**SUPPORTING INFORMATION**

# Novel triterpenoid derivatives from *Eucomis bicolor* Bak. (Hyacinthaceae: Hyacinthoideae)

Jaspreet K. Sihra,[a] Moses K. Langat,[a,b] Neil R. Crouch,[b,c] Jean-Marc Nuzillard,[d] Bertrand Plainchont[d] and Dulcie A. Mulholland,*[,a,b]

[a]*Department of Chemistry, Faculty of Engineering and Physical Sciences, University of Surrey, Guildford, GU2 7XH, Surrey, UK: d.mulholland@surrey.ac.uk. Tel +44 (0)1483 686751*
[b]*School of Chemistry and Physics, University of KwaZulu-Natal, Durban, 4041, South Africa*
[c]*Biodiversity Economy Unit, South African National Biodiversity Institute, PO Box 52099, Berea Road 4007, Durban, South Africa.*
[d]*Pharmacognosy Laboratory, University of Reims, Moulin de la House, BP 1039, 51687 Reims Cedex 2, France.*

**CONTENTS:** **Page**

## S.1 Structures of all compounds isolated from *Eucomis bicolor.*

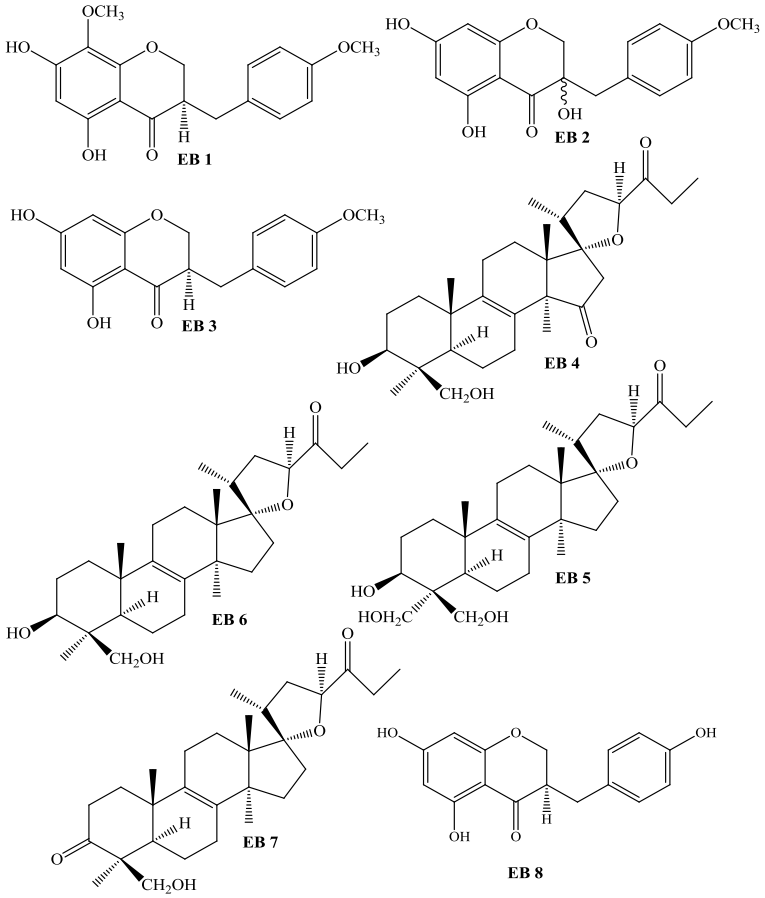



**Figure S1.1 Known compounds isolated from the dichloromethane extract (EB 1-7) and methanol extract (EB 8) of *Eucomis bicolor***

**EB 1** = (*R*)-5,7-dihydroxy-8-methoxy-3-(4'-methoxybenzyl)-4-chromanone; **EB 2** =3,5,7-trihydroxy-3-(4'-methoxybenzyl)-4-chromanone; **EB 3** = (*R*)-5,7-dihydroxy-3-(4'-methoxybenzyl)-4-chromanone; **EB 4** = eucosterol; **EB 5** = (17*S*,23*S*)-23,17-epoxy-3β,28,29-trihydroxy-27-norlanost-8-en-24-one; **EB 6** = 15-deoxoeucosterol; **EB 7** = 3-dehydro-15-deoxoeucosterol; **EB 8 =** (*R*)-5,7-dihydroxy-3-(4'-hydroxybenzyl)-4-chromanone.

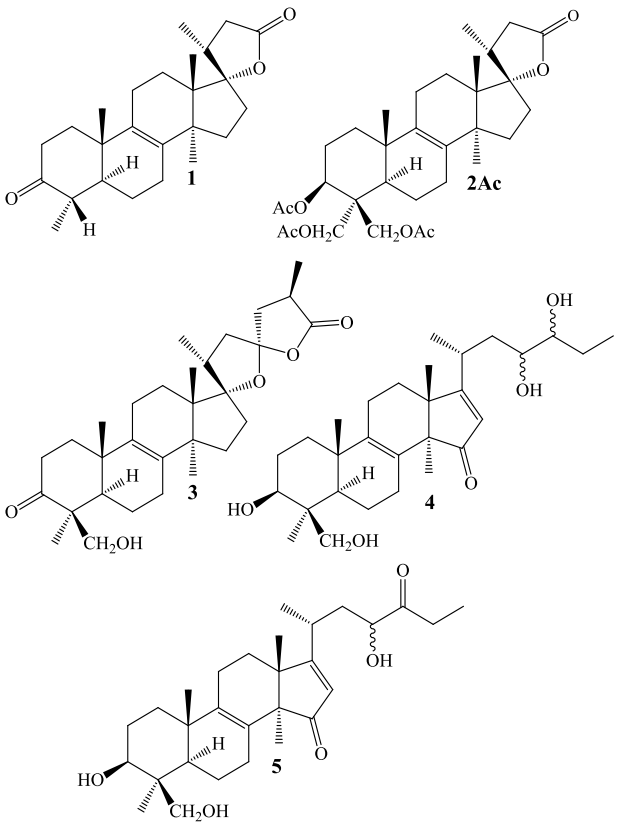



**Figure S 1.2: New compounds isolated from the dichloromethane extract of *Eucomis bicolor.***

**1** = (17*S*)-24,25,26,27,29-pentanor-3-oxolanost-8-en-23,17-olide; **2Ac** = (17*S*)-24,25,26,27-tetranor-3β,28,29-triacetoxylanost-8-en-23,17-olide; **3** = (17*S*,23*S*)-23,17-epoxy-29-hydroxy-3-oxolanost-8-en-27,23-olide; **4** = 3β,23*S*,24ε,29-tetrahydroxy-27-norlanosta-8,16-dien-15-one; **5** = 3β,23*S*,29-trihydroxy-27-norlanosta-8,16-diene-15,24-dione.

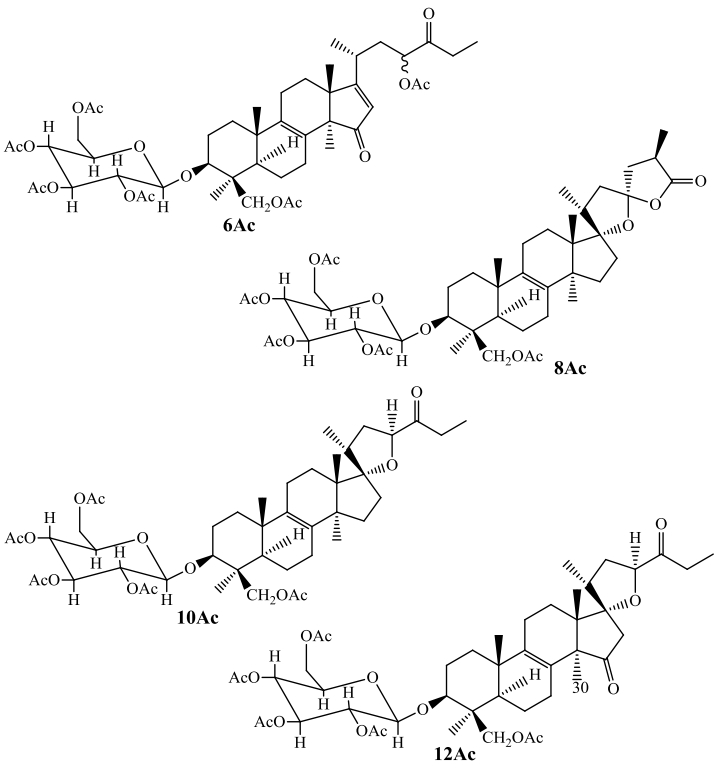



**Figure S 1.3: Monosaccharide triterpenoid derivaives isolated from the methanol extract of *Eucomis bicolor* (after acetylation of a complex mixture)**

**6Ac =** 23*S*,29-diacetoxy-3β-[2',3',4',6'-tetra-*O*-acetyl-*O*-β-D-glucopyranoside]-27-norlanosta-8,16-diene-15,24-dione; **8Ac** = (17*S*,23*S*)-29-acetoxy-23,17-epoxy-3β-[2',3',4',6'-tetra-*O*-acetyl-*O*-β-D-glucopyranoside]-lanost-8-en-27,23-olide; **10Ac** = (17*S*,23*S*)-29-acetoxy-23,17-epoxy-3β-[2',3',4',6'-tetra-*O*-acetyl-*O*-β-D-glucopyranoside]-27-norlanost-8-en-24-one; **12Ac** = (17*S*,23*S*)-29-acetoxy-23,17-epoxy-3β-[2',3',4',6'-tetra-*O*-acetyl-*O*-β-D-glucopyranoside]-27-norlanost-8-ene-15,24-dione.

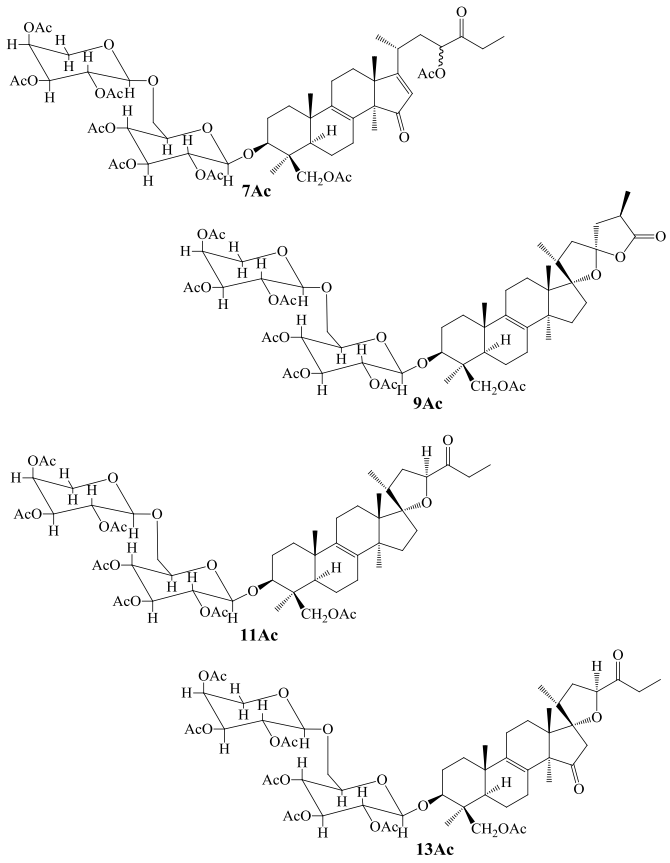



**Figure S 1.3: Disaccharide triterpenoid derivatives isolated from the methanol extract of *Eucomis bicolor* (after acetylation of a complex mixture).**

**7Ac** = 23*S*,29-diacetoxy-3β-[2',3',4'-tri-*O*-acetyl-*O*-β-D-glucopyranoside-(1''→6')-2'',3'',4''-tri-*O*-acetyl-β-D-arabinopyranosyl]-27-norlanosta-8,16-diene-15,24-dione); **9Ac** = (17*S*,23*S*)-29-acetoxy-23,17-epoxy-3β-[2',3',4'-tri-*O*-acetyl-*O*-β-D-glucopyranoside-(1''→6')-2'',3'',4''-tri-*O*-acetyl-*O*-β-D-arabinopyranosyl]-lanost-8-en-27,23-olide); **11Ac** = (17*S*,23*S*)-29-acetoxy-23,17-epoxy-3β-[2',3',4'-tri-*O*-acetyl-*O*-β-D-glucopyranoside-(1''→6')-2'',3'',4''-tri-*O*-acetyl-*O*-β-D-arabinopyranosyl]-27-norlanost-8-en-24-one); **13Ac** = (17*S*,23*S*)-29-acetoxy-23,17-epoxy-3β-[2',3',4'-tri-*O*-acetyl-*O*-β-D-glucopyranoside-(1''→6')-2',3',4'-tri-*O*-acetyl-*O*-β-D-arabinopyranosyl]-27-norlanost-8-ene-15,24-dione.

