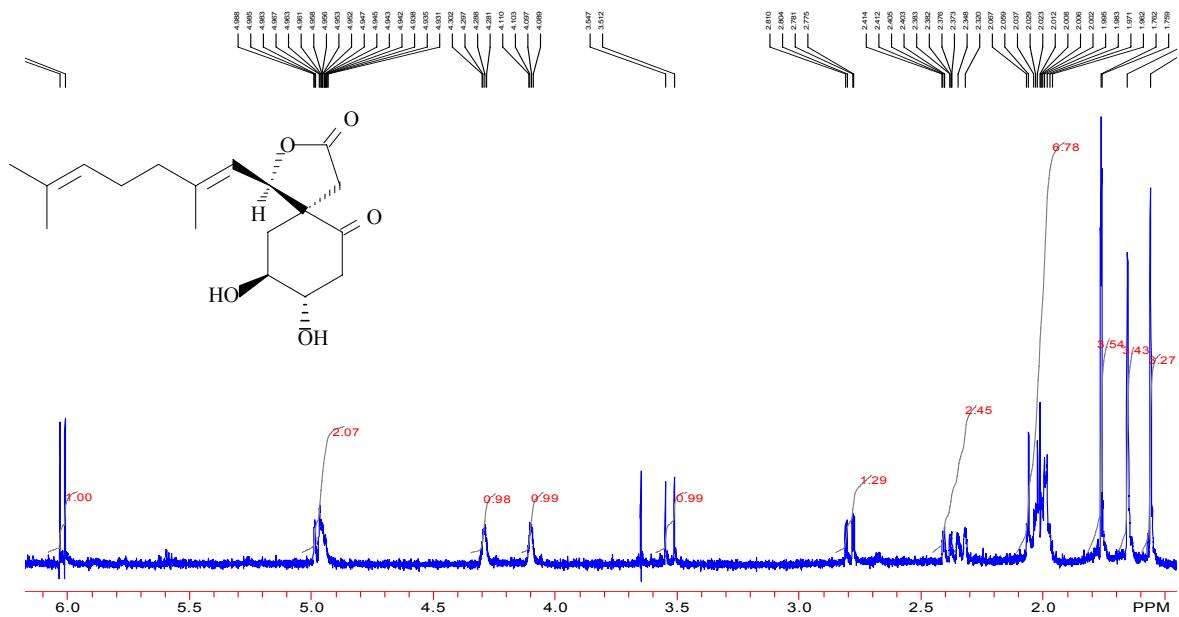


Figure F1. ^1H NMR Spectrum of Miliusane IV (**6**), CDCl_3

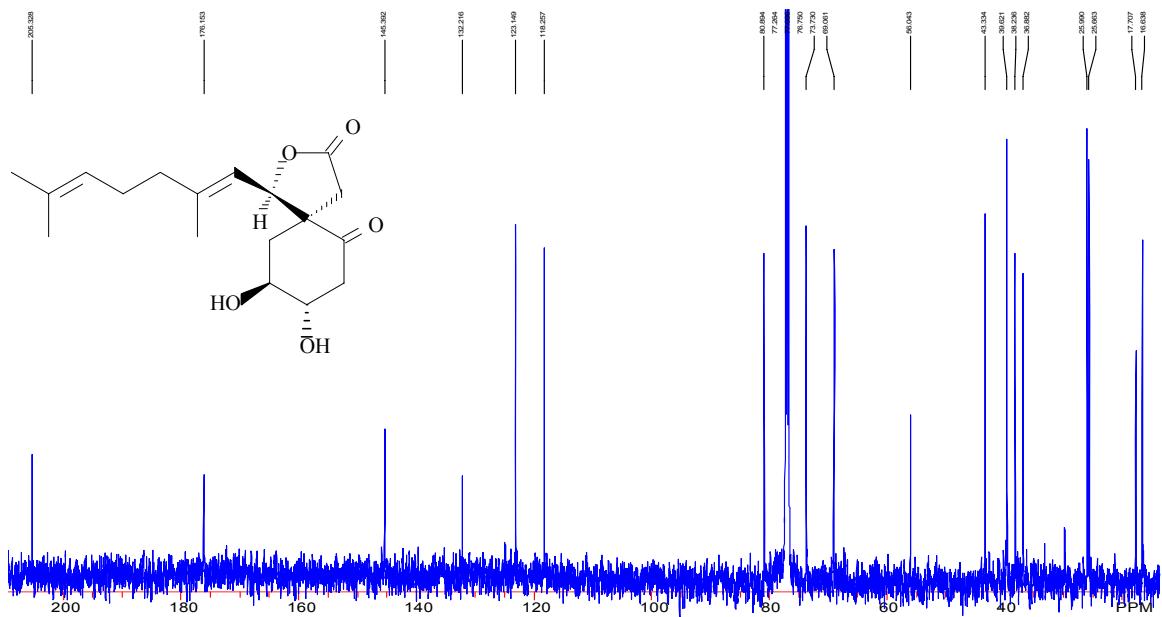


Figure F2. ^{13}C NMR Spectrum of Miliusane IV (**6**), CDCl_3

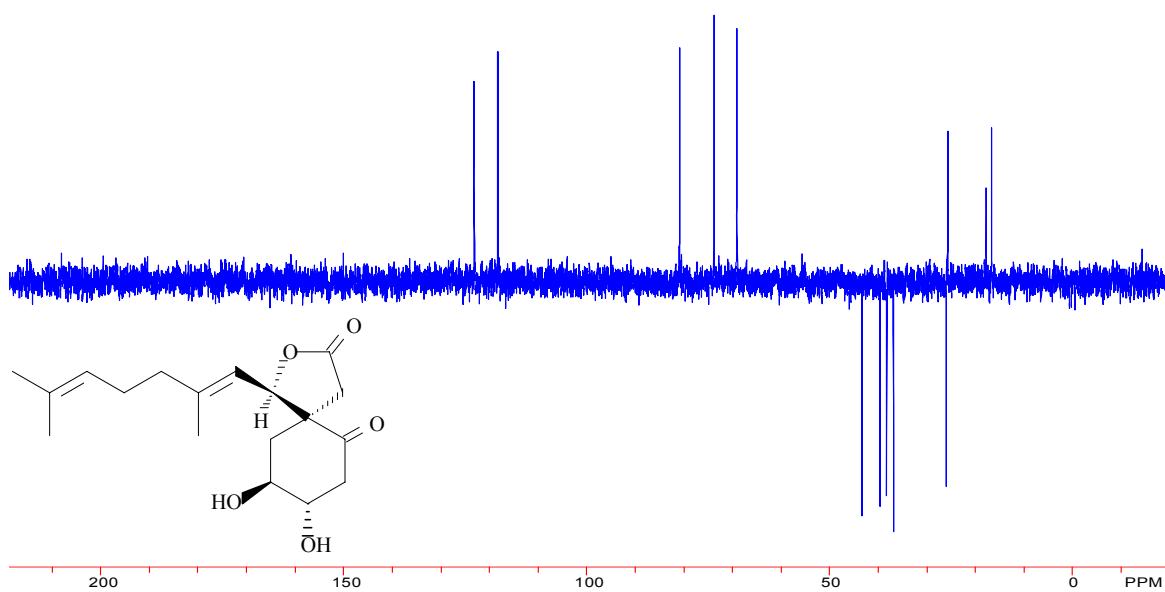


Figure F3. DEPT-135 Spectrum of Miliusane IV (**6**), CDCl_3

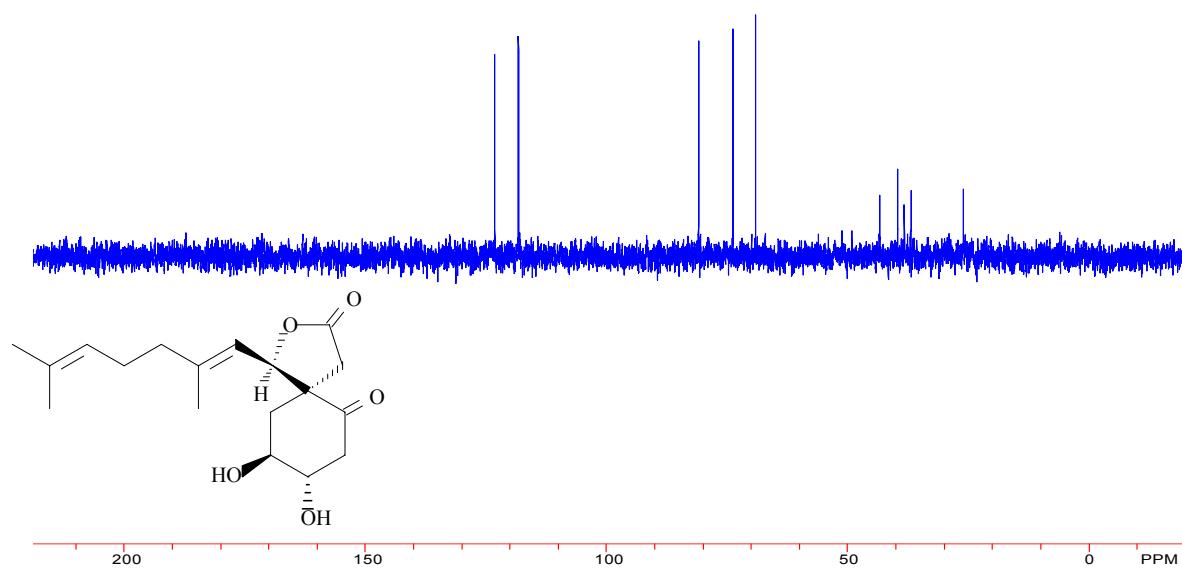


Figure F4. DEPT-90 Spectrum of Miliusane IV (**6**), CDCl_3

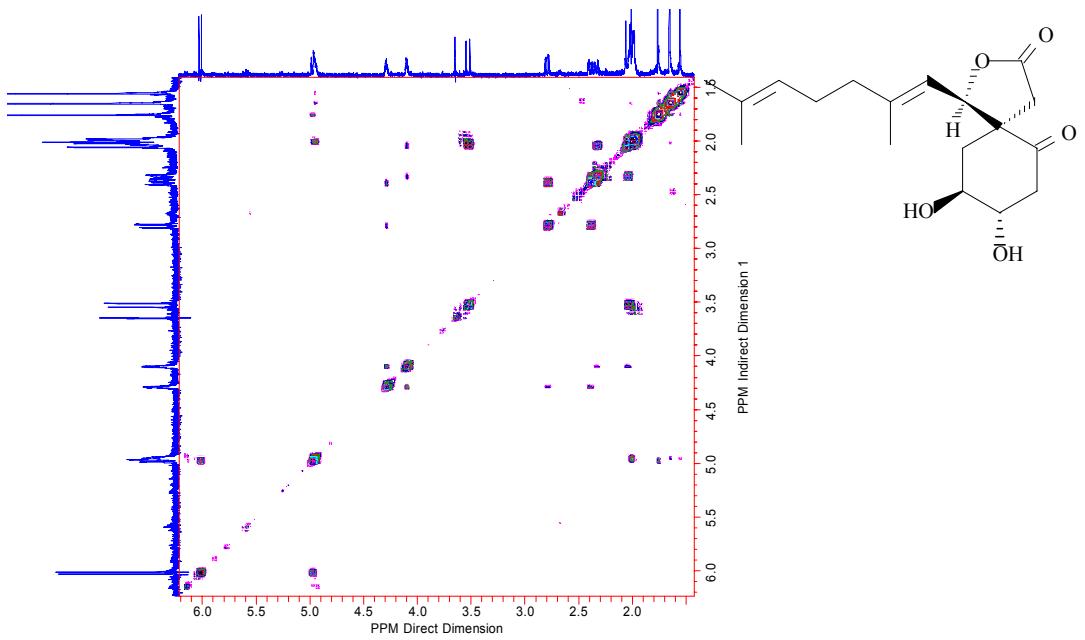


Figure F5. ^1H - ^1H COSY Spectrum of Miliusane IV (**6**), CDCl_3

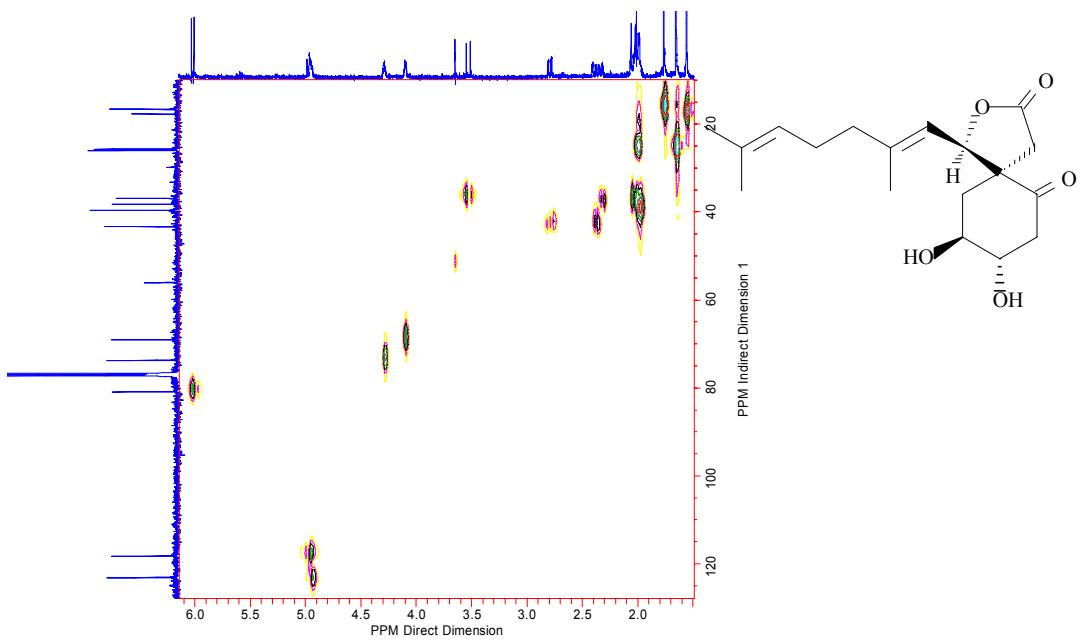


Figure F6. HMQC Spectrum of Miliusane IV (**6**), CDCl_3

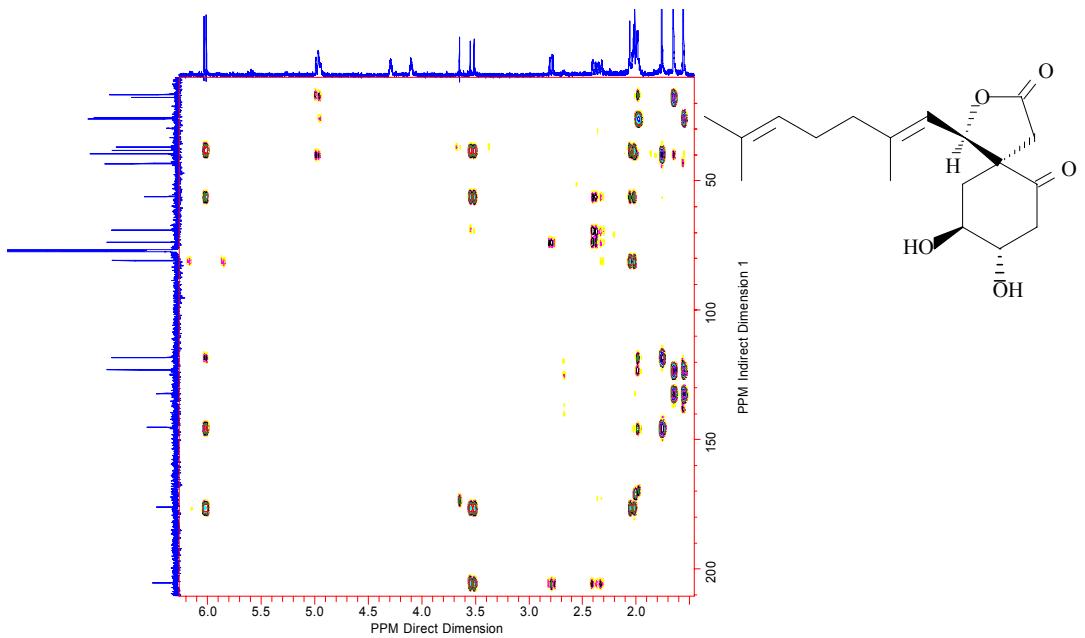


Figure F7. HMBC Spectrum of Miliusane IV (**6**), CDCl_3

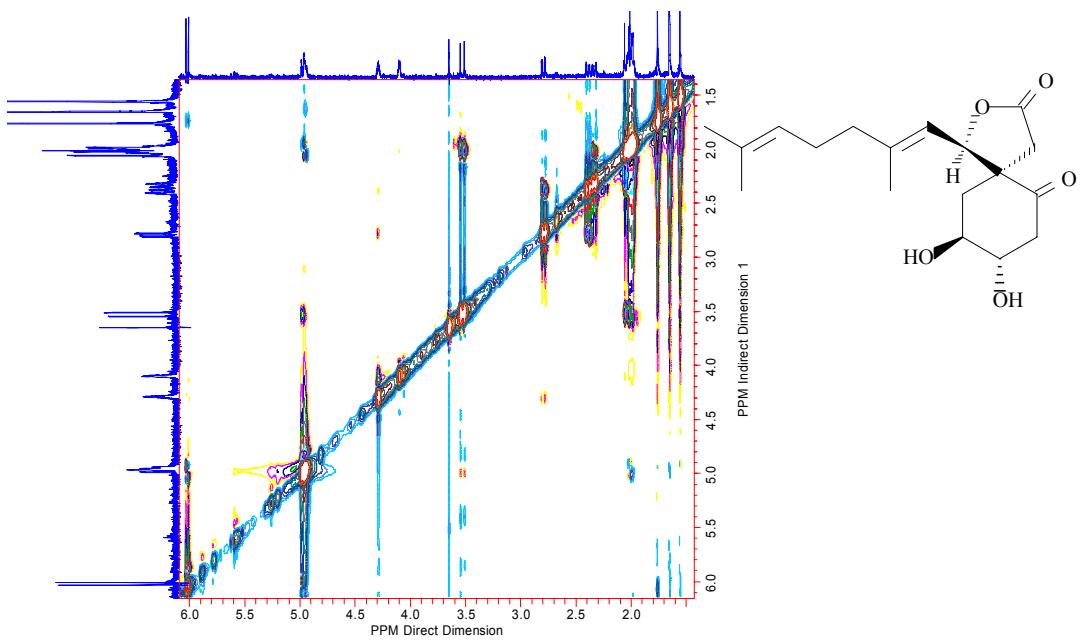


Figure F8. ROESY Spectrum of Miliusane IV (**6**), CDCl_3

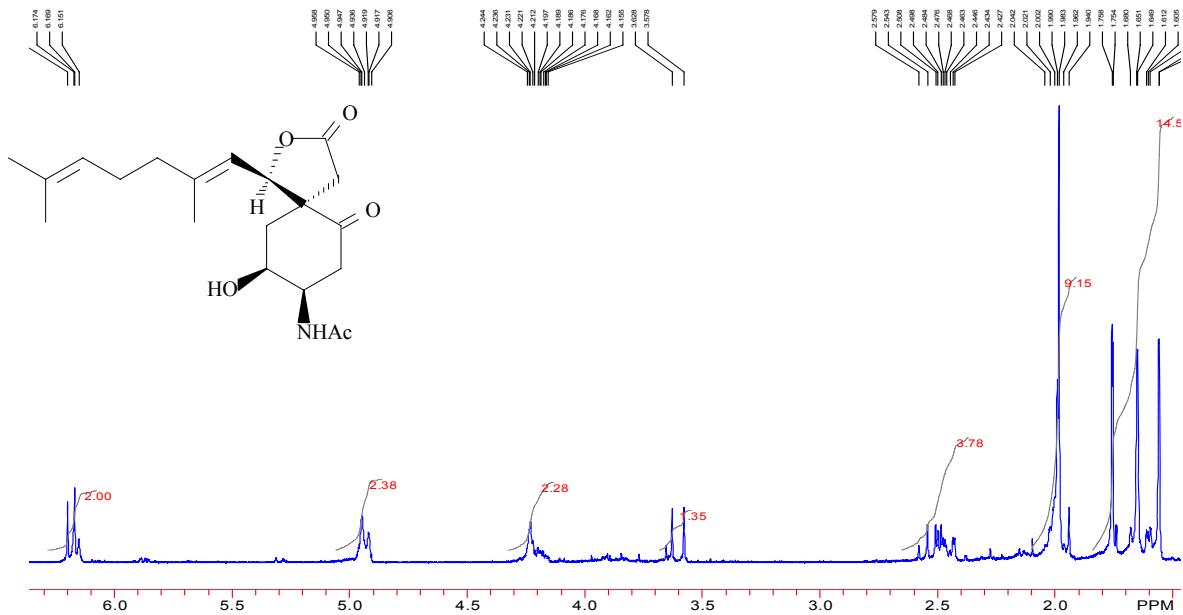


Figure G1. ^1H NMR Spectrum of Miliusane V (7), CDCl_3

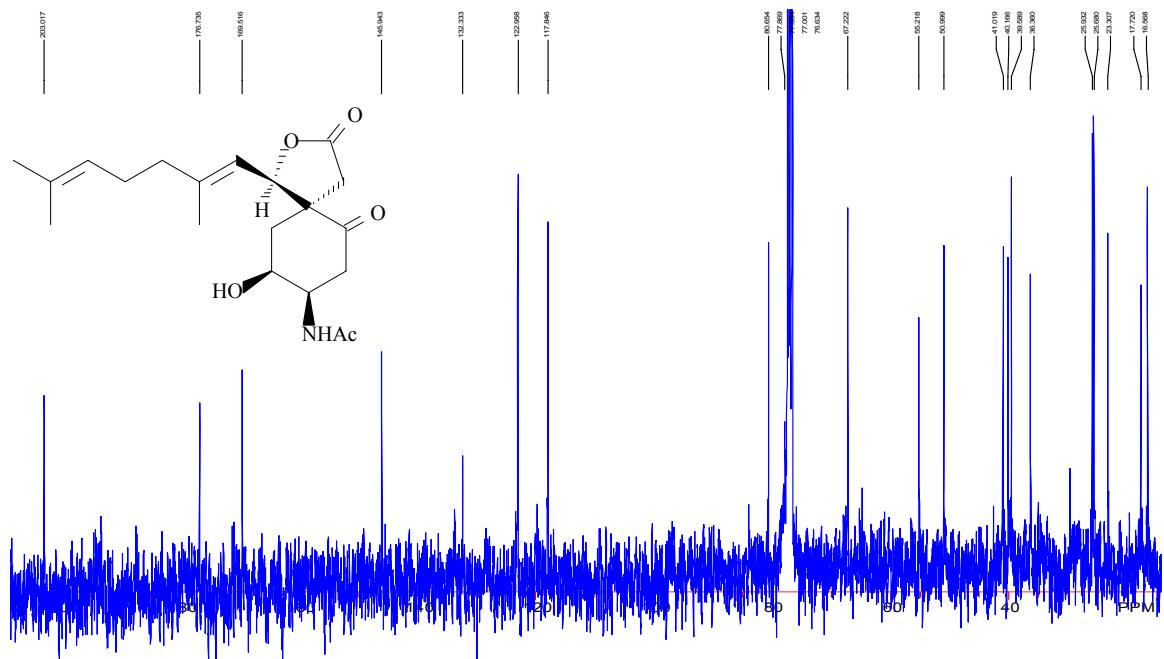


Figure G2. ^{13}C NMR Spectrum of Miliusane V (7), CDCl_3

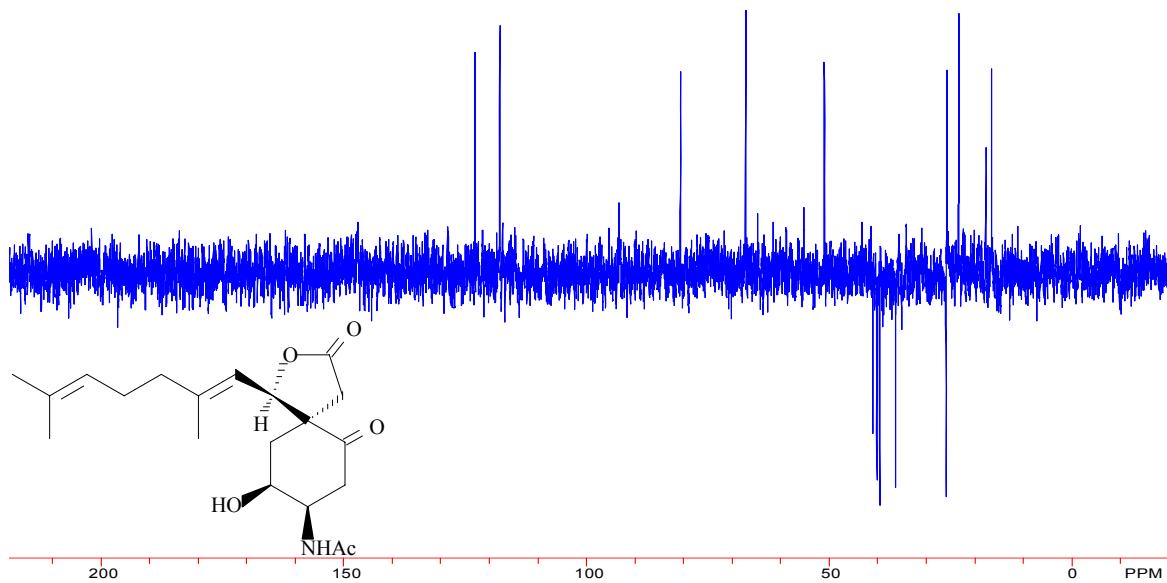


Figure G3. DEPT-135 Spectrum of Miliusane V (7), CDCl_3

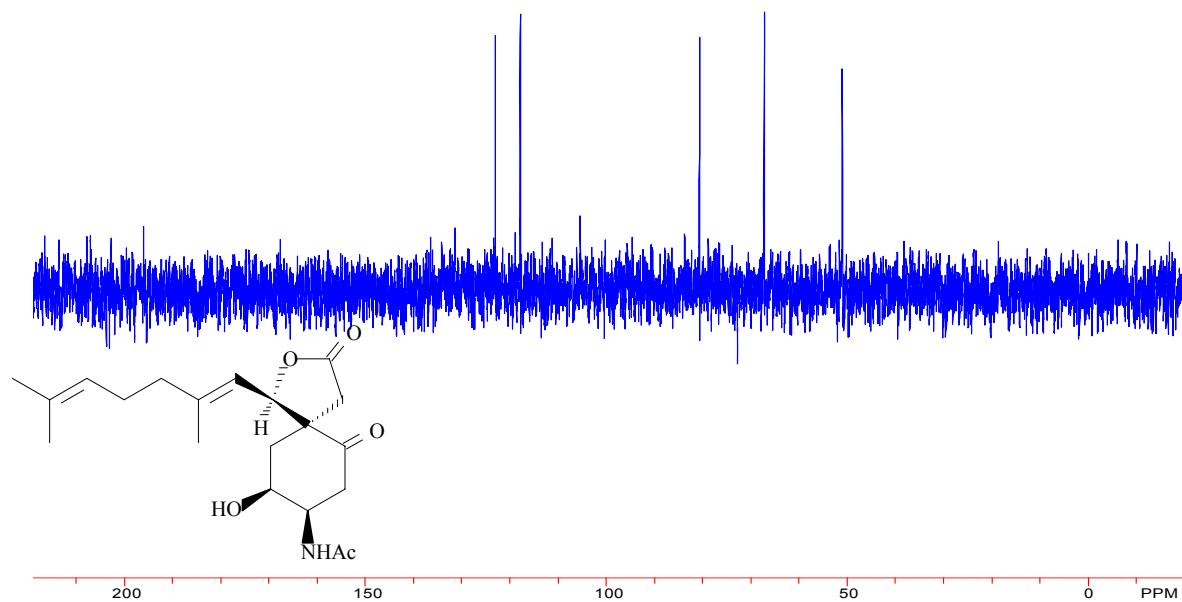


Figure G4. DEPT-90 Spectrum of Miliusane V (7), CDCl_3

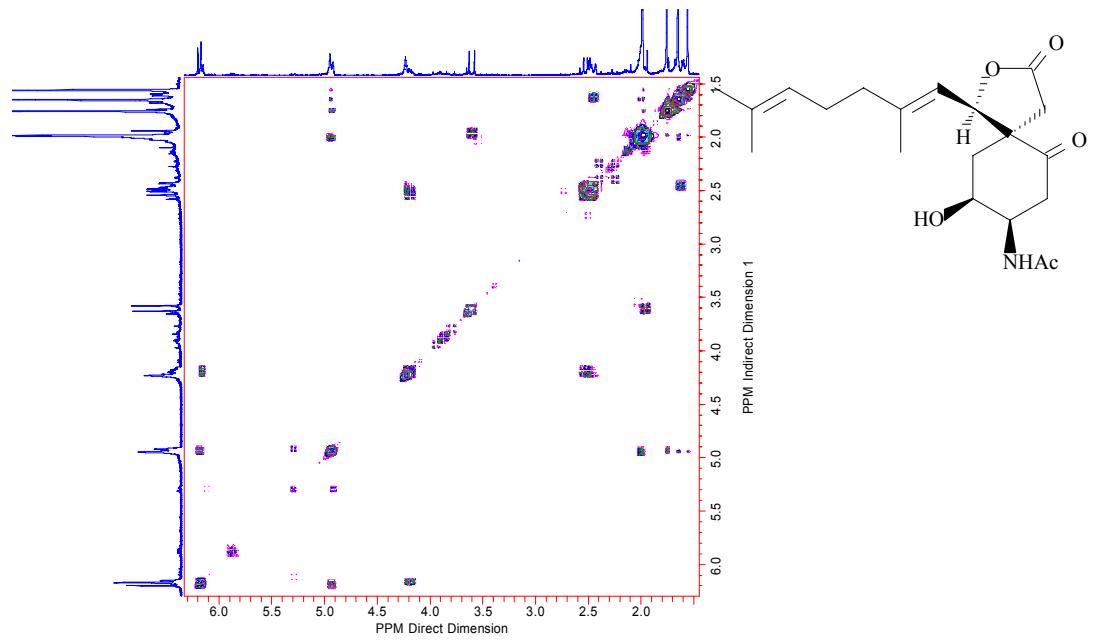


Figure G5. ^1H - ^1H COSY Spectrum of Miliusane V (**7**), CDCl_3

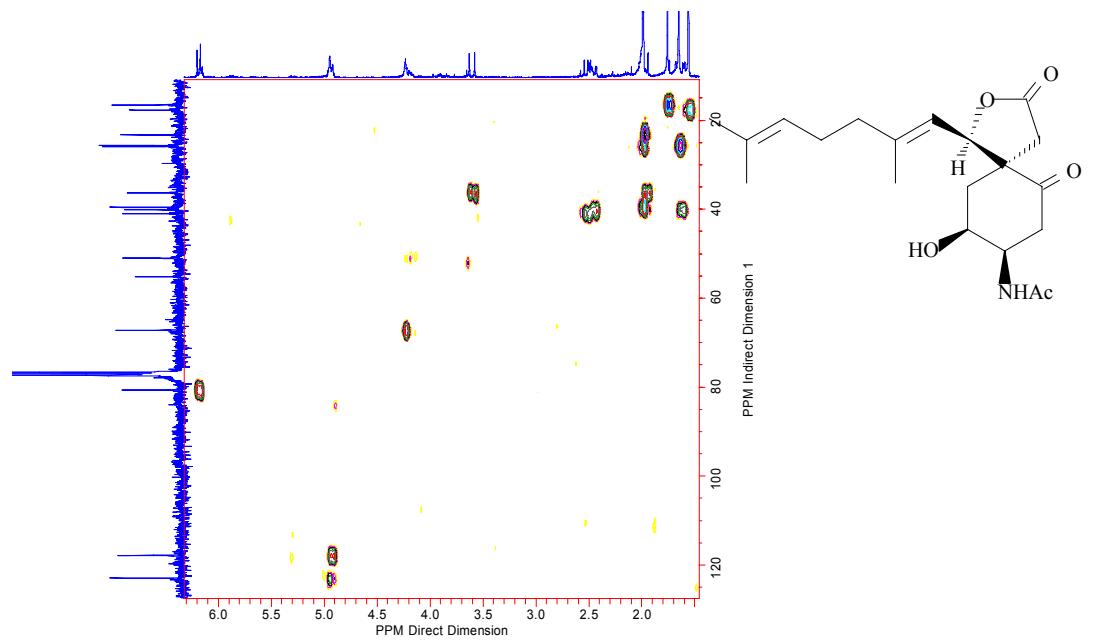


Figure G6. HMQC Spectrum of Miliusane V (**7**), CDCl_3

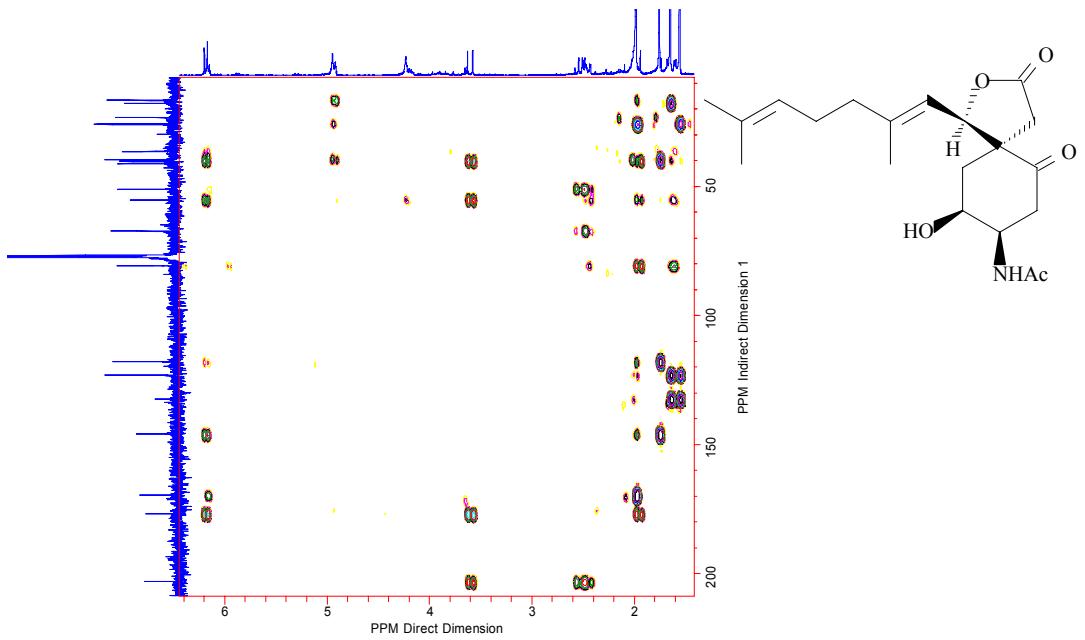


Figure G7. HMBC Spectrum of Miliusane V (7), CDCl_3

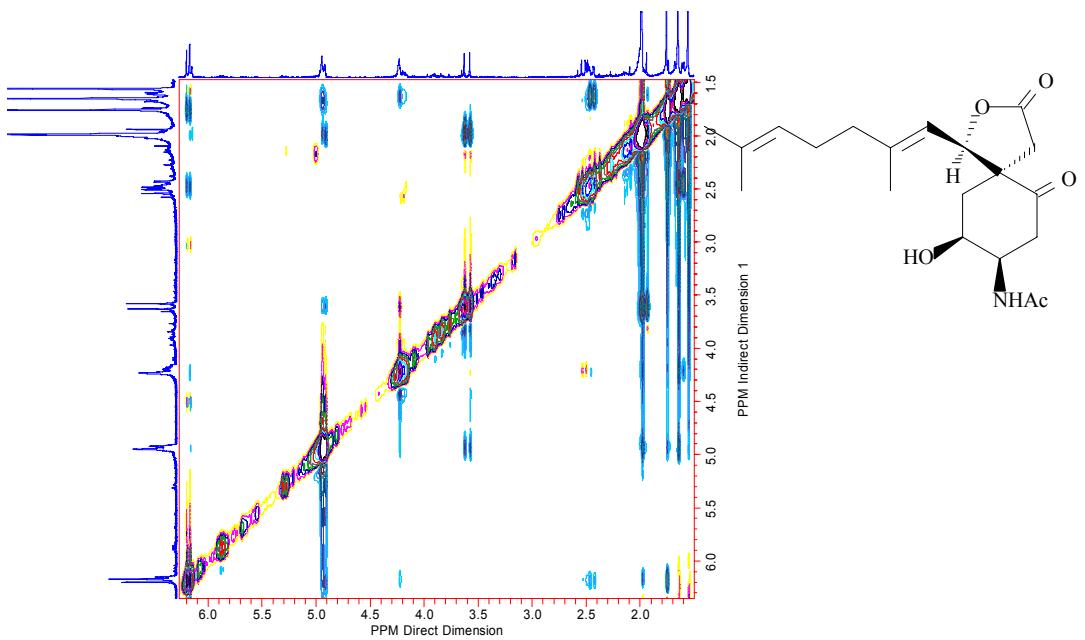


Figure G8. ROESY Spectrum of Miliusane V (7), CDCl_3

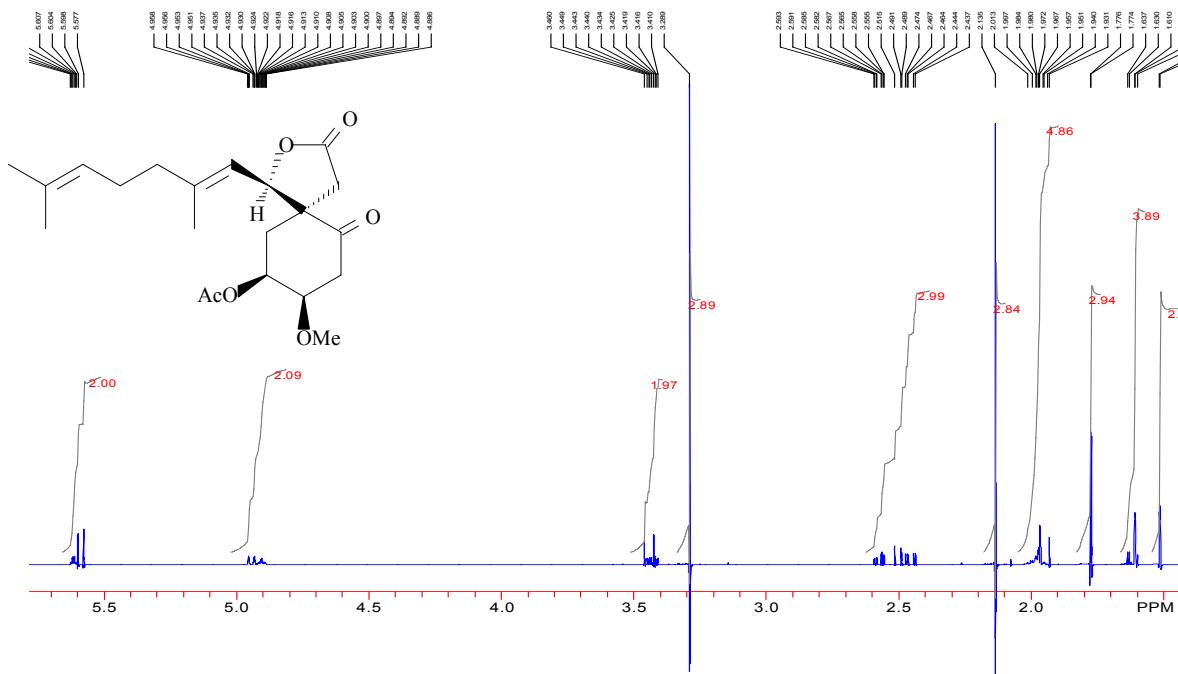


