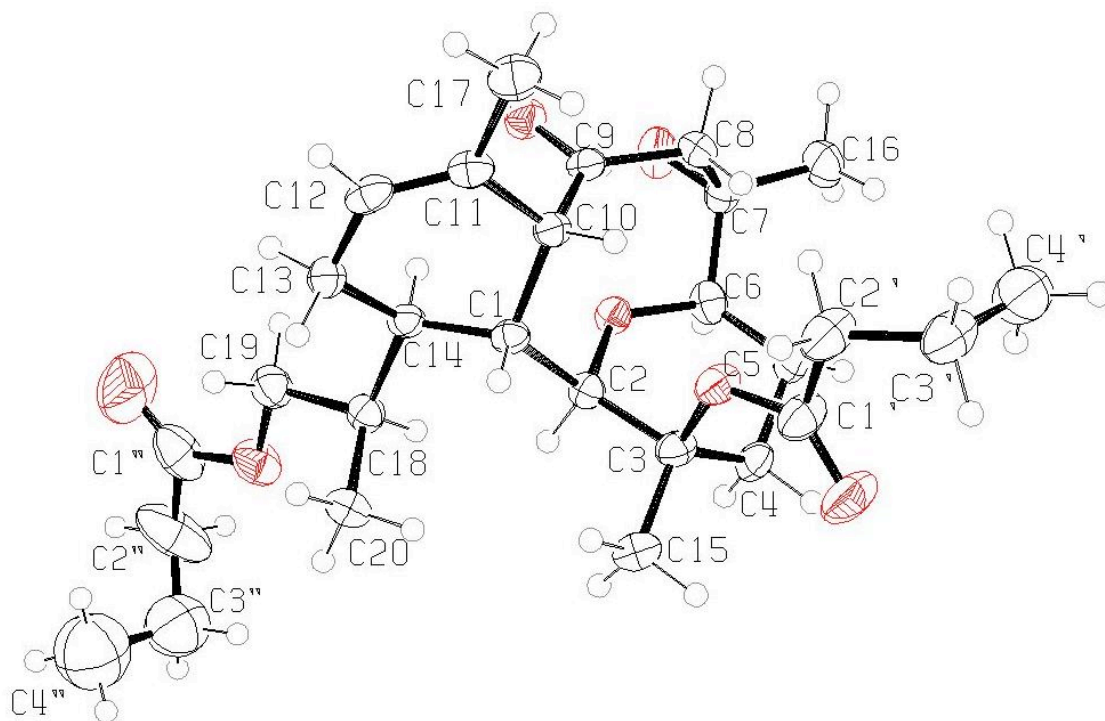


**Details of X-Ray Diffraction Analysis of Australin E (1) and  $^1\text{H}$  and  $^{13}\text{C}$  1D NMR spectra of Australins E-H (1-4)****Details of the X-ray Diffraction Analysis of Australin E (1)****Data Collection**

A colorless needle crystal of  $\text{C}_{28}\text{H}_{44}\text{O}_7$  having approximate dimensions of 0.12 x 0.25 x 0.50 mm was mounted on a glass fiber. All measurements were made on a Bruker X8 APEX II diffractometer with graphite monochromated Mo- $\text{K}\alpha$  radiation.

The data were collected at a temperature of  $-100.0 \pm 0.1^\circ\text{C}$  to a maximum  $2\theta$  value of  $51.86^\circ$ .

Data were collected in a series of  $\phi$  and  $\omega$  scans in  $0.50^\circ$  oscillations with 12.0 second exposures.

The crystal-to-detector distance was 36.00 mm.

**Data Reduction**

Of the 19162 reflections that were collected, 5292 were unique ( $R_{\text{int}} = 0.0231$ ); equivalent reflections were merged. Data were collected and integrated using the Bruker SAINT<sup>1</sup> software package. The linear absorption coefficient,  $\mu$ , for Mo-K $\alpha$  radiation is  $0.084 \text{ mm}^{-1}$ . Data were corrected for absorption effects using the multi-scan technique (SADABS<sup>2</sup>), with minimum and maximum transmission coefficients of 0.87 and 0.99, respectively. The data were corrected for Lorentz and polarization effects.