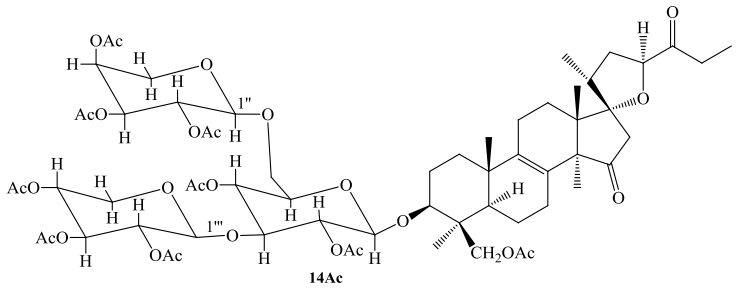
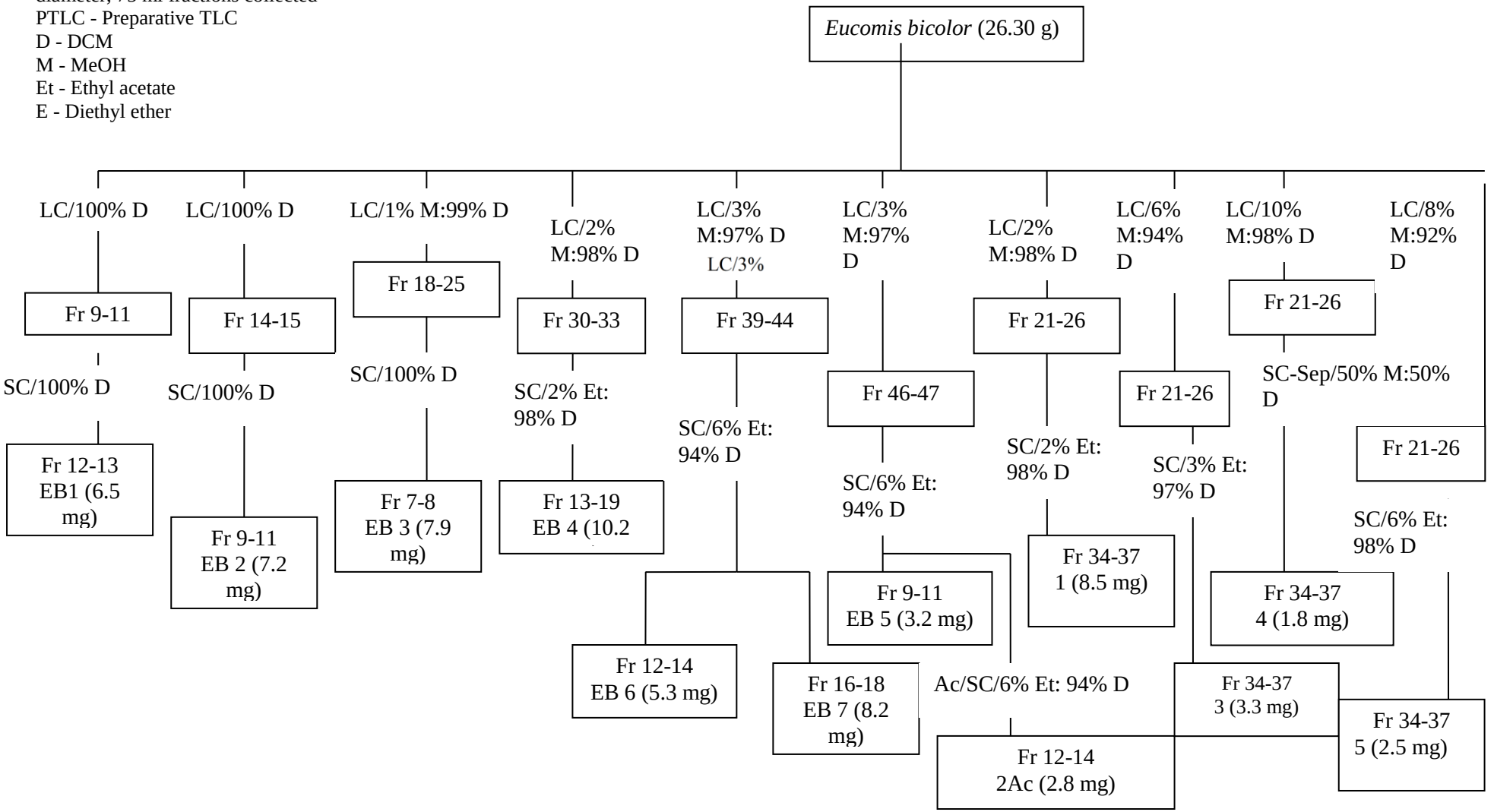



**Figure S 1.4: Trisaccharide triterpenoid derivative isolated from the methanol extract of *Eucomis bicolor* after acetylation.**

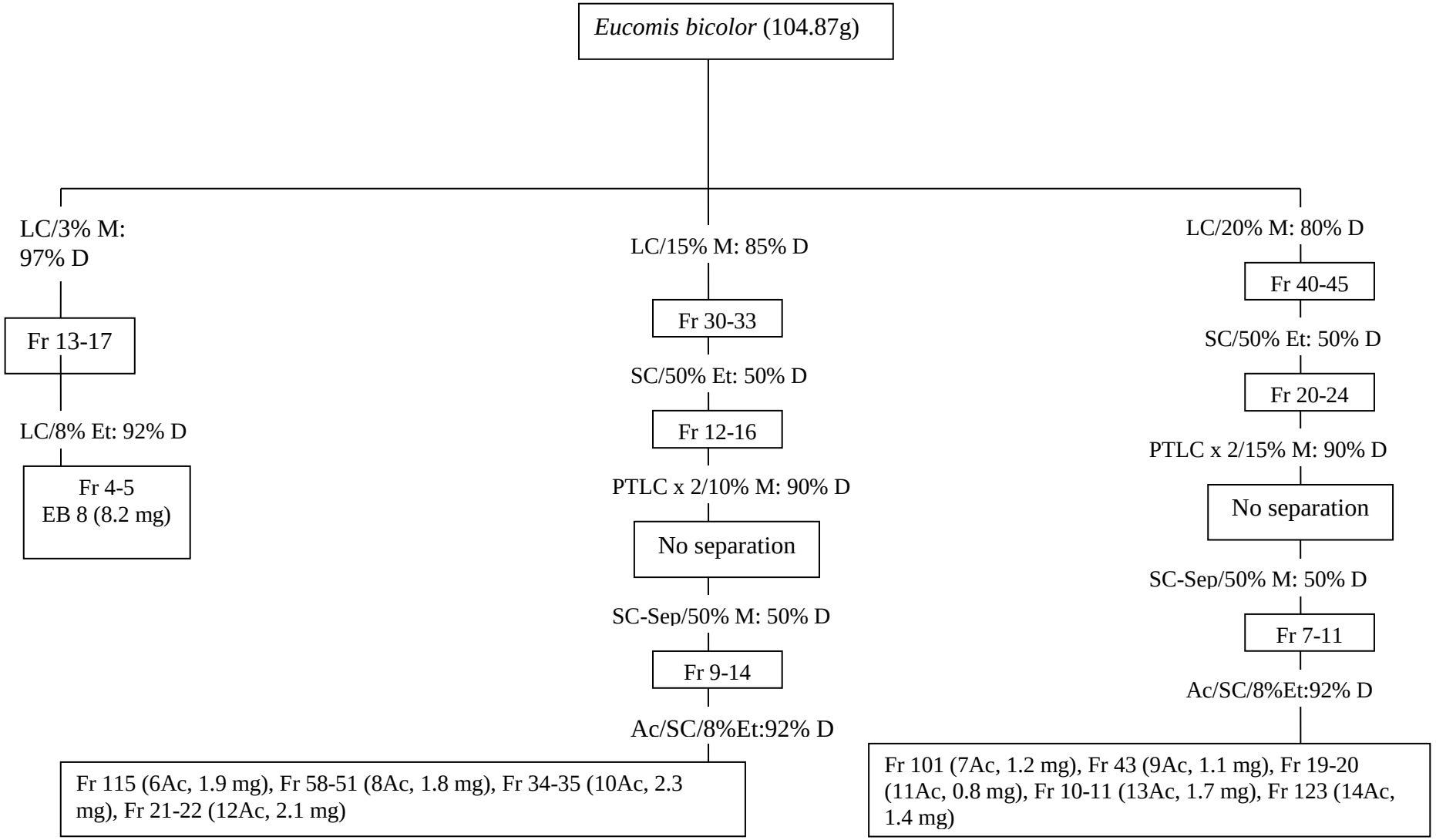
**14Ac** = (17*S*,23*S*)-29-acetoxy-23,17-epoxy-3β-[2',3',4'-tri-*O*-acetyl-*O*-β-D-glucopyranoside-(6'→1'')-2'',3'',4''-tri-*O*-acetyl-*O*-β-D-arabinopyranosyl--(3'→1''')-2''',3''',4'''-tri-*O*-acetyl-*O*-β-D-xylopyranosyl]-27-norlanost-8-ene-15,24-dione.

SC - small column-silica, 1 cm diameter, 2 ml fractions collected
SC-Sephedex – small column-sephedex, 1cm diameter, 2 ml fractions collected
LC - large column-silica, 5 cm diameter, 75 ml fractions collected
PTLC - Preparative TLC
D - DCM
M - MeOH
Et - Ethyl acetate
E - Diethyl ether

**S.2.1: Flow chart of the compounds isolated from the dichloromethane extract of *Eucomis bicolor* Bak.**



**S. 2.2 Flow chart of the compounds isolated from the methanol extract of *Eucomis bicolor* Bak.**



**S.3 Details for the LSD input file**

```
; The LSD input file starts below the row of stars.
; The lines must be copied in a text editor
; such as textedit (Mac), gedit (Linux) or notepad++ (Windows)
; and saved as simple text.

; ***************************

; Compound 1
ELIM 1 4 ; 1 HMBC correlation can be 4J

; LSD atom numbering (Hs excepted)
MULT 1 C 2 0 ; Atom 1 is a C, is hybridized sp2, and has 0 H atom attached
MULT 2 C 2 0
MULT 3 C 2 0
MULT 4 C 2 0
MULT 5 C 3 0
MULT 6 C 3 0
MULT 7 C 3 1
MULT 8 C 3 0
MULT 9 C 3 1
MULT 10 C 3 1
MULT 11 C 3 2
MULT 12 C 3 2
MULT 13 C 3 2
MULT 14 C 3 2
MULT 15 C 3 0
MULT 16 C 3 2
MULT 17 C 3 3
MULT 18 C 3 2
MULT 19 C 3 2
MULT 20 C 3 2
MULT 21 C 3 2
MULT 22 C 3 3
MULT 23 C 3 3
MULT 24 C 3 3
MULT 25 C 3 3
MULT 26 O 2 0
MULT 27 O 2 0
MULT 28 O 3 0

; the SHIX commands are ignored by LSD
; and were added as documentation
SHIX 1  213.1 ; The chemical shift of C-1
is 213.1 ppm
SHIX 2  177.1
SHIX 3  135.8
SHIX 4  133.1
SHIX 5   98.4
SHIX 6   50.9
SHIX 7   49.7
SHIX 8   49.0
SHIX 9   45.2
SHIX 10  41.9
SHIX 11  39.3
SHIX 12  39.2
SHIX 13  38.0
SHIX 14  37.1
SHIX 15  36.8
SHIX 16  31.5
SHIX 17  25.9
SHIX 18  25.4
SHIX 19  24.7
SHIX 20  22.1
SHIX 21  21.5
SHIX 22  18.2
SHIX 23  17.9
SHIX 24  17.7
SHIX 25  11.7

; H atom numbering
HSQC 1 1 ; see below about HMBC (11
18 21) 5
HSQC 2 2 ; idem
HSQC 3 3 ; idem
HSQC 4 4 ; idem
HSQC 5 5 ; idem
HSQC 6 6 ; idem
HSQC 7 7
HSQC 8 8 ; idem
HSQC 9 9
HSQC 10 10
HSQC 11 11
HSQC 12 12
HSQC 13 13
```

```
HSQC 14 14
HSQC 15 15 ; idem
HSQC 16 16
HSQC 17 17
HSQC 18 18 ; C-18 is bound to H-18 (even though there are two of them)
HSQC 19 19
HSQC 20 20
HSQC 21 21
HSQC 22 22
HSQC 23 23
HSQC 24 24
HSQC 25 25


COSY 7 9 ; H-7 and H-9 correlate in the COSY spectum (strong intensity, 3J)
COSY 7 20
COSY 10 12
COSY 13 14
COSY 23 10
COSY 25 9

BOND 1 26 ; C=O ketone
BOND 2 27 ; C=O lactone
BOND 2 28 ; C-O lactone
BOND 5 28 ; O-C lactone
BOND 3 4 ; C=C

HMBC 1 13 ; C-1 correlates with H-13 in the HMBC spectrum
HMBC 1 9
HMBC 1 14
HMBC 1 25

HMBC 2 12
HMBC 2 10
HMBC 2 12

HMBC 3 20 ; low intensity
HMBC 3 16 ; idem
HMBC 3 17

HMBC 4 14 ; idem
HMBC 4 19 ; idem
HMBC 4 24

; C-5 correlates with H-11 or H-18 or H-21
; This should be written HMBC 5 (11 18 21).
; The LSD syntax does not allow it, sorry.
; This is equivalent to HMBC (11 18 21) 5
; because of the numbering of Hs according to Cs using HSQC data.
; this forces to "invent" an H-5 that does not exist
; and explains why a few quaternary Cs (among which C-5)
; are declared in the HSQC section.
Therefore:
HMBC (11 18 21) 5
HMBC 5 12
HMBC 5 16
HMBC 5 23
HMBC 5 22

HMBC (11 18 21) 6
HMBC 6 16
HMBC 6 19
HMBC 6 17
HMBC 6 22

HMBC 7 9
HMBC (14 18) 7
HMBC 7 20
HMBC 7 24
HMBC 7 25

HMBC 8 19
HMBC (11 18 21) 8
HMBC 8 19
HMBC (16 20) 8
HMBC 8 17
HMBC 8 22

HMBC 9 13
HMBC (16 20) 9
HMBC 9 7
HMBC 9 25

HMBC 10 12
HMBC (11 18 21) 10
HMBC 10 12
HMBC 10 23

HMBC 11 16

HMBC 12 10
HMBC 12 23
```