Figure H1. ¹H NMR Spectrum of Miliusane VI (8), CDCl₃

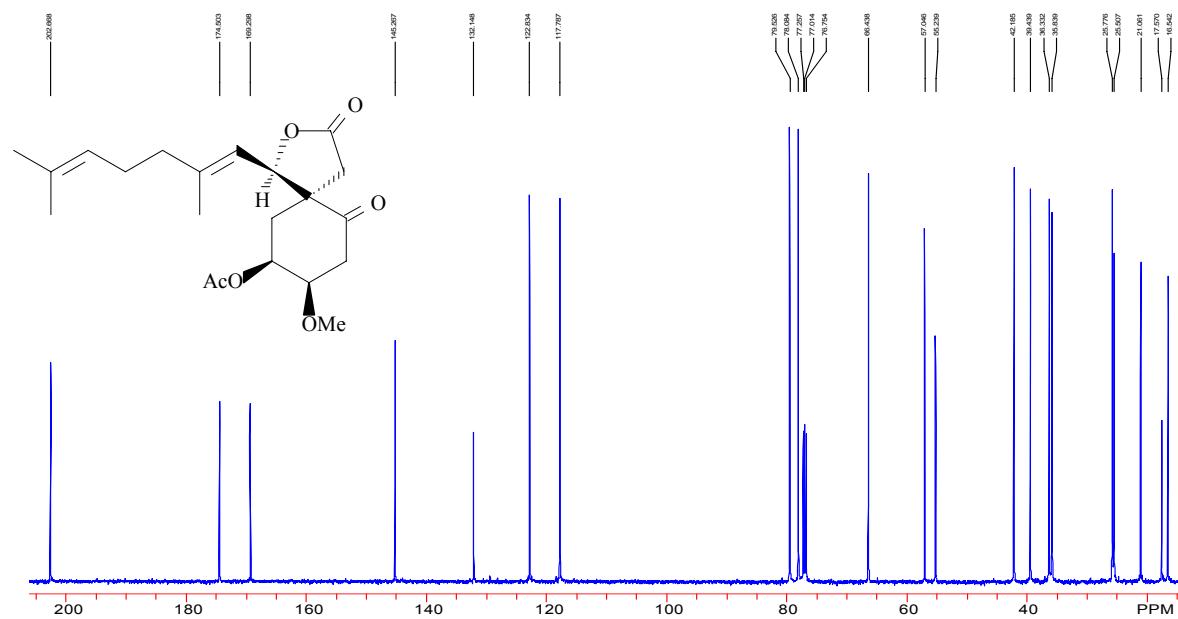


Figure H2. ¹³C NMR Spectrum of Miliusane VI (8), CDCl₃

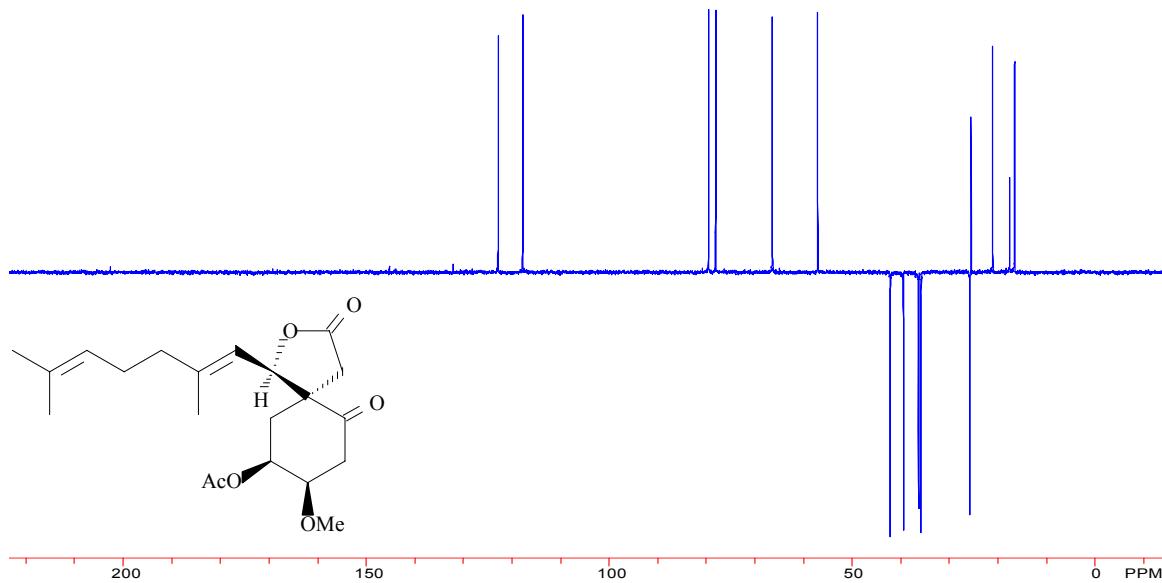


Figure H3. DEPT-135 Spectrum of Miliusane VI (**8**), CDCl_3

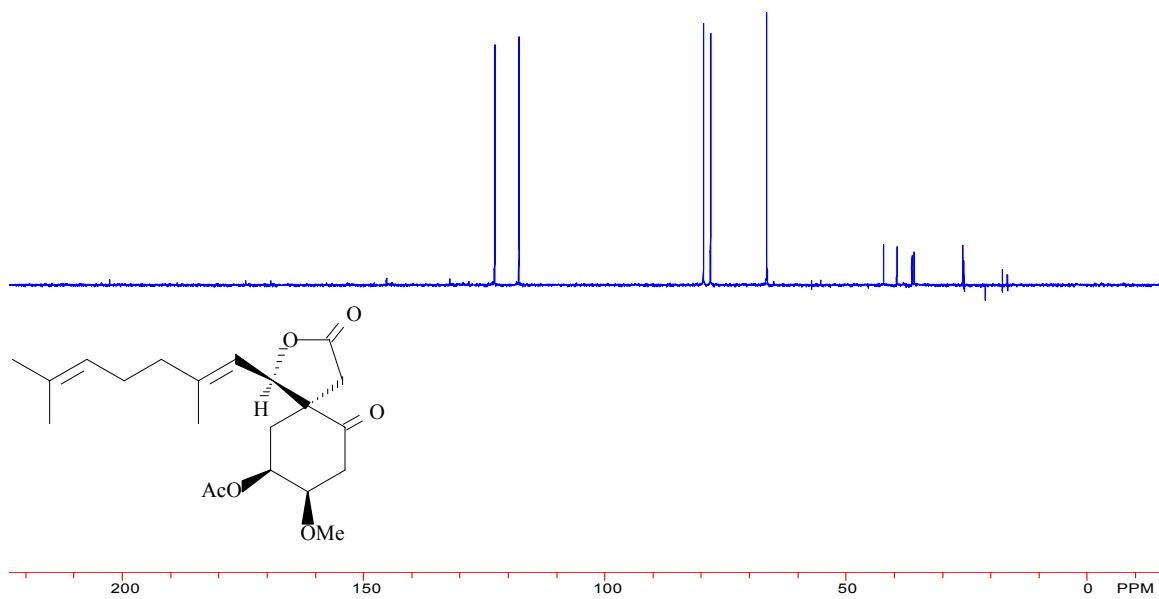


Figure H4. DEPT-90 Spectrum of Miliusane VI (**8**), CDCl_3

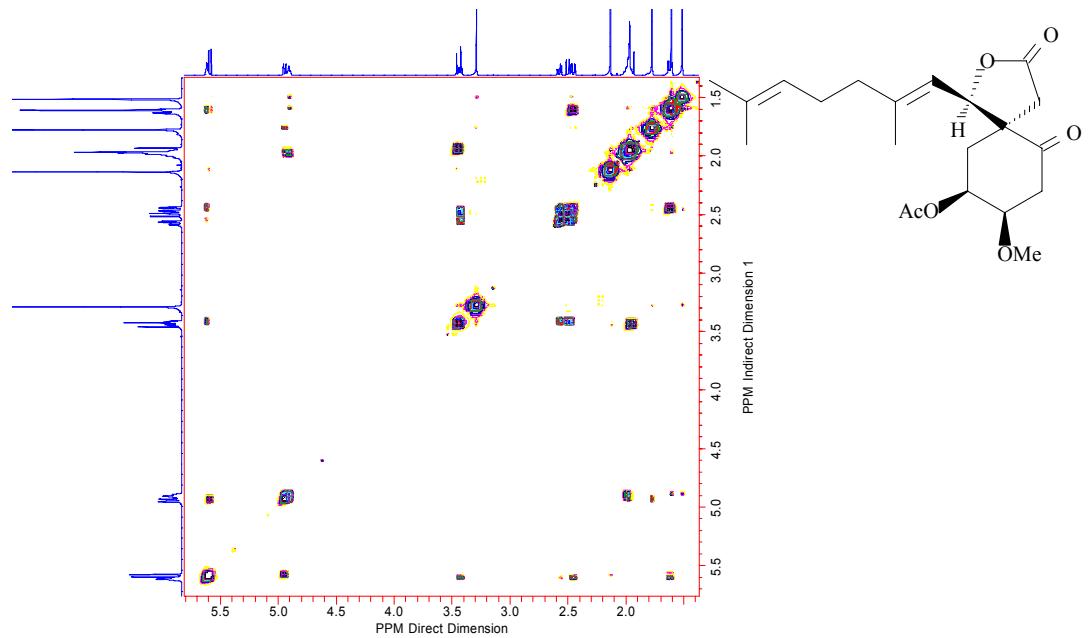


Figure H5. ^1H - ^1H COSY Spectrum of Miliusane VI (**8**), CDCl_3

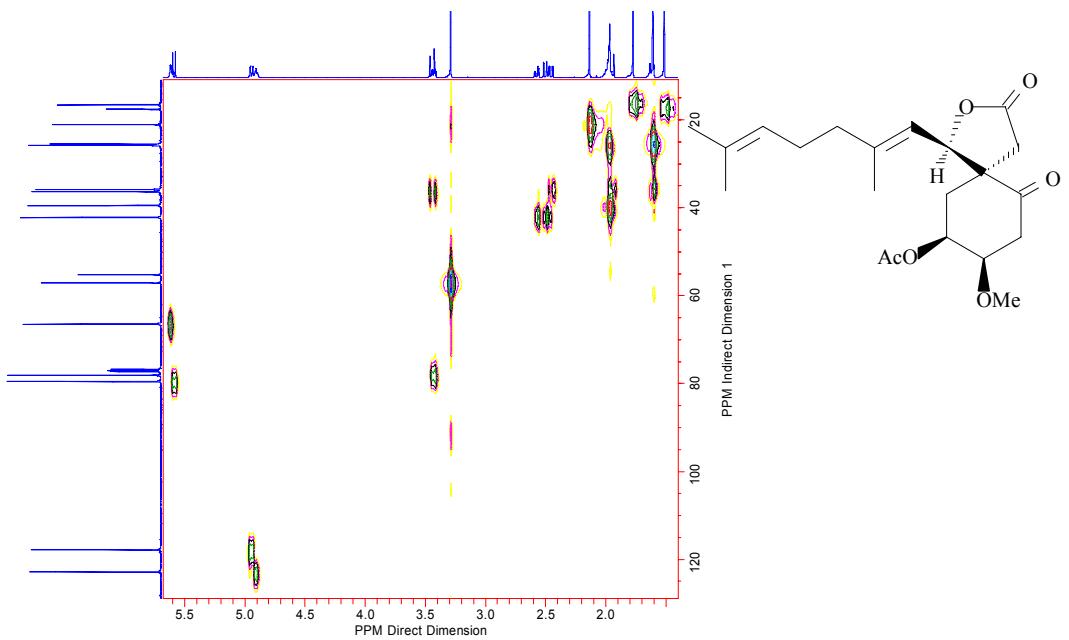


Figure H6. HMQC Spectrum of Miliusane VI (**8**), CDCl_3

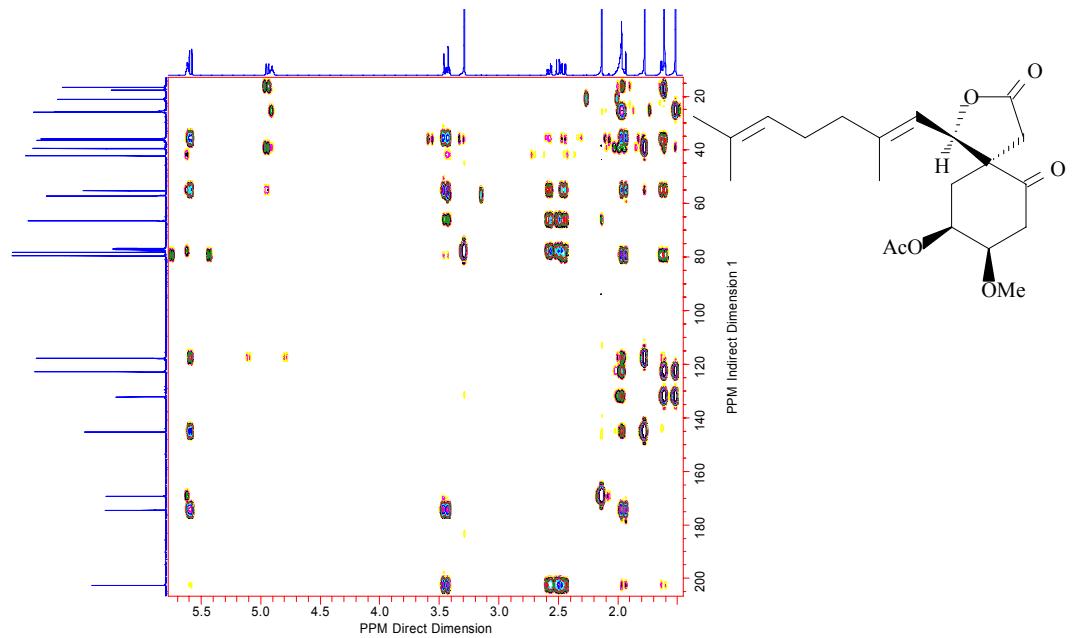


Figure H7. HMBC Spectrum of Miliusane VI (**8**), CDCl_3

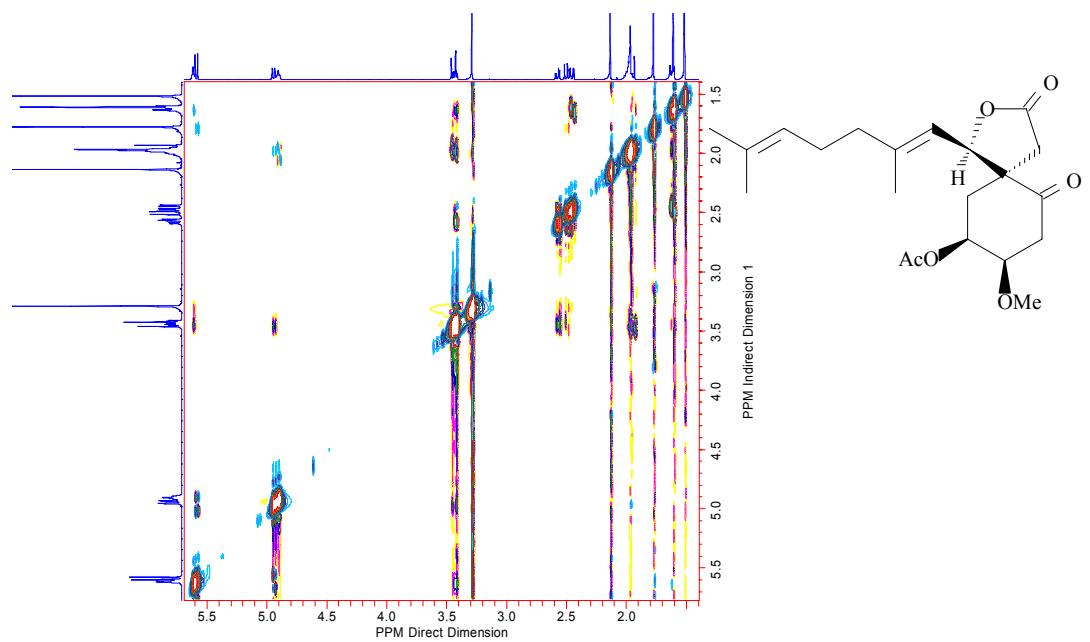


Figure H8. ROESY Spectrum of Miliusane VI (**8**), CDCl_3

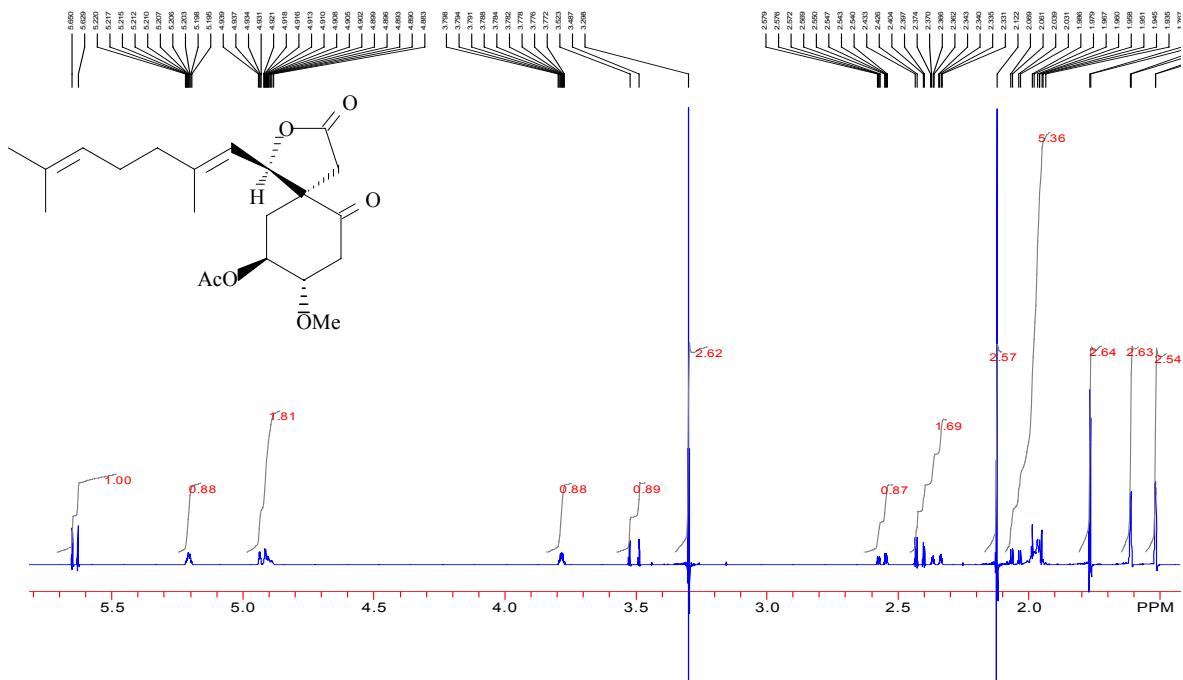


Figure 11. ^1H NMR Spectrum of Miliusane VII (**9**), CDCl_3

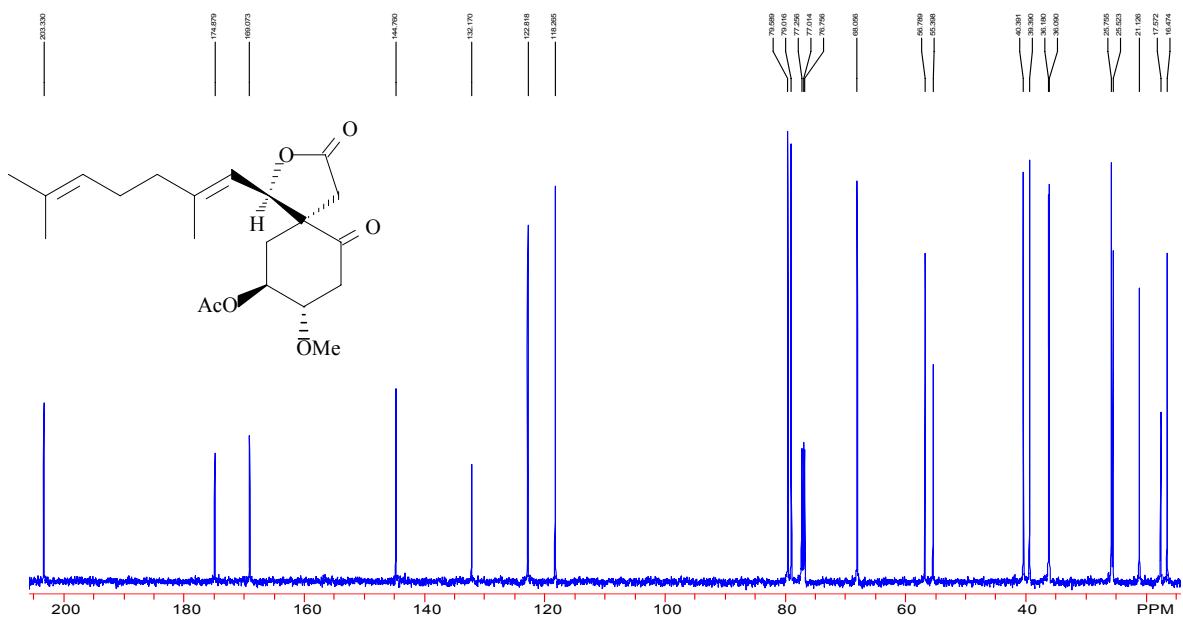


Figure 12. ^1H NMR Spectrum of Miliusane VII (**9**), CDCl_3

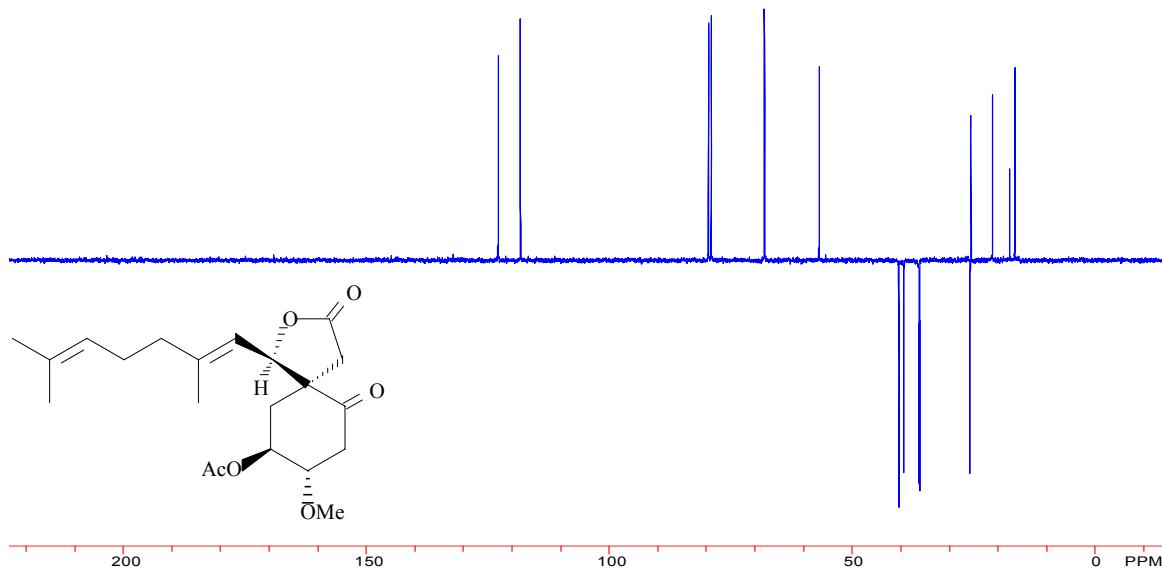


Figure I3. DEPT-135 Spectrum of Miliusane VII (**9**), CDCl_3

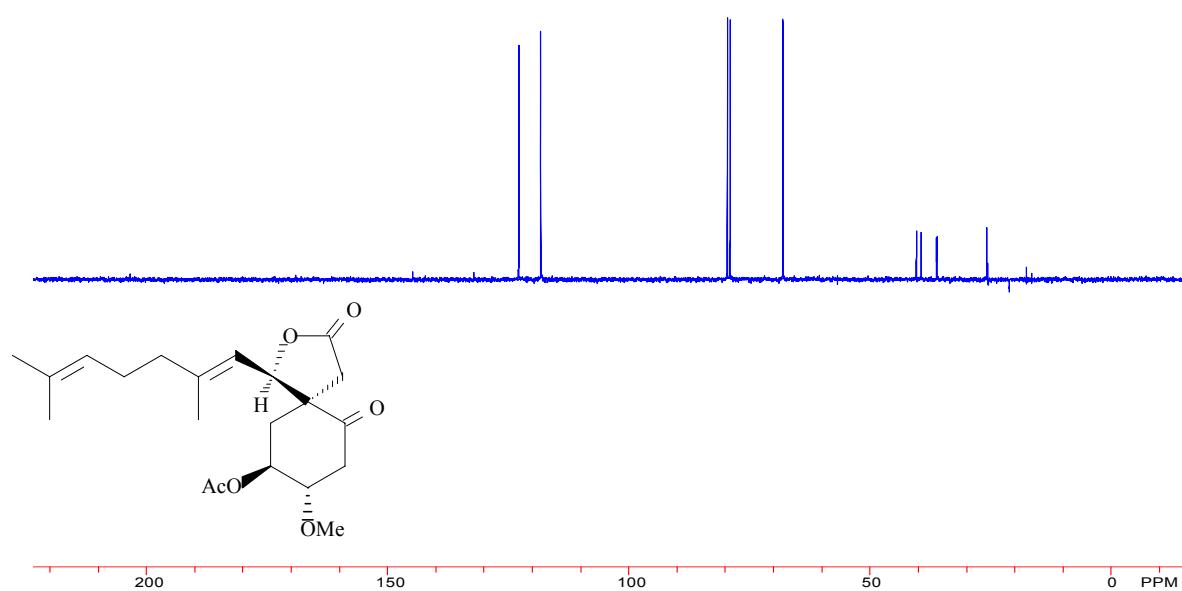


Figure I4. DEPT-90 Spectrum of Miliusane VII (**9**), CDCl_3

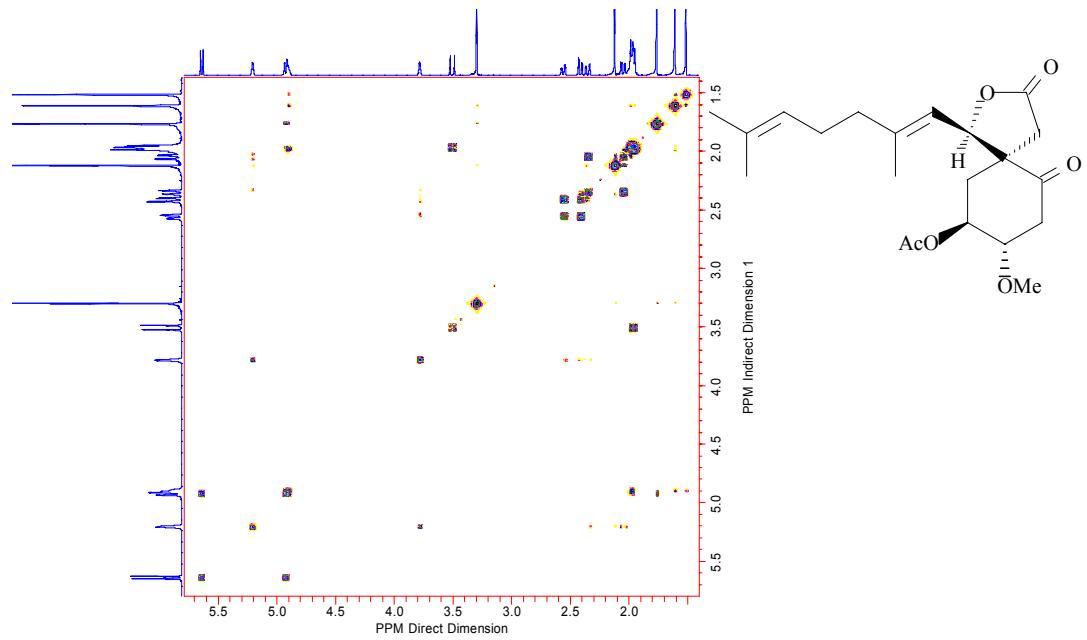


Figure I5. ^1H - ^1H COSY Spectrum of Miliusane VII (**9**), CDCl_3

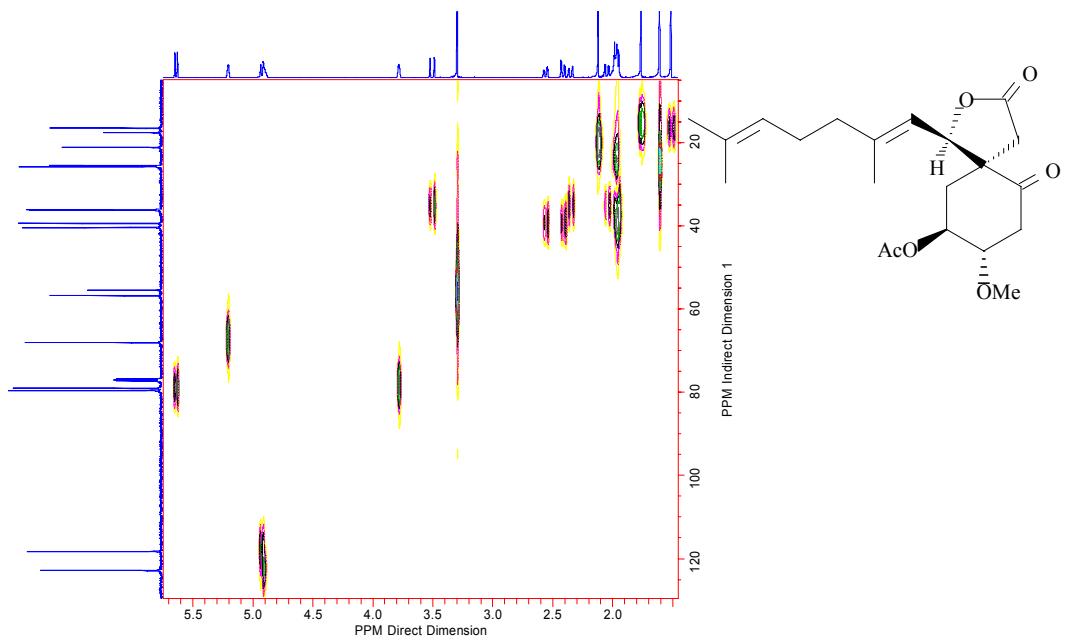


Figure I6. HMQC Spectrum of Miliusane VII (**9**), CDCl_3

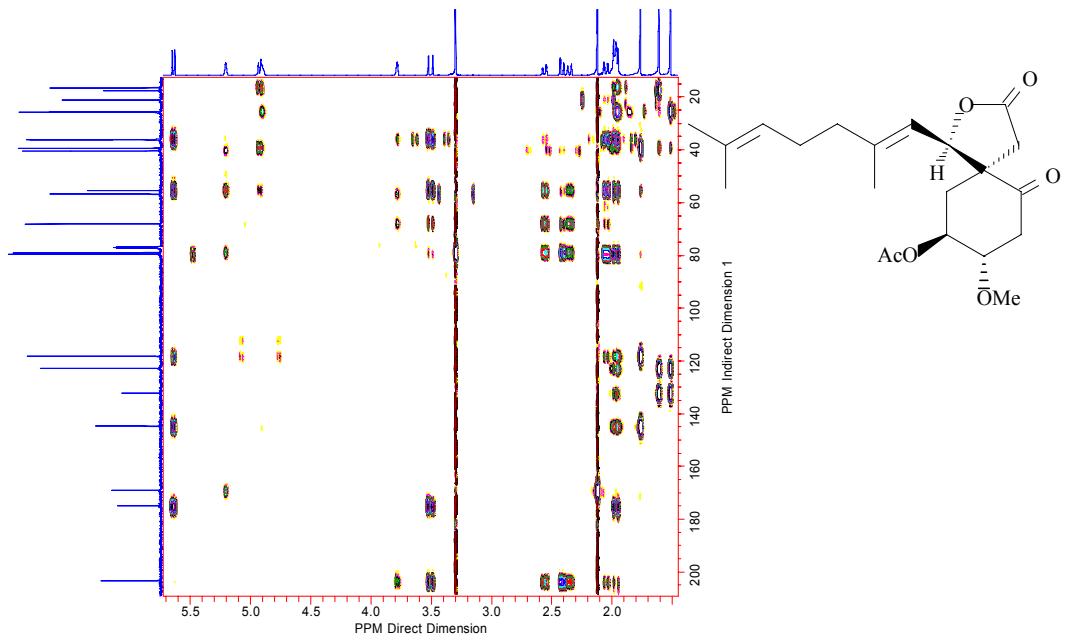


Figure I7. HMBC Spectrum of Miliusane VII (**9**), CDCl_3

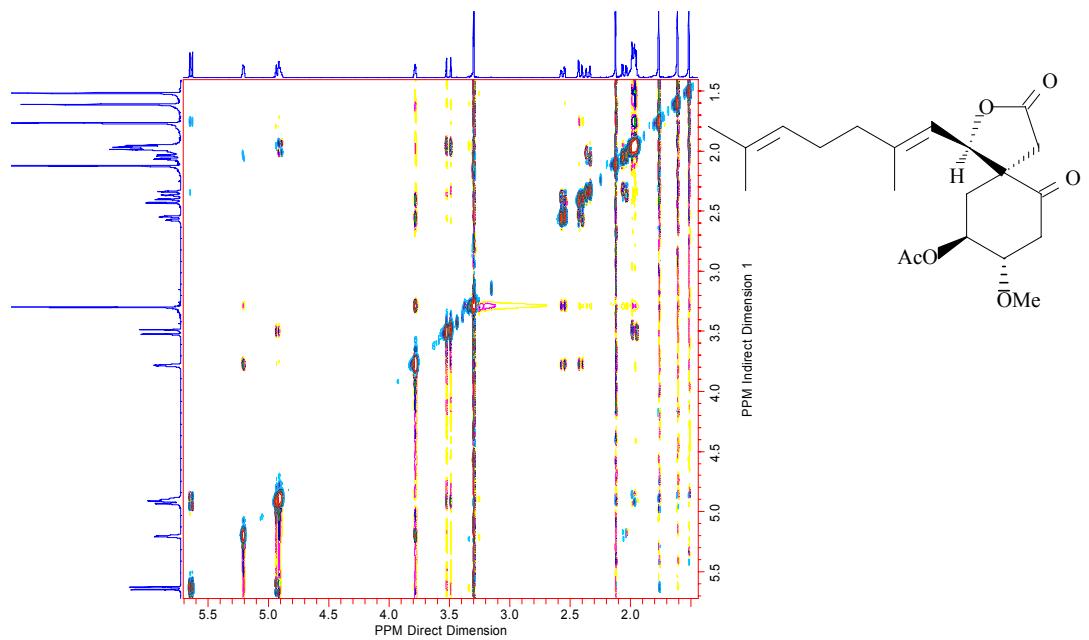
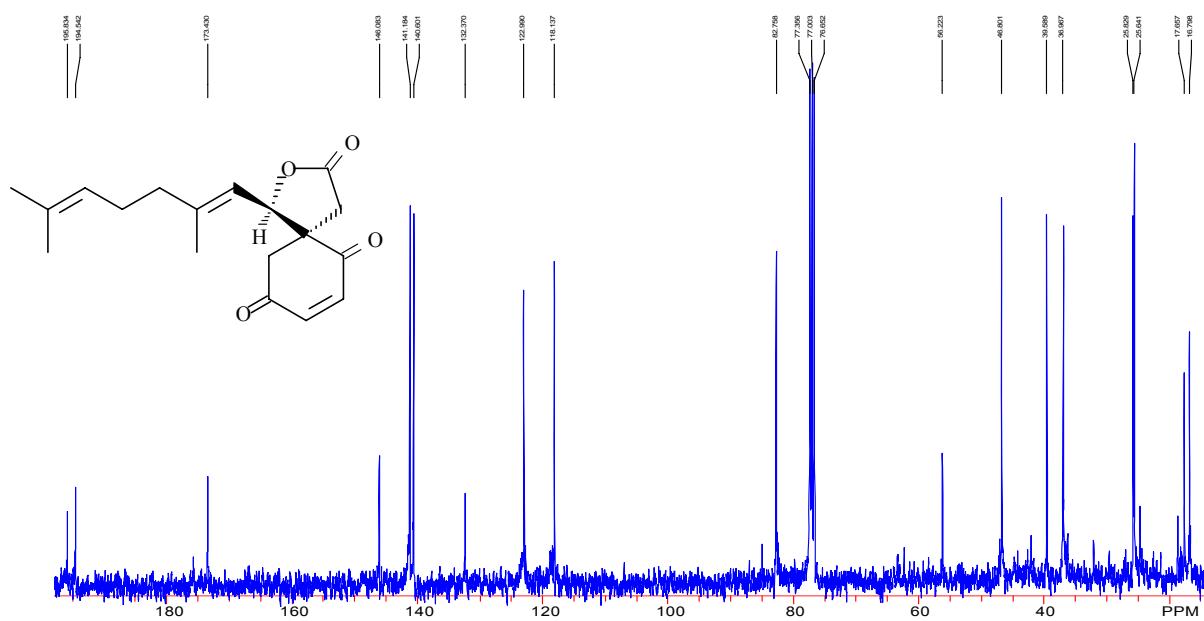
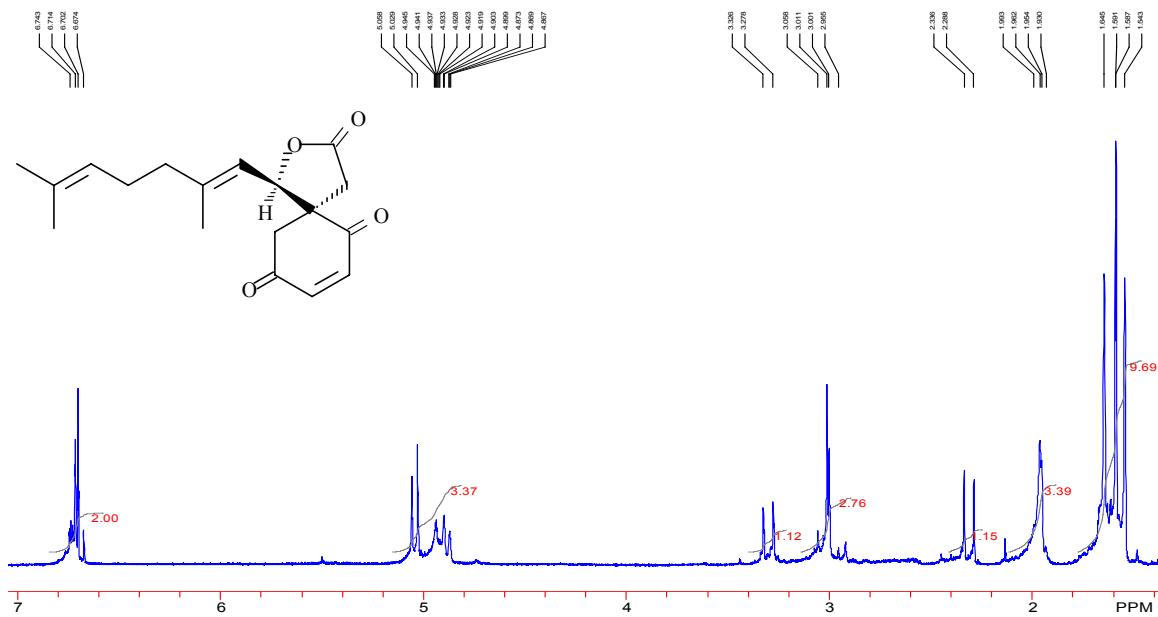