### Structure Solution and Refinement

The structure was solved by direct methods<sup>3</sup>. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were placed in calculated positions but were not refined. The final cycle of full-matrix least-squares refinement<sup>4</sup> on  $F^2$  was based on 5292 reflections and 327 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.052$$

$$wR2 = [ \Sigma ( w (F_o^2 - F_c^2)^2 ) / \Sigma w(F_o^2)^2 ]^{1/2} = 0.098$$

The standard deviation of an observation of unit weight<sup>5</sup> was 1.05. The weighting scheme was based on counting statistics. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.609 and  $-0.381 \text{ e}^{-}/\text{\AA}^3$ , respectively.

Neutral atom scattering factors were taken from Cromer and Waber<sup>6</sup>. Anomalous dispersion effects were included in  $F_{\text{calc}}$ <sup>7</sup>; the values for  $\Delta f'$  and  $\Delta f''$  were those of Creagh and McAuley<sup>8</sup>. The values for the mass attenuation coefficients are those of Creagh and Hubbell<sup>9</sup>. All refinements were performed using the SHELXTL<sup>10</sup> crystallographic software package of Bruker-AXS.

### References

(1) SAINT. Version 7.03A. Bruker AXS Inc., Madison, Wisconsin, USA. (1997-2003).

(2) SADABS. Bruker Nonius area detector scaling and absorption correction - V2.10, Bruker AXS Inc., Madison, Wisconsin, USA (2003).

(3) SIR97 - Altomare A., Burla M.C., Camalli M., Cascarano G.L., Giacovazzo C., Guagliardi A., Moliterni A.G.G., Polidori G., Spagna R. (1999) J. Appl. Cryst. 32, 115-119.

(4) Least Squares function minimized:

$$\sum w(F_o^2 - F_c^2)^2$$

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

$$[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$$

where:  $N_o$  = number of observations

$N_v$  = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J. ; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) SHELXTL Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA. (1997).

## EXPERIMENTAL DETAILS

### *A. Crystal Data*

Empirical Formula	C <sub>28</sub> H <sub>44</sub> O <sub>7</sub>
Formula Weight	492.63
Crystal Color, Habit	colorless, needle
Crystal Dimensions	0.12 X 0.25 X 0.50 mm
Crystal System	monoclinic
Lattice Type	primitive

Lattice Parameters	a = 9.3711(14) Å
	b = 13.5349(17) Å
	c = 10.9891(17) Å
	$\alpha = 90^\circ$
	$\beta = 99.142(7)^\circ$
	$\gamma = 90^\circ$
	V = 1376.1 (3) Å <sup>3</sup>
Space Group	P 21/b
Z value	2
D <sub>calc</sub>	1.189 · 10 <sup>-3</sup> g/cm <sup>3</sup>
F <sub>000</sub>	536.00
$\mu(\text{MoK}\alpha)$	0.84 cm <sup>-1</sup>