HMBC 13 14

HMBC 14 13
HMBC 14 24

HMBC 15 13
HMBC 15 14
HMBC 15 20
HMBC 15 7
HMBC 15 24

HMBC (11 18 21) 16
HMBC 16 17

HMBC 17 16

HMBC 18 20

HMBC 19 22

HMBC 20 9
HMBC (14 18) 20
HMBC 20 7

HMBC 21 19

HMBC 22 19

HMBC 23 12
HMBC 23 10

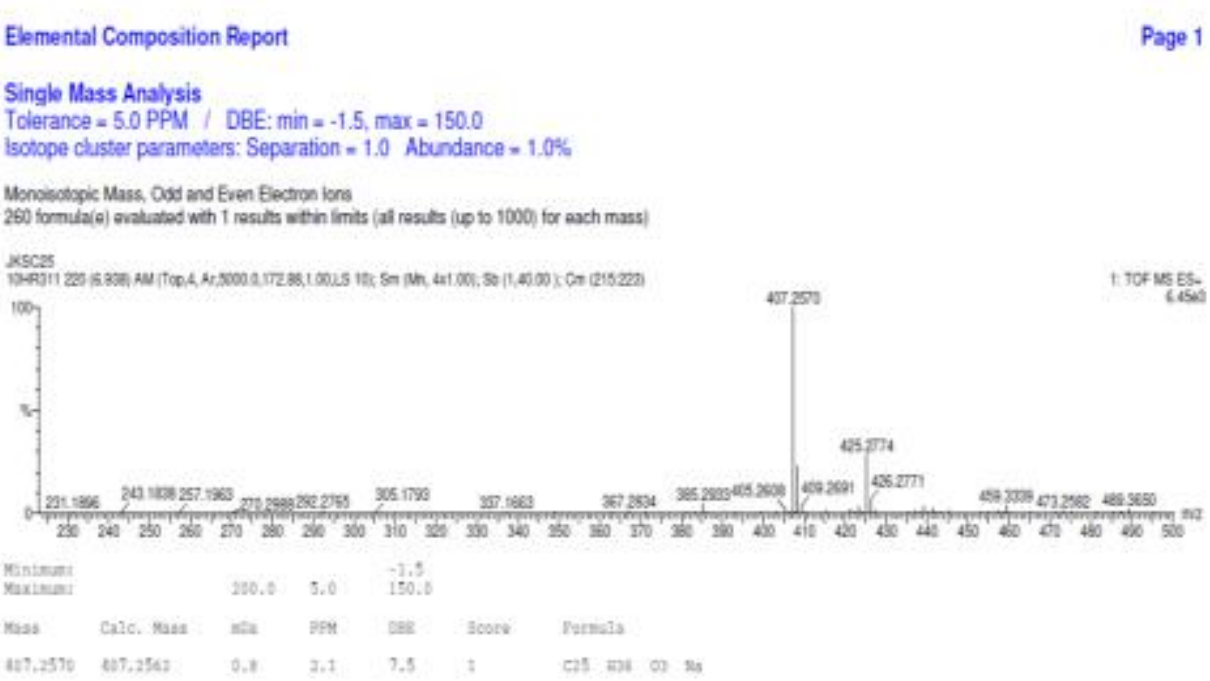
HMBC 24 14
HMBC 24 7

HMBC 25 13
HMBC 25 9

QUAT L1 ; L1 is the list of quaternary carbons
LIST L2 17 22 24 ; L2 is the list of the indexes of methyl singlets
PROP L2 1 L1 ; each singlet methyl has exactly one quaternary carbon as neighbor

CH L3 ; L3 is the list of methine carbons
LIST L4 23 25 ; L4 is the list of the indexes of methyl doublets
PROP L4 1 L3 ; each doublet methyl has exactly one methine carbon as neighbor

## S.4 NMR spectra for compounds 1-5 and 6Ac-14Ac. (See Figures S1.1, S 1.2, S 1.3 and S 1.4 for names and structures).



Elemental Composition Report Page 1

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 150.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

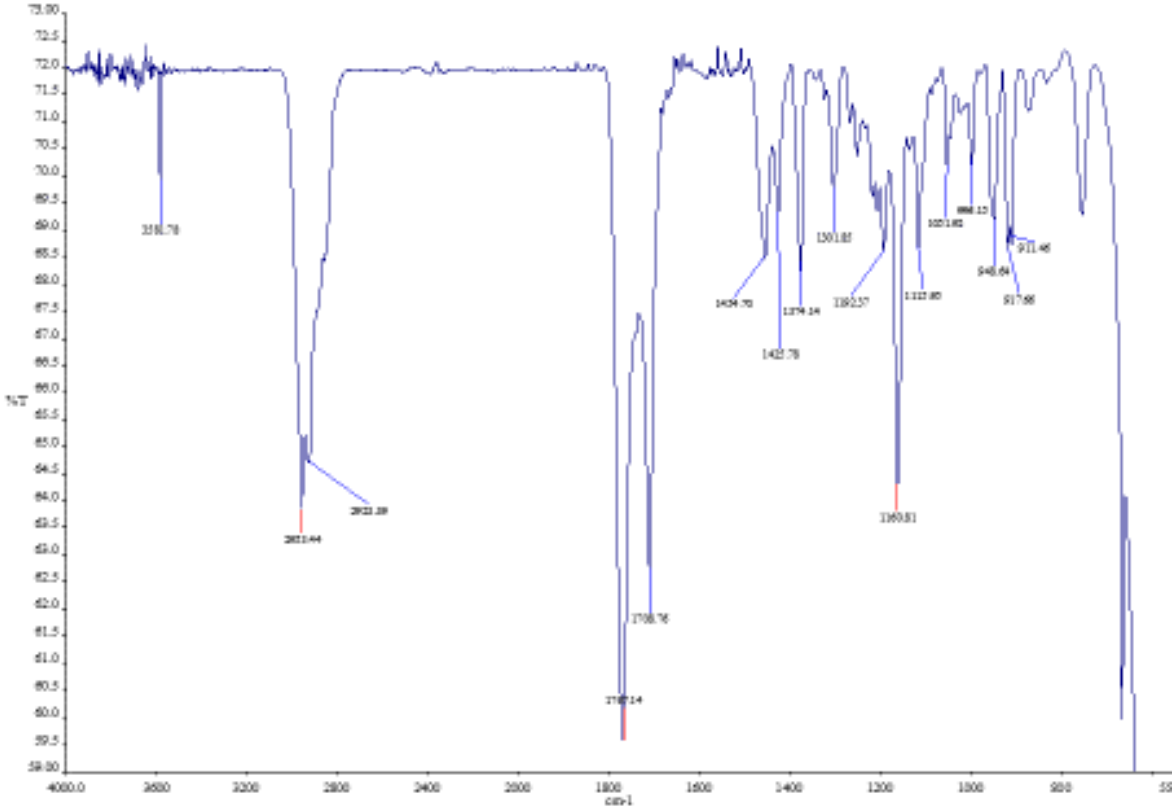
Monoisotopic Mass, Odd and Even Electron Ions
260 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

JKSC25
10HR311 220 (6.938) AM (Top,4, Ar,5000.0,172.86,1.00,LS 10); Sm (Mn, 4x1.00); Sb (1,40.00 ); Cm (215:223) 1: TOF MS ES+ 6.45e3

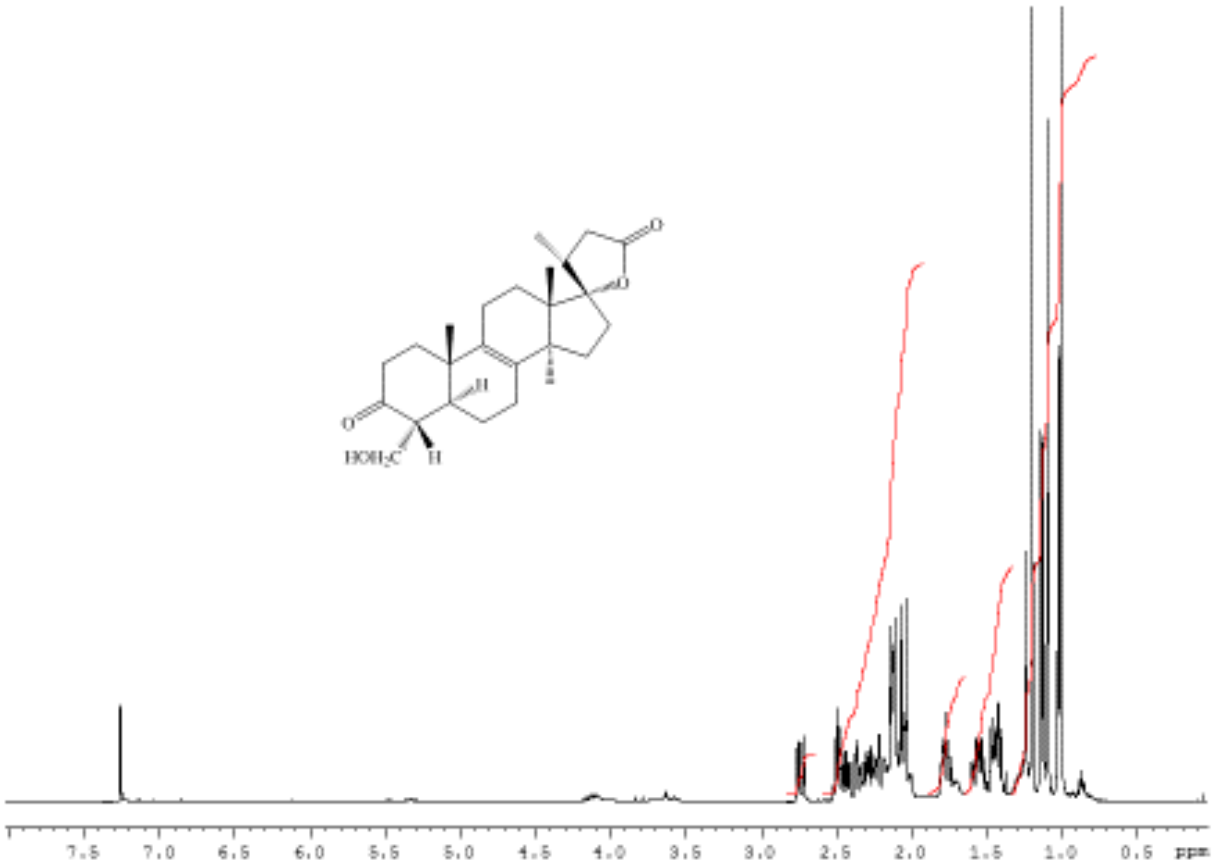


| | | | | | | |
|---|---|---|---|---|---|---|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 200.0 | 5.0 | 150.0 | | |
| Mass | Calc. Mass | mDa | PPM | DBE | Score | Formula |
| 407.2570 | 407.2562 | 0.8 | 2.1 | 7.5 | 1 | C25 H36 O3 Na |

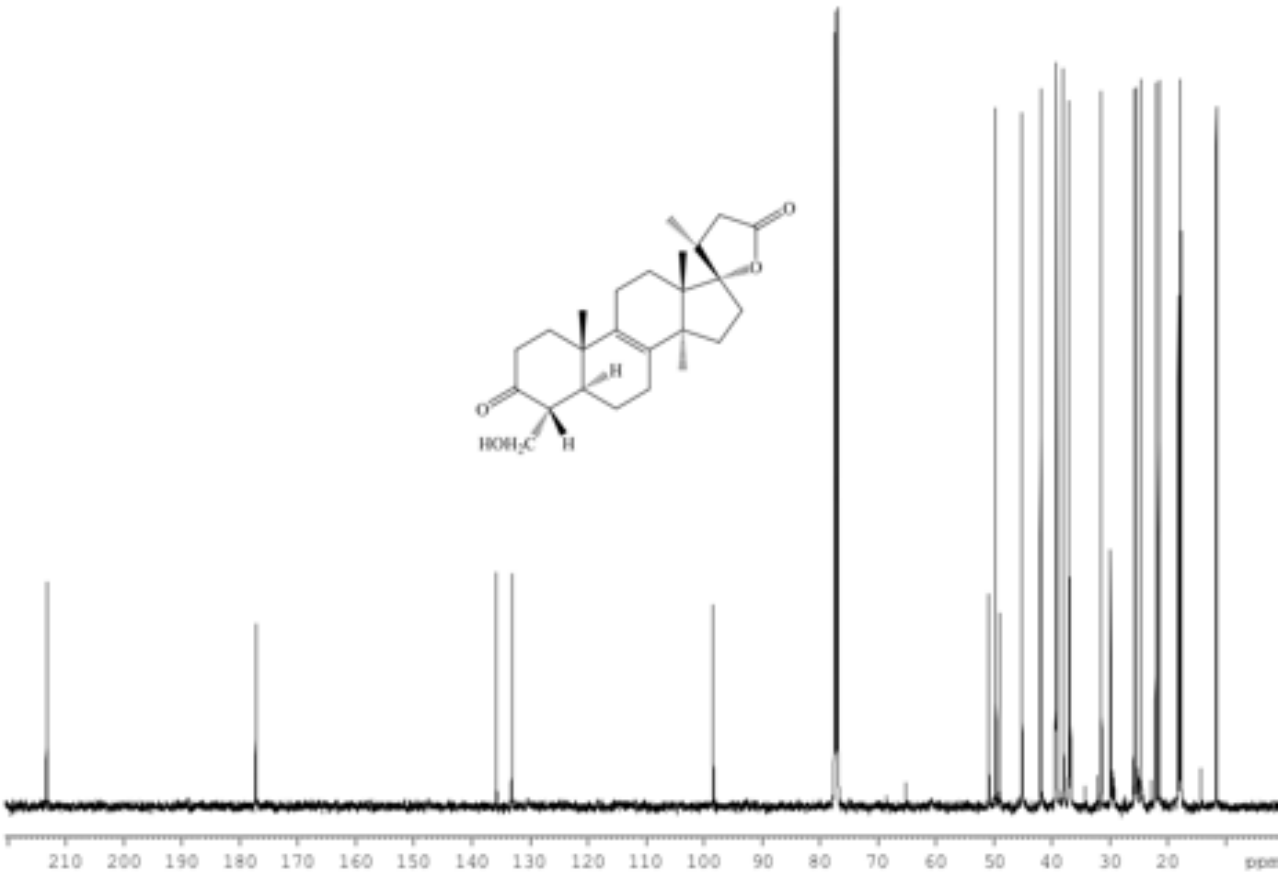
**Spectrum S.3.1.1: Mass spectrum for compound 1**