Figure I8. ROESY Spectrum of Miliusane VII (**9**), CDCl_3



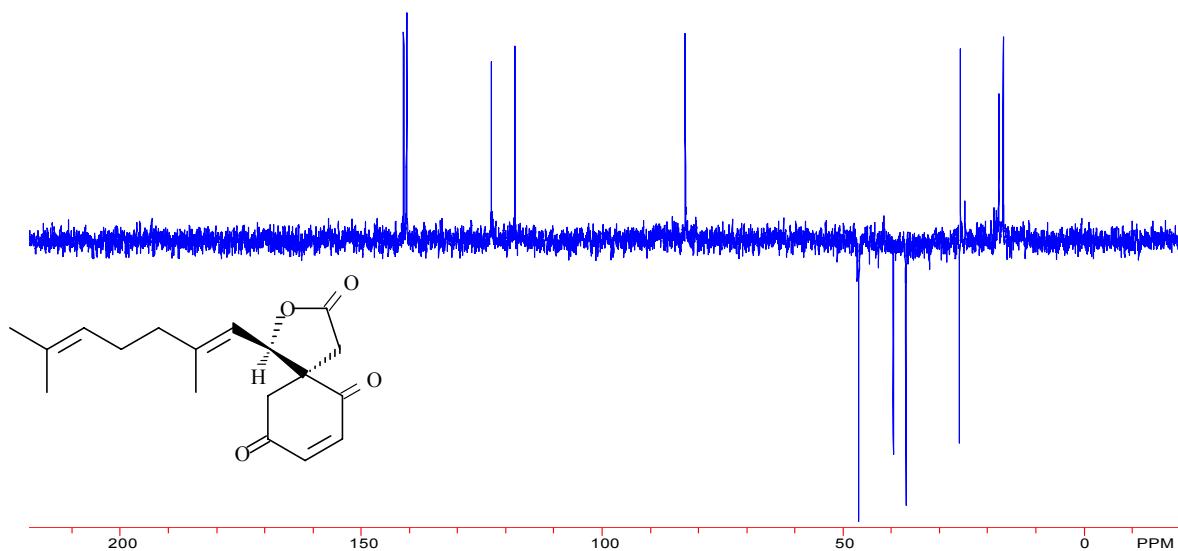


Figure J3. DEPT-135 Spectrum of Miliusane VIII (**10**), CDCl₃

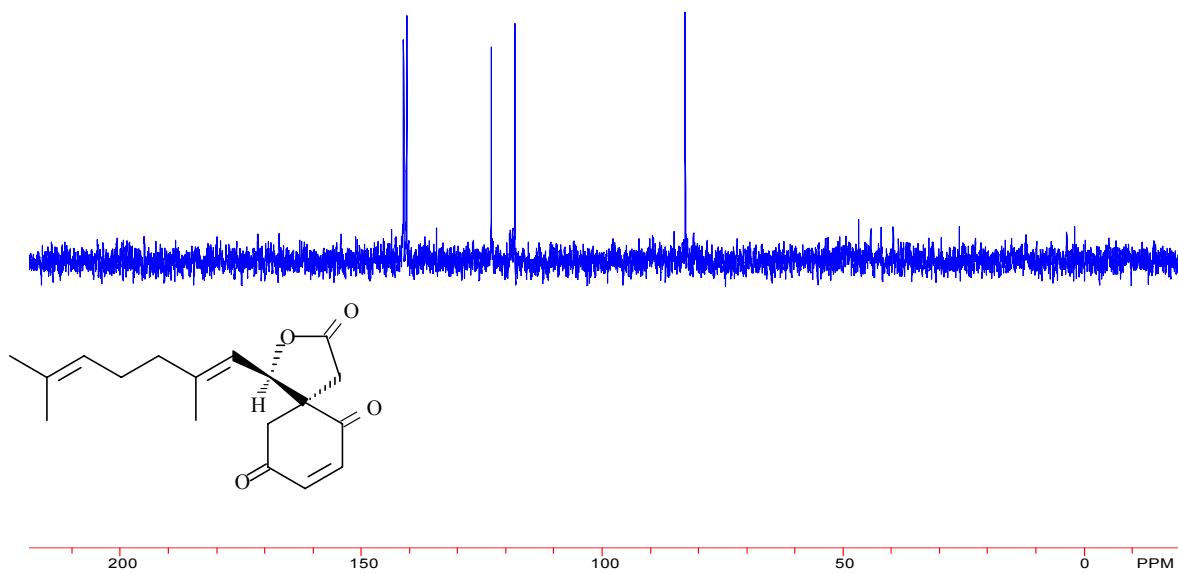


Figure J4. DEPT-90 Spectrum of Miliusane VIII (**10**), CDCl₃

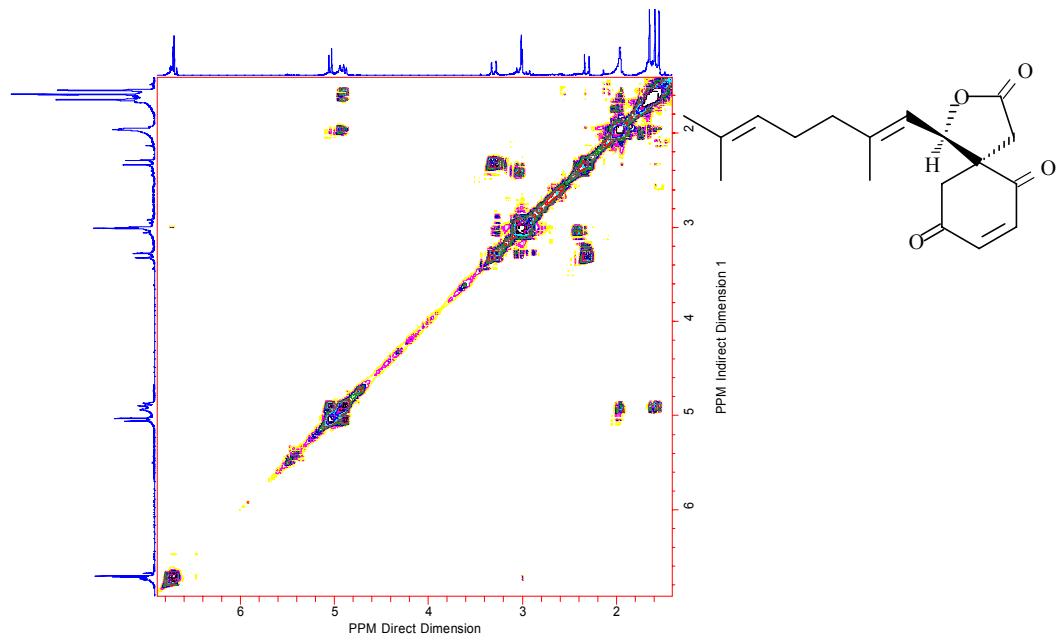


Figure J5. ^1H - ^1H COSY Spectrum of Miliusane VIII (**10**), CDCl_3

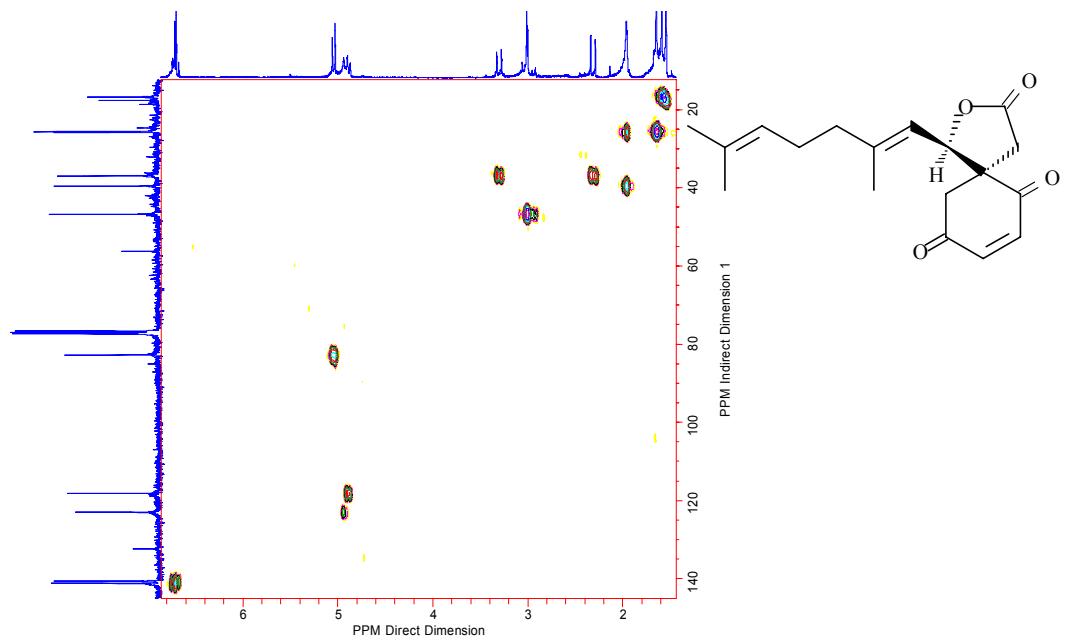


Figure J6. HMQC Spectrum of Miliusane VIII (**10**), CDCl_3

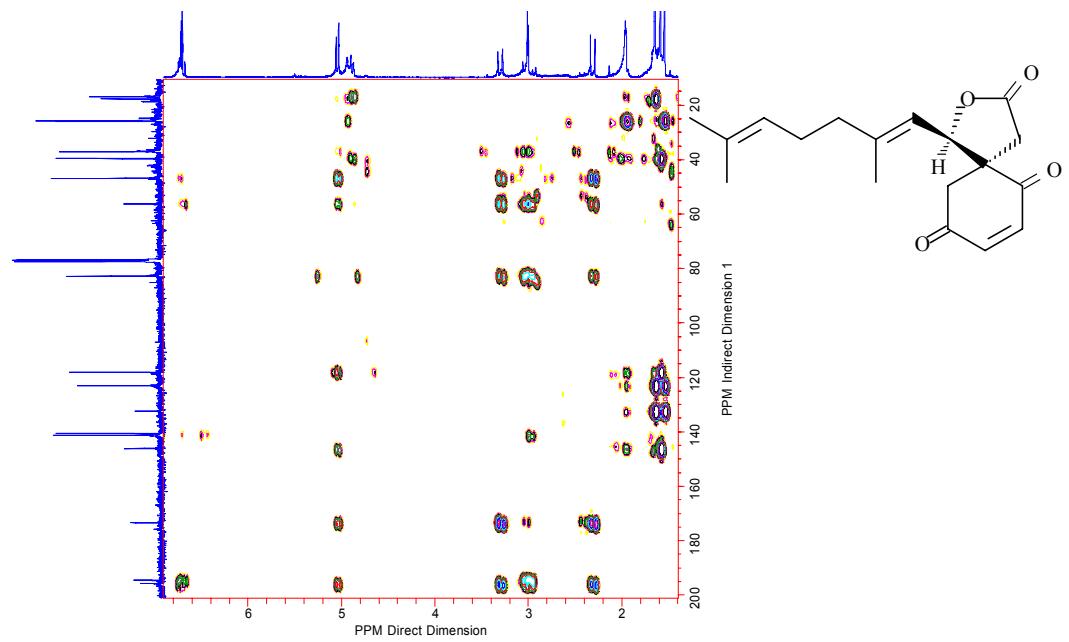


Figure J7. HMBC Spectrum of Miliusane VIII (**10**), CDCl_3

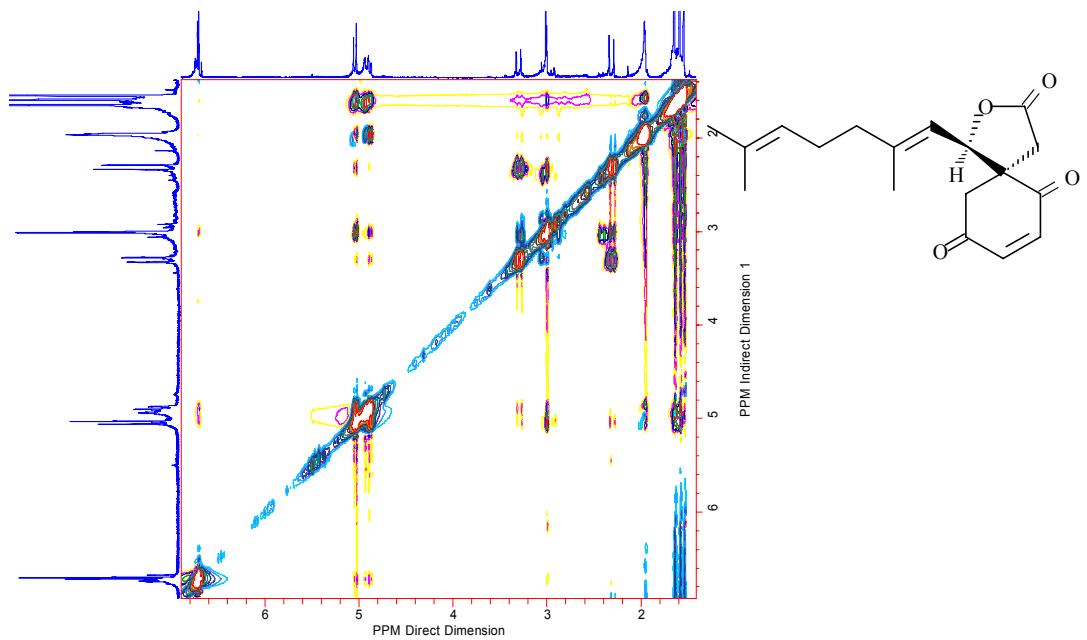


Figure J8. ROESY Spectrum of Miliusane VIII (**10**), CDCl_3

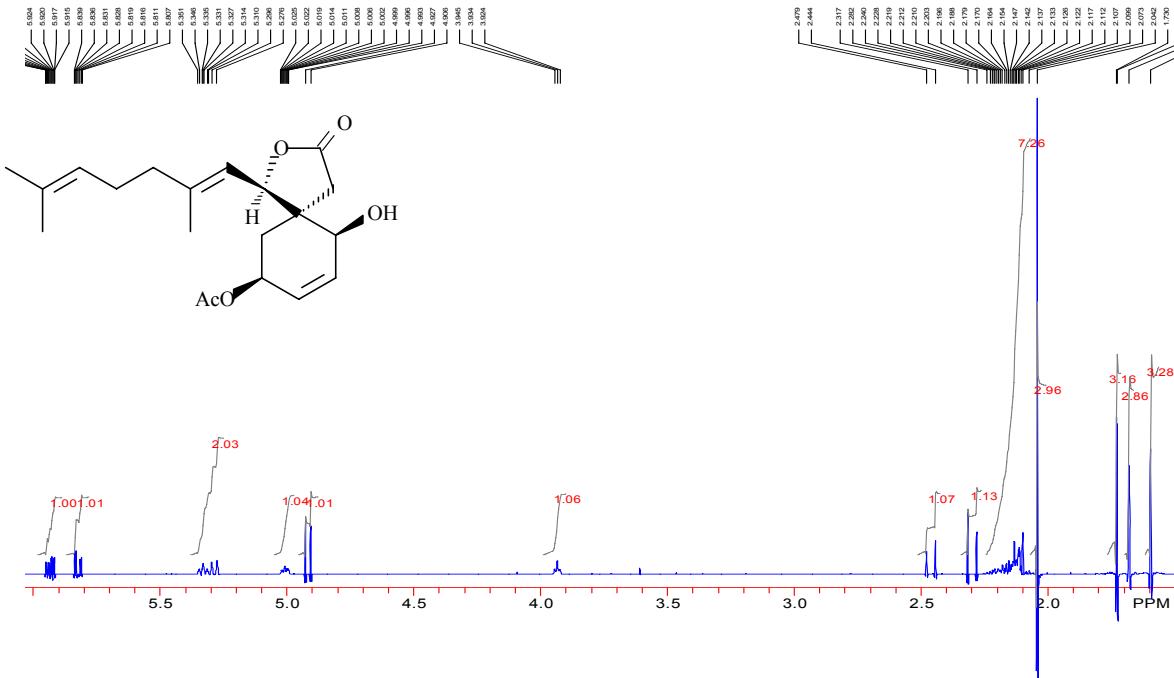


Figure K1. ¹H NMR Spectrum of Miliusane IX (11), CDCl₃

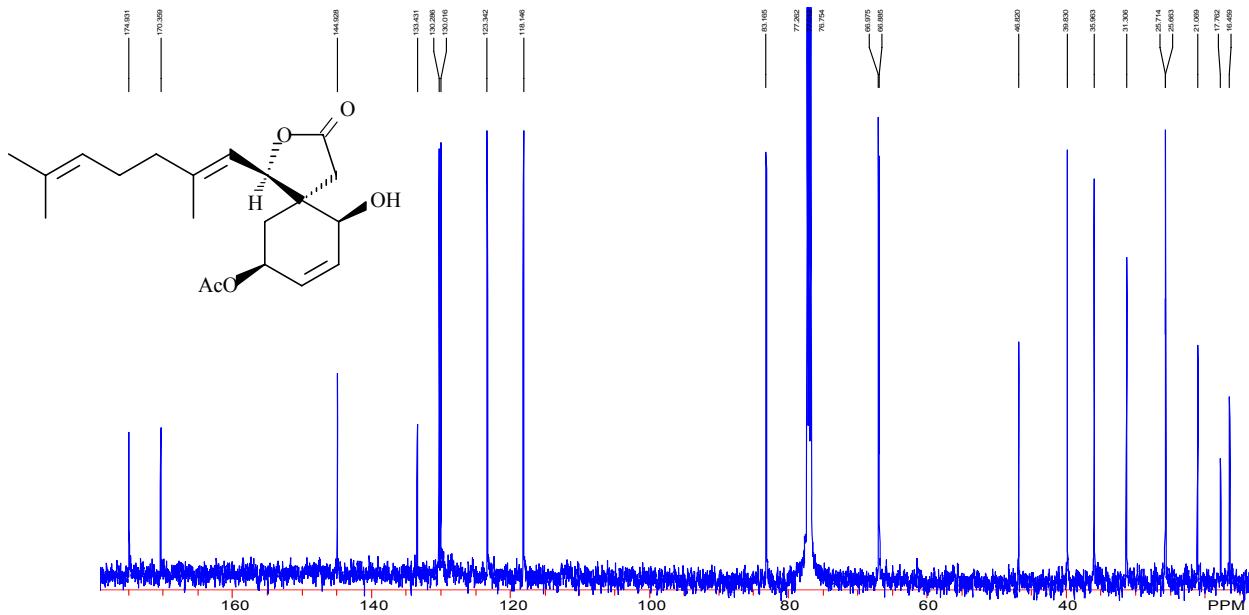


Figure K2. ¹³C NMR Spectrum of Miliusane IX (11), CDCl₃

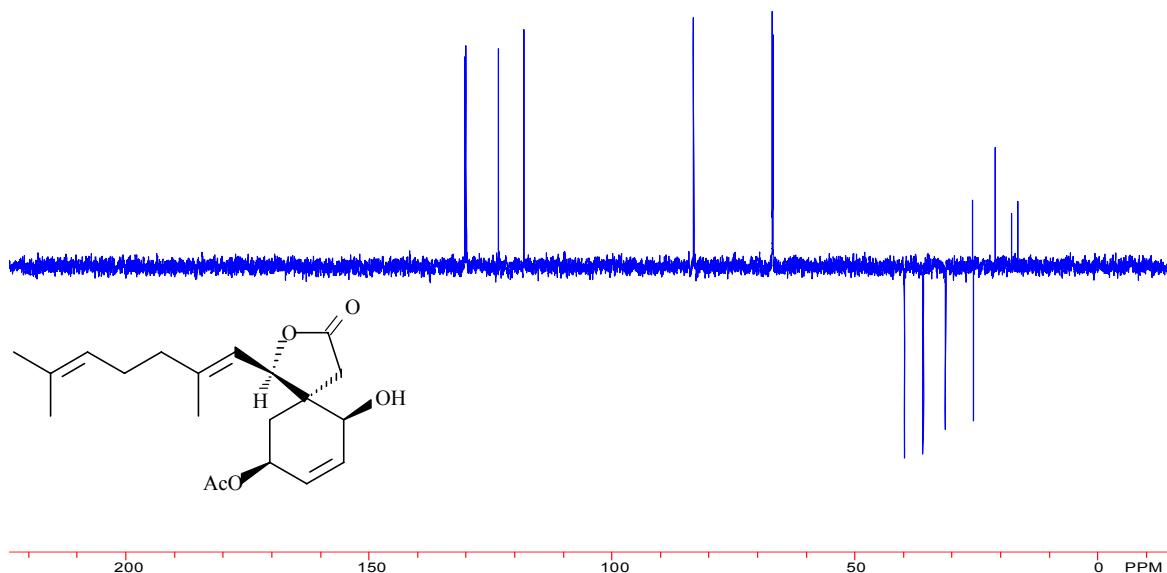


Figure K3. DEPT-135 Spectrum of Miliusane IX (**11**), CDCl_3

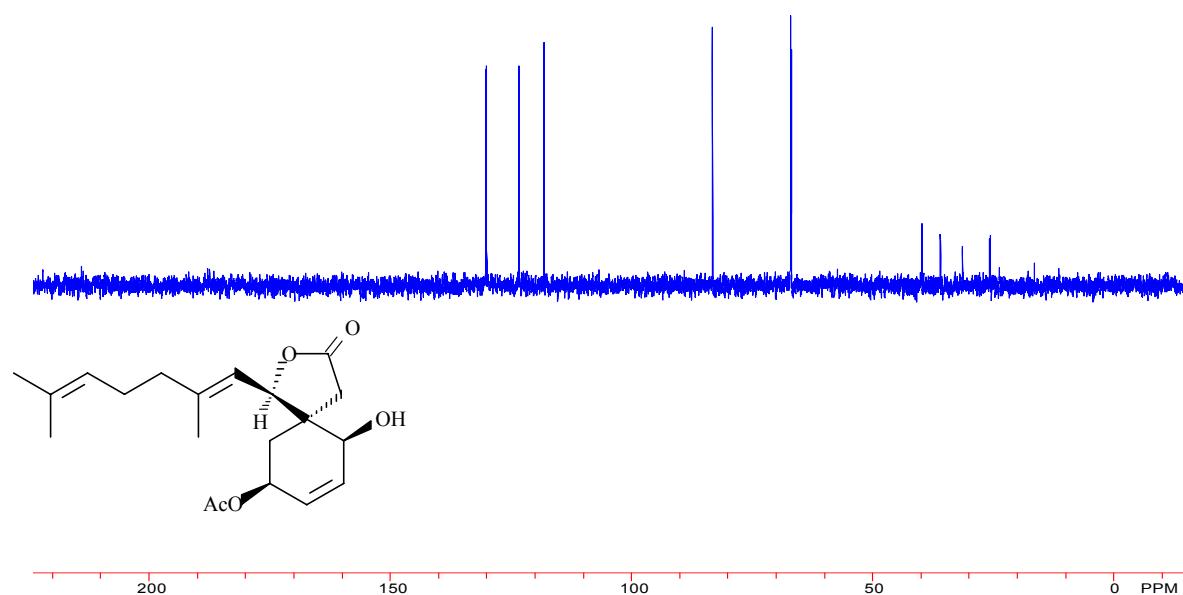


Figure K4. DEPT-90 Spectrum of Miliusane IX (**11**), CDCl_3

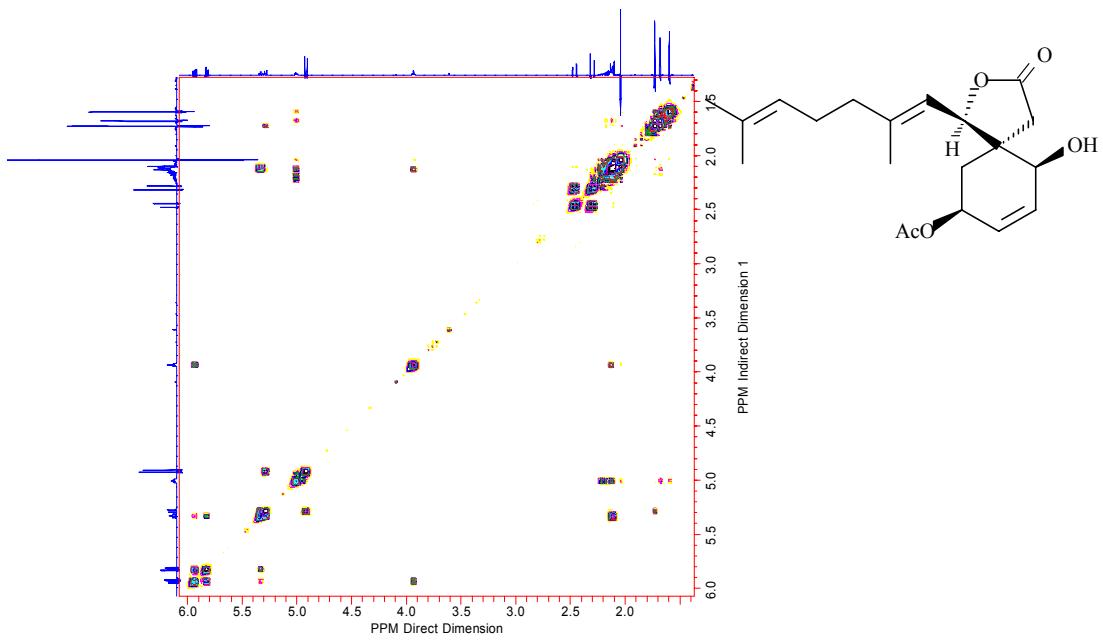


Figure K5. ^1H - ^1H COSY Spectrum of Miliusane IX (**11**), CDCl_3

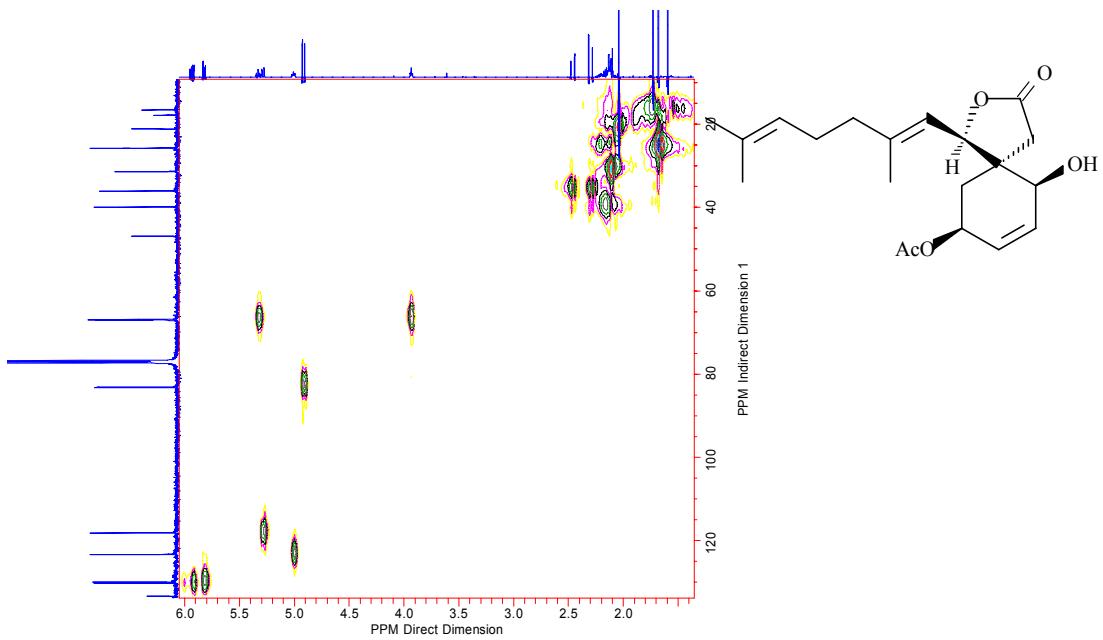


Figure K6. HMQC Spectrum of Miliusane IX (**11**), CDCl_3

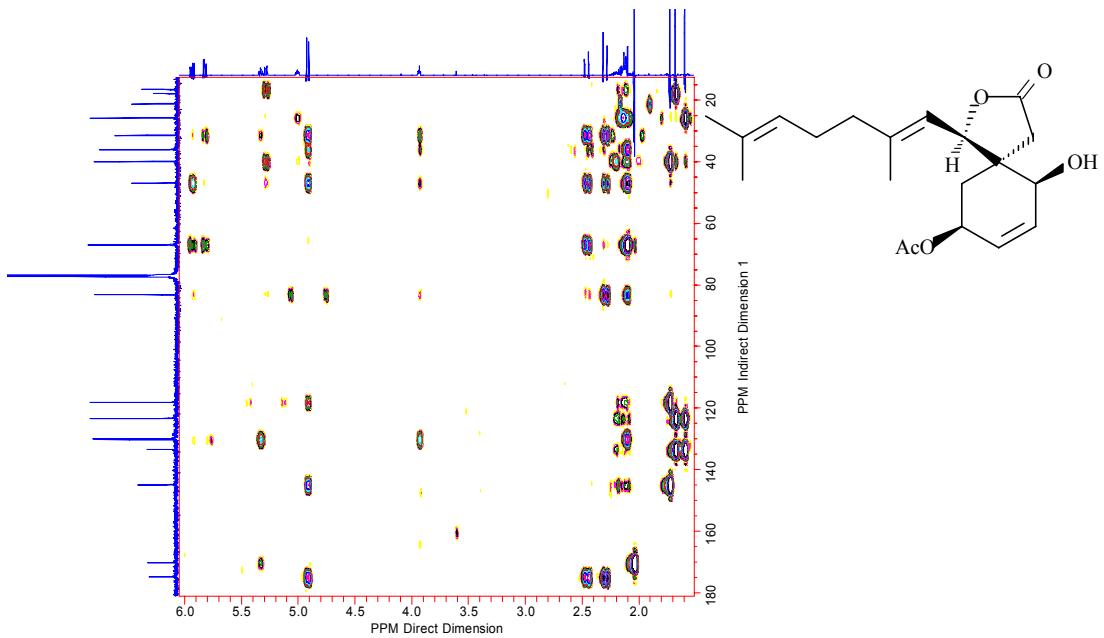


Figure K7. HMBC Spectrum of Miliusane IX (**11**), CDCl_3

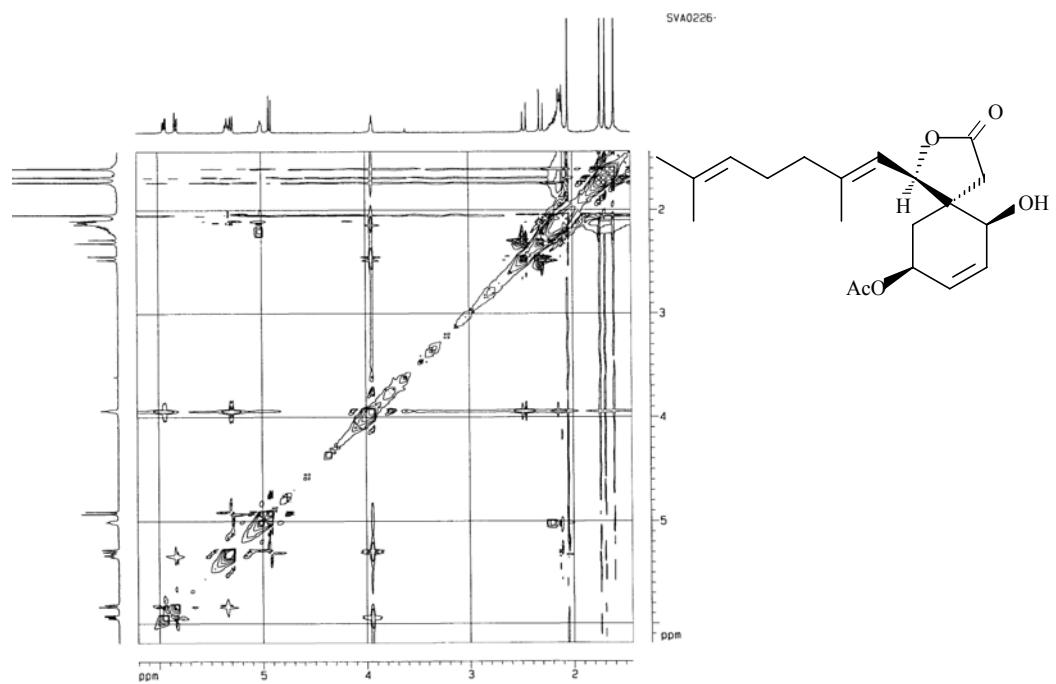


Figure K8. ROESY Spectrum of Miliusane IX (**11**), CDCl_3

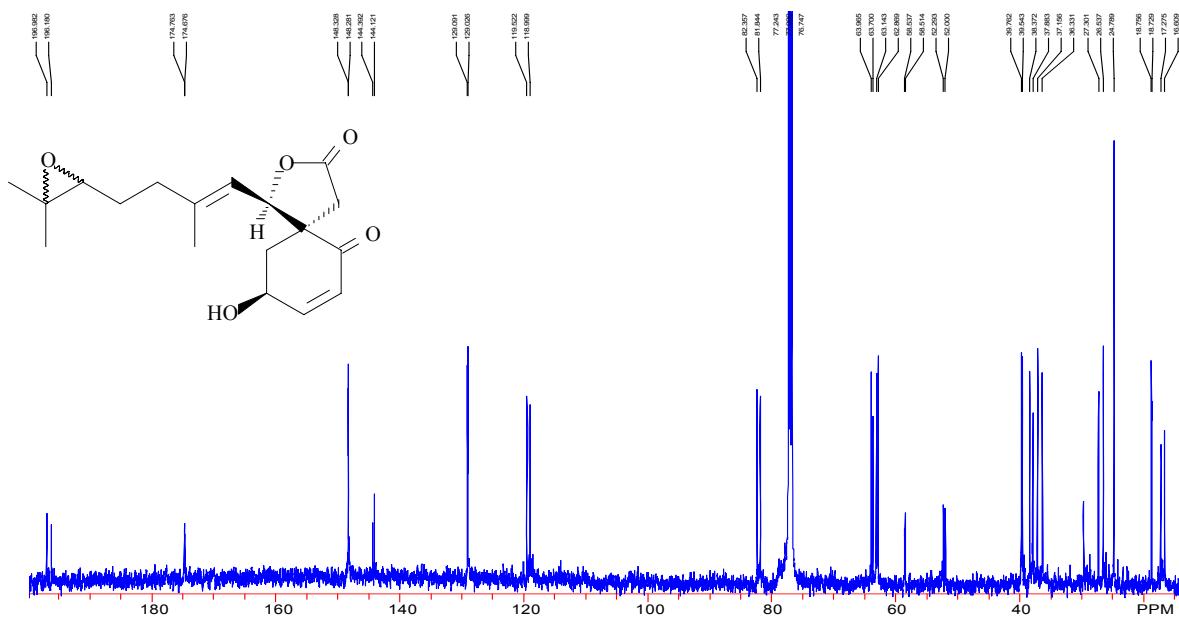
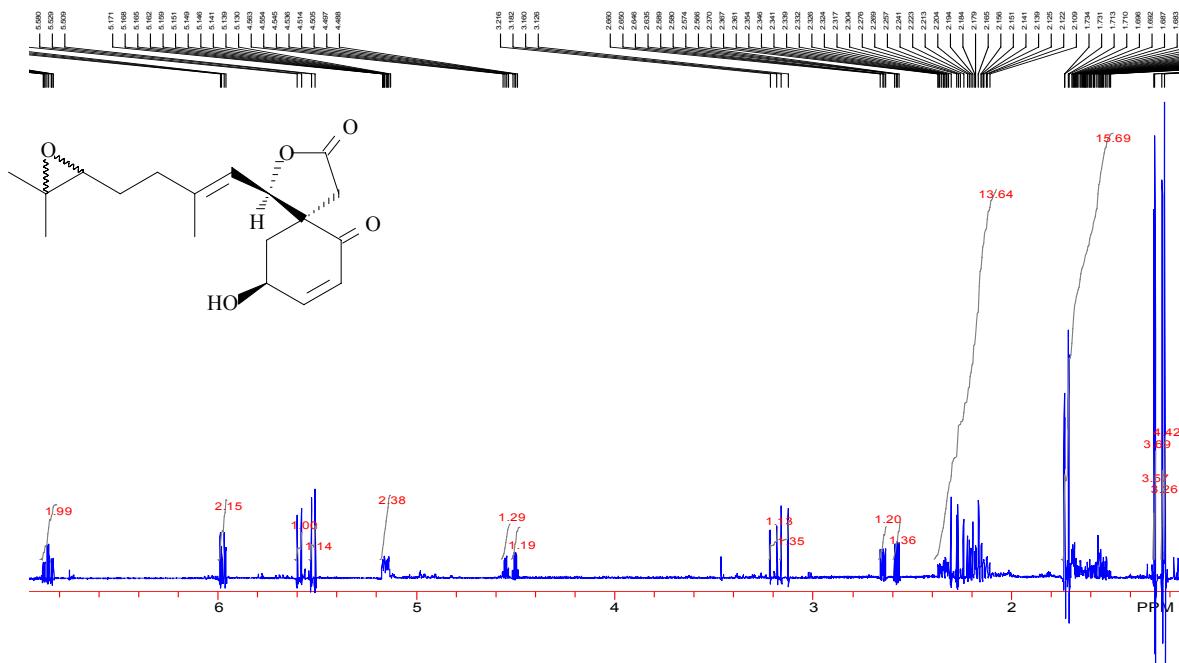