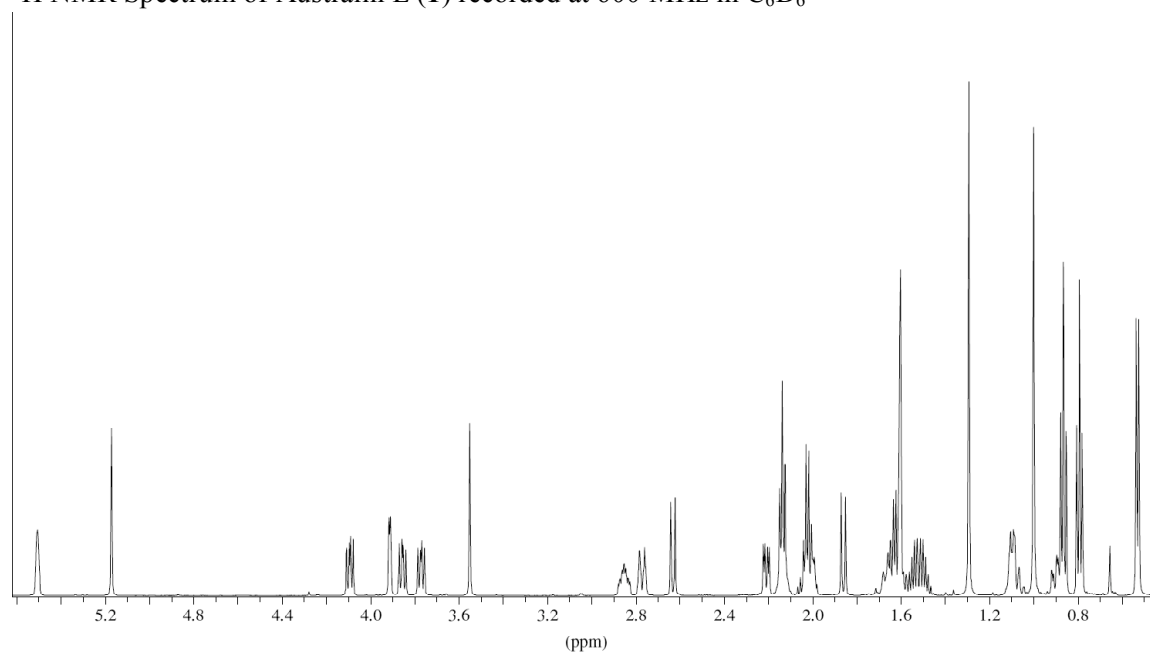
### *B. Intensity Measurements*

Diffractometer	Bruker X8 APEX II
Radiation	MoK $\alpha$ ( $\lambda = 0.71073$ Å)
	Graphite monochromated
Data Images	1444 exposures @ 12 seconds
Detector Position	36 mm
$2\theta_{\text{max}}$	51.86°
No. of Reflections Measured	Total: 19162
	Unique: 5292 ( $R_{\text{int}} = 0.0231$ )
Corrections	Absorption ( $T_{\text{min}} = 0.87$ ; $T_{\text{max}} = 0.99$ )
	Lorentz-polarization

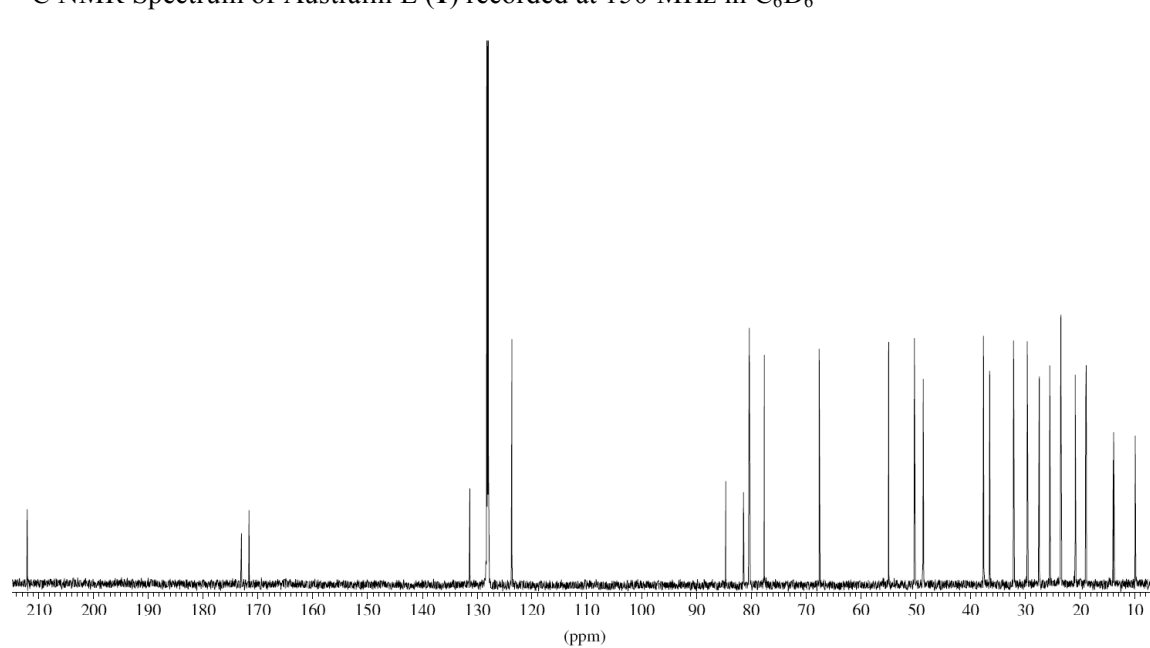
### *C. Structure Solution and Refinement*