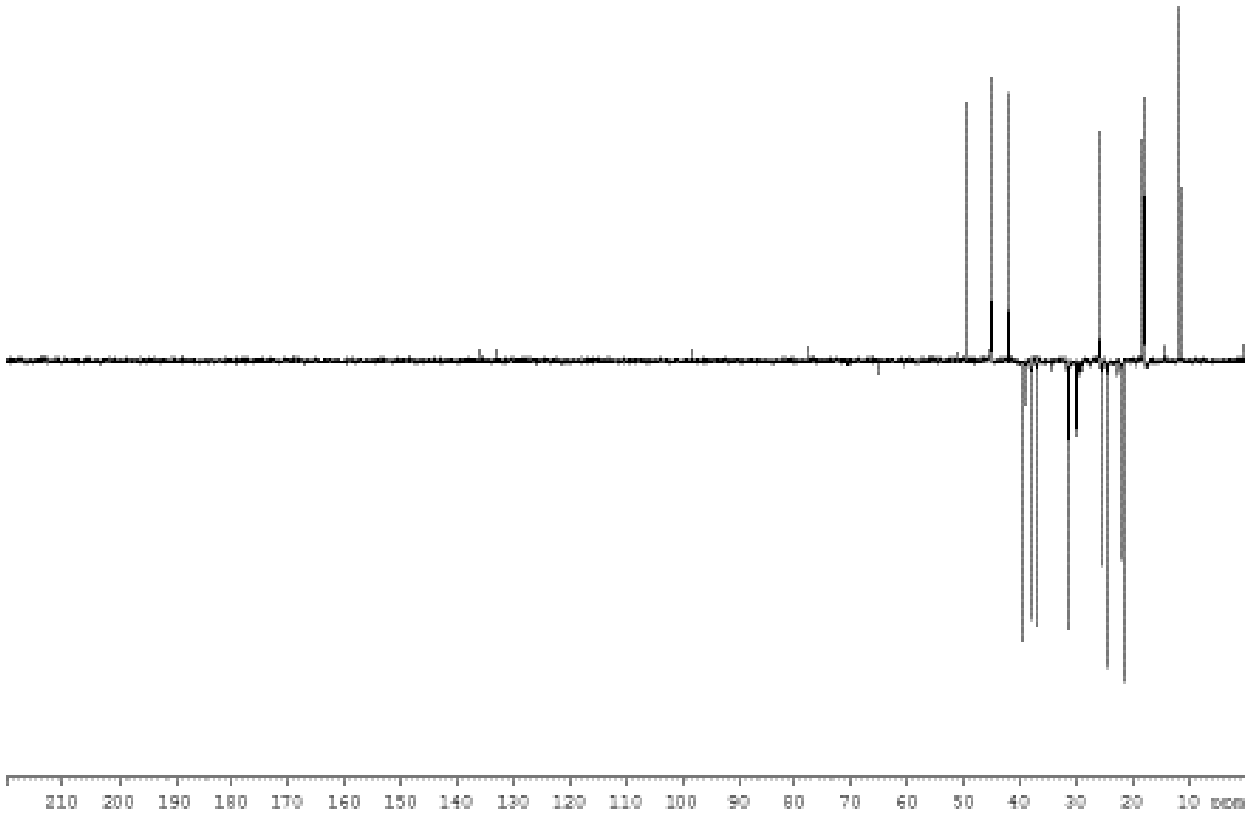
Spectrum S.3.1.2: FTIR spectrum for compound 1