Figure L2. ^{13}C NMR Spectrum of Miliusane X/XI (12/13), CDCl_3

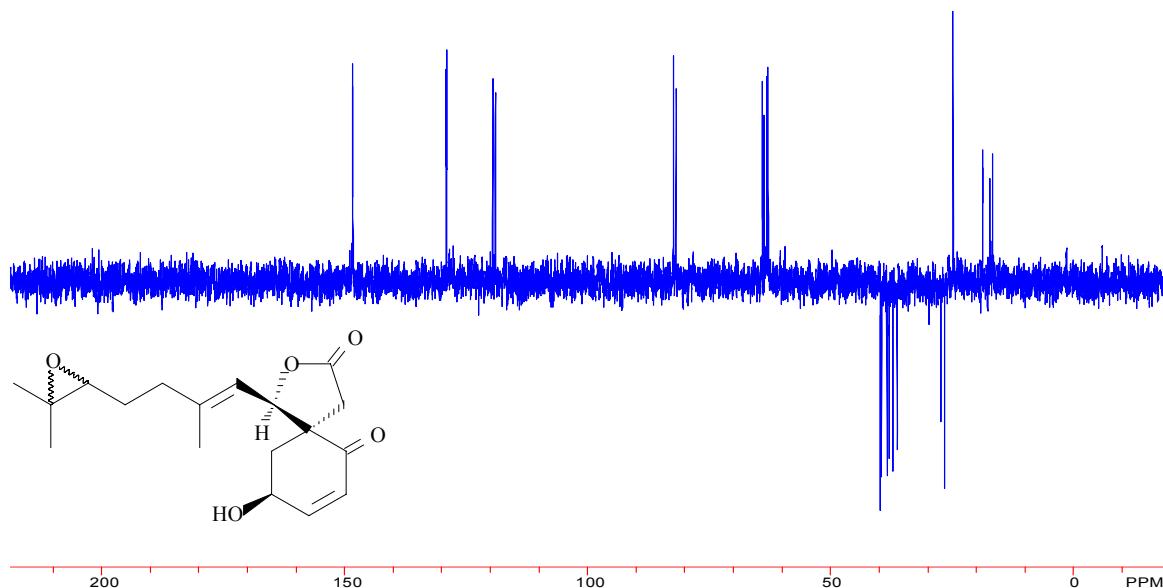


Figure L3. DEPT-135 Spectrum of Miliusane X/XI (**12/13**), CDCl₃

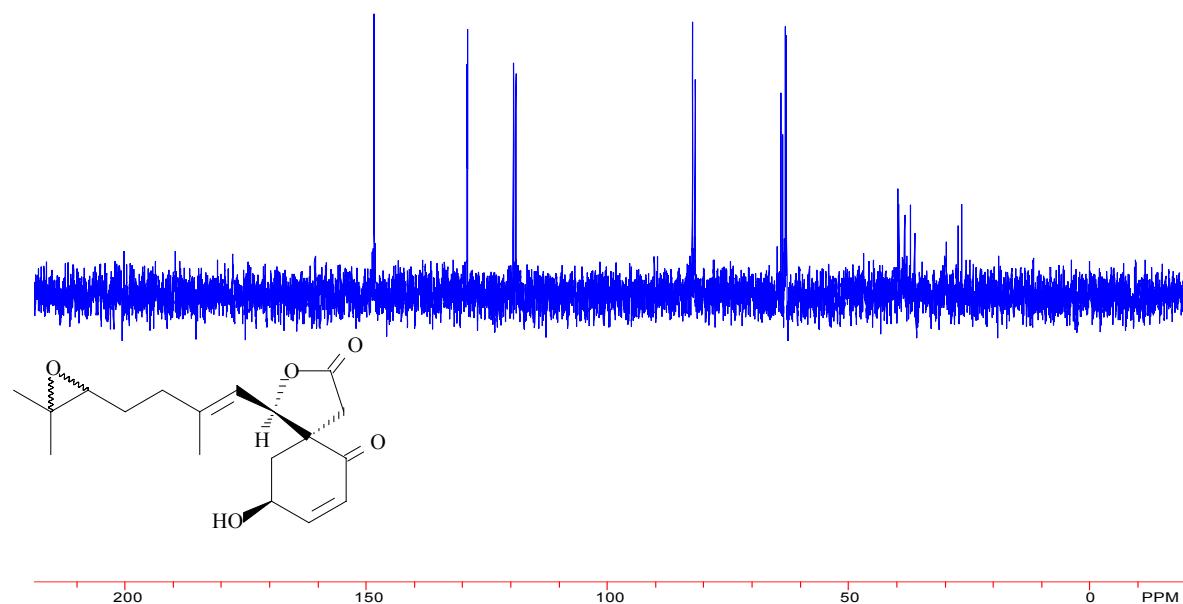


Figure L4. DEPT-90 Spectrum of Miliusane X/XI (**12/13**), CDCl₃

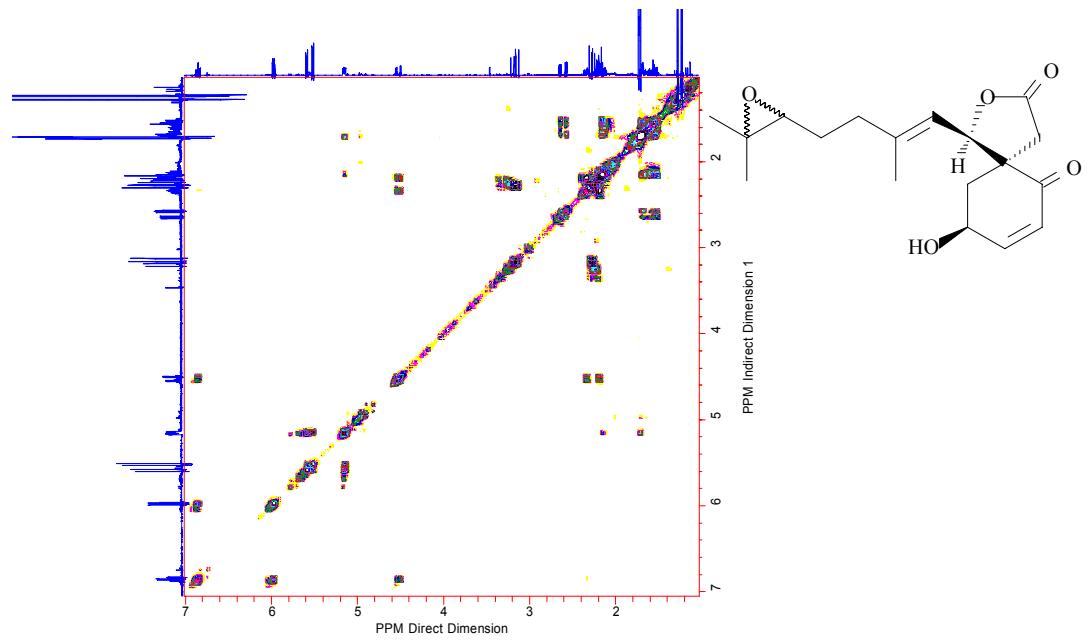


Figure L5. ^1H - ^1H COSY Spectrum of Miliusane X/XI (**12/13**), CDCl_3

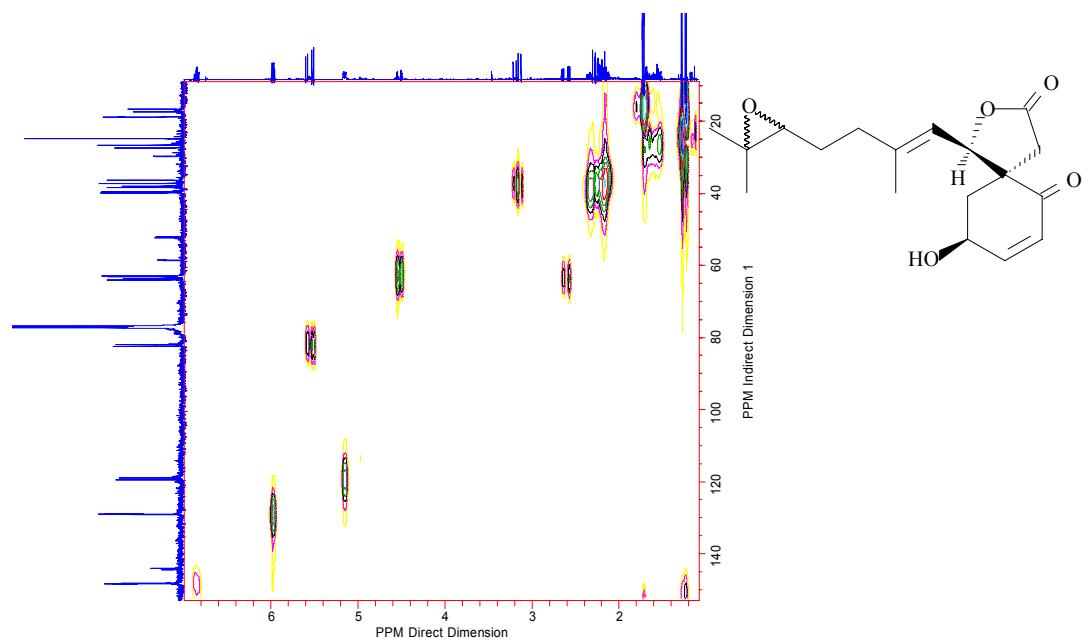


Figure L6. HMQC Spectrum of Miliusane X/XI (**12/13**), CDCl_3

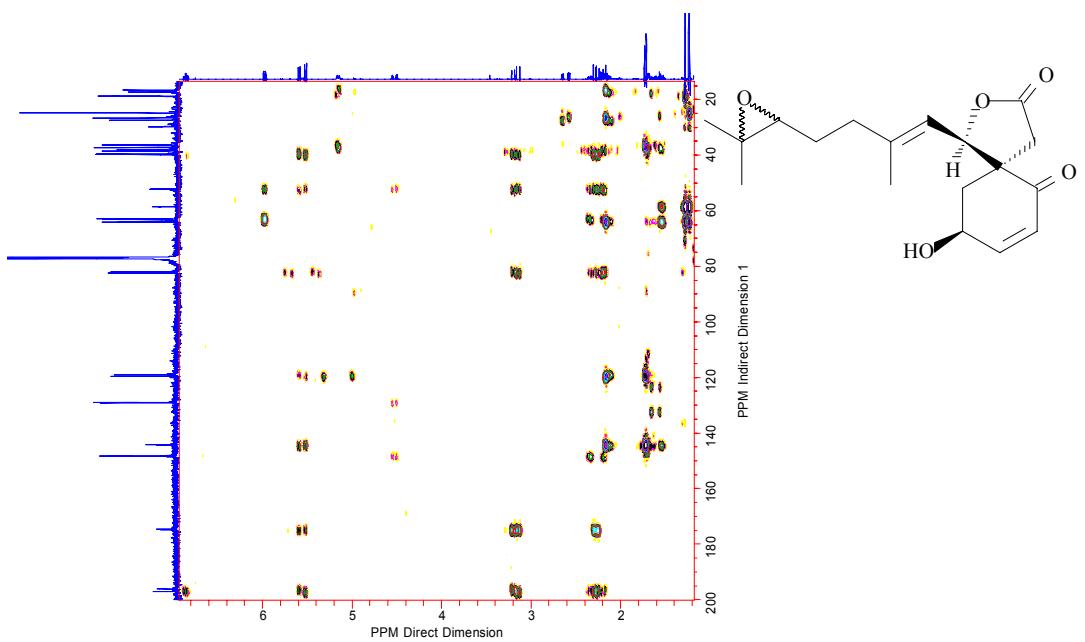


Figure L7. HMBC Spectrum of Miliusane X/XI (**12/13**), CDCl_3

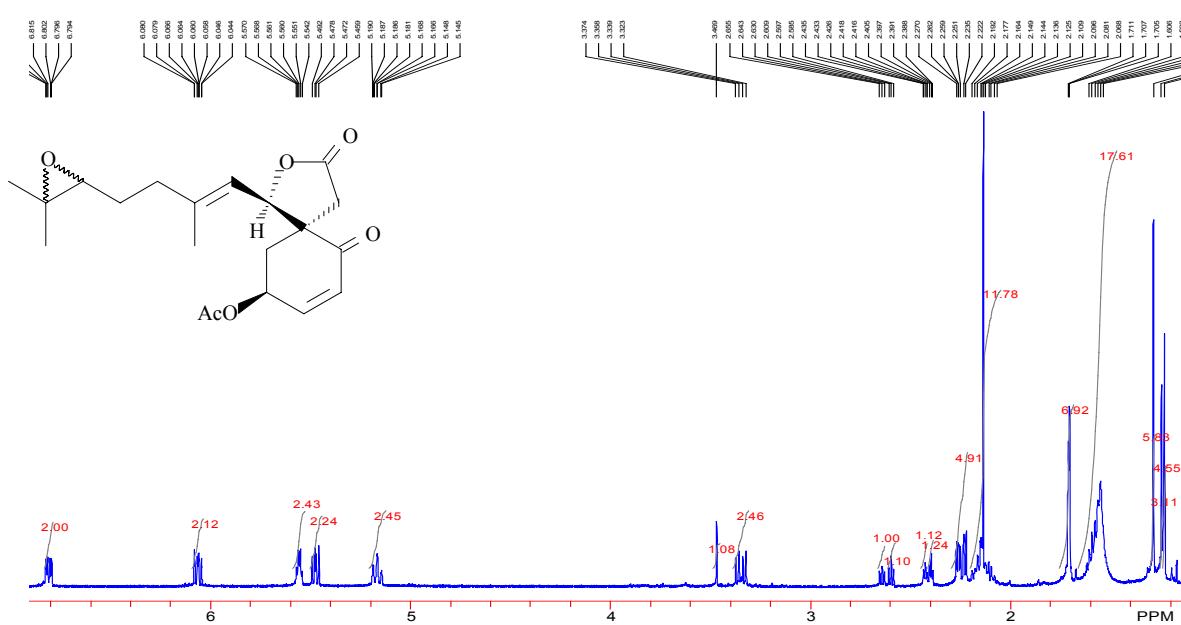


Figure M1. ^1H NMR Spectrum of Miliusane XII/XIII (**14/15**), CDCl_3

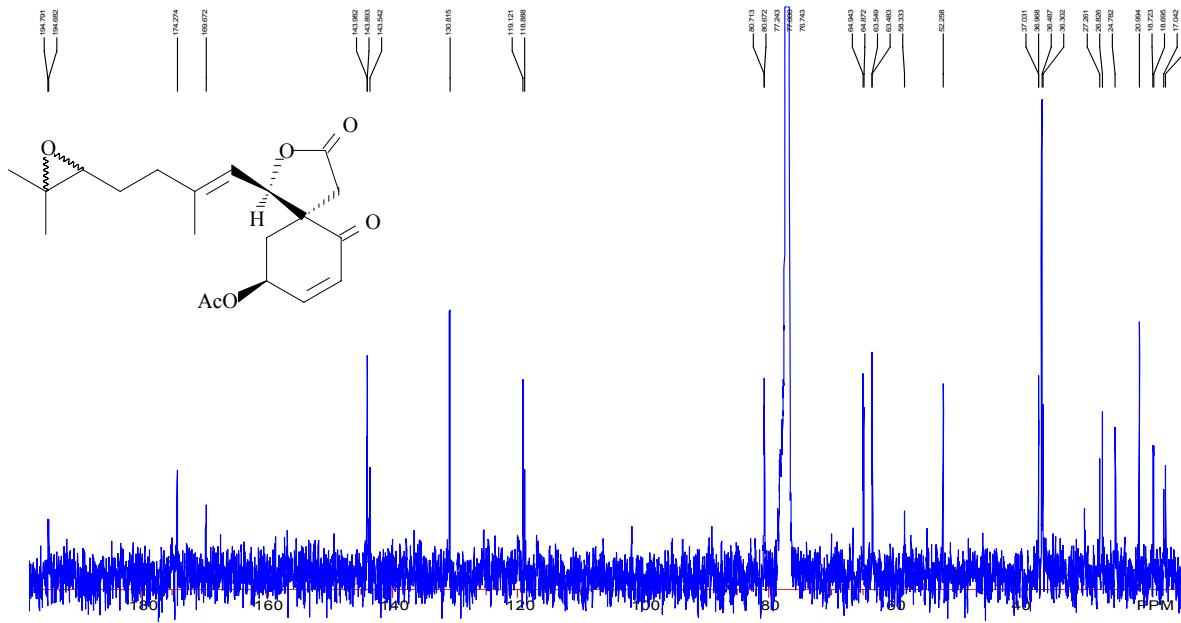


Figure M2. ^{13}C NMR Spectrum of Miliusane XII/XIII (**14/15**), CDCl_3

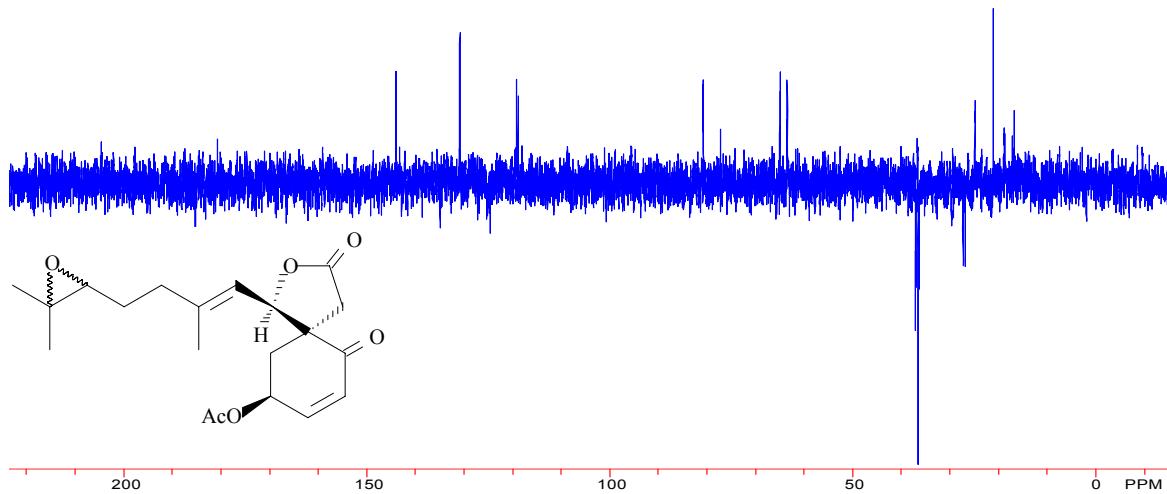


Figure M3. DEPT-135 Spectrum of Miliusane XII/XIII (**14/15**), CDCl_3

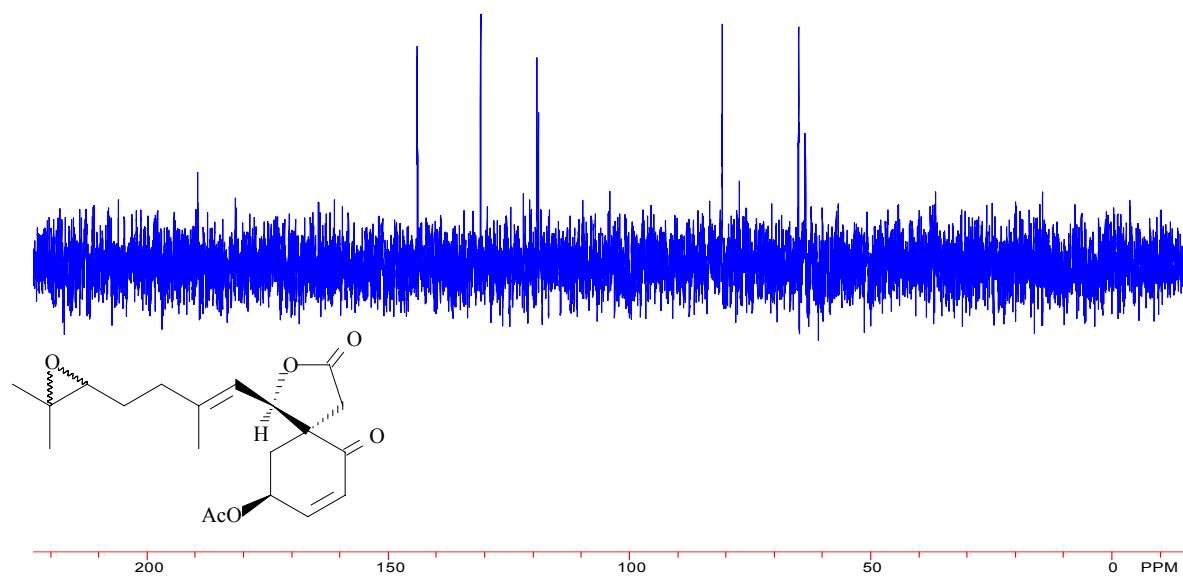


Figure M4. DEPT-90 Spectrum of Miliusane XII/XIII (**14/15**), CDCl_3

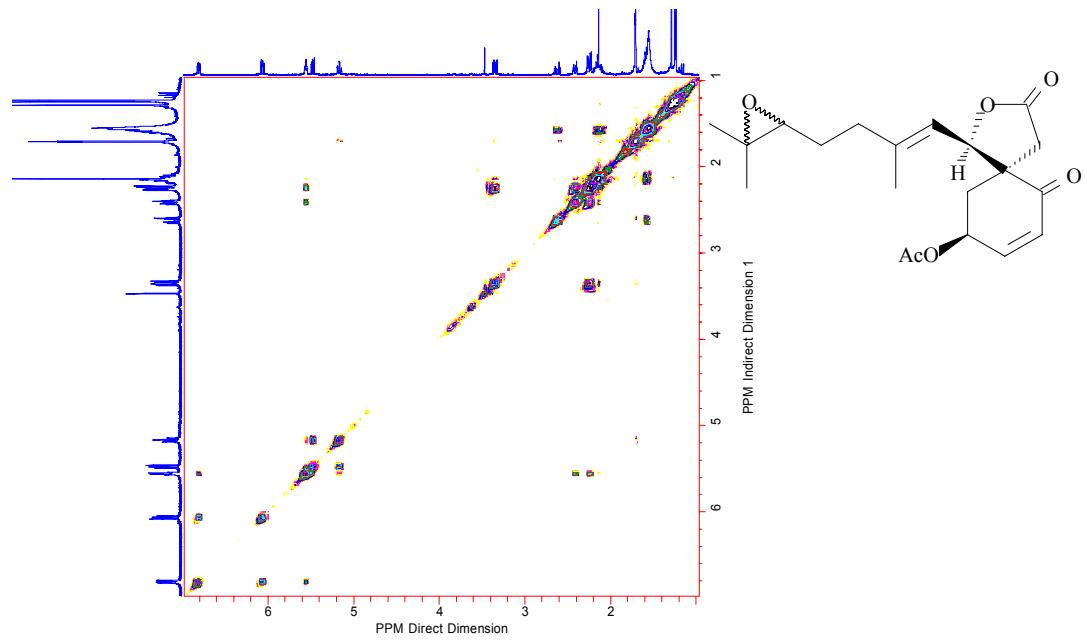


Figure M5. ¹H-¹H COSH Spectrum of Miliusane XII/XIII (**14/15**), CDCl_3

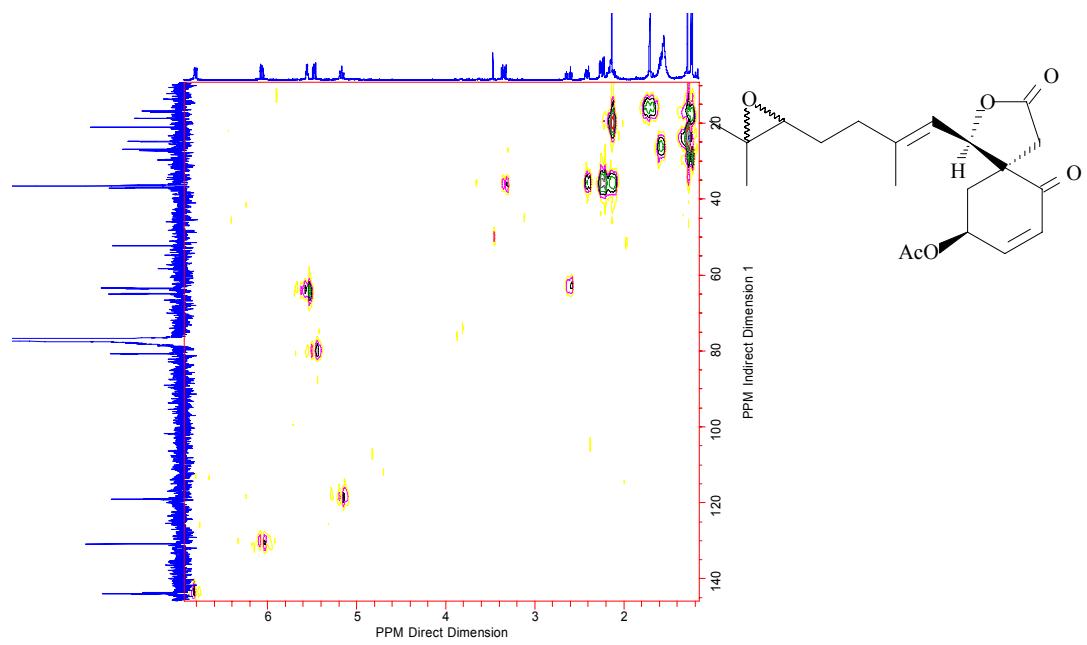


Figure M6. HMQC Spectrum of Miliusane XII/XIII (**14/15**), CDCl_3

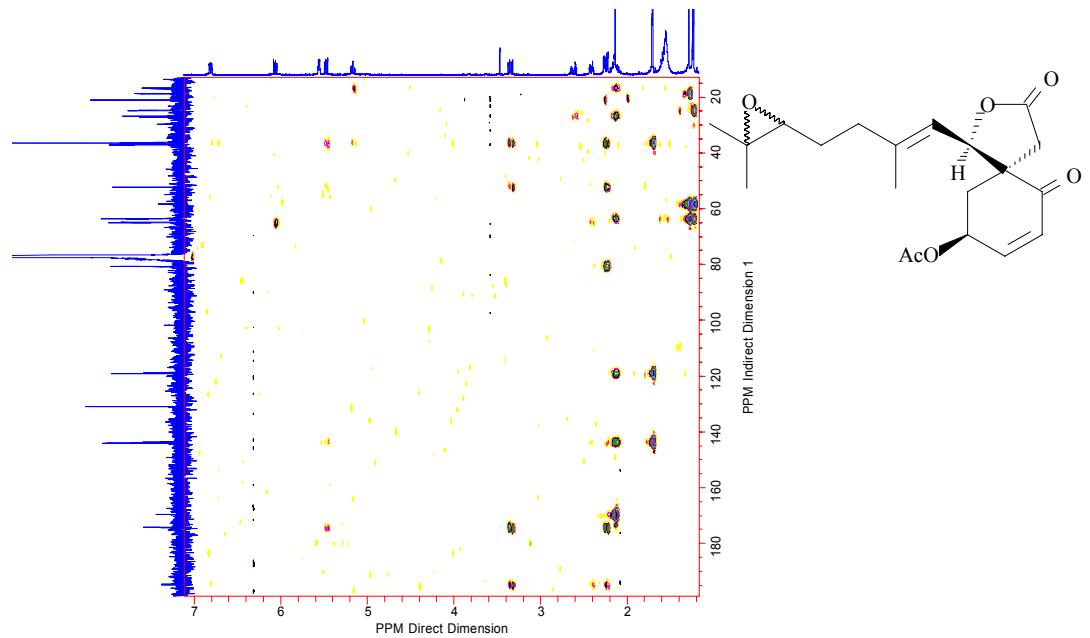


Figure M7. HMBC Spectrum of Miliusane XII/XIII (**14/15**), CDCl_3

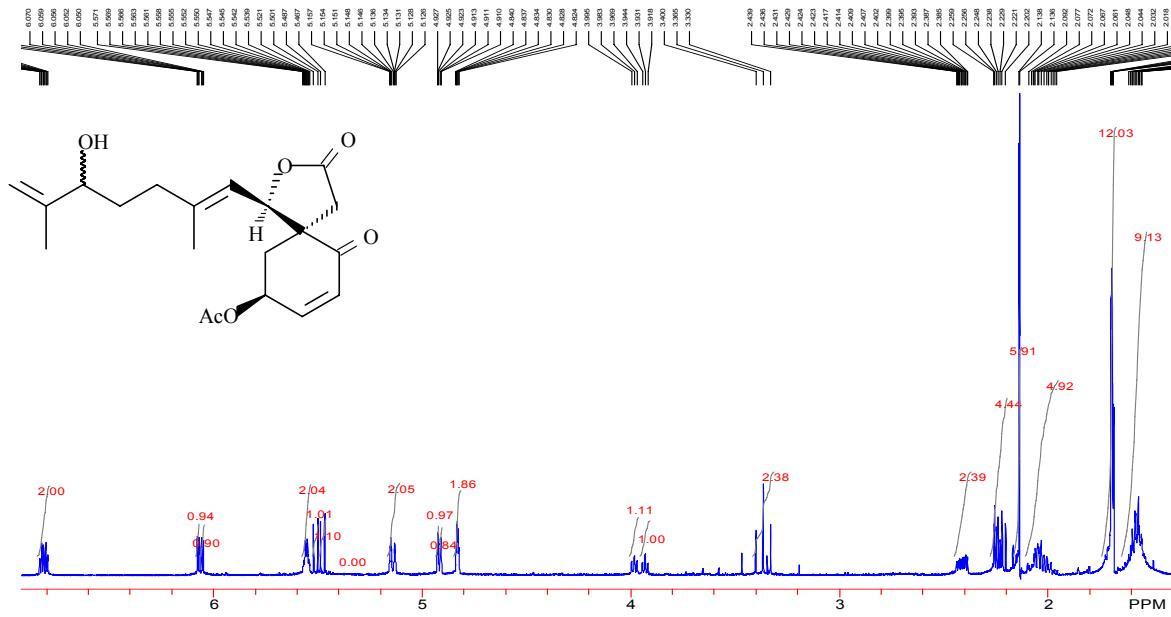


Figure N1. ^1H NMR Spectrum of Miliusane XIV/XV (**16/17**), CDCl_3

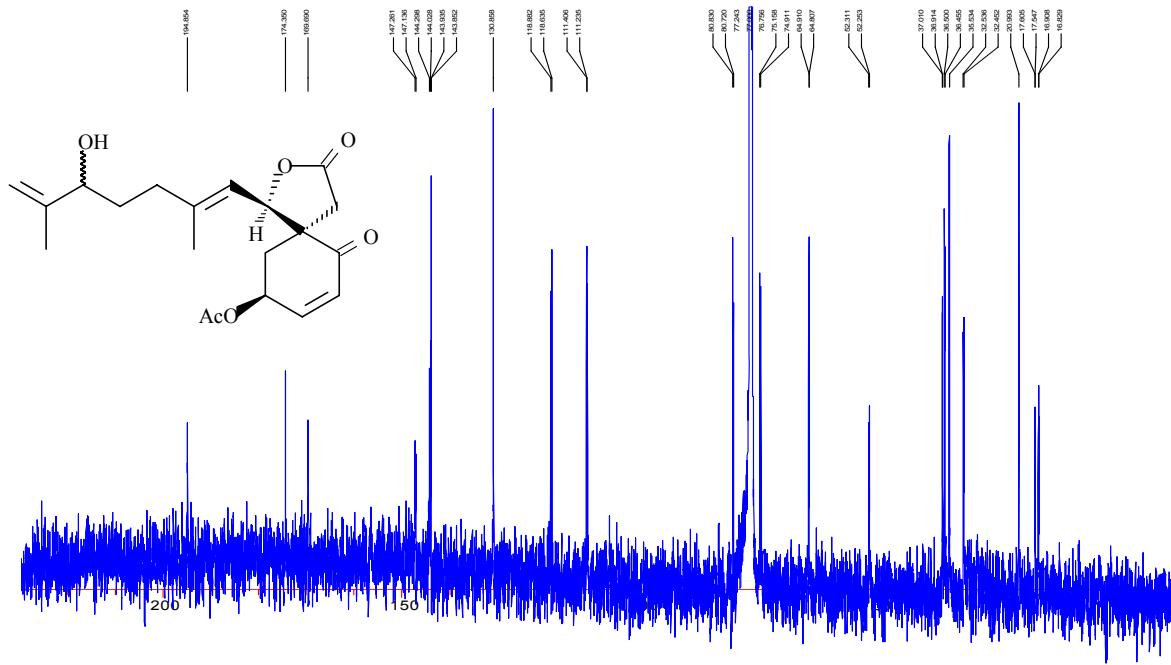


Figure N2. ^{13}C NMR Spectrum of Miliusane XIV/XV (**16/17**), CDCl_3

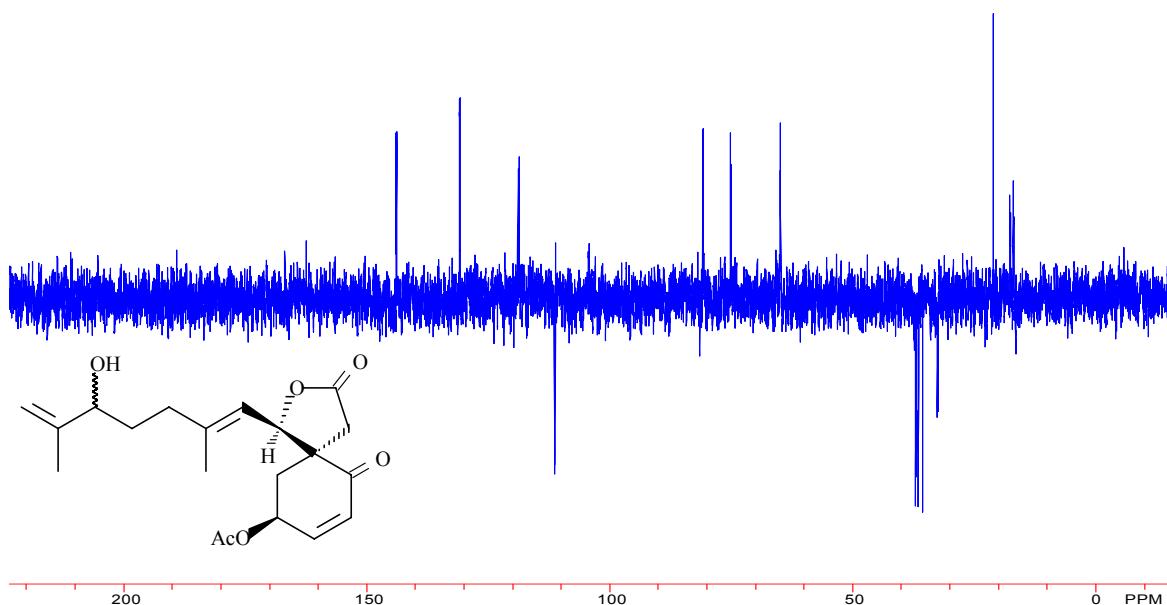


Figure N3. DEPT-135 Spectrum of Miliusane XIV/XV (**16/17**), CDCl_3

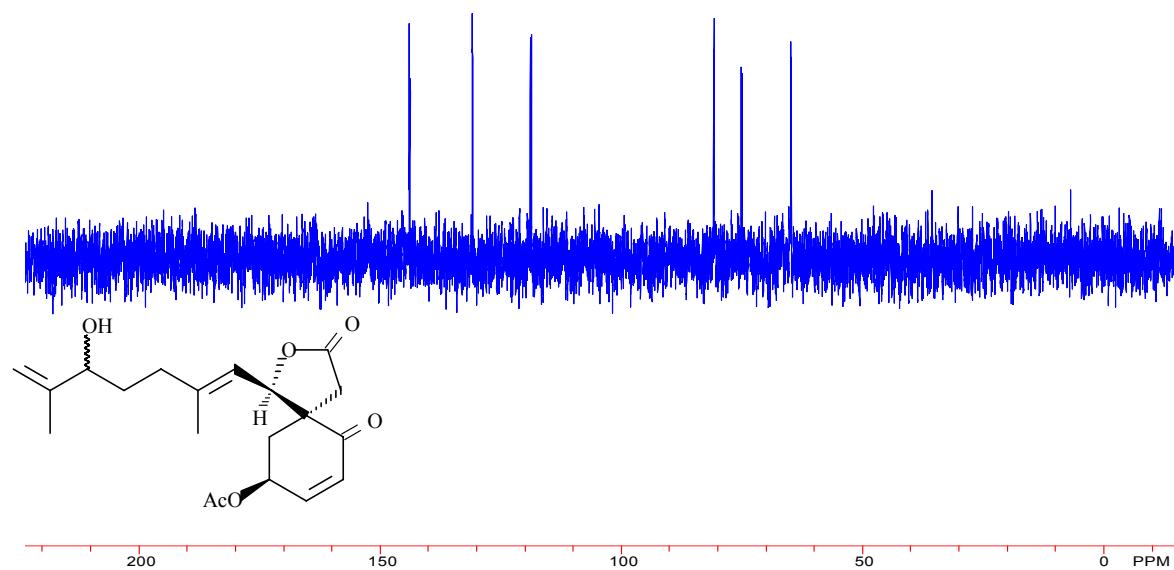


Figure N4. DEPT-90 Spectrum of Miliusane XIV/XV (**16/17**), CDCl_3