Structure Solution	Direct Methods (SHELXL)
Refinement	Full-matrix least-squares on $F^2$
Function Minimized	$\Sigma w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w=1/(\sigma^2(F_o^2)+(0.0465P)^2+ 0.3397P)$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ( $I>0.00\sigma(I)$ )	5292
No. Variables	327
Reflection/Parameter Ratio	16.18
Residuals (refined on $F^2$ , all data): R1; wR2	0.0577; 0.1444
Goodness of Fit Indicator	1.058
No. Observations ( $I>2.00\sigma(I)$ )	4752
Residuals (refined on F): R1; wR2	0.0502; 0.1374
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.609 e <sup>-</sup> /Å <sup>3</sup>
Minimum peak in Final Diff. Map	-0.381 e <sup>-</sup> /Å <sup>3</sup>

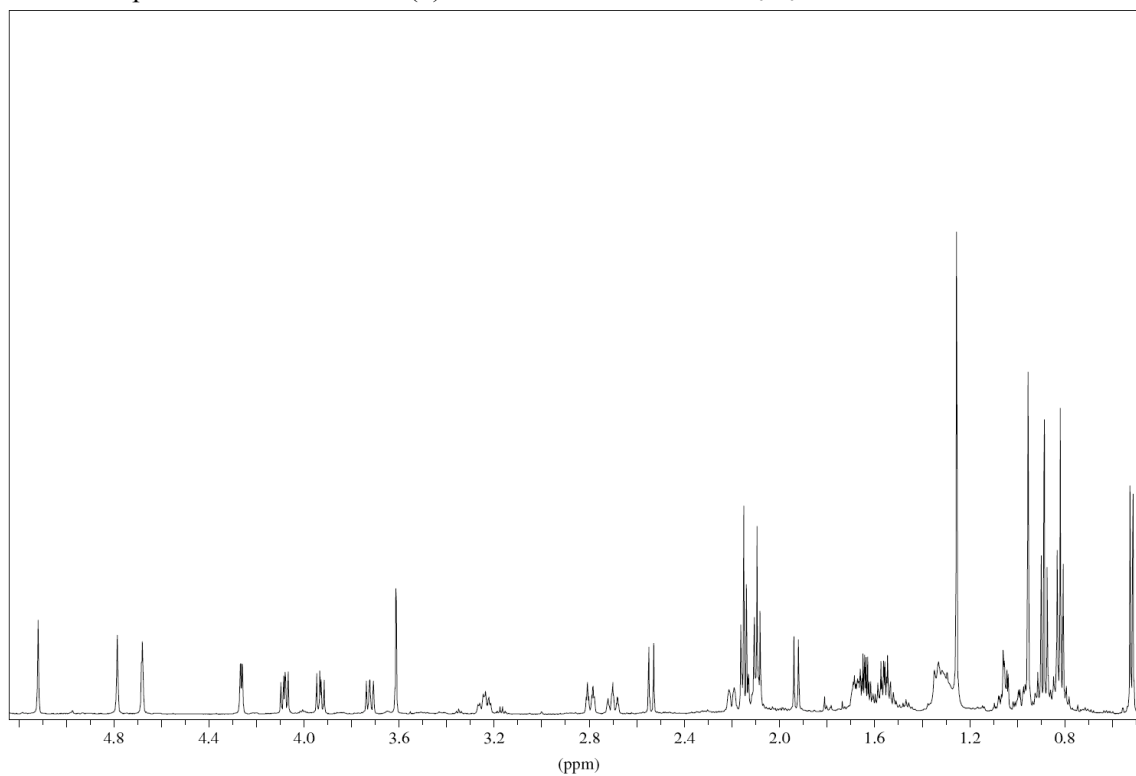
$^1\text{H}$  NMR Spectrum of Australin E (**1**) recorded at 600 MHz in  $\text{C}_6\text{D}_6$