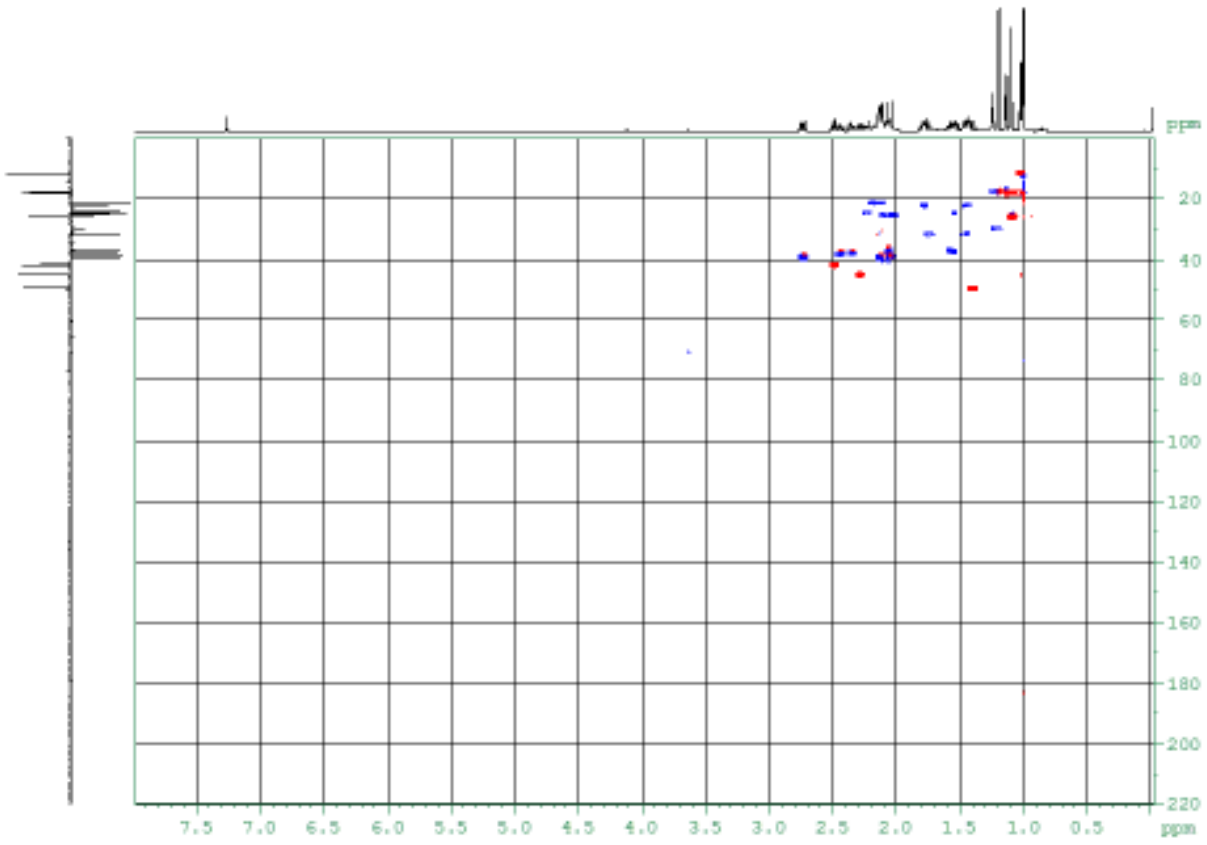

Spectrum S.3.1.3: $^1$H NMR spectrum for compound 1 in $CDCl_3$



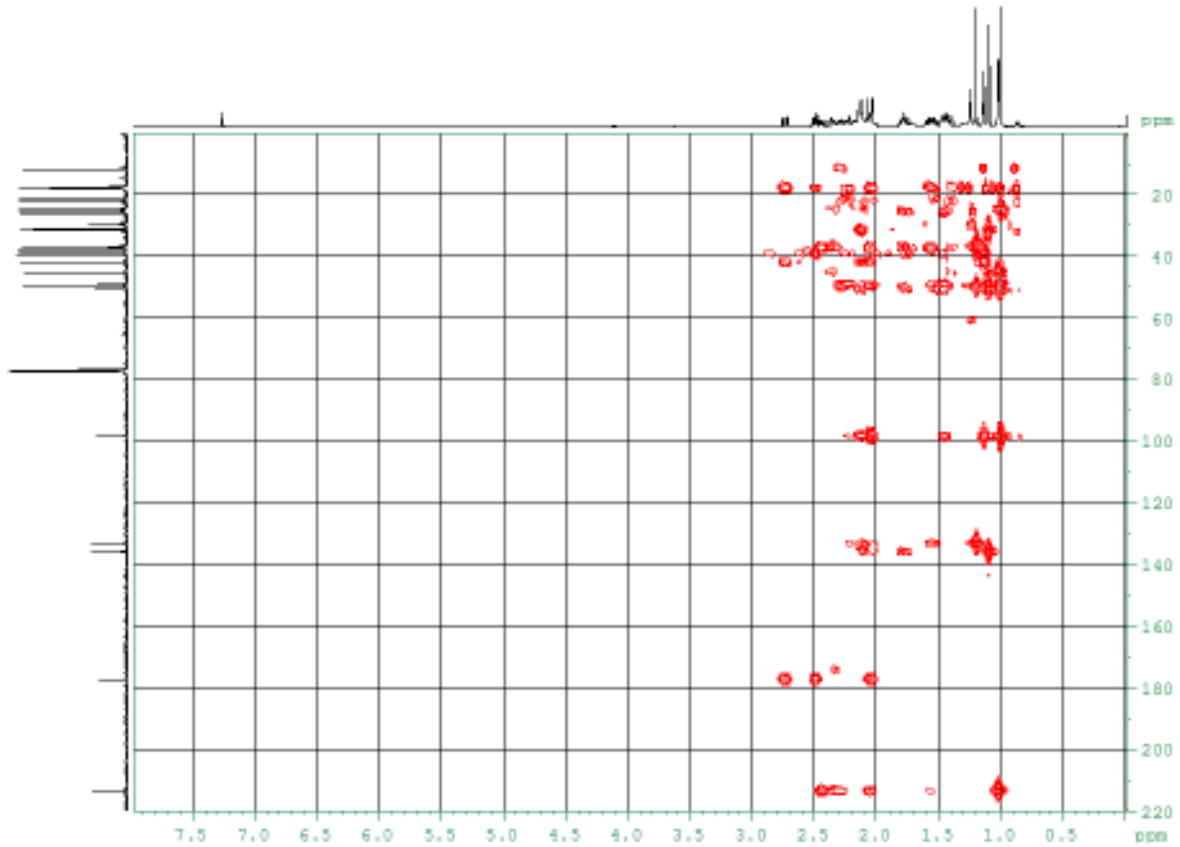

**Spectrum S.3.1.4: $^{13}C$ NMR spectrum for compound 1 in $CDCl_3$**



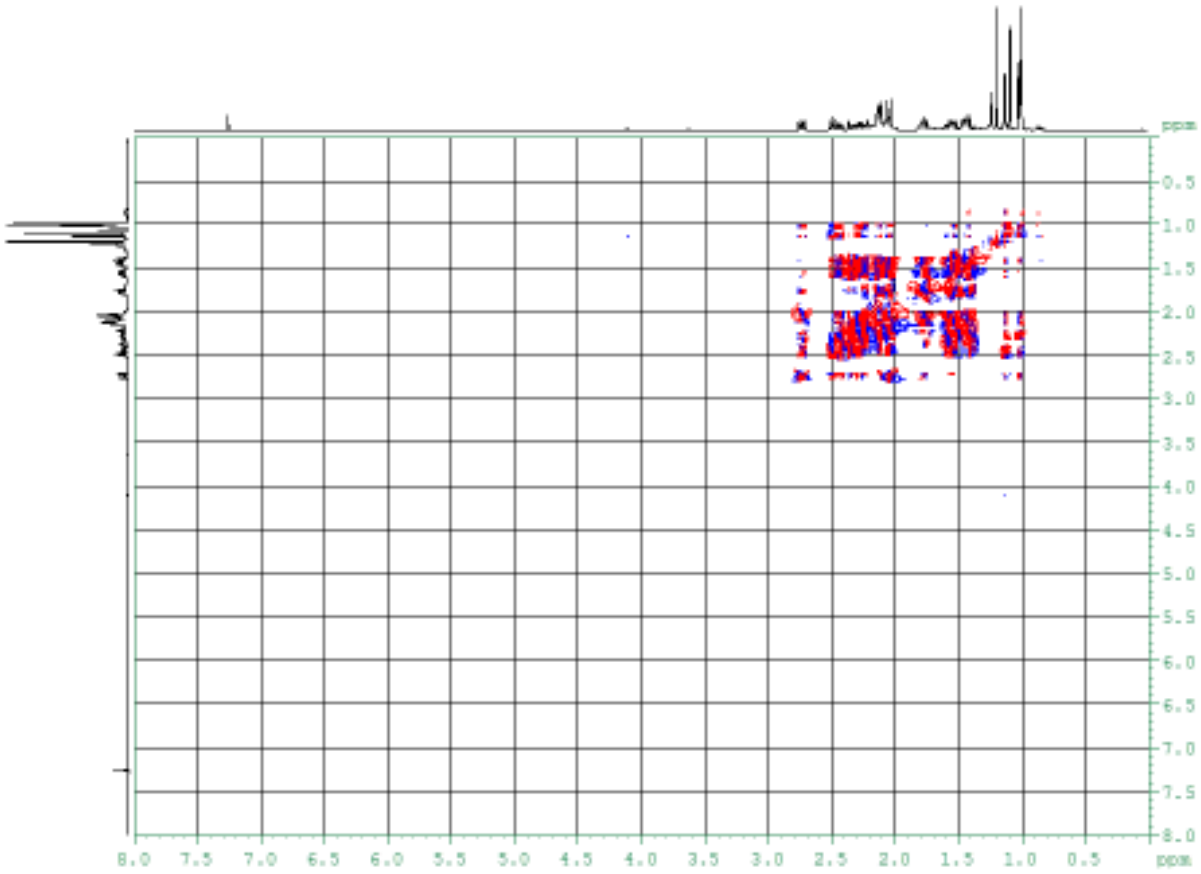

**Spectrum S.3.1.5: DEPT spectrum for compound 1 in $CDCl_3$**



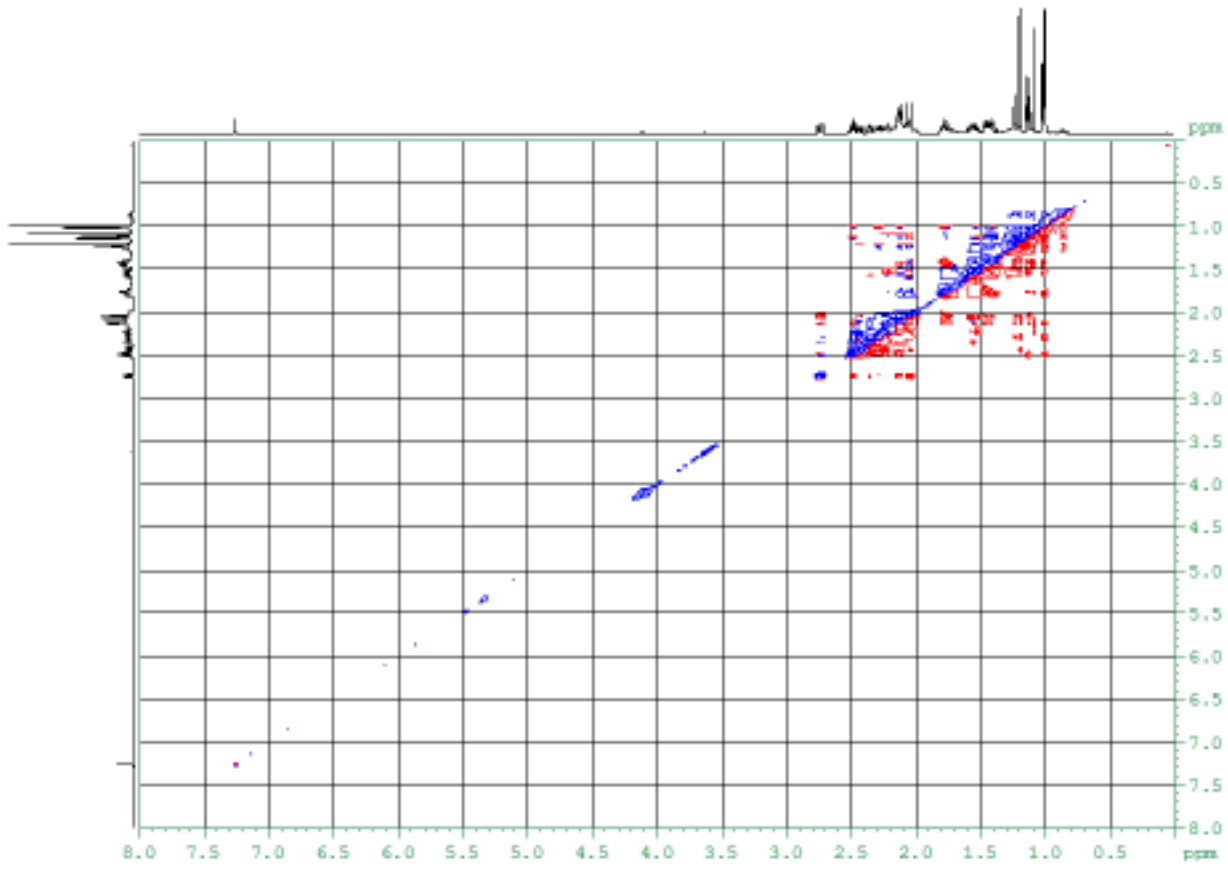

**Spectrum S.3.1.6: HSQCDEPT spectrum for compound 1 in $CDCl_3$**



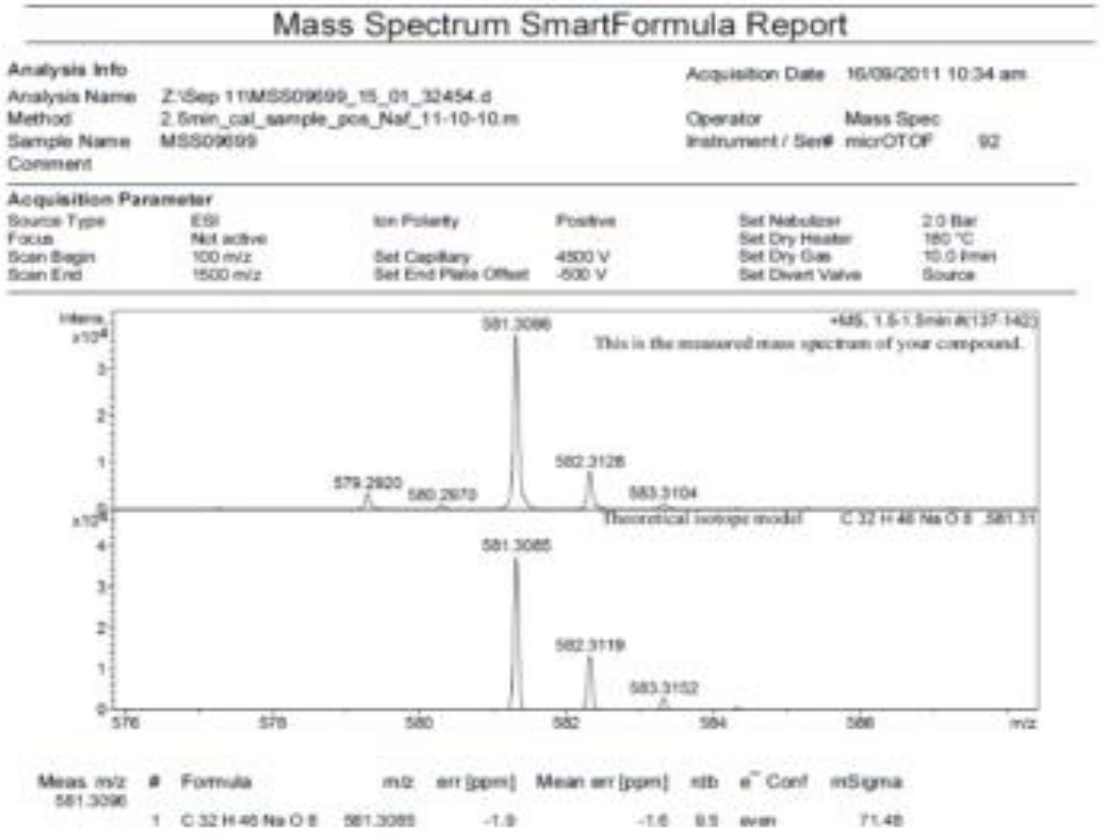

**Spectrum S.3.1.7: HMBC spectrum for compound 1 in $CDCl_3$**



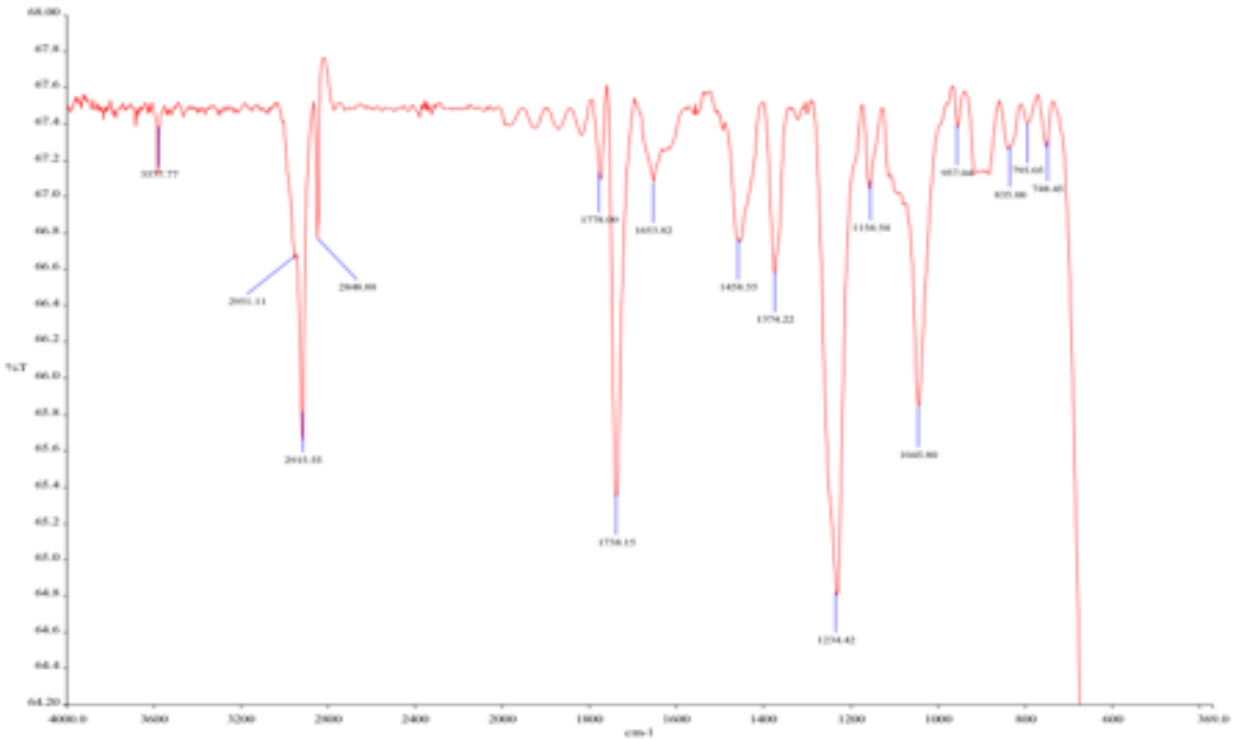