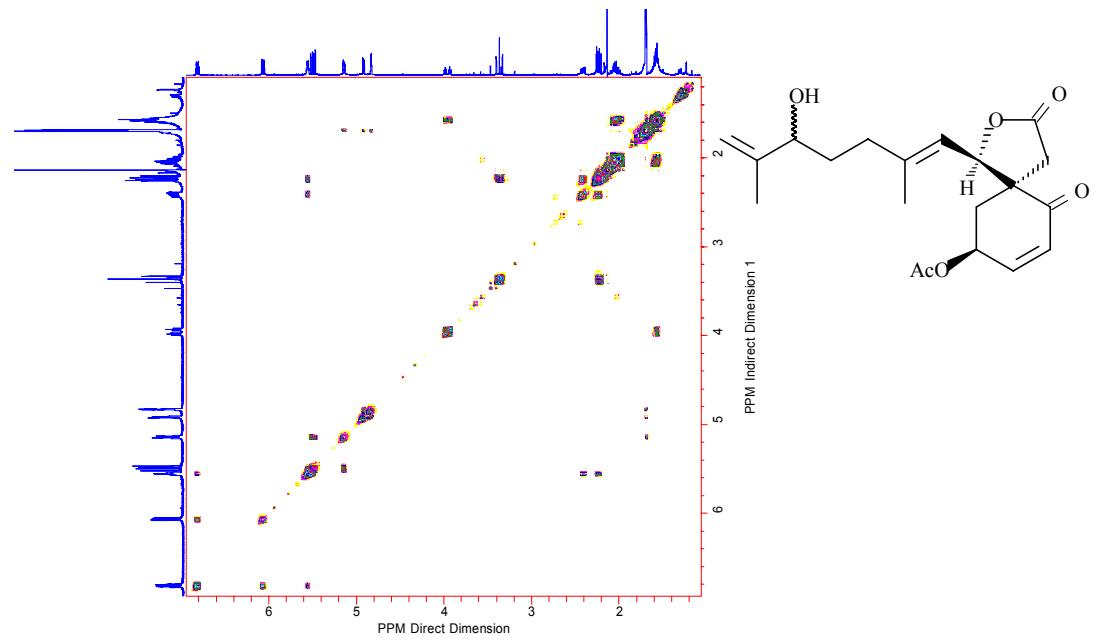


Figure N5. ^1H - ^1H COSY Spectrum of Miliusane XIV/XV (**16/17**), CDCl_3

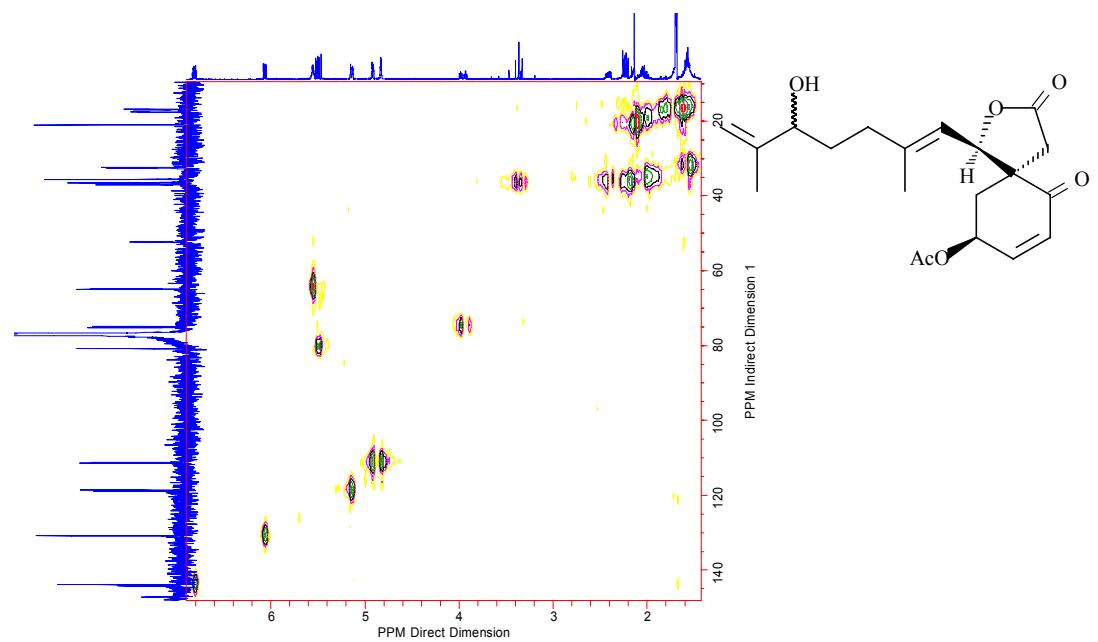


Figure N6. HMQC Spectrum of Miliusane XIV/XV (**16/17**), CDCl_3

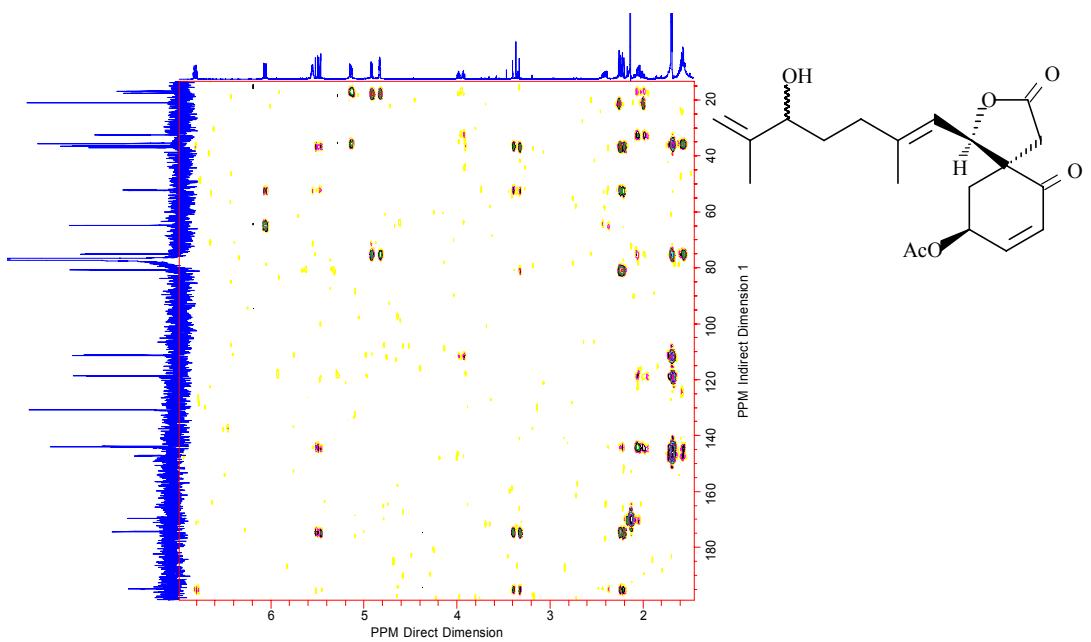


Figure N7. HMBC Spectrum of Miliusane XIV/XV (**16/17**), CDCl_3

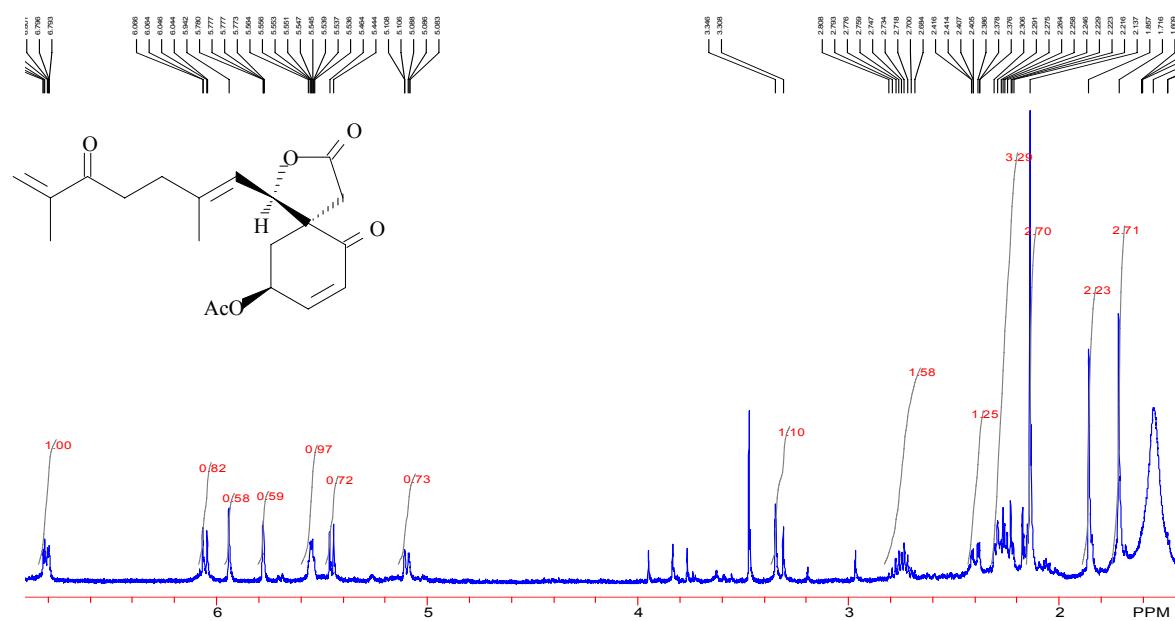


Figure O1. ¹H NMR Spectrum of Miliusane XVI (**18**), CDCl₃

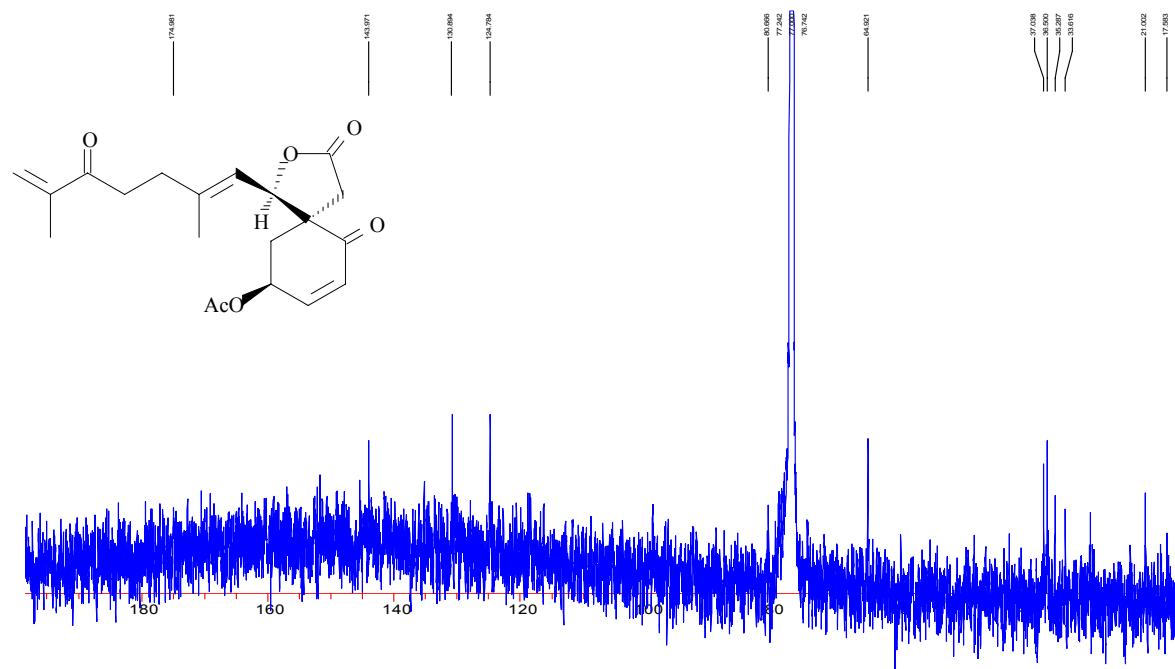


Figure O2. ¹³C NMR Spectrum of Miliusane XVI (**18**), CDCl₃

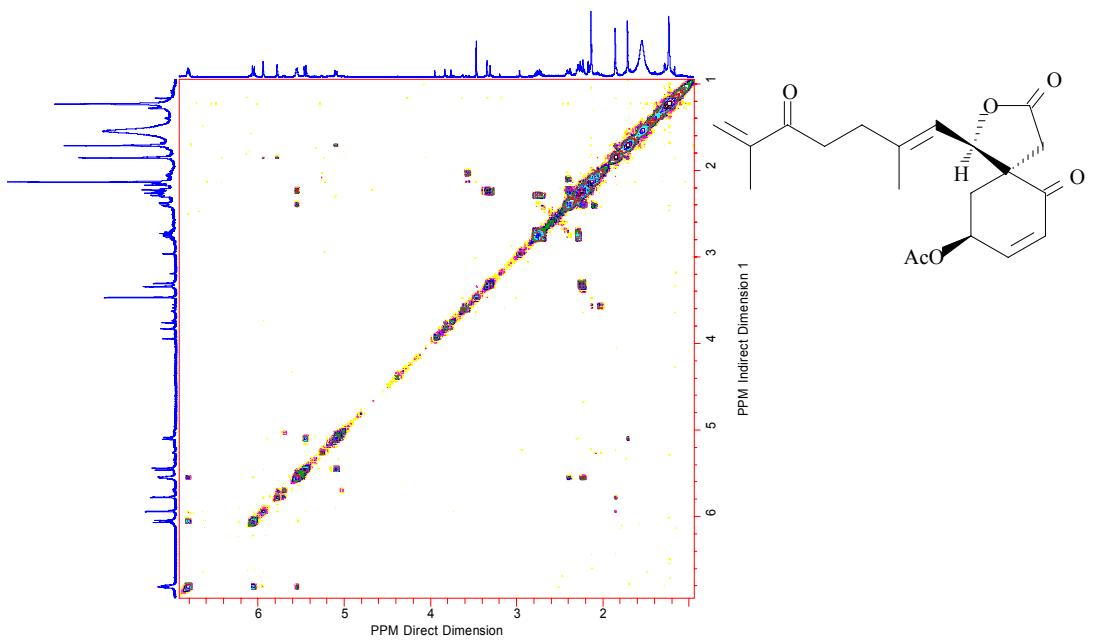


Figure O3. ^1H - ^1H COSY Spectrum of Miliusane XVI (**18**), CDCl_3

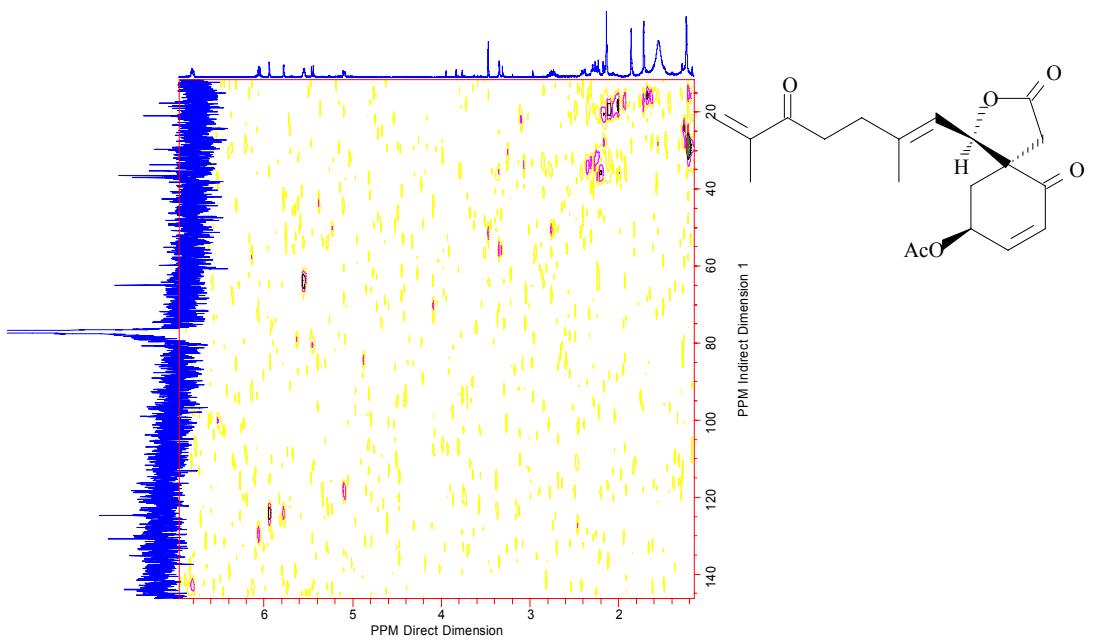


Figure O4. HMQC Spectrum of Miliusane XVI (**18**), CDCl_3

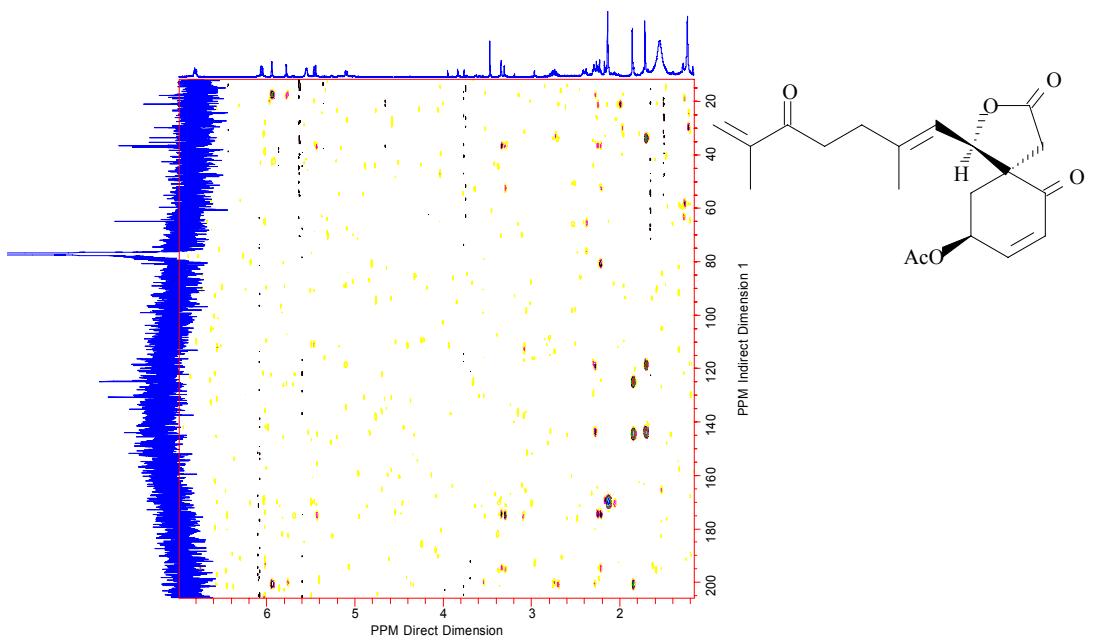


Figure O5. HMBC Spectrum of Miliusane XVI (**18**), CDCl_3

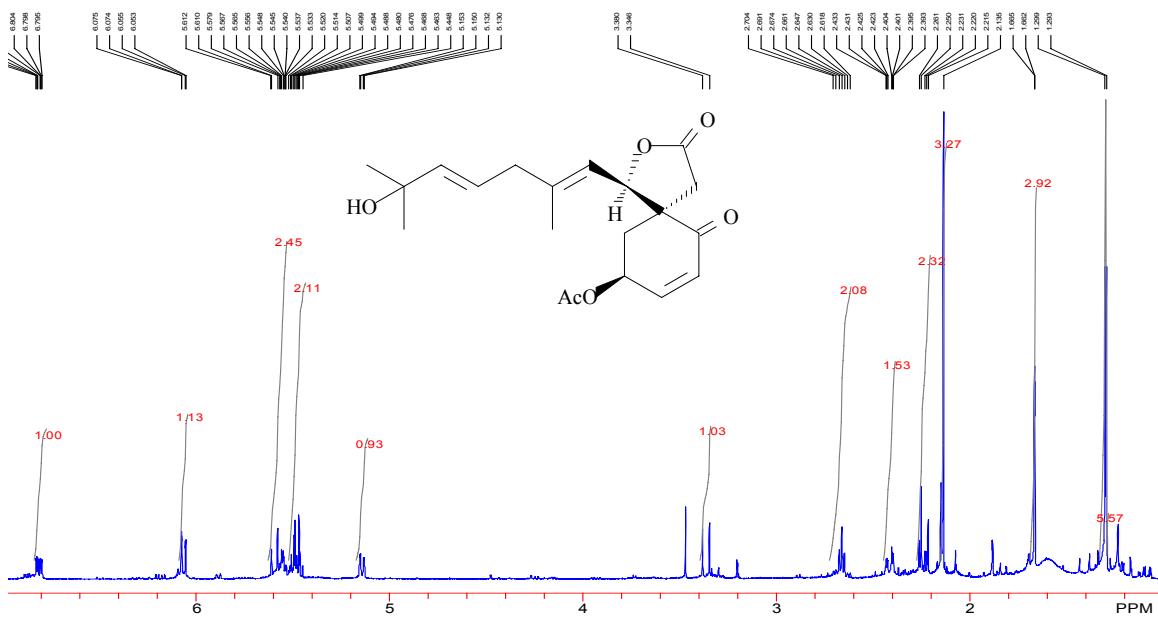


Figure P1. ¹H NMR Spectrum of Miliusane XVII (**19**), CDCl₃

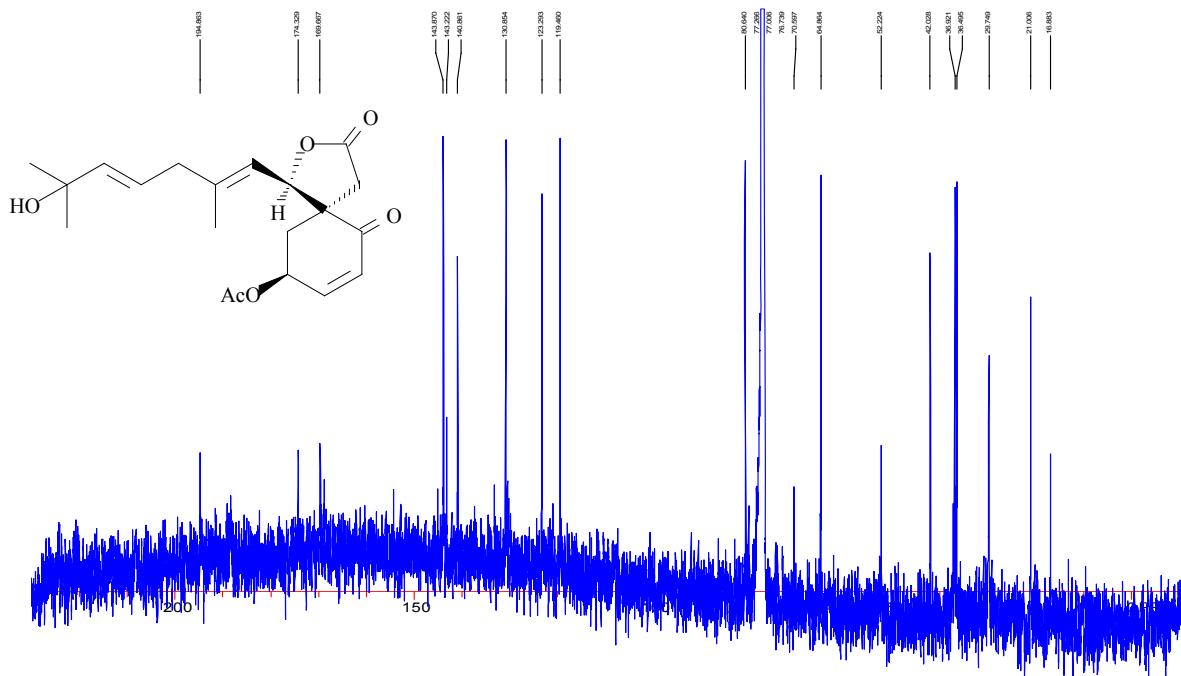


Figure P2. ¹³C NMR Spectrum of Miliusane XVII (**19**), CDCl₃

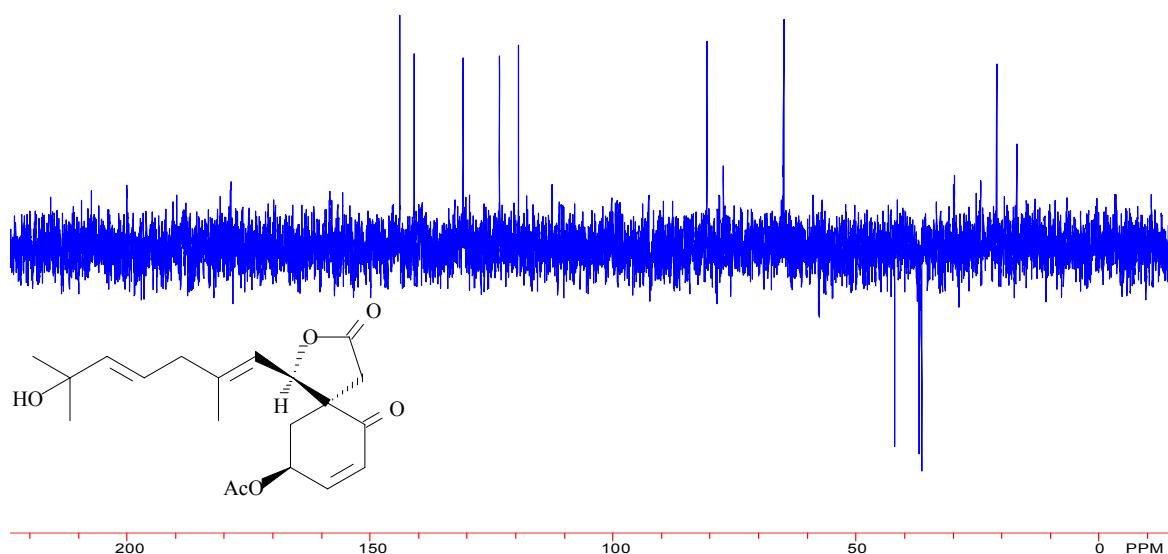


Figure P3. DEPT-135 Spectrum of Miliusane XVII (**19**), CDCl_3

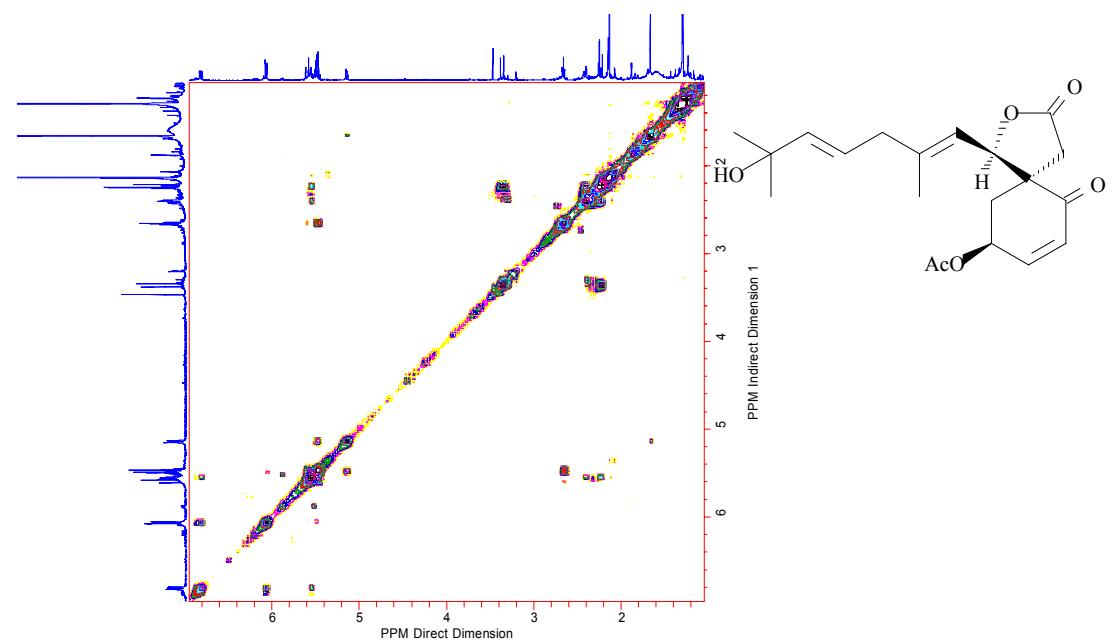


Figure P4. ^1H - ^1H COSY Spectrum of Miliusane XVII (**19**), CDCl_3

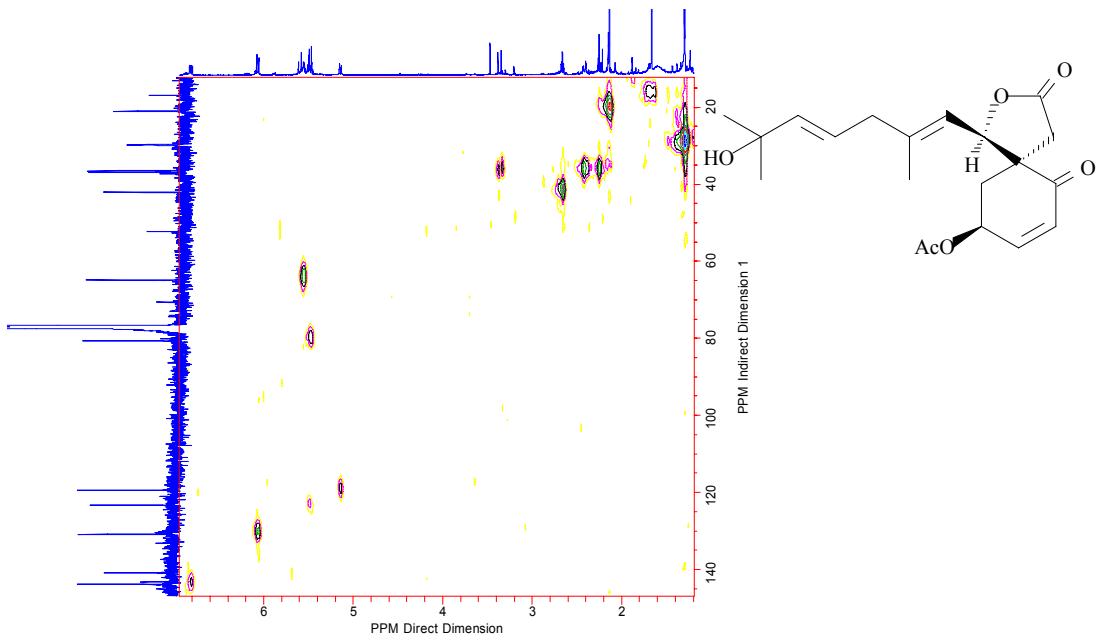


Figure P5. HMQC Spectrum of Miliusane XVII (**19**), CDCl_3

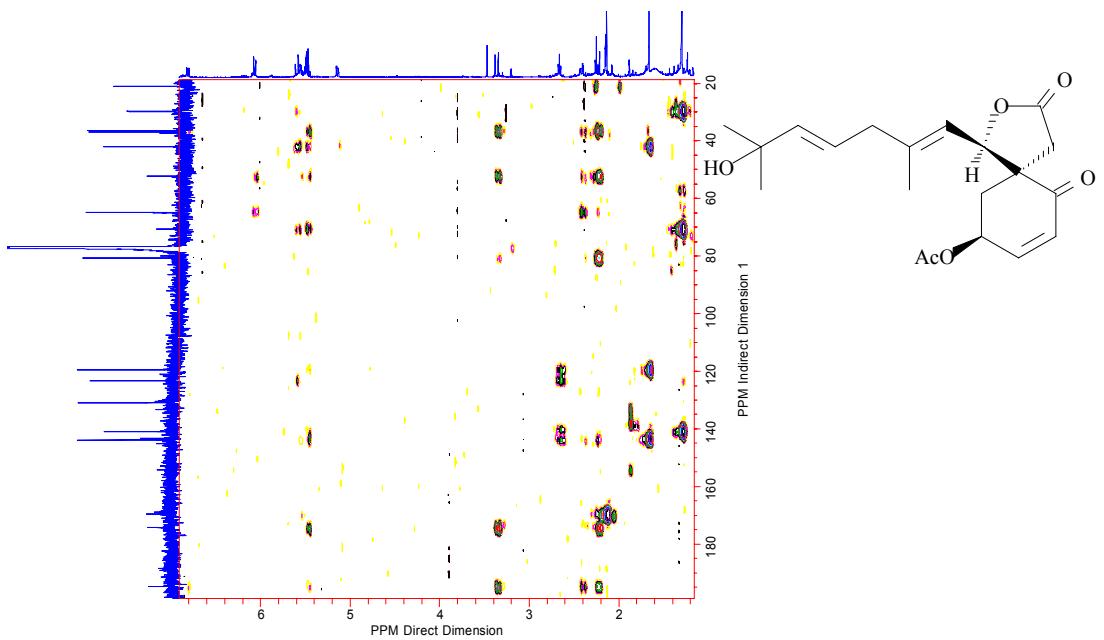


Figure P6. HMBC Spectrum of Miliusane XVII (**19**), CDCl_3

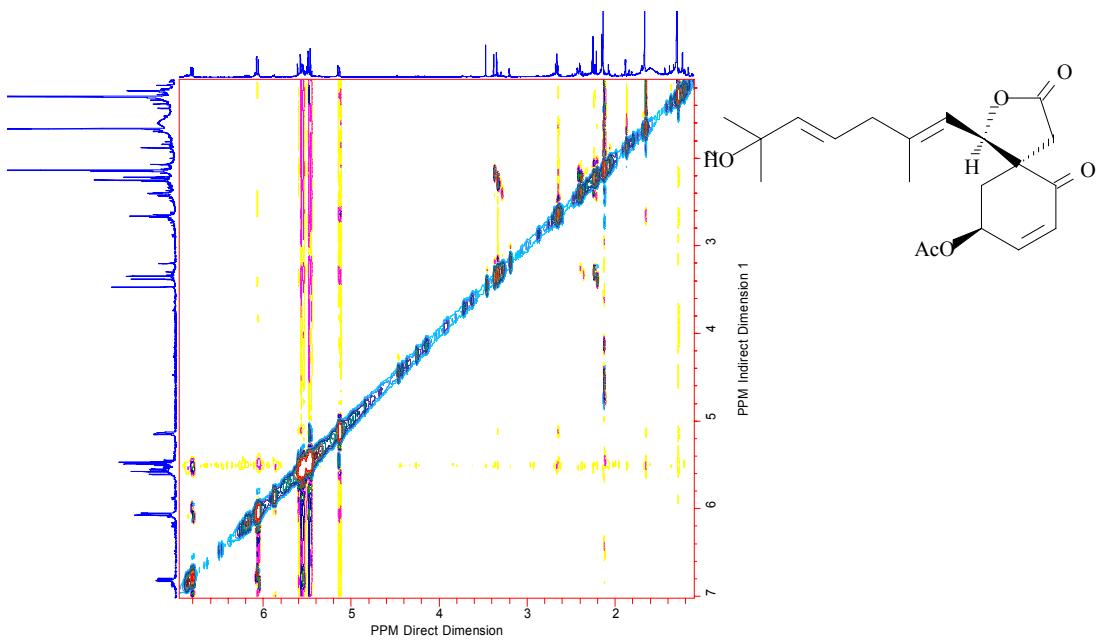


Figure P7. ROESY Spectrum of Miliusane XVII (**19**), CDCl_3

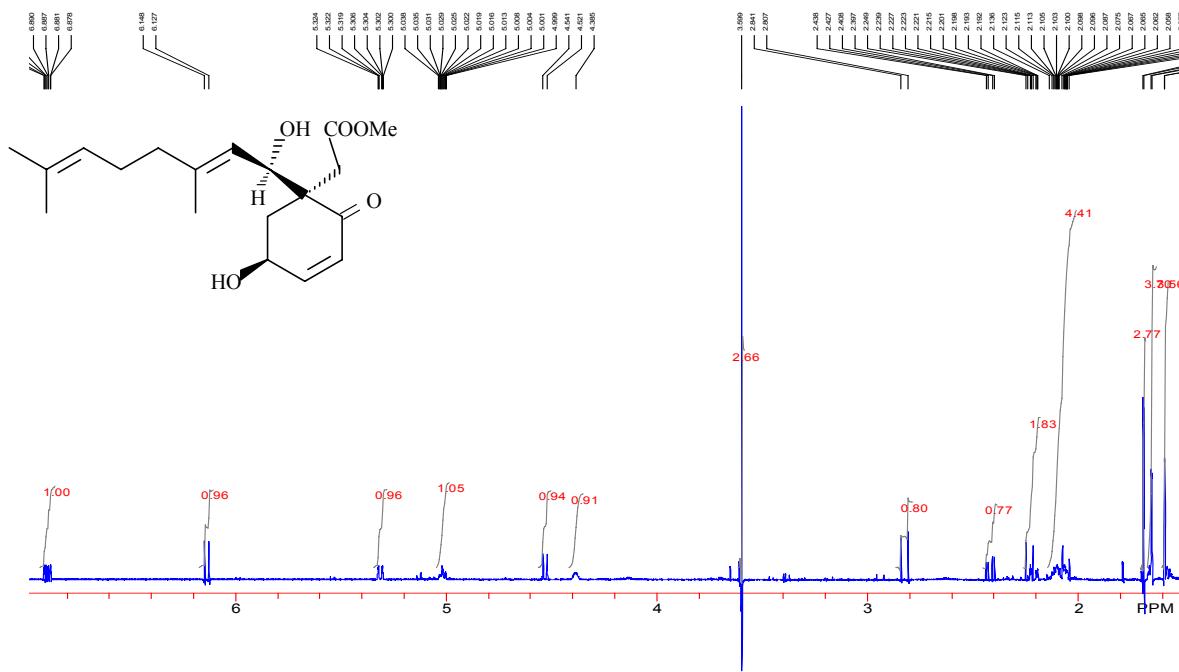


Figure Q1. ^1H NMR Spectrum of Miliusane XVIII (**20**), CDCl_3

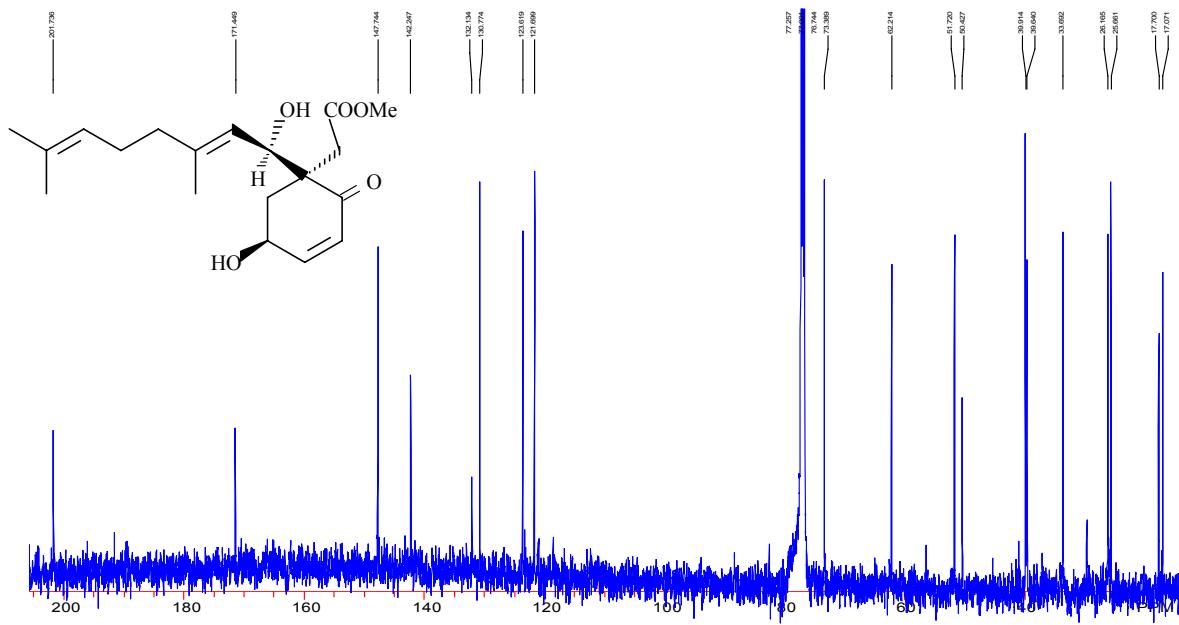


Figure Q2. ^{13}C NMR Spectrum of Miliusane XVIII (**20**), CDCl_3