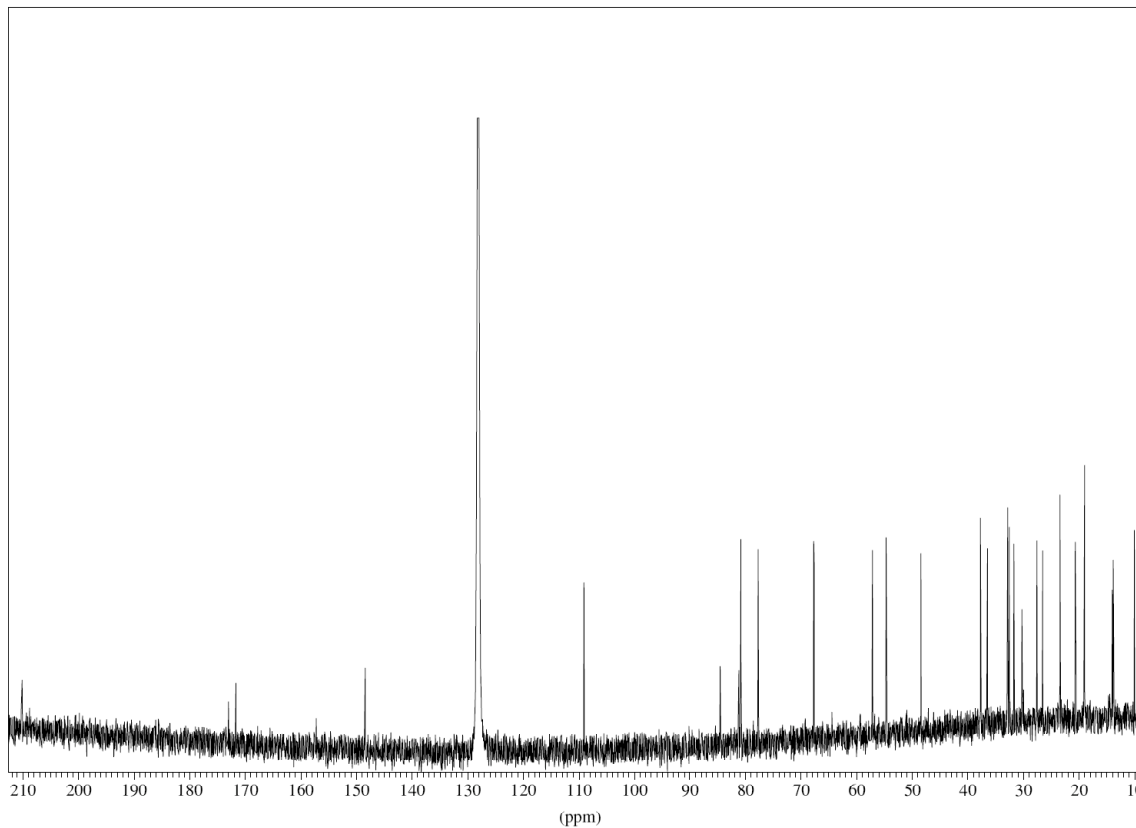
$^{13}\text{C}$  NMR Spectrum of Australin E (**1**) recorded at 150 MHz in  $\text{C}_6\text{D}_6$