**Spectrum S.3.1.8: COSY spectrum for compound 1 in $CDCl_3$**



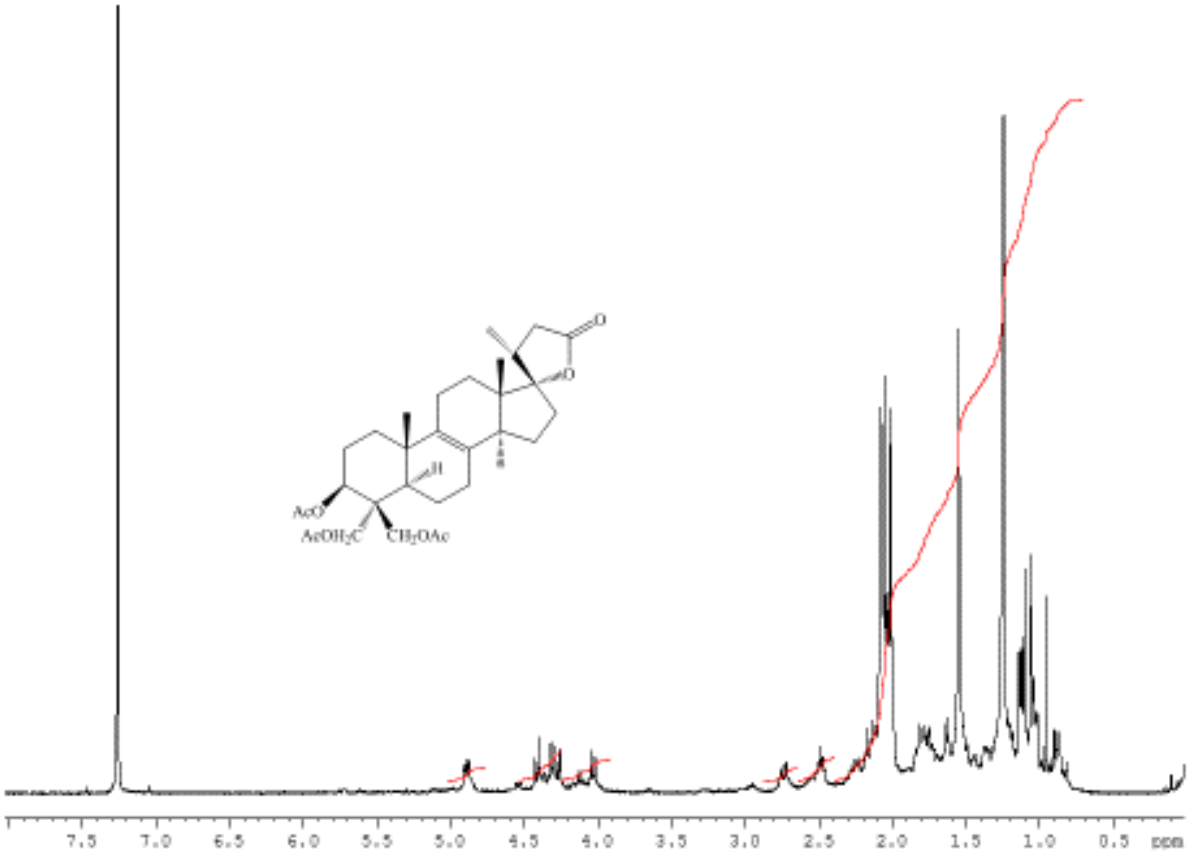

**Spectrum S.3.1.9: NOESY spectrum for compound 1 in $CDCl_3$**



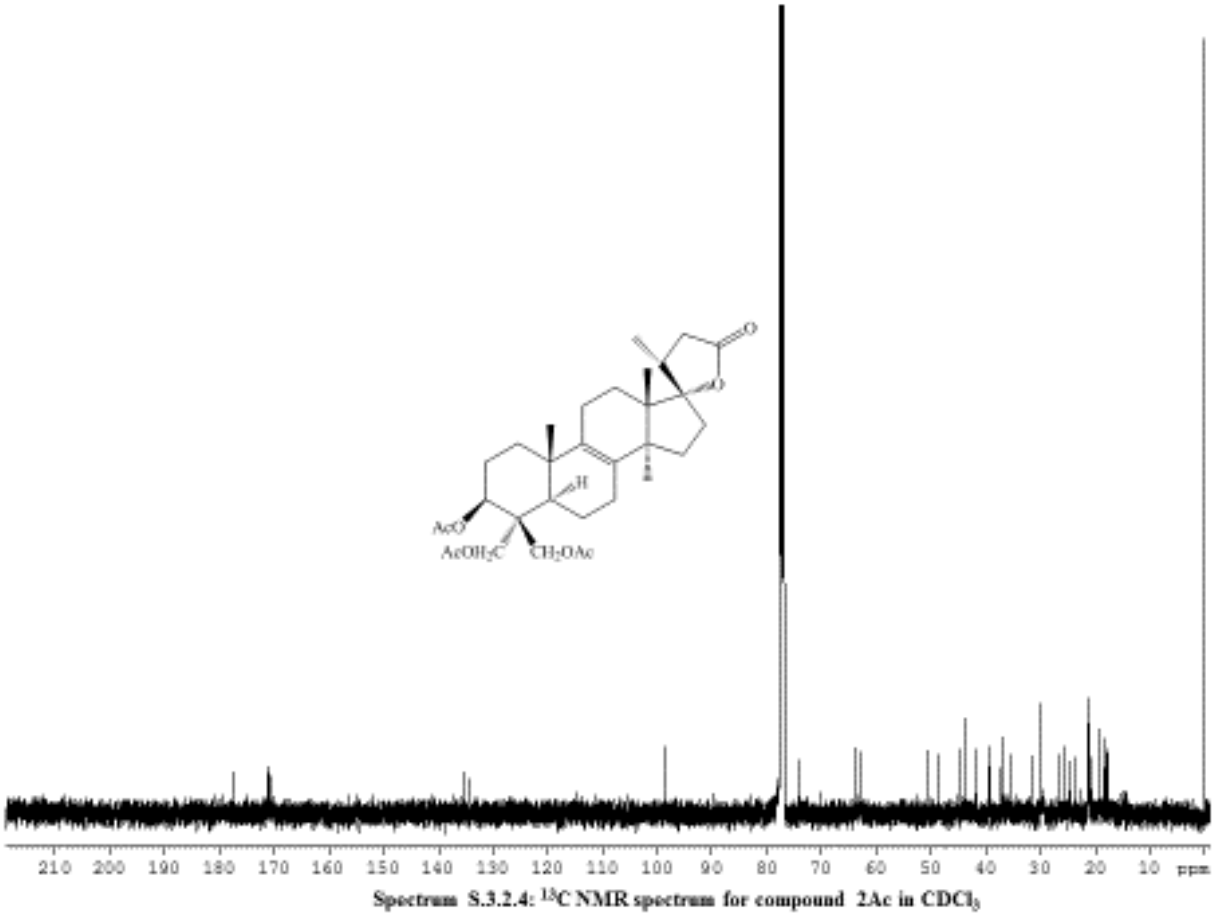

**Spectrum S.3.2.1: Mass spectrum for compound 2Ac**



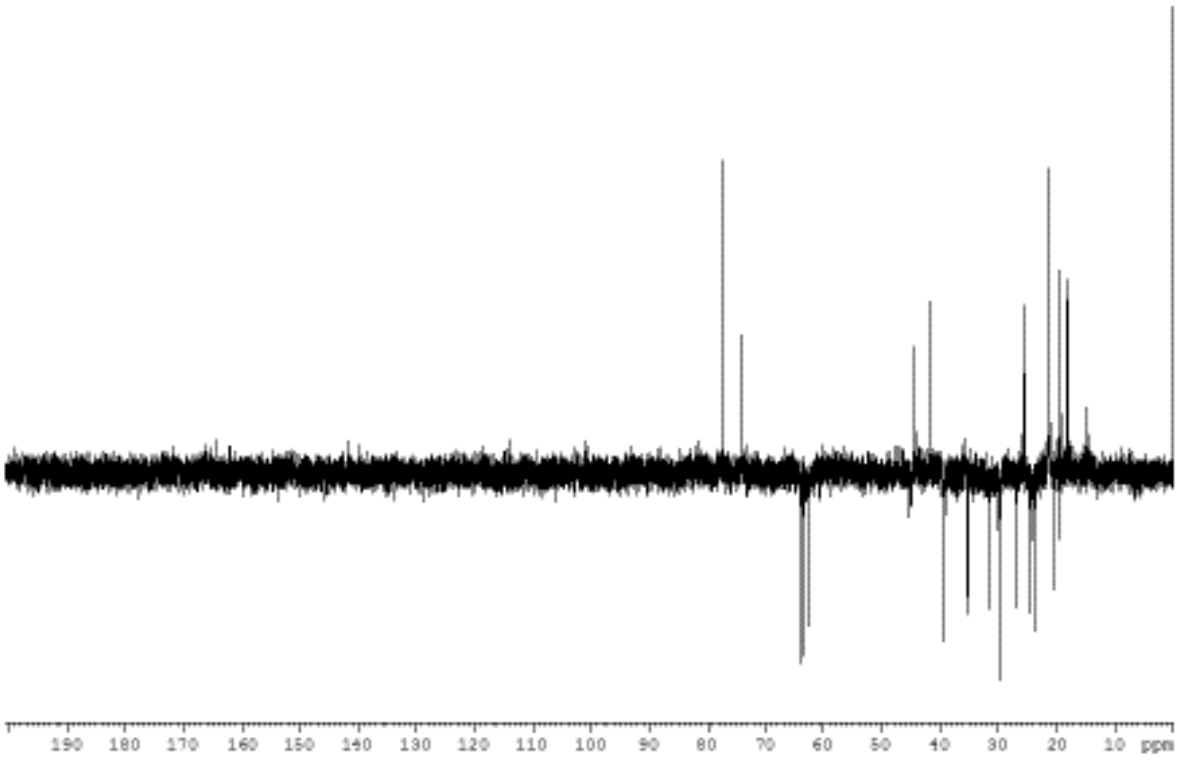

**Spectrum S.3.2.2: FTIR spectrum for compound 2Ac**



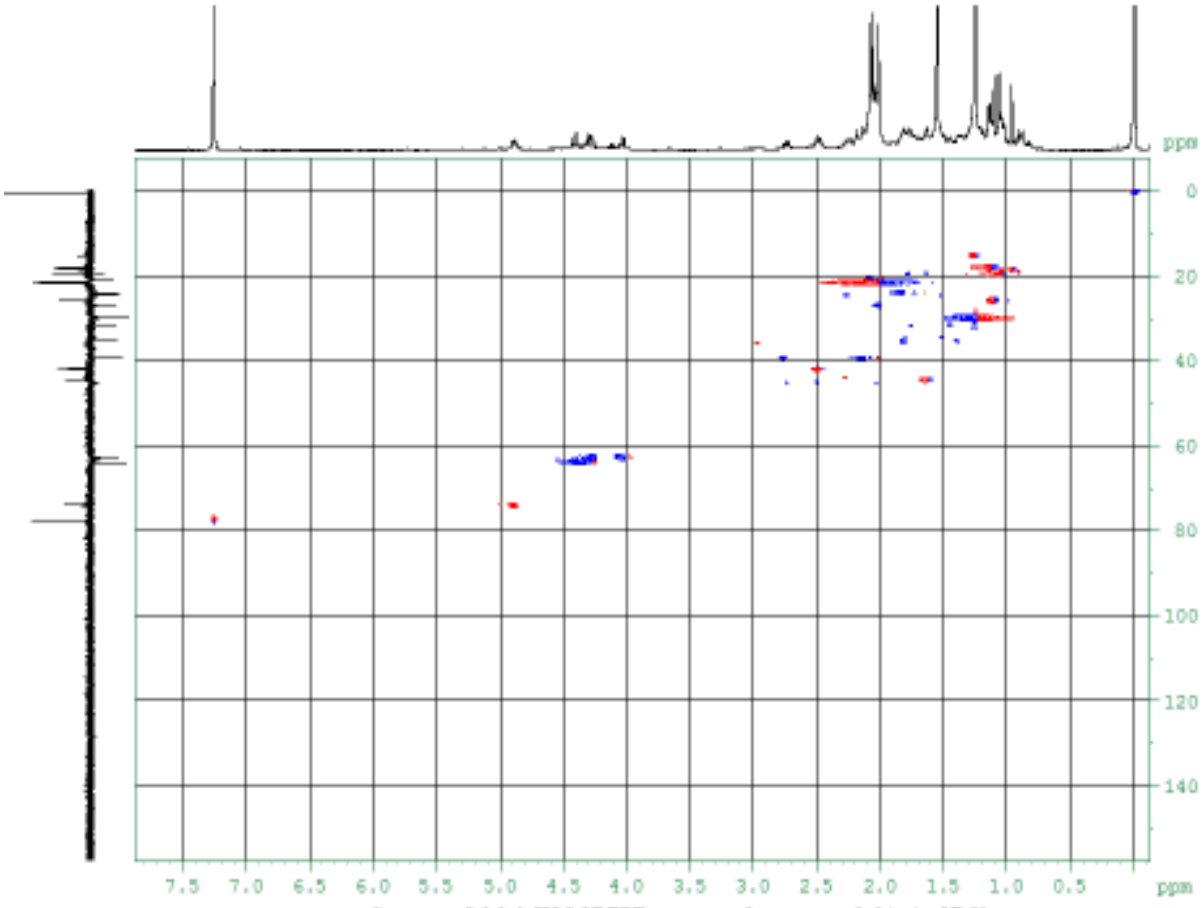

**Spectrum S.3.2.3: $^{1}H$ NMR spectrum for compound 2Ac in $CDCl_3$**



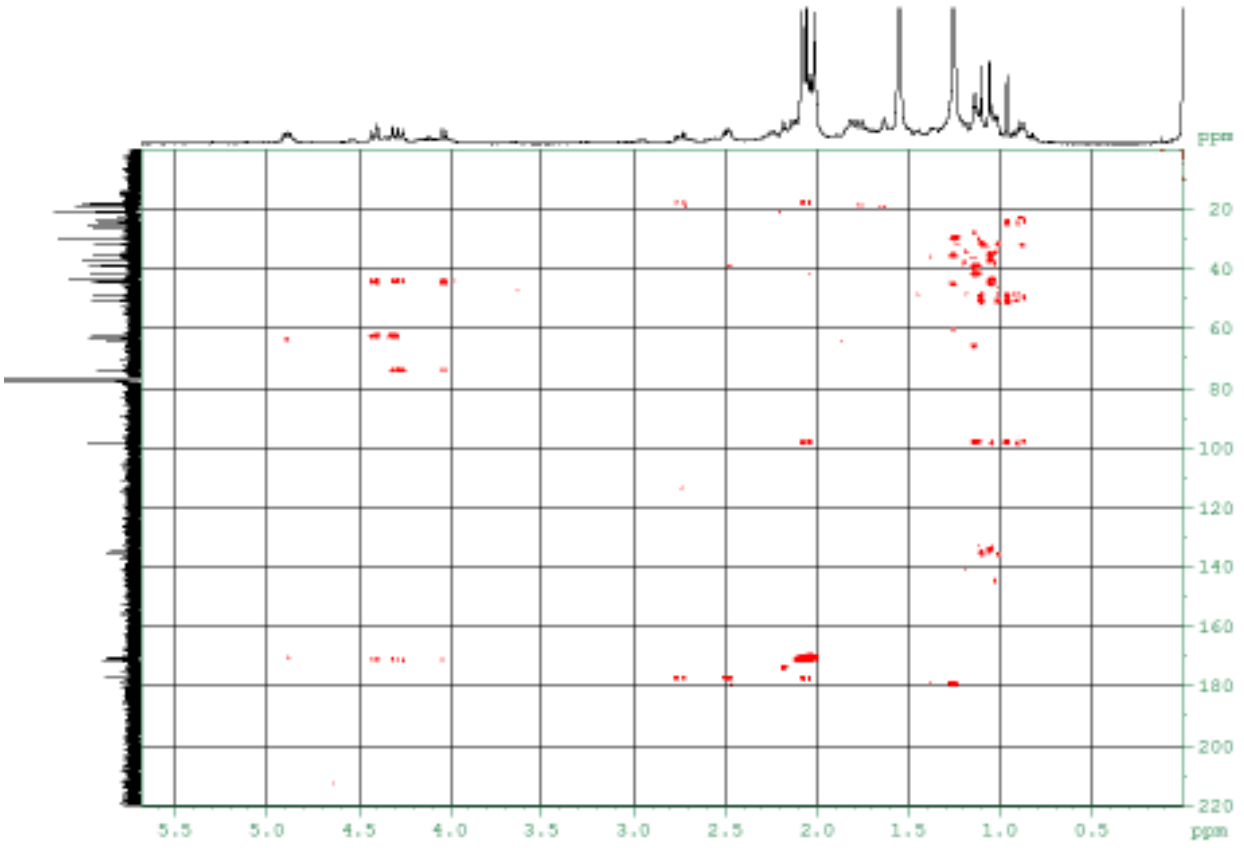