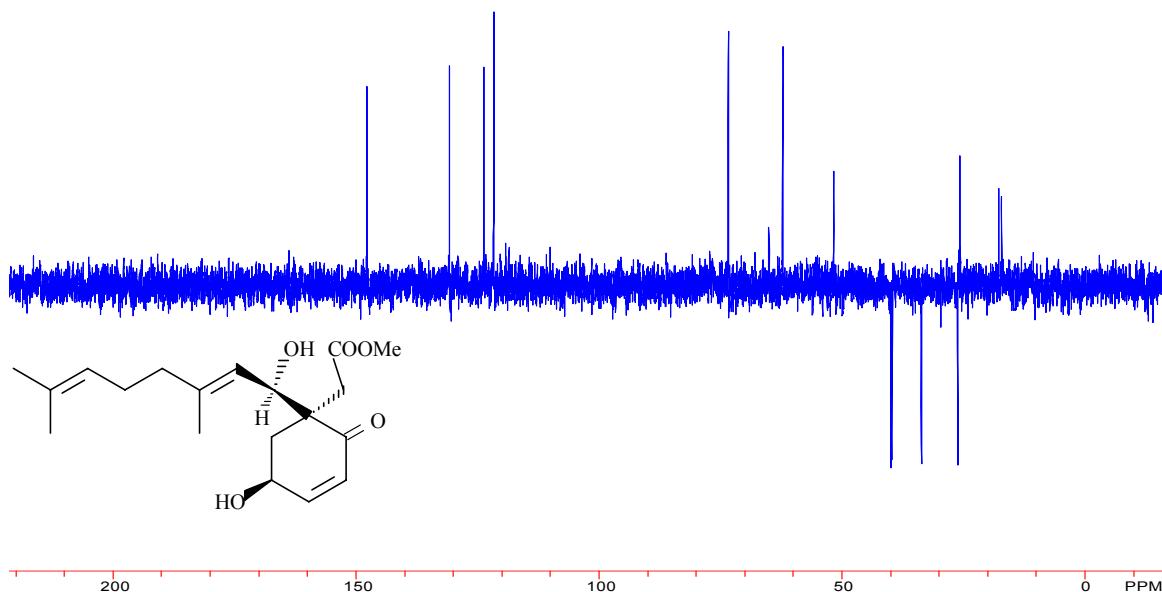


Figure Q3. DEPT-135 Spectrum of Miliusane XVIII (**20**), CDCl_3

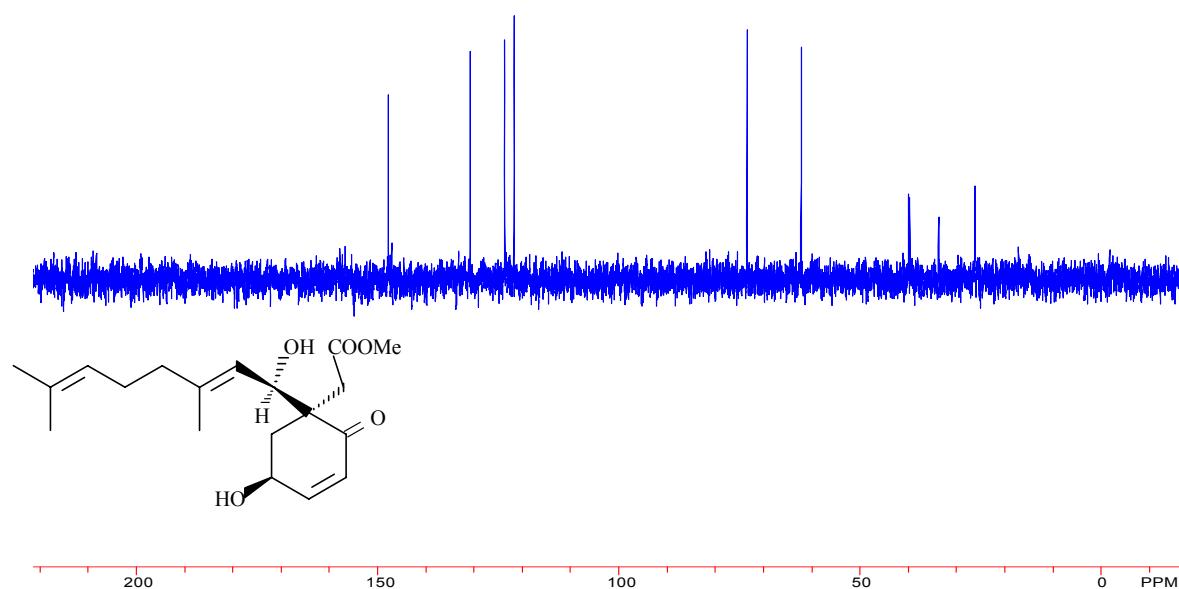


Figure Q4. DEPT-90 Spectrum of Miliusane XVIII (**20**), CDCl_3

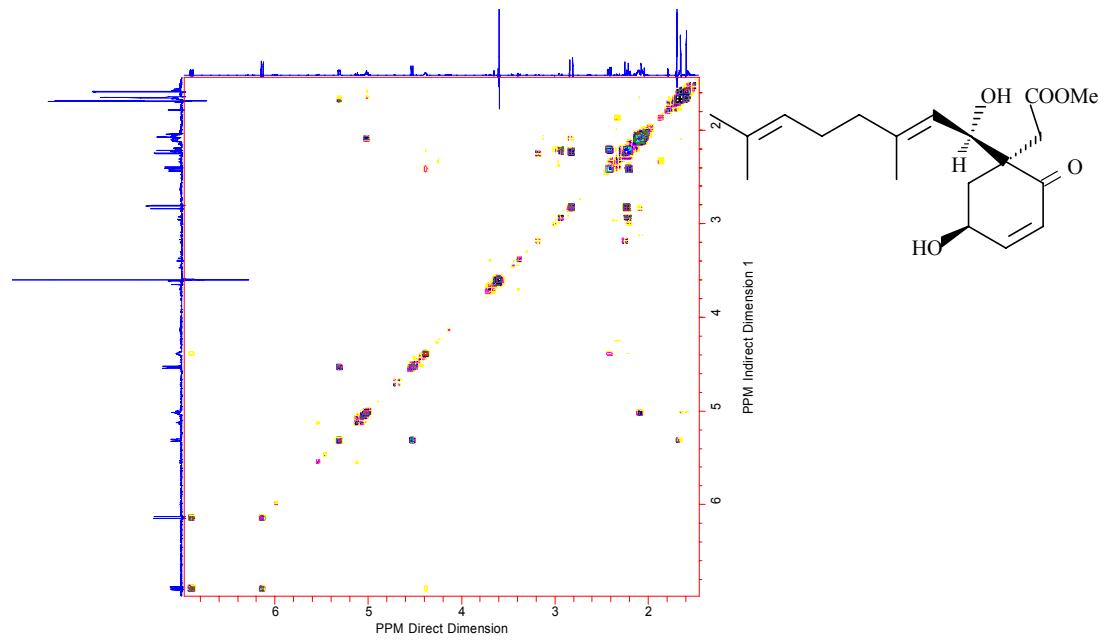


Figure Q5. ^1H - ^1H COSY Spectrum of Miliusane XVIII (**20**), CDCl_3

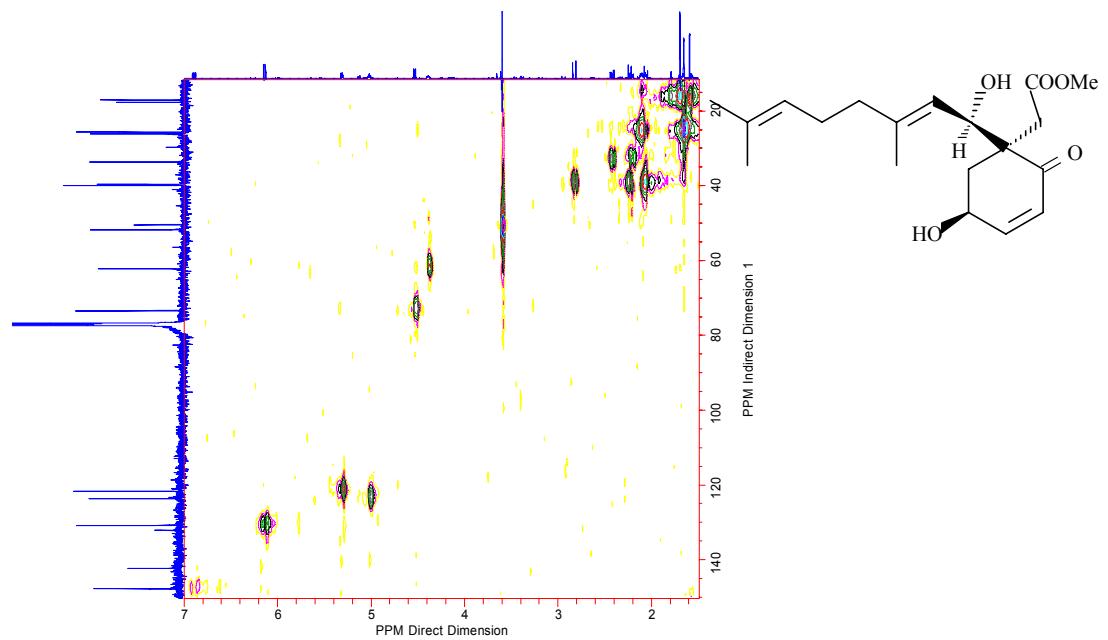


Figure Q6. HMQC Spectrum of Miliusane XVIII (**20**), CDCl_3

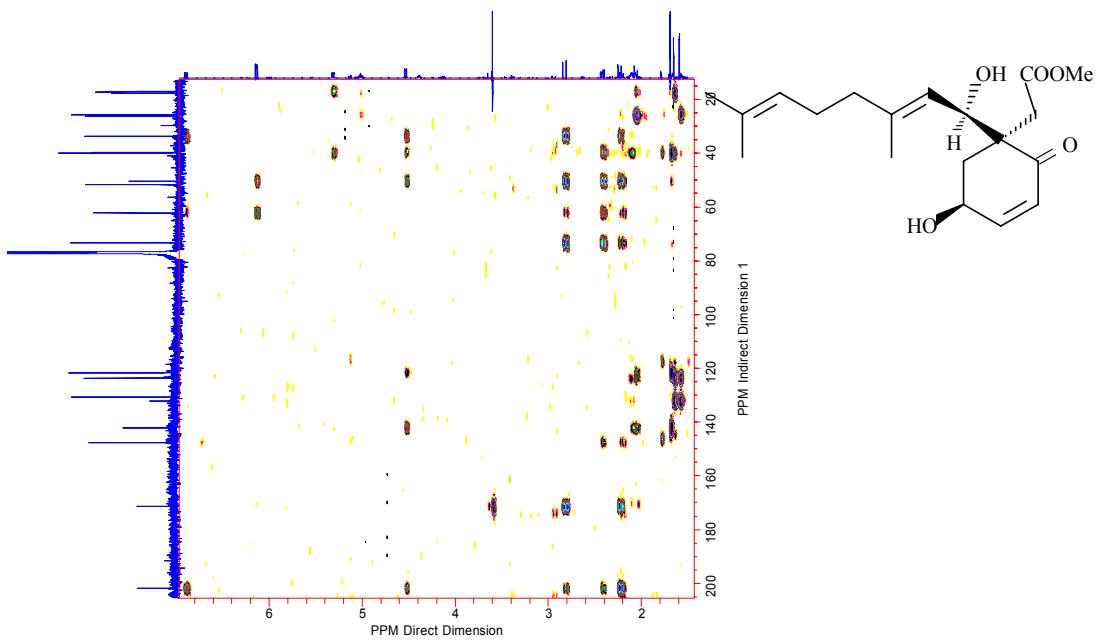


Figure Q7. HMBC Spectrum of Miliusane XVIII (**20**), CDCl_3

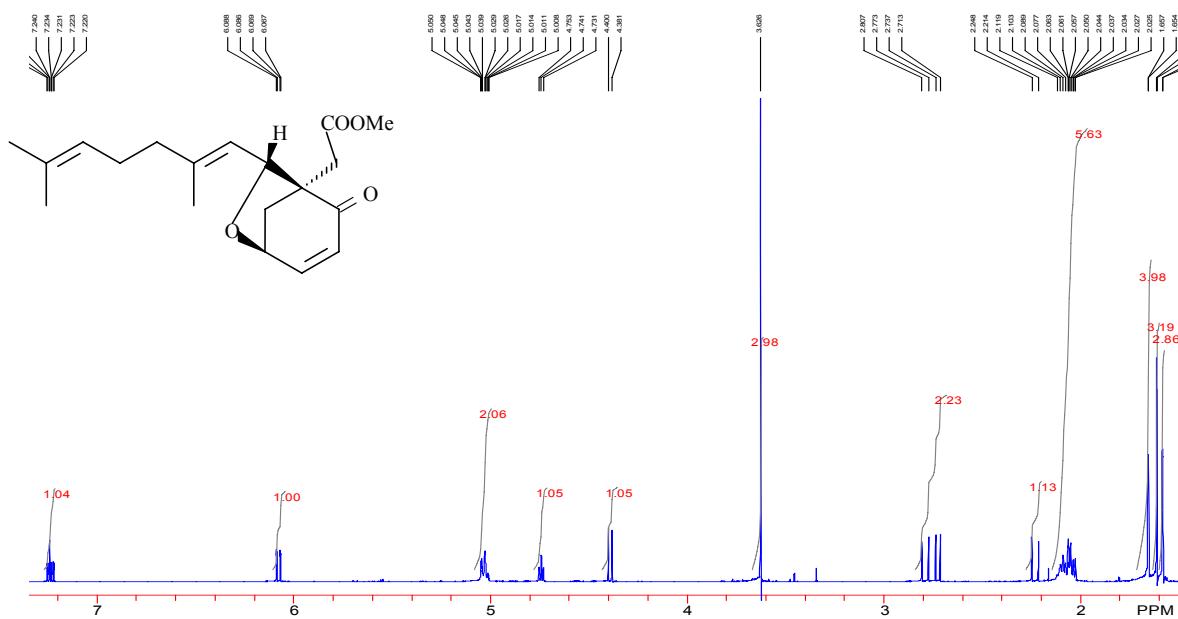


Figure R1. ^1H NMR Spectrum of Miliusane XIX (**21**), CDCl_3

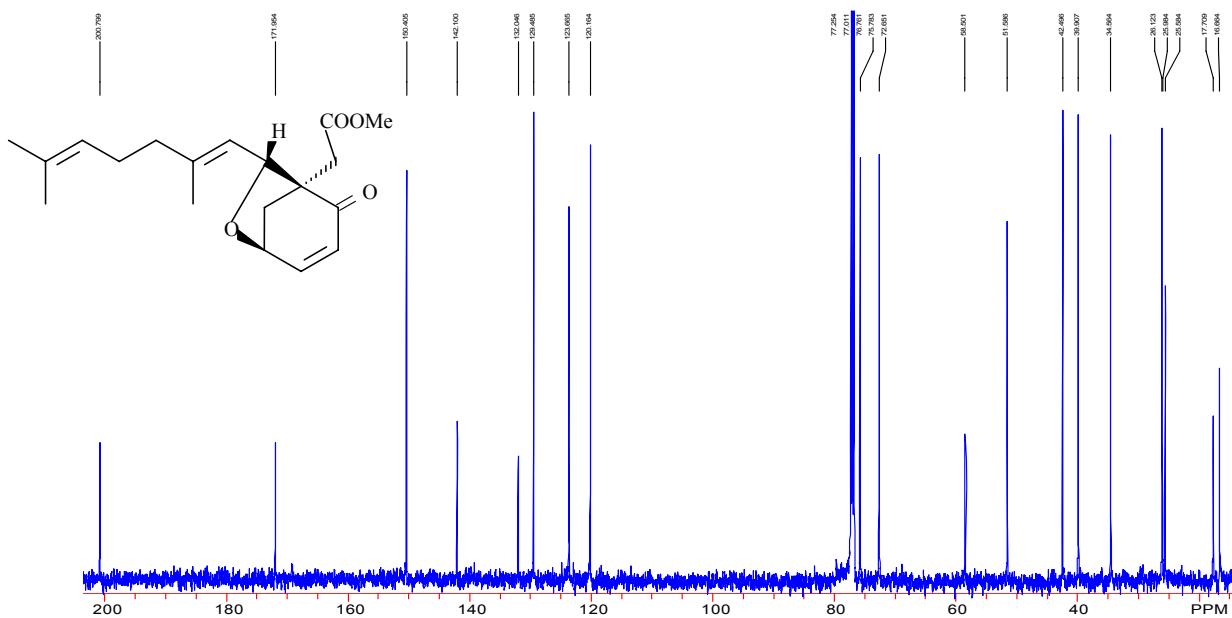


Figure R2. ^{13}C NMR Spectrum of Miliusane XIX (**21**), CDCl_3

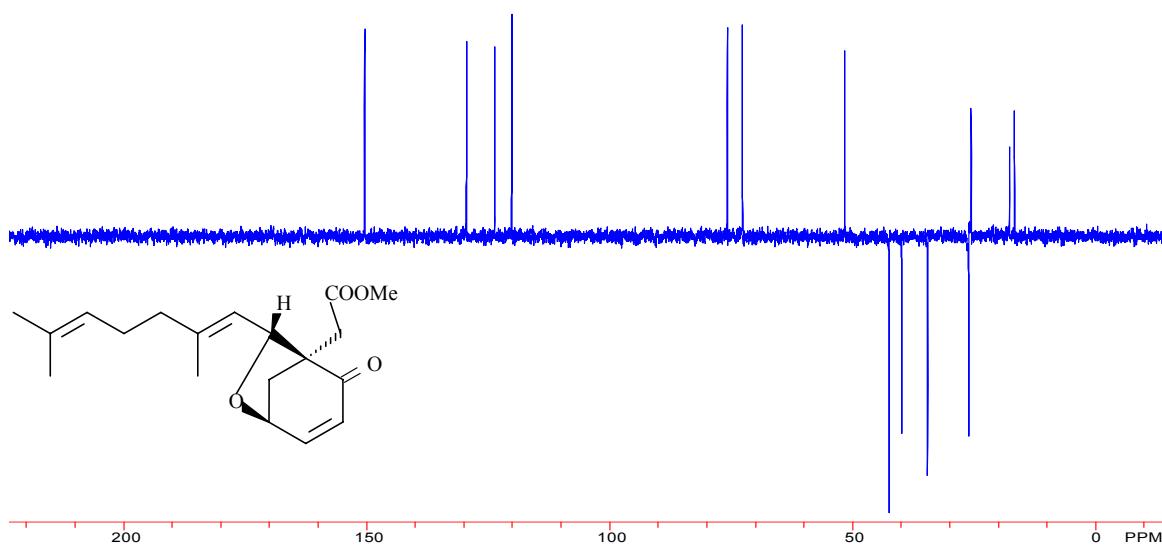


Figure R3. DEPT-135 Spectrum of Miliusane XIX (**21**), CDCl₃

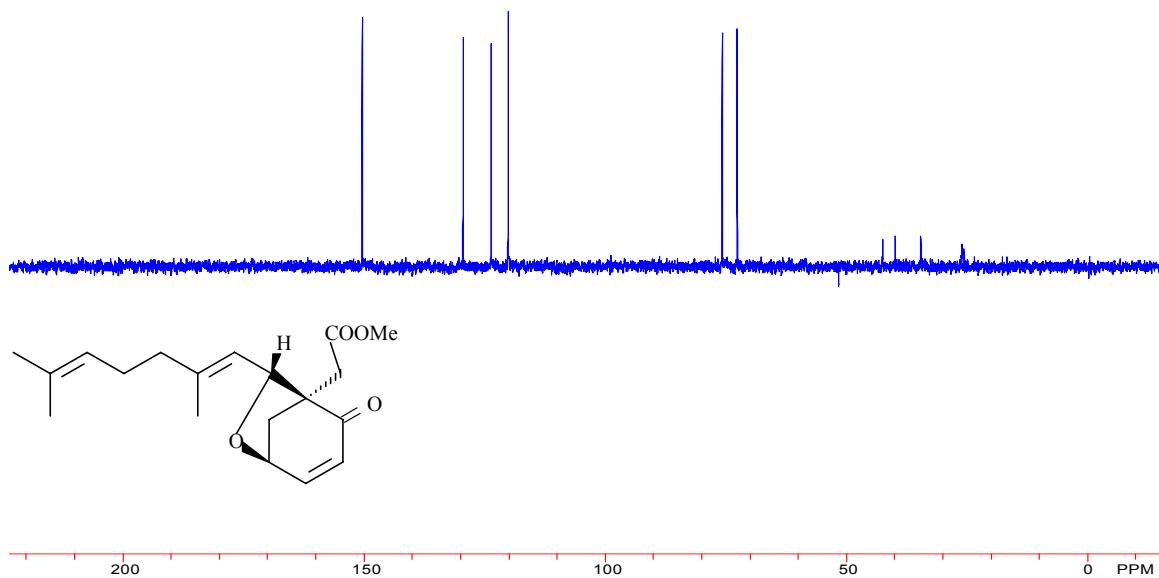


Figure R4. DEPT-90 Spectrum of Miliusane XIX (**21**), CDCl₃

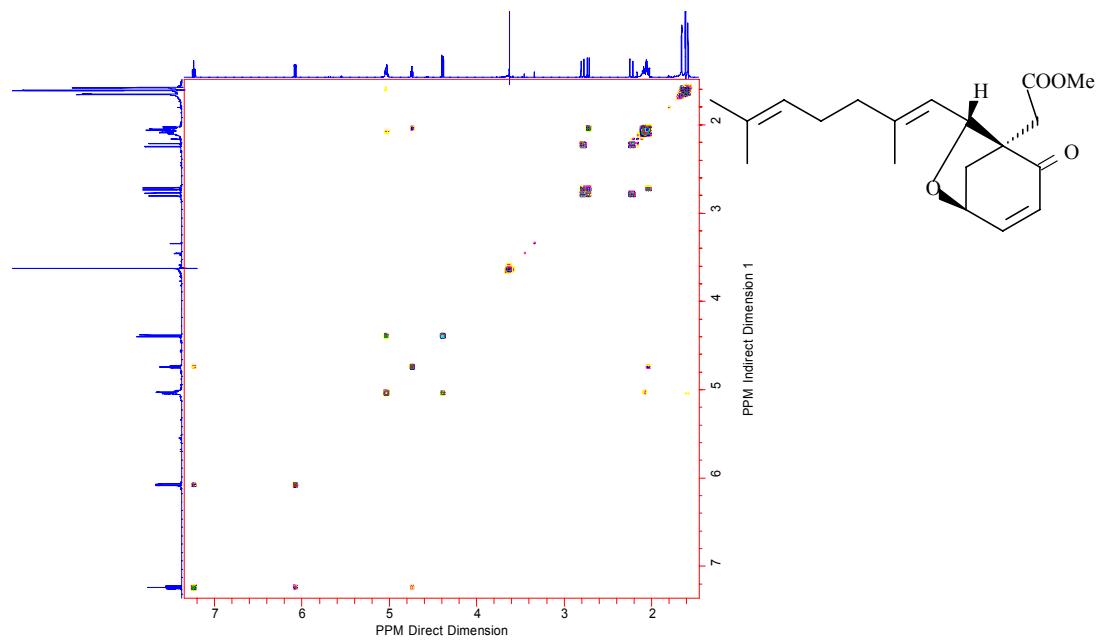


Figure R5. ^1H - ^1H COSY Spectrum of Miliusane XIX (**21**), CDCl_3

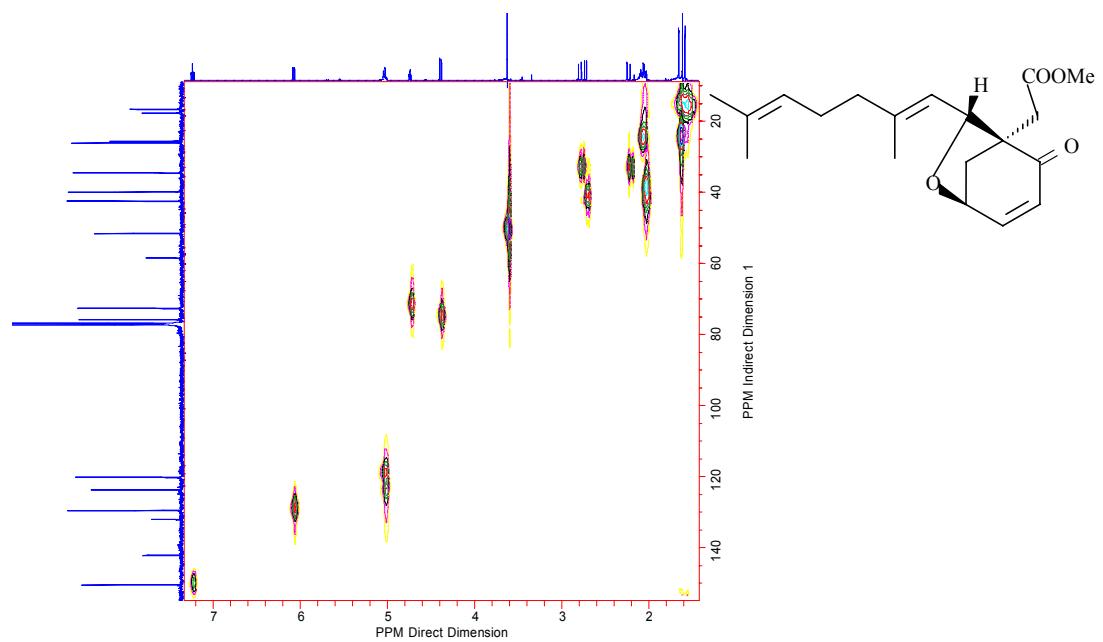


Figure R6. HMQC Spectrum of Miliusane XIX (**21**), CDCl_3

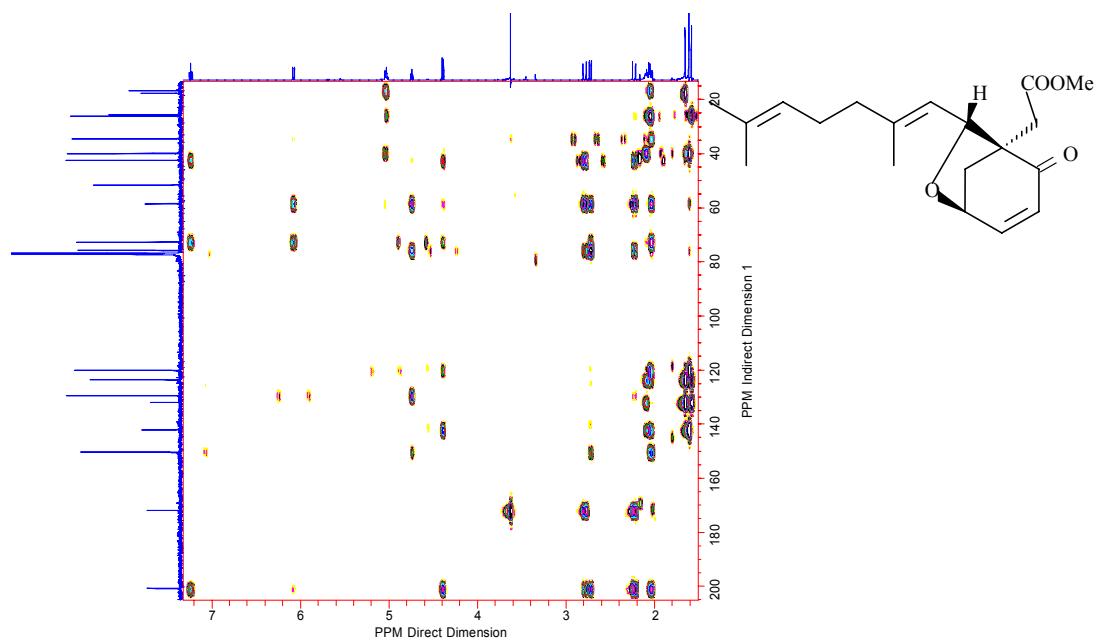


Figure R7. HMBC Spectrum of Miliusane XIX (**21**), CDCl_3

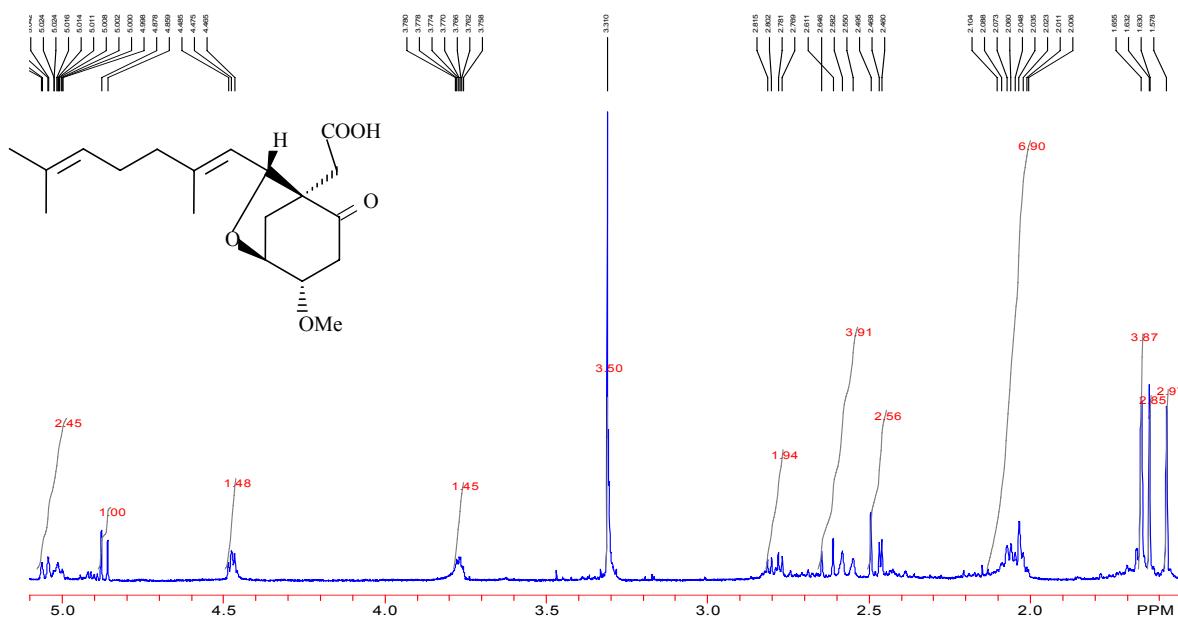


Figure S1. ^1H NMR Spectrum of Miliusane XX (**22**), CDCl_3

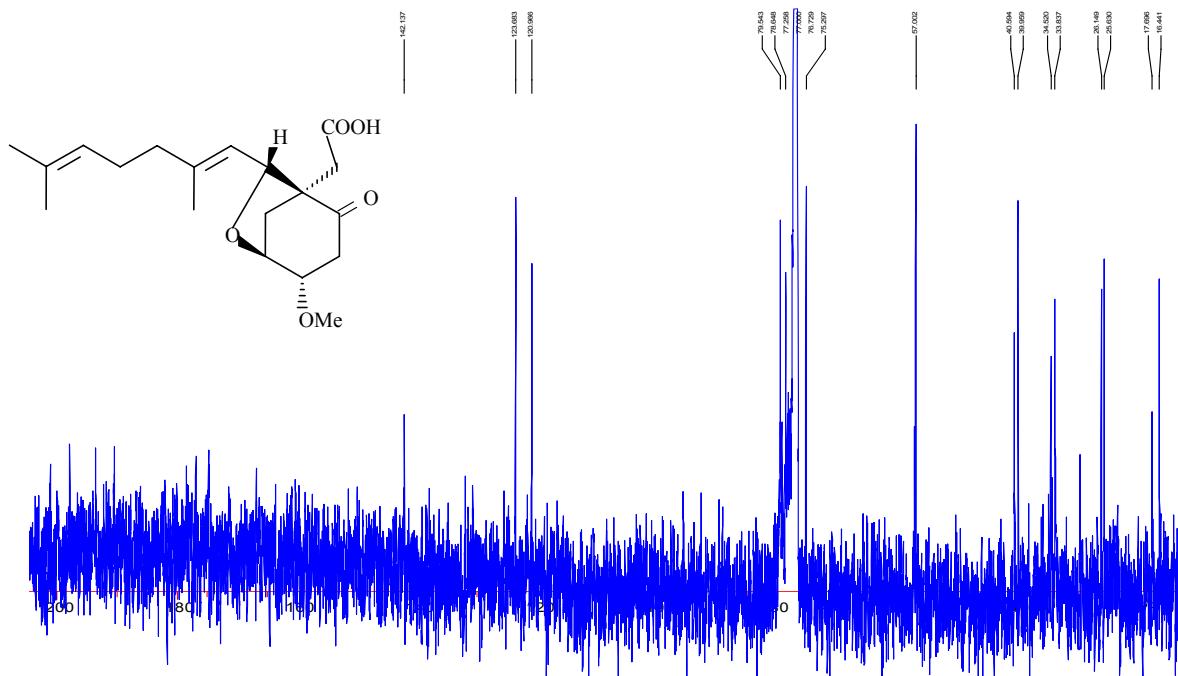


Figure S2. ^{13}C NMR Spectrum of Miliusane XX (**22**), CDCl_3

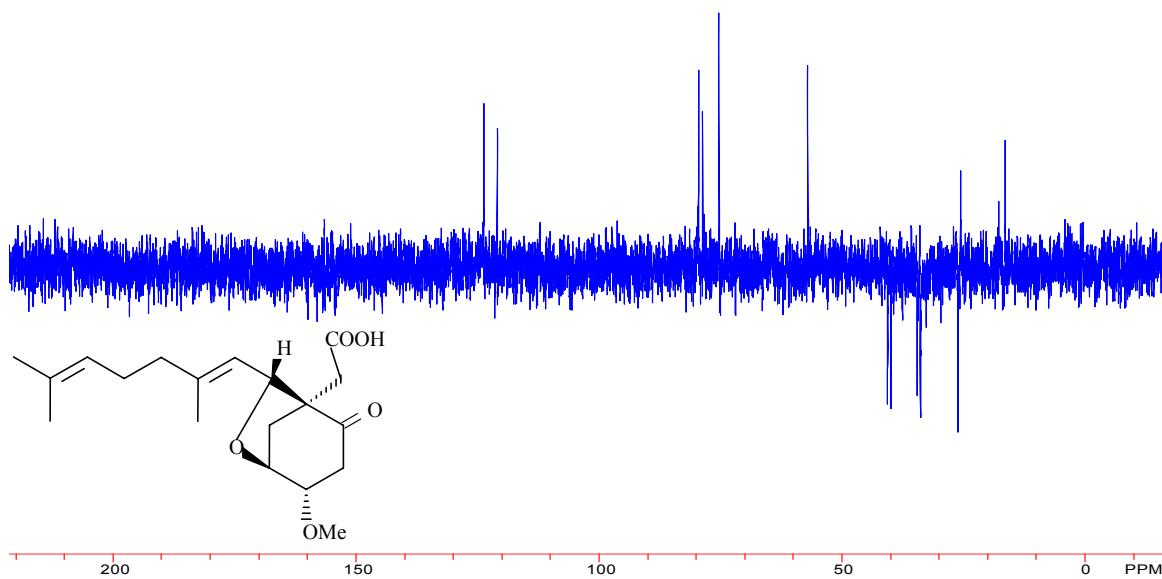


Figure S3. DEPT-135 Spectrum of Miliusane XX (**22**), CDCl_3

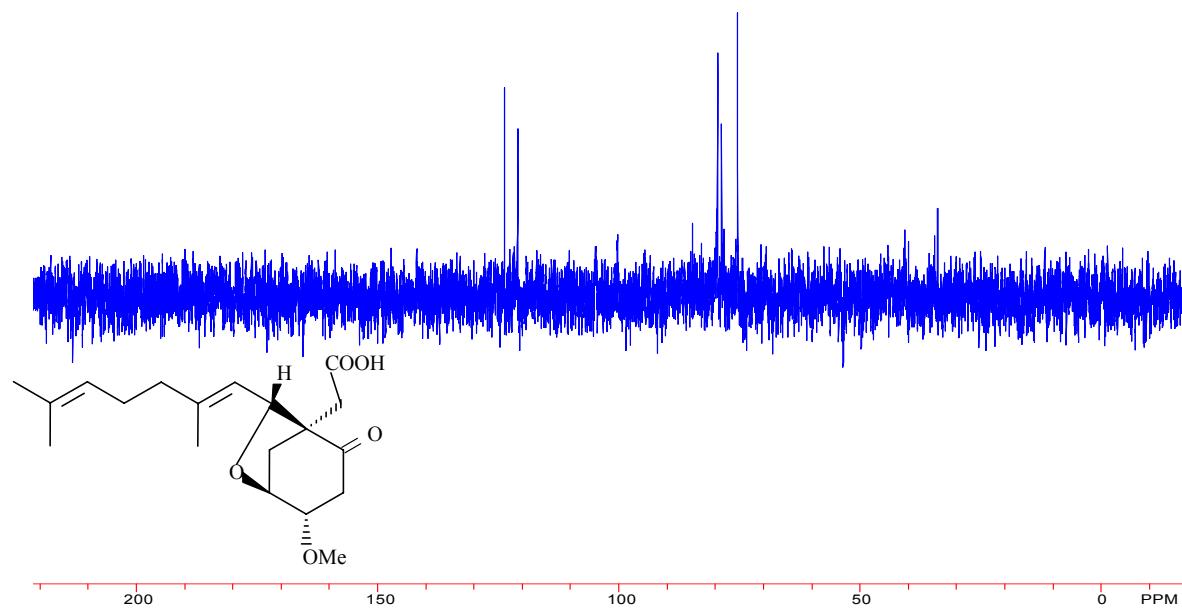


Figure S4. DEPT-90 Spectrum of Miliusane XX (**22**), CDCl_3

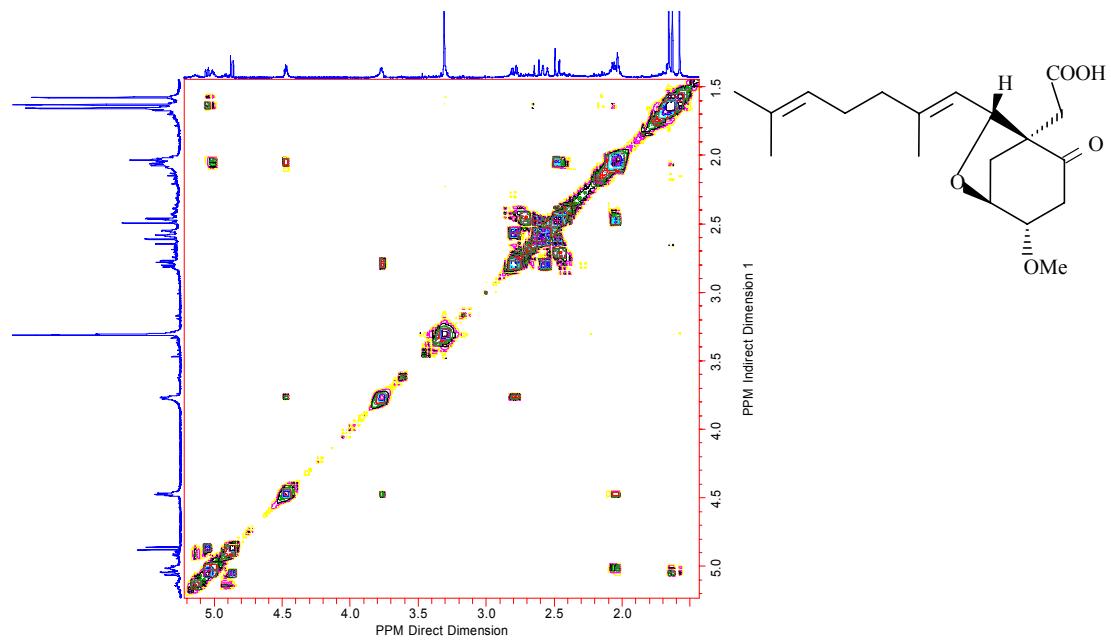


Figure S5. ^1H - ^1H COSY Spectrum of Miliusane XX (**22**), CDCl_3