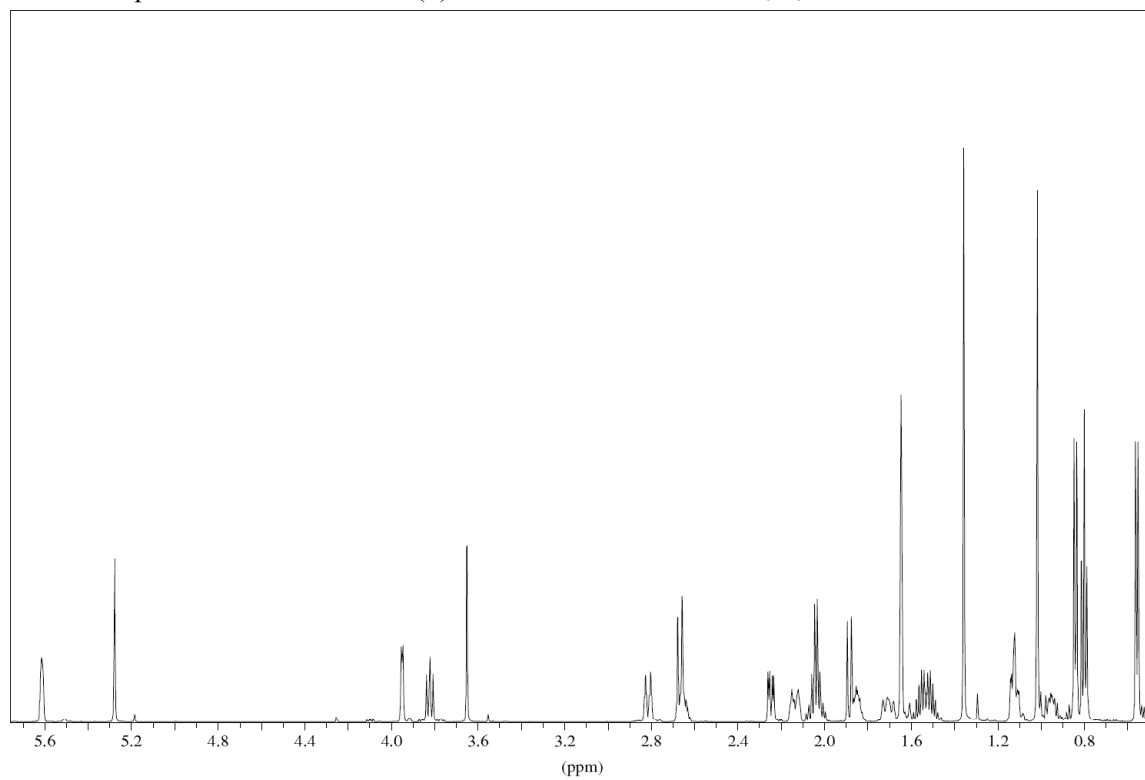
$^1\text{H}$  NMR Spectrum of Australin F (**2**) recorded at 600 MHz in  $\text{C}_6\text{D}_6$



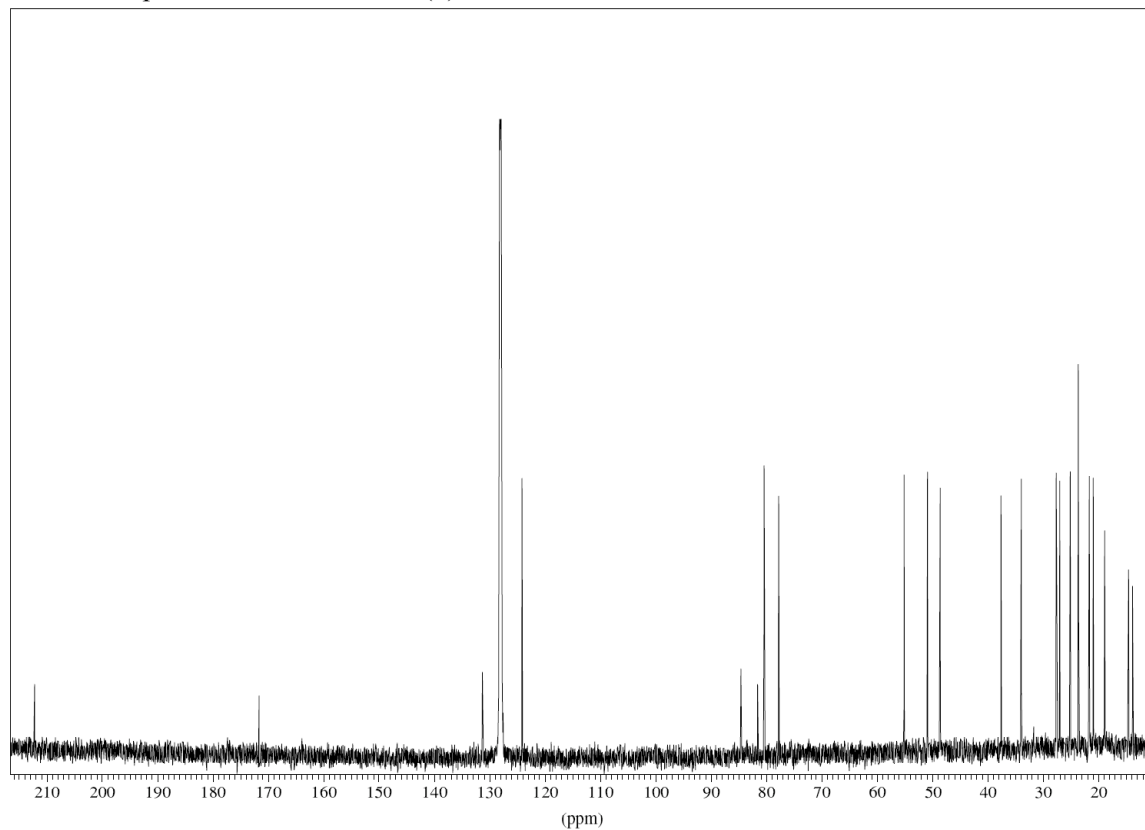
$^{13}\text{C}$  NMR Spectrum of Australin F (**2**) recorded at 150 MHz in  $\text{C}_6\text{D}_6$