**Spectrum S.3.2.4: $^{13}C$ NMR spectrum for compound 2Ac in $CDCl_3$**



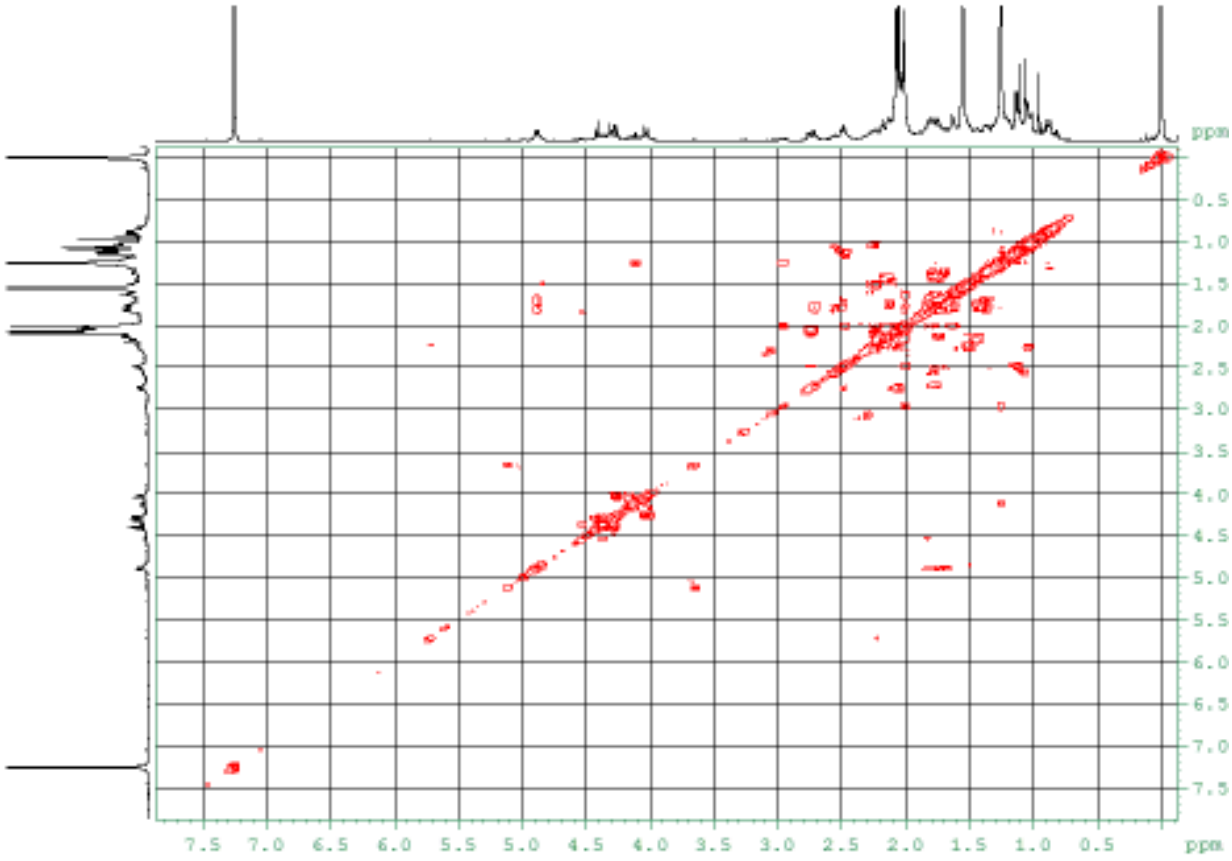

**Spectrum S.3.2.5: DEPT spectrum for compound 2Ac in $CDCl_3$**



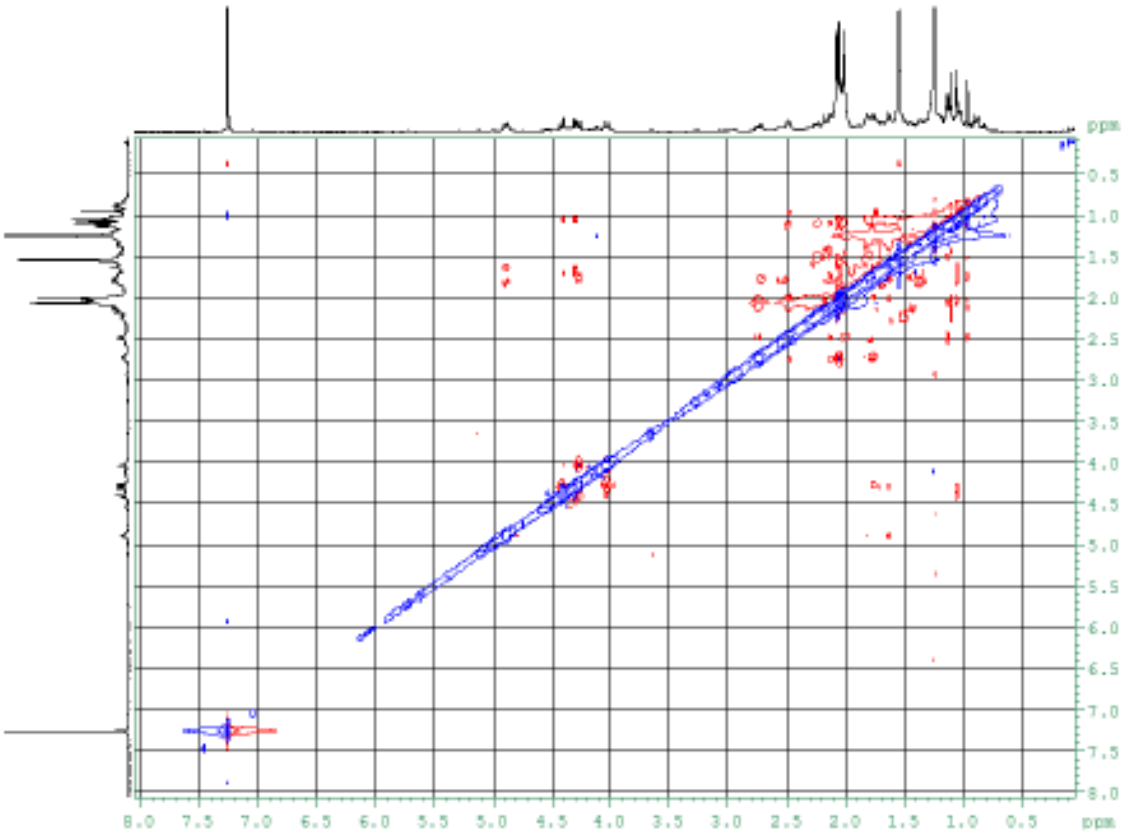

**Spectrum S.3.2.6: HSQCDEPT spectrum for compound 2Ac in $CDCl_3$**



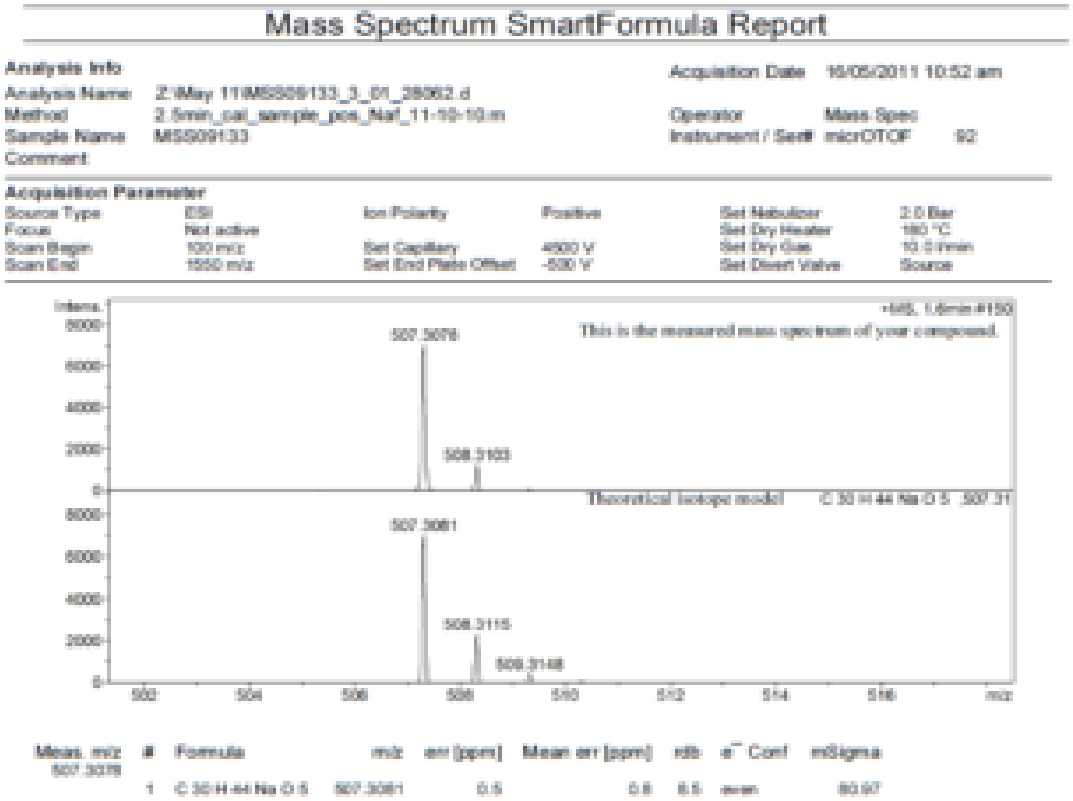

**Spectrum S.3.2.7: HMBC spectrum for compound 2Ac in $CDCl_3$**



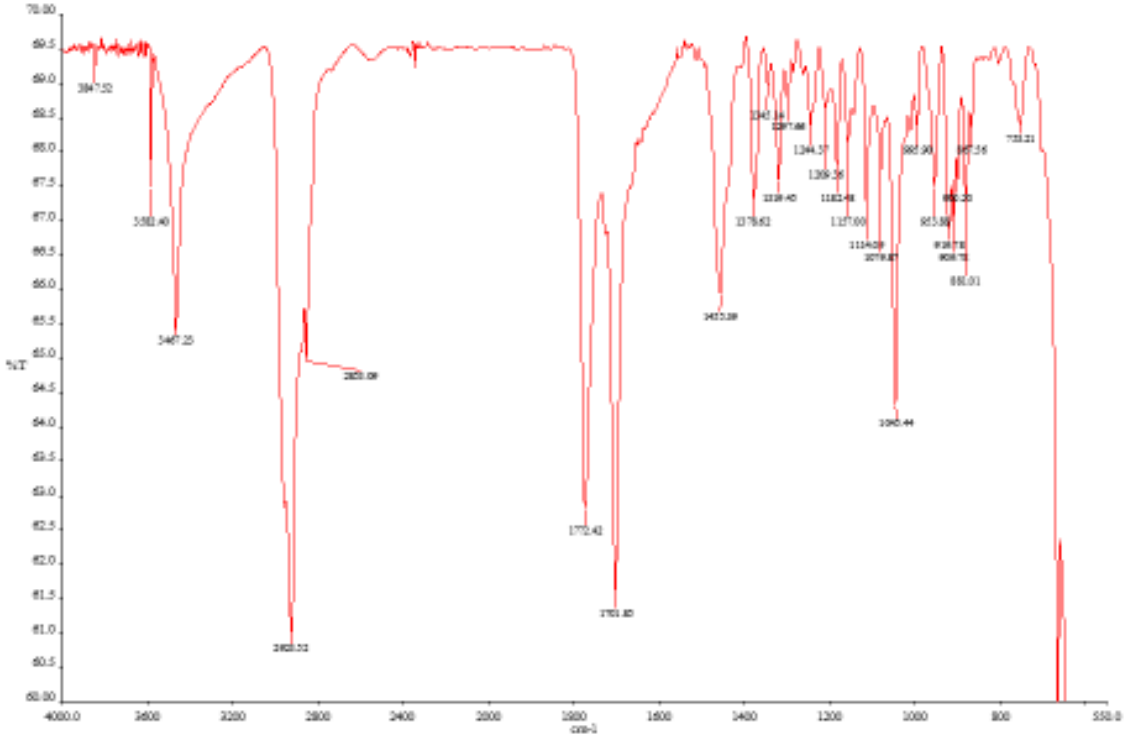

**Spectrum S.3.2.8: COSY spectrum for compound 2Ac in $CDCl_3$**



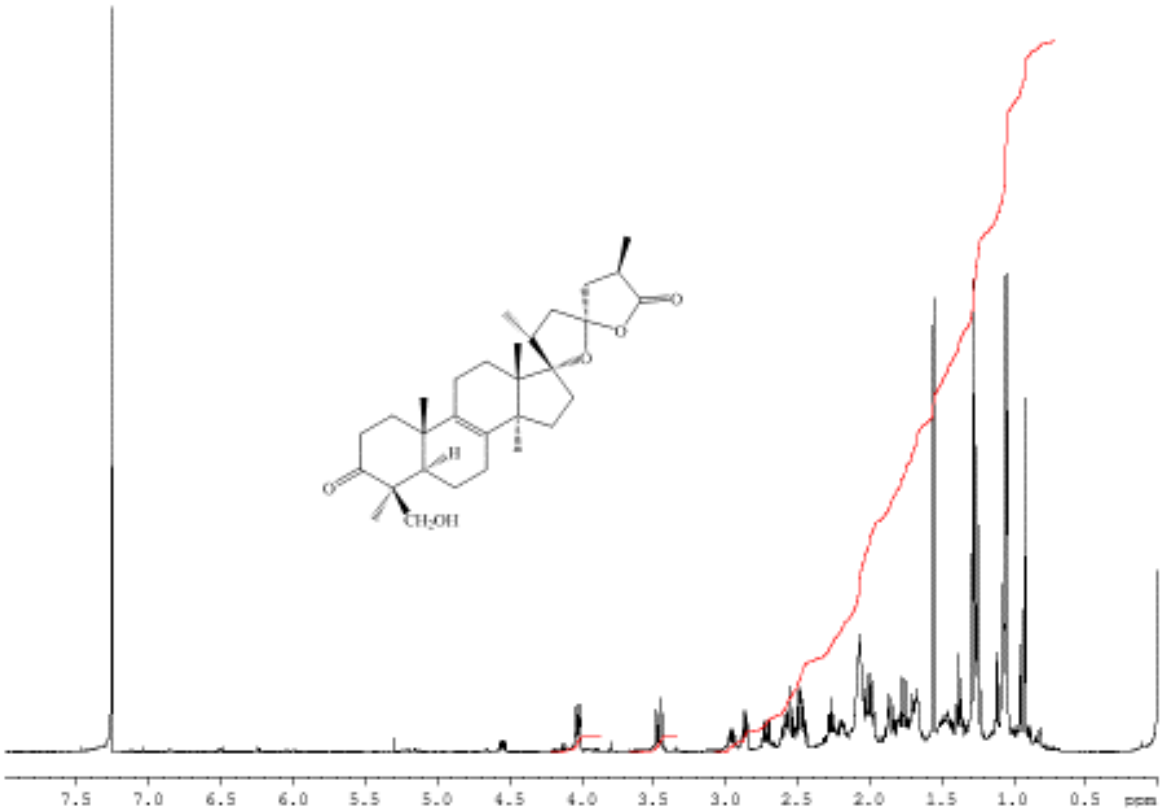