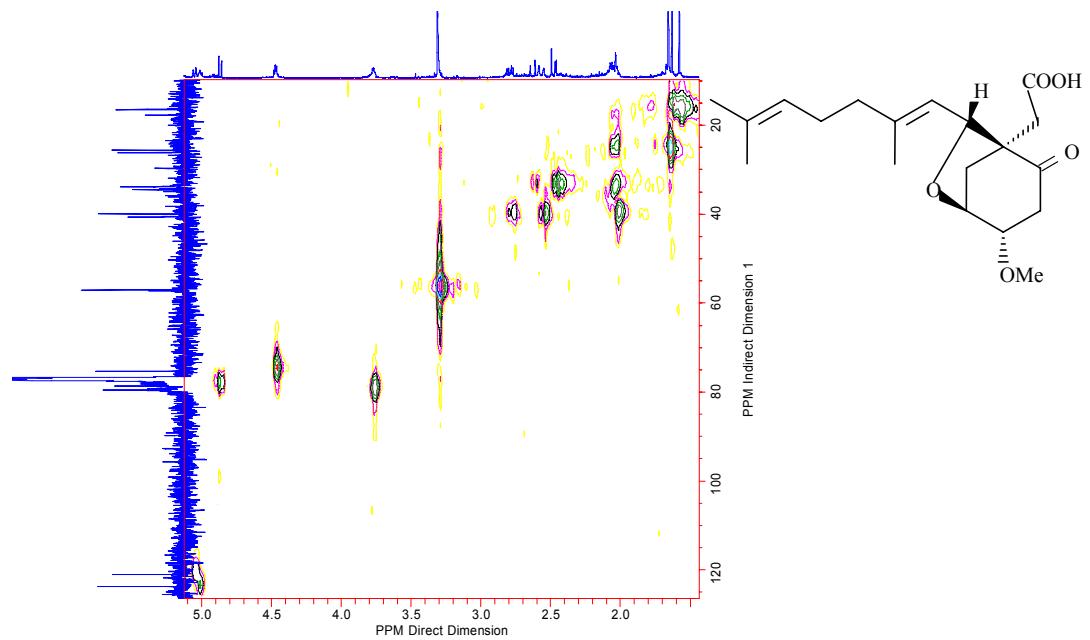


Figure S6. HMQC Spectrum of Miliusane XX (**22**), CDCl_3

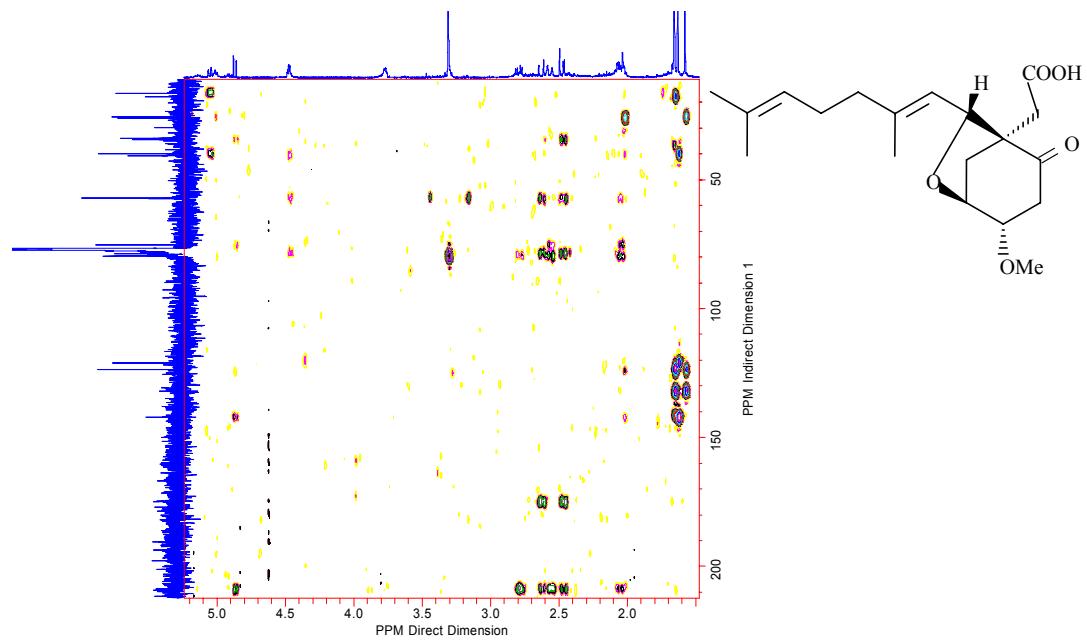


Figure S7. HMBC Spectrum of Miliusane XX (**22**), CDCl_3

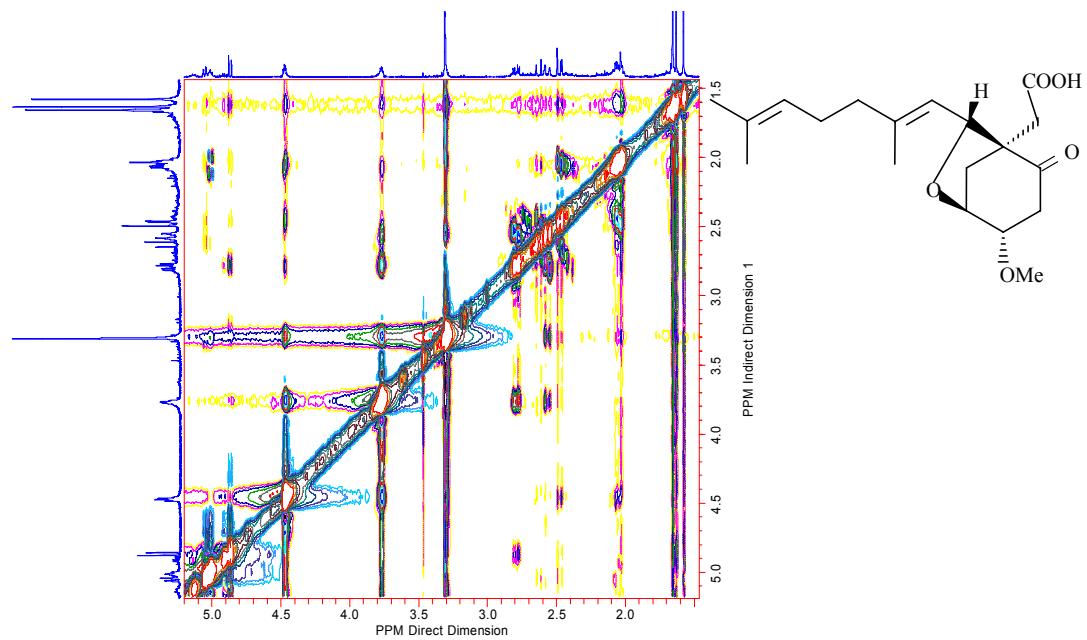


Figure S8. ROESY Spectrum of Miliusane XX (**22**), CDCl_3

SPECTRAL DATA OF MOSHER'S ESTERS OF MILIUSOL (2) AND MILIUSANE I

(3).

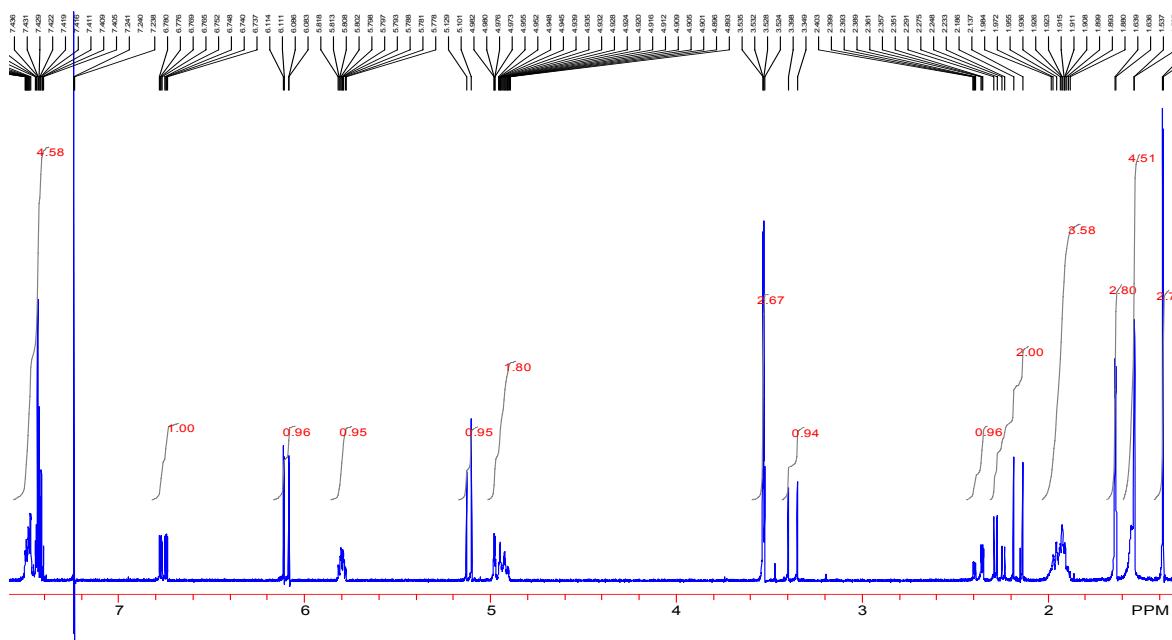


Figure T1. ^1H NMR Spectrum of (*S*)-MTPA ester of Miliusol (2), CDCl_3

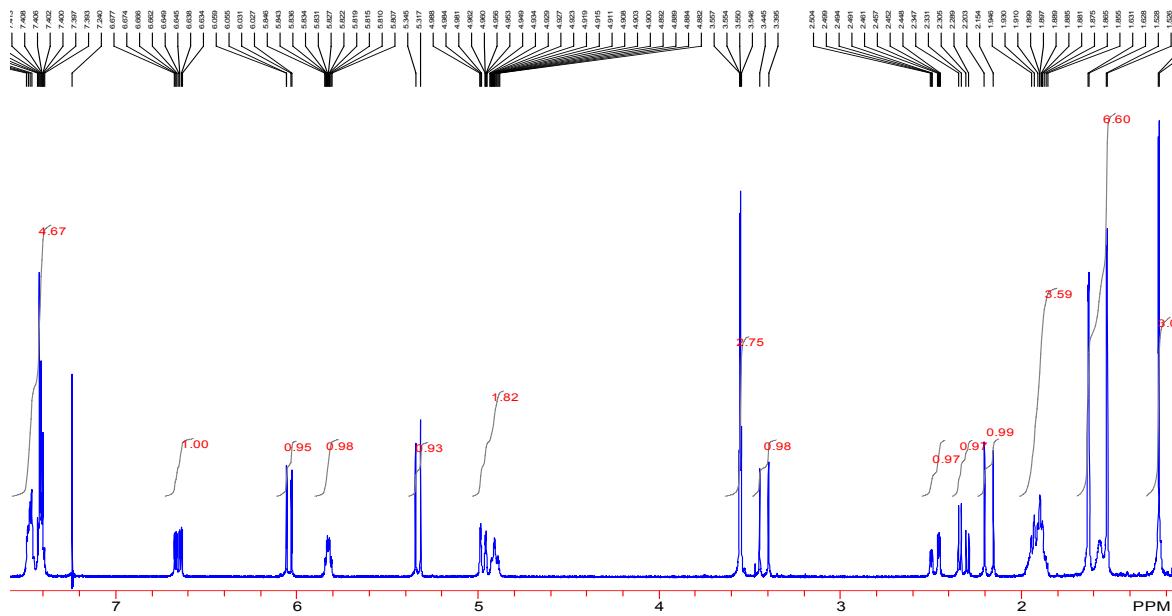
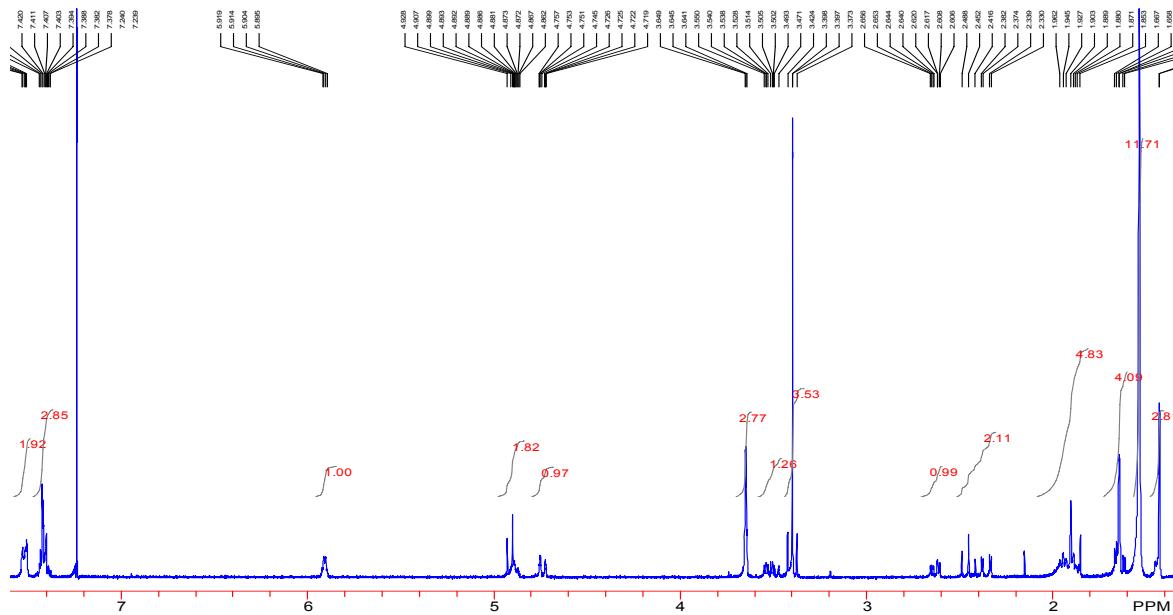
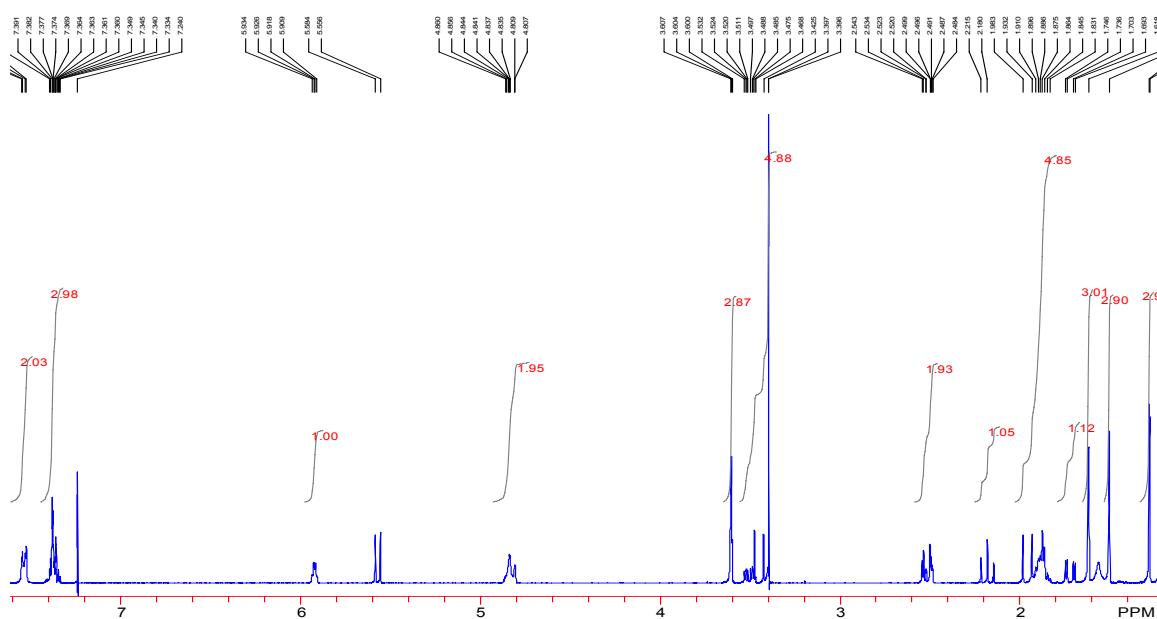


Figure T2. ^1H NMR Spectrum of (*R*)-MTPA ester of Miliusol (**2**), CDCl_3 **Figure T3.** ^1H NMR Spectrum of (*S*)-MTPA ester of Miliusane I (**3**), CDCl_3 **Figure T4.** ^1H NMR Spectrum of (*R*)-MTPA ester of Miliusane I (**3**), CDCl_3

X-ray Crystallography Data of Miliusate (1) (SVA0226-K005)

A colorless crystal of miliusate measuring roughly 0.1mm on edge was crystallized from MeOH solution. Data collection was carried out with a Rigaku RAPID area detector using a Cu sealed tube and low-temperature cooling system. Data processing was carried out using the CrystalClear package. The non-hydrogen atoms were located using the SIR92 package, and structure analysis was carried out using the CrystalStructure package. The compound crystallized in space group P2₁2₁2₁ (No. 19) with cell parameters a = 6.457(5)Å, b = 8.865(6)Å, c = 33.91(3)Å, V = 1940.8(24)Å³, Z = 4, D_{calc} = 1.186 g/cm³, λ = 1.5418Å, μ(CuKα) = 6.891 mm⁻¹, and F(000) = 744. A total of 18,762 observations were measured yielding 2009 averaged, unique reflections to 2θ = 144.2°; 1679 reflections have intensities greater than 3σ. The structure was refined by full-matrix least-squares on F to R(3σ) = 0.082, R(all) = 0.105, and GOF = 0.932.