$^1\text{H}$  NMR Spectrum of Australin G (**3**) recorded at 600 MHz in  $\text{C}_6\text{D}_6$

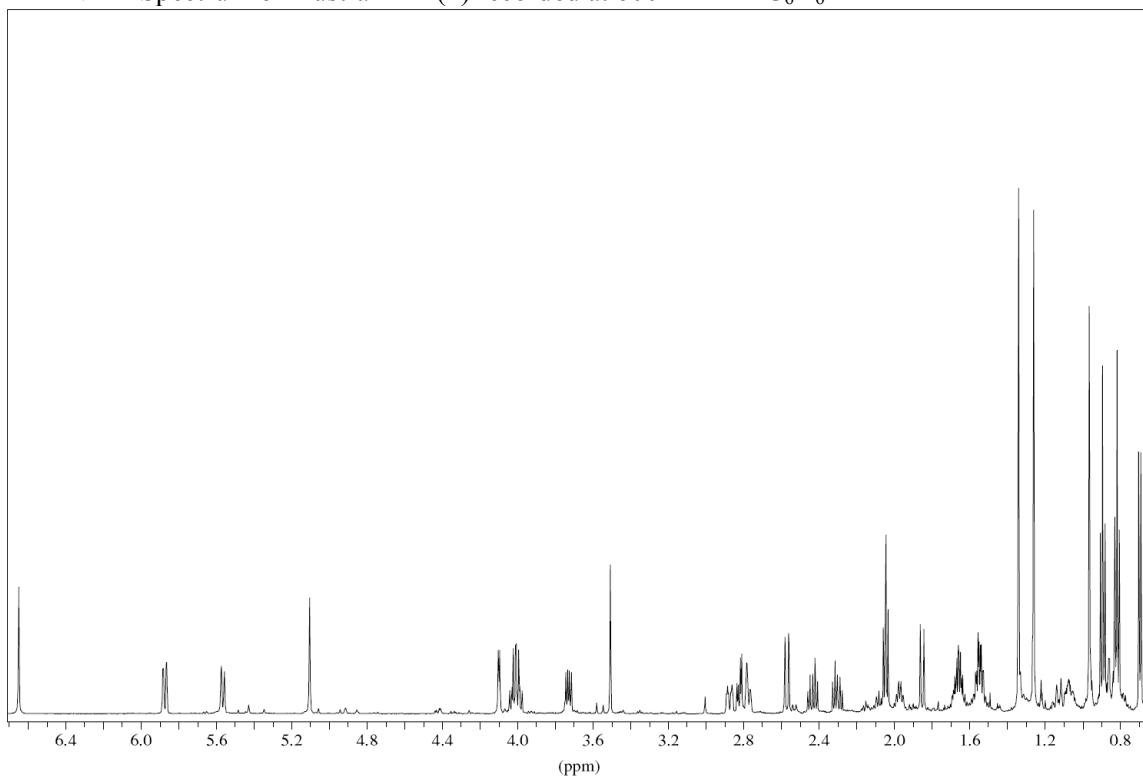


$^{13}\text{C}$  NMR Spectrum of Australin G (**3**) recorded at 150 MHz in  $\text{C}_6\text{D}_6$





$^1\text{H}$  NMR Spectrum of Australin H (**4**) recorded at 600 MHz in  $\text{C}_6\text{D}_6$



$^{13}\text{C}$  NMR Spectrum of Australin H (**4**) recorded at 150 MHz in  $\text{C}_6\text{D}_6$