EXPERIMENTAL

Data Collection

A colorless prism crystal of C₂₀H₂₆O₅ having approximate dimensions of 0.10 x 0.10 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Cu-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

$$\begin{aligned} a &= 6.457(5) \text{ \AA} \\ b &= 8.865(6) \text{ \AA} \\ c &= 33.91(2) \text{ \AA} \\ V &= 1940.8(24) \text{ \AA}^3 \end{aligned}$$

For Z = 0 and F.W. = 346.42, the calculated density is 0.00 g/cm³. The systematic absences of:

$$\begin{aligned} h00: h &\pm 2n \\ 0k0: k &\pm 2n \\ 00l: l &\pm 2n \end{aligned}$$

uniquely determine the space group to be:

$$\text{P2}_1\text{2}_1\text{2}_1 (\#19)$$

The data were collected at a temperature of -170 ± 1°C to a maximum 2θ value of 144.2°. A total of 145 oscillation images were collected. A sweep of data was done using ω

oscillations from 0.0 to 180.0° in 5.0° steps. The exposure rate was 360.0 [sec./°]. The detector swing angle was -0.19°. A second sweep was performed using ω oscillations from 0.0 to 180.0° in 5.0° steps. The exposure rate was 480.0 [sec./°]. The detector swing angle was -0.19°. Another sweep was performed using ω oscillations from 0.0 to 180.0° in 5.0° steps. The exposure rate was 480.0 [sec./°]. The detector swing angle was -0.19°. Another sweep was performed using ω oscillations from 0.0 to 180.0° in 5.0° steps. The exposure rate was 480.0 [sec./°]. The detector swing angle was -0.19°. Another sweep was performed using ω oscillations from 30.0 to 32.0° in 2.0° steps. The exposure rate was 60.0 [sec./°]. The detector swing angle was -0.19°. Another sweep was performed using ω oscillations from 0.0 to 180.0° in 5.0° steps. The exposure rate was 360.0 [sec./°]. The detector swing angle was -0.19°. Another sweep was performed using ω oscillations from 0.0 to 180.0° in 5.0° steps. The exposure rate was 480.0 [sec./°]. The detector swing angle was -0.19°. Another sweep was performed using ω oscillations from 0.0 to 180.0° in 5.0° steps. The exposure rate was 480.0 [sec./°]. The detector swing angle was -0.19°. Another sweep was performed using ω oscillations from 0.0 to 180.0° in 5.0° steps. The exposure rate was 480.0 [sec./°]. The detector swing angle was -0.19°. Another sweep was performed using ω oscillations from 30.0 to 32.0° in 2.0° steps. The exposure rate was 60.0 [sec./°]. The detector swing angle was -0.19°. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 18762 reflections that were collected, 2009 were unique ($R_{int} = 0.050$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Cu-K α radiation is 6.891 cm $^{-1}$. was applied which resulted in transmission factors ranging from 0.52 to 1.00. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction¹ was applied (coefficient = 0.626550).

Structure Solution and Refinement

The structure was solved by direct methods² and expanded using Fourier techniques.³ The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement⁴ on F was based on 1679 observed reflections ($I > 3.00\sigma(I)$) and 228 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||F_O| - |F_C|| / \Sigma |F_O| = 0.082$$

$$R_w = [\Sigma w (|F_O| - |F_C|)^2 / \Sigma w |F_O|^2]^{1/2} = 0.105$$

The standard deviation of an observation of unit weight⁵ was 0.93. A Robust-resistant weighting scheme was used.⁶ Plots of $\Sigma w (|F_O| - |F_C|)^2$ versus $|F_O|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.36 and -0.40 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber.⁷ Anomalous dispersion effects were included in Fcalc;⁸ the values for Δf and $\Delta f'$ were those of Creagh and McAuley.⁹ The values for the mass attenuation coefficients are those of Creagh and Hubbell.¹⁰ All calculations were performed using the CrystalStructure^{11, 12} crystallographic software package.

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- (1) Larson, A.C. Crystallographic Computing. F.R. Ahmed, Ed. Munksgaard, Copenhagen (equation 22, with V replaced by the cell volume), **1970**, 291-294.
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- (3) DIRDIF99: Beurskens, P.T.; Admiraal, G.; Beurskens, G.; Bosman, W.P.; de Gelder, R.; Israel, R.; Smits, J. M. M. The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands, **1999**.
- (4) Least Squares function minimized:

$$\Sigma w(|F_O|-|F_C|)^2 \quad \text{where } w = \text{Least Squares weights.}$$

- (5) Standard deviation of an observation of unit weight:

$$[\Sigma w(|F_O|-|F_C|)^2/(N_O-N_V)]^{1/2}$$

where: N_O = number of observations
 N_V = number of variables

- (6) Carruthers, J.R.; Watkin, D.J. *Acta Cryst.* **1979**, A35, 698-699.
- (7) Cromer, D. T.; Waber, J. T. In: International Tables for X-ray Crystallography. The Kynoch Press, Birmingham, England, **1974**; Vol. IV, Table 2.2 A.
- (8) Ibers, J. A.; Hamilton, W. C. *Acta Crystallogr.* **1964**, 17, 781.
- (9) Creagh, D. C.; McAuley, W. J. In: International Tables for Crystallography. Wilson, A. J. C. Ed., Kluwer Academic Publishers, Boston, **1992**; Vol. C, Table 4.2.6.8, pages 219-222.
- (10) Creagh, D. C.; Hubbell, J.H. In: International Tables for Crystallography. Wilson, A. J. C. Ed., Kluwer Academic Publishers, Boston, **1992**; Vol. C, Table 4.2.4.3, pages 200-206.
- (11) CrystalStructure 3.10: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2002).

(12) CRYSTALS Issue 10: Watkin, D. J.; Prout, C. K.; Carruthers, J. R.; Betteridge, P.W. Chemical Crystallography Laboratory, Oxford, UK.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₂₀ H ₂₆ O ₅
Formula Weight	346.42
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 6.457(5) Å b = 8.865(6) Å c = 33.91(2) Å V = 1940.8(24) Å ³
Space Group	P2 ₁ 2 ₁ 2 ₁ (#19)
Z value	4
D _{calc}	1.186 g/cm ³
F ₀₀₀	744
μ(CuKα)	6.891 cm ⁻¹

B. Intensity Measurements

Diffractionometer	Rigaku/MSC RAPID
Radiation	CuKα ($\lambda = 1.54180 \text{ \AA}$) graphite monochromated
Detector Aperture	0 mm x 0 mm
Data Images	145 exposures
ω oscillation Range	0.0 - 180.0°
Exposure Rate	360.0 sec./°
ω oscillation Range	0.0 - 180.0°
Exposure Rate	480.0 sec./°
ω oscillation Range	0.0 - 180.0°
Exposure Rate	480.0 sec./°
ω oscillation Range	0.0 - 180.0°
Exposure Rate	480.0 sec./°
ω oscillation Range	30.0 - 32.0°
Exposure Rate	60.0 sec./°

ω oscillation Range	0.0 - 180.0°
Exposure Rate	360.0 sec./°
ω oscillation Range	0.0 - 180.0°
Exposure Rate	480.0 sec./°
ω oscillation Range	0.0 - 180.0°
Exposure Rate	480.0 sec./°
ω oscillation Range	0.0 - 180.0°
Exposure Rate	480.0 sec./°
ω oscillation Range	30.0 - 32.0°
Exposure Rate	60.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	144.2°
No. of Reflections Measured	Total: 18762 Unique: 2009 ($R_{\text{int}} = 0.050$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.5249 - 1.0000) Secondary Extinction (coefficient: 6.26550e-001)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F
Function Minimized	$\Sigma w (F_O - F_C)^2$
Least Squares Weights	Chebychev polynomial with 3
parameters	8.936, 10.306, 6.362,
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00\sigma(I)$)	1679
No. Variables	228
Reflection/Parameter Ratio	7.36
Residuals: $R (I > 3.00\sigma(I))$	0.082
Residuals: $R_w (I > 3.00\sigma(I))$	0.105
Goodness of Fit Indicator	0.93
Flack Parameter:	0.0(5)
Max Shift/Error in Final Cycle	0.01
Maximum peak in Final Diff. Map	0.36 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.40 e ⁻ /Å ³

Table 1a. Atom coordinates and B_{eq}

atom	x	y	z	B _{eq}
C(1)	0.0474(6)	0.5159(4)	-0.19902(14)	3.51(8)
C(2)	-0.0785(6)	0.3838(4)	-0.21262(13)	3.12(8)
O(2)	-0.1007(6)	0.3461(3)	-0.24697(9)	4.00(7)
O(3)	-0.1658(4)	0.3127(3)	-0.18214(8)	3.09(6)
C(3)	-0.3072(6)	0.1881(4)	-0.19063(12)	3.17(7)
C(4)	-0.5177(7)	0.2348(4)	-0.17641(17)	4.10(9)
C(5)	-0.6550(6)	0.1349(4)	-0.16287(15)	3.96(9)
C(6)	-0.6102(6)	-0.0288(4)	-0.16226(11)	2.83(7)
O(6)	-0.7478(4)	-0.1187(3)	-0.15544(8)	3.25(6)
C(7)	-0.3890(5)	-0.0785(4)	-0.17123(12)	2.68(7)
C(8)	-0.2308(5)	0.0506(4)	-0.16871(11)	2.66(7)
C(9)	-0.3922(5)	-0.1609(4)	-0.21078(11)	2.71(7)
O(9)	-0.4719(4)	-0.4273(3)	-0.22131(9)	3.12(5)
C(10)	-0.4249(6)	-0.3253(4)	-0.19976(11)	2.76(7)
O(10)	-0.3903(4)	-0.3460(3)	-0.16112(8)	2.97(5)
C(11)	-0.3180(6)	-0.2068(4)	-0.14231(11)	2.93(7)
C(12)	-0.3954(6)	-0.1976(4)	-0.10098(12)	2.95(7)
C(13)	-0.2925(7)	-0.1500(5)	-0.06953(13)	3.55(9)
C(14)	-0.0663(9)	-0.1099(7)	-0.0694817)	4.88(12)
C(15)	-0.4068(7)	-0.1315(6)	-0.03101(13)	3.86(9)
C(16)	-0.4564(12)	0.0334(7)	-0.02205(18)	5.74(14)
C(17)	-0.5951(10)	0.0541(7)	0.01362(17)	5.15(12)
C(18)	-0.5414(11)	0.0861(6)	0.04923(19)	5.51(13)
C(19)	-0.6962(16)	0.0998(7)	0.08263(19)	6.87(17)
C(20)	-0.3176(20)	0.1092(13)	0.06183(29)	9.82(32)

Table 1b. Hydrogen Atom Coordinates and B(iso).

H(1)	0.1074(6)	0.5670(4)	-0.22082(14)	4.34(12)
H(2)	0.1534(6)	0.4807(4)	-0.18199(14)	4.33(12)
H(3)	-0.0405(6)	0.5835(4)	-0.18501(14)	4.32(12)
H(4)	-0.3099(6)	0.1688(4)	-0.21819(13)	3.83(11)
H(5)	-0.5525(7)	0.3388(4)	-0.17715(17)	4.92(13)
H(6)	-0.7842(7)	0.1697(4)	-0.15308(15)	4.77(13)
H(7)	-0.2118(6)	0.0774(4)	-0.14184(11)	3.21(11)
H(8)	-0.1024(6)	0.0191(4)	-0.17961(11)	3.24(11)
H(9)	-0.2638(5)	-0.1504(4)	-0.22428(10)	3.26(11)
H(10)	-0.5010(5)	-0.1243(4)	-0.22705(10)	3.26(11)
H(11)	-0.1711(6)	-0.2096(4)	-0.14175(11)	3.52(11)
H(12)	-0.5350(6)	-0.2282(4)	-0.09696(12)	3.55(11)
H(13)	0.0140(9)	-0.1941(7)	-0.07771(17)	5.99(15)
H(14)	-0.0248(9)	-0.0804(7)	-0.04371(17)	5.99(15)
H(15)	-0.0450(9)	-0.0284(7)	-0.08723(17)	5.99(15)

H(16)	-0.3219(7)	-0.1690(6)	-0.01022(13)	4.67(12)
H(17)	-0.5327(7)	-0.1865(6)	-0.03170(13)	4.67(12)
H(18)	-0.5220(12)	0.0746(7)	-0.04474(18)	6.90(17)
H(19)	-0.3302(12)	0.0857(7)	-0.01734(18)	6.90(17)
H(20)	-0.7395(10)	0.0415(7)	0.00931(17)	6.23(14)
H(21)	-0.793(2)	0.1774(7)	0.07613(19)	8.46(23)
H(22)	-0.632(2)	0.1233(7)	0.10700(19)	8.45(23)
H(23)	-0.768(2)	0.0066(7)	0.08490(19)	8.45(23)
H(24)	-0.242(2)	0.0192(13)	0.05671(29)	12.4(5)
H(25)	-0.314(2)	0.1307(13)	0.08923(29)	12.4(5)
H(26)	-0.257(2)	0.1904(13)	0.04761(29)	12.4(5)

$$B_{eq} = \frac{8}{3} \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U11	U22	U33	U12	U13	U23
C(1)	0.039(2)	0.036(2)	0.058(3)	-0.001(1)	0.005(2)	0.003(2)
C(2)	0.034(2)	0.035(1)	0.050(3)	0.0022(13)	0.006(1)	-0.001(1)
O(2)	0.054(2)	0.050(1)	0.048(2)	-0.006(1)	0.0127(12)	0.0006(13)
O(3)	0.0407(13)	0.0331(11)	0.044(2)	-0.0083(10)	-0.0004(10)	0.0008(10)
C(3)	0.038(2)	0.034(2)	0.049(2)	-0.008(1)	-0.001(2)	0.003(1)
C(4)	0.039(2)	0.033(2)	0.083(3)	-0.002(1)	-0.007(2)	0.009(2)
C(5)	0.041(2)	0.032(2)	0.078(3)	0.004(1)	0.002(2)	0.003(2)
C(6)	0.032(2)	0.035(2)	0.040(2)	-0.0009(13)	0.0007(13)	0.0018(13)
O(6)	0.0337(13)	0.0404(12)	0.049(2)	-0.0001(10)	0.0029(12)	0.0026(10)
C(7)	0.025(2)	0.034(2)	0.043(2)	-0.0025(11)	0.0029(13)	0.0022(13)
C(8)	0.030(2)	0.0311(13)	0.040(2)	-0.0037(12)	0.0011(12)	0.0012(13)
C(9)	0.034(2)	0.031(1)	0.038(2)	-0.0029(13)	0.0006(12)	0.0020(13)
O(9)	0.0399(13)	0.0330(10)	0.046(2)	0.0004(10)	-0.0001(11)	-0.0038(10)
C(10)	0.033(2)	0.035(1)	0.037(2)	0.0004(13)	0.0033(12)	-0.0010(13)
O(10)	0.042(1)	0.0309(10)	0.040(2)	0.0008(10)	-0.0022(10)	-0.0006(10)
C(11)	0.042(2)	0.033(1)	0.036(2)	0.0008(12)	-0.0002(13)	-0.002(1)
C(12)	0.037(2)	0.035(1)	0.040(2)	-0.0010(13)	0.000(1)	0.004(1)
C(13)	0.050(2)	0.045(2)	0.040(2)	0.005(2)	0.001(2)	0.004(2)
C(14)	0.054(3)	0.087(3)	0.045(3)	-0.001(3)	-0.008(2)	-0.004(2)
C(15)	0.054(2)	0.064(2)	0.029(2)	0.002(2)	0.004(2)	0.003(2)
C(16)	0.099(4)	0.074(3)	0.045(3)	0.009(3)	0.016(3)	-0.006(2)
C(17)	0.073(3)	0.070(3)	0.053(3)	0.003(3)	0.003(3)	-0.009(2)
C(18)	0.096(4)	0.053(2)	0.060(4)	-0.010(3)	0.016(3)	-0.008(2)
C(19)	0.139(6)	0.068(3)	0.055(4)	-0.007(4)	0.029(4)	-0.020(3)
C(20)	0.148(8)	0.150(10)	0.075(6)	-0.070(8)	-0.007(5)	-0.005(5)

$$\text{The general temperature factor expression: } \exp(-2\pi^2(a^*a^2U_{11}h^2 + b^*b^2U_{22}k^2 + c^*c^2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Table 3. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
C(1)	C(2)	1.498(5)	C(2)	O(2)	1.220(5)
C(2)	O(3)	1.335(5)	O(3)	C(3)	1.461(4)
C(3)	C(4)	1.500(6)	C(3)	C(8)	1.510(5)
C(4)	C(5)	1.335(6)	C(5)	C(6)	1.479(5)
C(6)	O(6)	1.216(5)	C(6)	C(7)	1.525(5)
C(7)	C(8)	1.537(4)	C(7)	C(9)	1.527(5)
C(7)	C(11)	1.570(5)	C(9)	C(10)	1.519(4)
O(9)	C(10)	1.201(4)	C(10)	O(10)	1.342(5)
O(10)	C(11)	1.466(4)	C(11)	C(12)	1.490(6)
C(12)	C(13)	1.326(6)	C(13)	C(14)	1.503(7)
C(13)	C(15)	1.509(6)	C(15)	C(16)	1.527(8)
C(16)	C(17)	1.516(8)	C(17)	C(18)	1.288(9)
C(18)	C(19)	1.516(9)	C(18)	C(20)	1.52(1)

Table 4. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950(6)	C(1)	H(2)	0.948(6)
C(1)	H(3)	0.952(6)	C(3)	H(4)	0.950(6)
C(4)	H(5)	0.949(5)	C(5)	H(6)	0.949(6)
C(8)	H(7)	0.949(5)	C(8)	H(8)	0.950(5)
C(9)	H(9)	0.951(5)	C(9)	H(10)	0.950(5)
C(11)	H(11)	0.949(6)	C(12)	H(12)	0.950(5)
C(14)	H(13)	0.950(8)	C(14)	H(14)	0.951(8)
C(14)	H(15)	0.951(9)	C(15)	H(16)	0.953(7)
C(15)	H(17)	0.948(7)	C(16)	H(18)	0.951(9)
C(16)	H(19)	0.951(10)	C(17)	H(20)	0.950(9)
C(19)	H(21)	0.955(11)	C(19)	H(22)	0.947(10)
C(19)	H(23)	0.952(10)	C(20)	H(24)	0.95(2)
C(20)	H(25)	0.95(1)	C(20)	H(26)	0.95(2)

Table 5. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	C(2)	O(2)	124.8(4)	C(1)	C(2)	O(3)	111.1(3)
O(2)	C(2)	O(3)	124.1(3)	C(2)	O(3)	C(3)	117.9(3)
O(3)	C(3)	C(4)	107.1(3)	O(3)	C(3)	C(8)	108.0(3)
C(4)	C(3)	C(8)	111.1(3)	C(3)	C(4)	C(5)	122.0(3)
C(4)	C(5)	C(6)	121.7(4)	C(5)	C(6)	O(6)	120.2(3)
C(5)	C(6)	C(7)	117.6(3)	O(6)	C(6)	C(7)	122.2(3)
C(6)	C(7)	C(8)	113.3(3)	C(6)	C(7)	C(9)	107.5(3)
C(8)	C(7)	C(9)	114.5(3)	C(6)	C(7)	C(11)	111.0(3)
C(8)	C(7)	C(11)	108.1(3)	C(9)	C(7)	C(11)	101.9(3)
C(3)	C(8)	C(7)	110.9(3)	C(7)	C(9)	C(10)	104.2(3)
C(9)	C(10)	O(9)	127.4(4)	C(9)	C(10)	O(10)	110.4(3)
O(9)	C(10)	O(10)	122.2(3)	C(10)	O(10)	C(11)	111.3(3)
C(7)	C(11)	O(10)	104.2(3)	C(7)	C(11)	C(12)	116.7(3)
O(10)	C(11)	C(12)	110.4(3)	C(11)	C(12)	C(13)	127.3(4)
C(12)	C(13)	C(14)	124.3(5)	C(12)	C(13)	C(15)	119.1(4)
C(14)	C(13)	C(15)	116.7(4)	C(13)	C(15)	C(16)	112.2(4)
C(15)	C(16)	C(17)	113.5(5)	C(16)	C(17)	C(18)	128.0(6)
C(17)	C(18)	C(19)	122.7(7)	C(17)	C(18)	C(20)	123.3(7)
C(19)	C(18)	C(20)	114.0(7)				

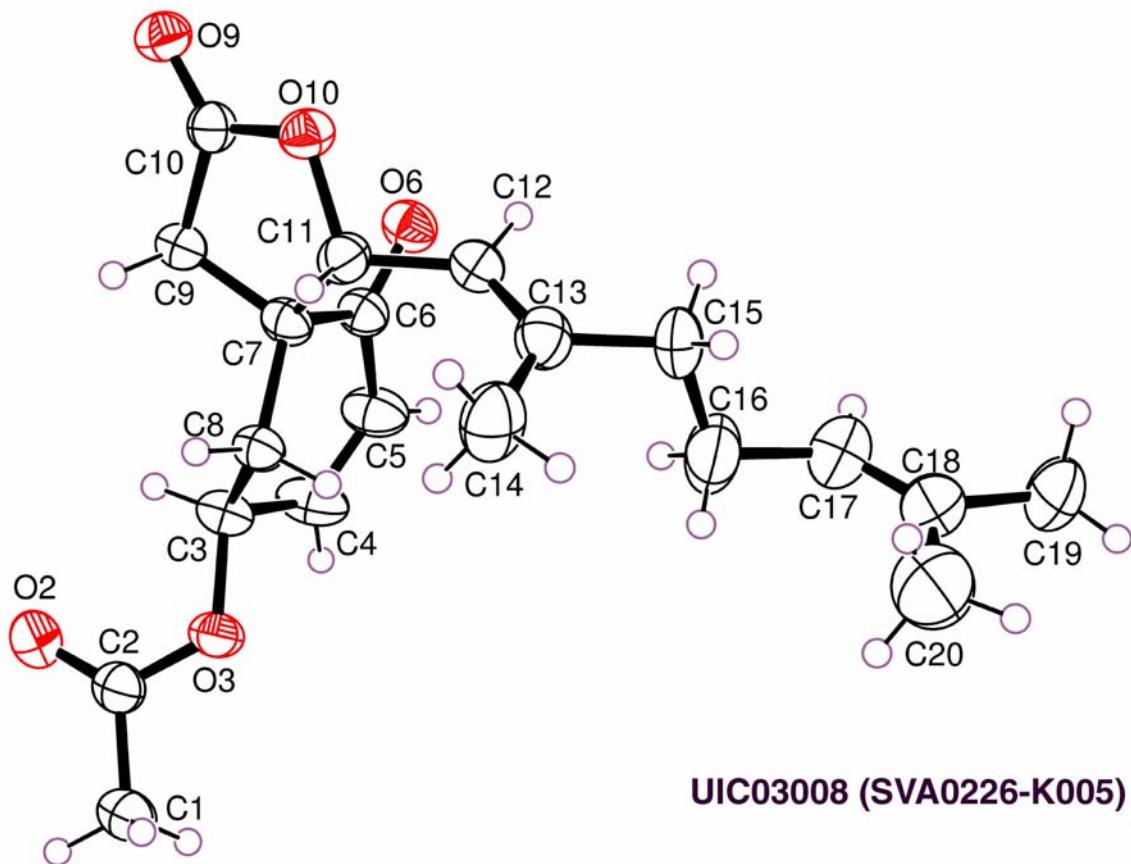
Table 6. Bond angles involving hydrogens ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(1)	H(1)	110.8(4)	C(2)	C(1)	H(2)	108.8(4)
H(1)	C(1)	H(2)	109.7(5)	C(2)	C(1)	H(3)	108.8(4)
H(1)	C(1)	H(3)	109.3(5)	H(2)	C(1)	H(3)	109.5(5)
O(3)	C(3)	H(4)	110.0(4)	C(4)	C(3)	H(4)	110.4(4)
C(8)	C(3)	H(4)	110.1(4)	C(3)	C(4)	H(5)	118.3(5)
C(5)	C(4)	H(5)	119.8(5)	C(4)	C(5)	H(6)	119.2(5)
C(6)	C(5)	H(6)	119.1(5)	C(3)	C(8)	H(7)	108.2(4)
C(7)	C(8)	H(7)	109.0(4)	C(3)	C(8)	H(8)	109.3(4)
C(7)	C(8)	H(8)	109.8(4)	H(7)	C(8)	H(8)	109.5(5)
C(7)	C(9)	H(9)	111.3(4)	C(10)	C(9)	H(9)	109.5(4)
C(7)	C(9)	H(10)	110.9(4)	C(10)	C(9)	H(10)	111.6(4)
H(9)	C(9)	H(10)	109.3(4)	C(7)	C(11)	H(11)	108.9(4)
O(10)	C(11)	H(11)	107.7(4)	C(12)	C(11)	H(11)	108.5(4)
C(11)	C(12)	H(12)	115.9(4)	C(13)	C(12)	H(12)	116.8(4)
C(13)	C(14)	H(13)	110.1(6)	C(13)	C(14)	H(14)	109.9(6)
H(13)	C(14)	H(14)	109.4(8)	C(13)	C(14)	H(15)	108.6(6)
H(13)	C(14)	H(15)	109.4(8)	H(14)	C(14)	H(15)	109.4(8)
C(13)	C(15)	H(16)	108.7(5)	C(16)	C(15)	H(16)	107.9(5)

C(13)	C(15)	H(17)	110.0(5)	C(16)	C(15)	H(17)	108.5(5)
H(16)	C(15)	H(17)	109.4(6)	C(15)	C(16)	H(18)	107.5(7)
C(17)	C(16)	H(18)	109.6(7)	C(15)	C(16)	H(19)	108.7(7)
C(17)	C(16)	H(19)	108.2(7)	H(18)	C(16)	H(19)	109.3(9)
C(16)	C(17)	H(20)	116.3(7)	C(18)	C(17)	H(20)	115.7(7)
C(18)	C(19)	H(21)	108.6(8)	C(18)	C(19)	H(22)	112.4(8)
H(21)	C(19)	H(22)	109.2(10)	C(18)	C(19)	H(23)	108.2(8)
H(21)	C(19)	H(23)	108.8(10)	H(22)	C(19)	H(23)	109.5(10)
C(18)	C(20)	H(24)	108.9(12)	C(18)	C(20)	H(25)	109.1(12)
H(24)	C(20)	H(25)	109.5(15)	C(18)	C(20)	H(26)	110.6(12)
H(24)	C(20)	H(26)	109.3(15)	H(25)	C(20)	H(26)	109.5(15)

Table 7. Torsion Angles($^{\circ}$)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(1)	C(2)	O(3)	C(3)	175.3(3)	O(2)	C(2)	O(3)	C(3)	-4.0(5)
C(2)	O(3)	C(3)	C(4)	-115.5(4)	C(2)	O(3)	C(3)	C(8)	124.7(4)
O(3)	C(3)	C(4)	C(5)	-148.7(4)	C(8)	C(3)	C(4)	C(5)	-31.0(6)
O(3)	C(3)	C(8)	C(7)	171.6(3)	C(4)	C(3)	C(8)	C(7)	54.4(4)
C(3)	C(4)	C(5)	C(6)	-2.0(6)	C(4)	C(5)	C(6)	O(6)	-168.9(4)
C(4)	C(5)	C(6)	C(7)	9.9(6)	C(5)	C(6)	C(7)	C(8)	15.7(5)
C(5)	C(6)	C(7)	C(9)	-111.9(4)	C(5)	C(6)	C(7)	C(11)	137.5(4)
O(6)	C(6)	C(7)	C(8)	-165.6(3)	O(6)	C(6)	C(7)	C(9)	66.9(4)
O(6)	C(6)	C(7)	C(11)	-43.7(4)	C(6)	C(7)	C(8)	C(3)	-47.3(4)
C(9)	C(7)	C(8)	C(3)	76.5(4)	C(11)	C(7)	C(8)	C(3)	-170.7(3)
C(6)	C(7)	C(9)	C(10)	-91.5(3)	C(8)	C(7)	C(9)	C(10)	141.6(3)
C(11)	C(7)	C(9)	C(10)	25.2(3)	C(6)	C(7)	C(11)	O(10)	86.5(3)
C(6)	C(7)	C(11)	C(12)	-35.5(4)	C(8)	C(7)	C(11)	O(10)	-148.7(3)
C(8)	C(7)	C(11)	C(12)	89.3(4)	C(9)	C(7)	C(11)	O(10)	-27.7(3)
C(9)	C(7)	C(11)	C(12)	-149.7(3)	C(7)	C(9)	C(10)	O(9)	166.4(4)
C(7)	C(9)	C(10)	O(10)	-14.6(4)	C(9)	C(10)	O(10)	C(11)	-4.0(4)
O(9)	C(10)	O(10)	C(11)	175.0(3)	C(10)	O(10)	C(11)	C(7)	20.4(4)
C(10)	O(10)	C(11)	C(12)	146.5(3)	C(7)	C(11)	C(12)	C(13)	-101.8(5)
O(10)	C(11)	C(12)	C(13)	139.5(4)	C(11)	C(12)	C(13)	C(14)	-6.4(7)
C(11)	C(12)	C(13)	C(15)	172.6(4)	C(12)	C(13)	C(15)	C(16)	-102.6(5)
C(14)	C(13)	C(15)	C(16)	76.5(6)	C(13)	C(15)	C(16)	C(17)	172.5(5)
C(15)	C(16)	C(17)	C(18)	97.6(7)	C(16)	C(17)	C(18)	C(19)	-178.4(6)
C(16)	C(17)	C(18)	C(20)	-0.1(12)					



Determination of Purity of the Target Compounds

The purity of the target compounds was determined by reversed-phase HPLC, which was carried out on a Waters 600E Delivery System pump, equipped with a Waters 996 photodiode detector, and an Alltima C18 column (5 μm, 250 mm × 10 mm).

Compound	Solvent System	Retention Time (min)	Purity (%)
1	A: MeCN/H ₂ O 50:50	41.60	98.55
	B: MeOH/H ₂ O 60:40	46.05	99.07
2	A: MeCN/H ₂ O 60:40	30.07	99.22
	B: MeOH/H ₂ O 80:20	23.79	99.54
3	A: MeCN/H ₂ O 60:40	36.33	99.96
	B: MeOH/H ₂ O 75:25	32.01	99.65
5	A: MeCN/H ₂ O 60:40	20.87	99.34
	B: MeOH/H ₂ O 75:25	18.31	99.69
8	A: MeCN/H ₂ O 55:45	40.78	99.37
	B: MeOH/H ₂ O 70:30	36.80	97.89
9	A: MeCN/H ₂ O 60:40	32.10	99.00
	B: MeOH/H ₂ O 85:15	24.87	98.40
20	A: MeCN/H ₂ O 70:30	19.36	96.75
	B: MeOH/H ₂ O 85:15	20.04	97.10
21	A: MeCN/H ₂ O 65:35	24.06	98.70
	B: MeOH/H ₂ O 80:20	20.47	98.82
2ab	A: MeCN/H ₂ O 70:30	31.27	99.58
	B: MeOH/H ₂ O 90:10	22.12	99.68
2ad	A: MeCN/H ₂ O 80:20	22.37	98.59
	B: MeOH/H ₂ O 90:10	21.00	98.72
2ae	A: MeCN/H ₂ O 80:20	23.70	99.10
	B: MeOH/H ₂ O 90:10	18.48	98.68
2af	A: MeCN/H ₂ O 80:20	26.02	99.68
	B: MeOH/H ₂ O 90:10	20.89	97.64
2ag	A: MeCN/H ₂ O 80:20	20.58	100
	B: MeOH/H ₂ O 90:10	17.95	98.65
2ah	A: MeCN/H ₂ O 80:20	20.32	97.26
	B: MeOH/H ₂ O 90:10	20.51	96.91
2ai	A: MeCN/H ₂ O 80:20	23.70	97.19
	B: MeOH/H ₂ O 90:10	19.84	97.71
2an	A: MeCN/H ₂ O 70:30	35.57	98.26
	B: MeOH/H ₂ O 80:20	29.32	98.91
2ay	A: MeCN/H ₂ O 80:20	30.60	96.96
	B: MeOH/H ₂ O 90:10	21.15	97.57
2ba	A: MeCN/H ₂ O 80:20	20.17	99.91
	B: MeOH/H ₂ O 90:10	18.51	99.90
2bh	A: MeCN/H ₂ O 80:20	25.97	97.75
	B: MeOH/H ₂ O 90:10	21.44	97.85
2bi	A: MeCN/H ₂ O 65:35	20.22	99.55
	B: MeOH/H ₂ O 85:15	14.68	99.61
2bk	A: MeCN/H ₂ O 100:0	17.97	98.46
	B: MeOH/H ₂ O 100:0	13.90	95.26