**Spectrum S.3.2.9: NOESY spectrum for compound 2Ac in $CDCl_3$**



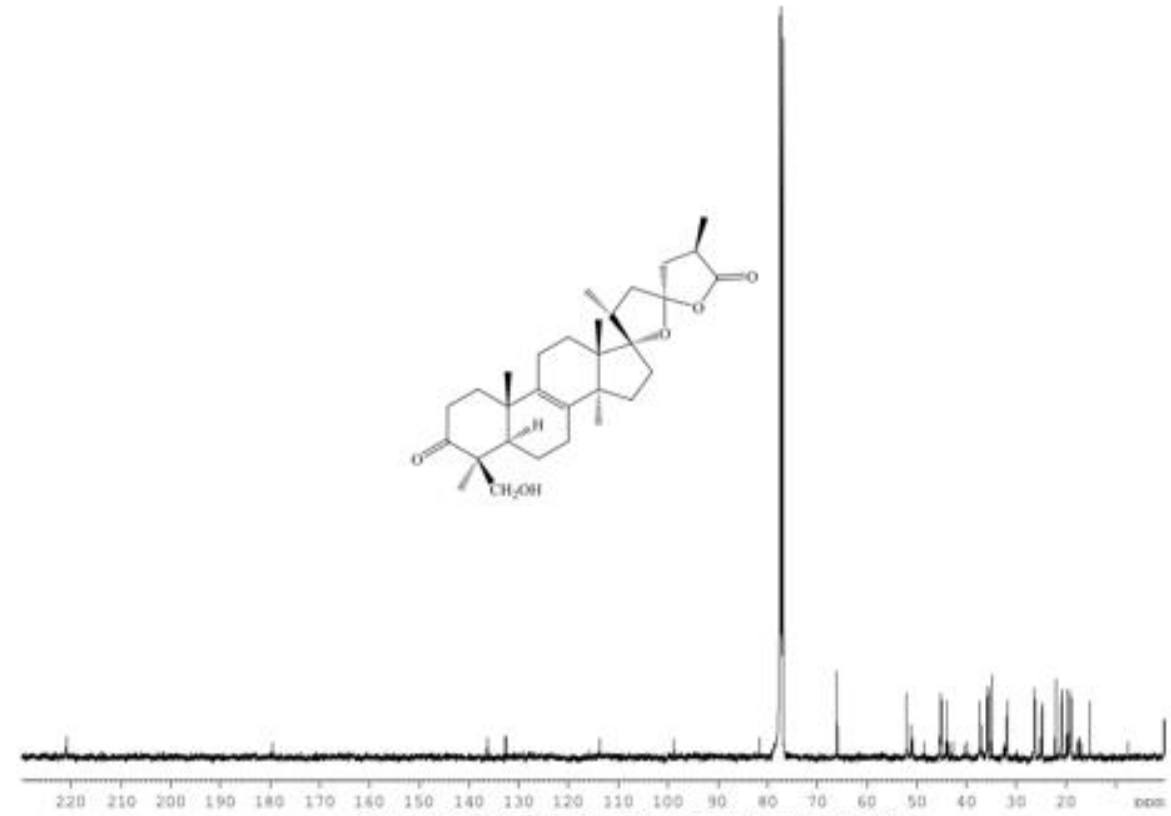

**Spectrum S.3.3.1: Mass spectrum for compound 3**



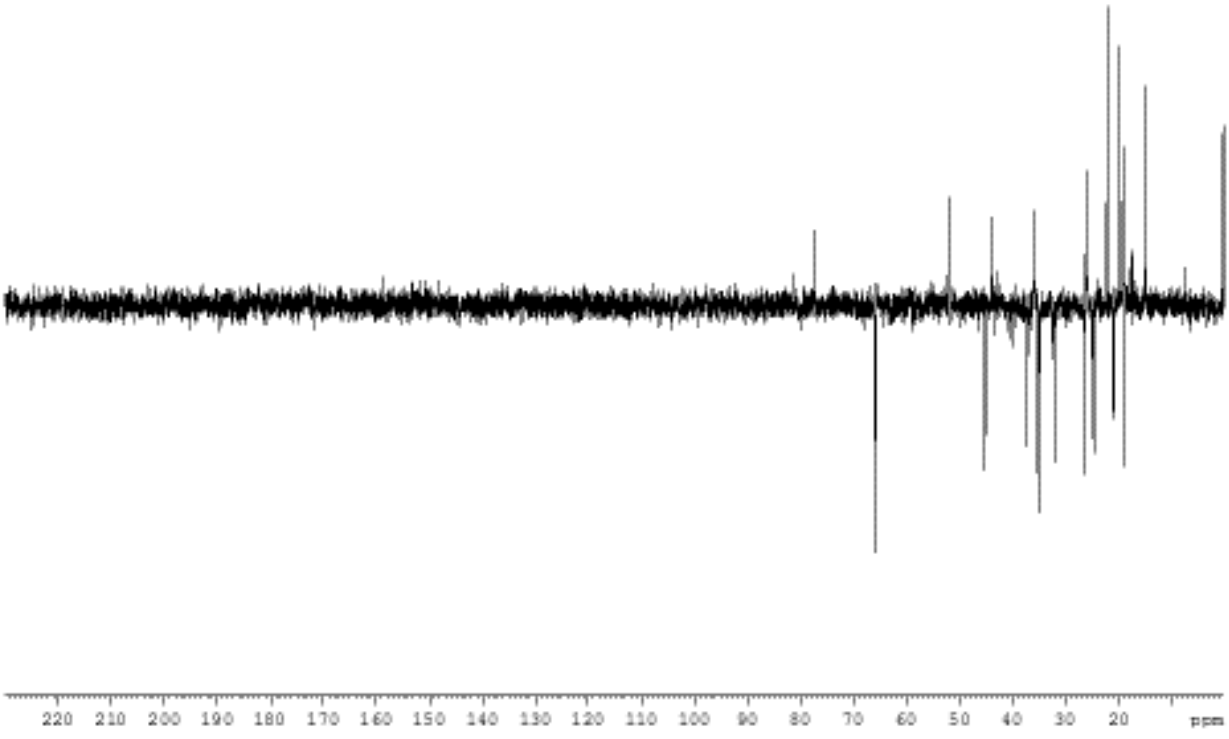
**Spectrum S.3.3.2: FTIR spectrum for compound 3**



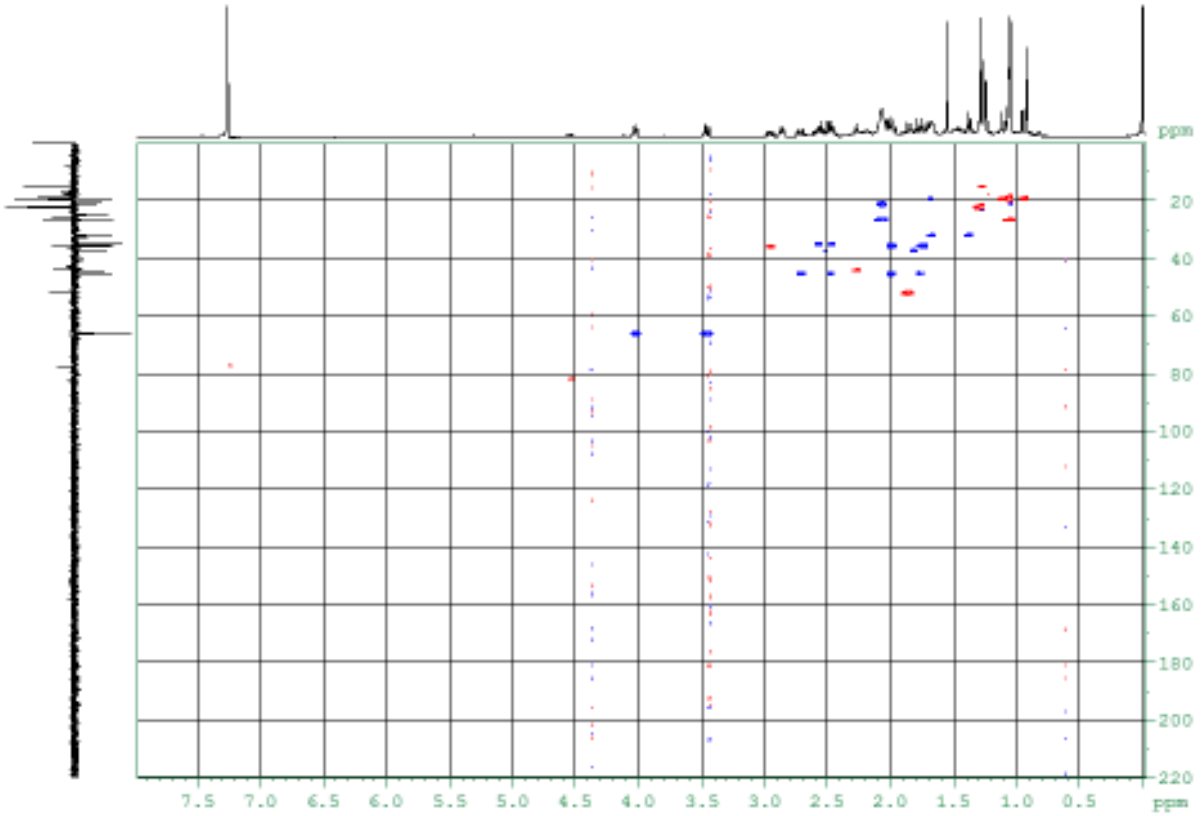

**Spectrum S.3.3.3: $^1$H NMR spectrum for compound 3 in $CDCl_3$**



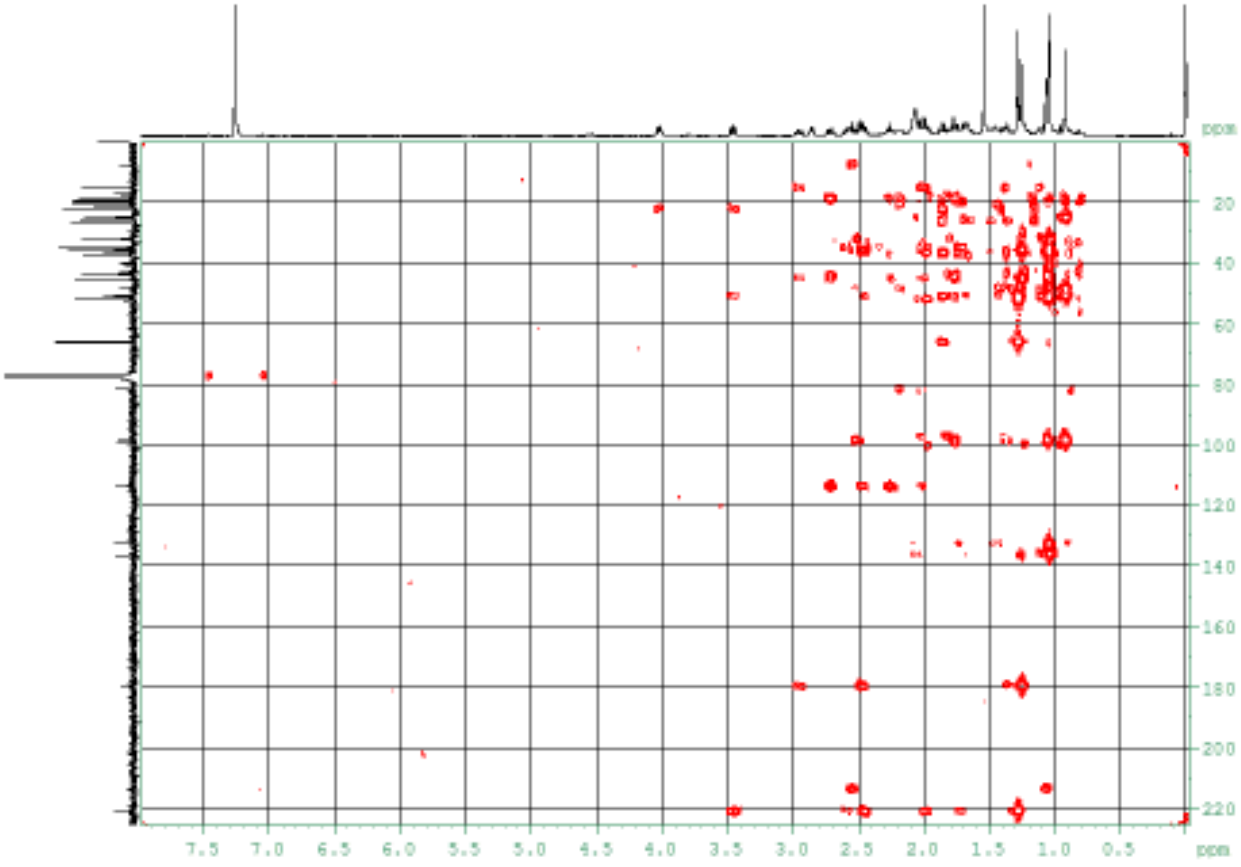

**Spectrum S.3.3.4: $^{13}C$ NMR spectrum for compound 3 in $CDCl_3$**



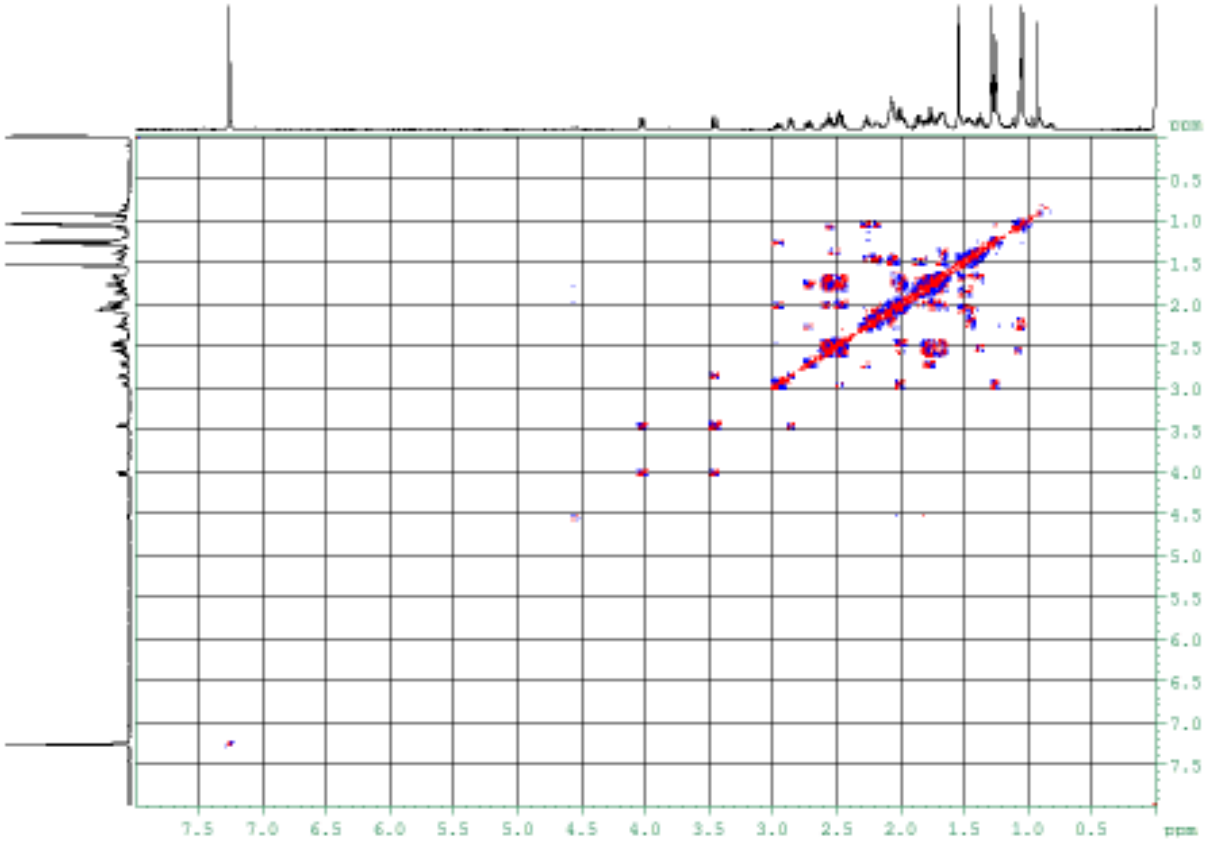

**Spectrum S.3.3.5: DEPT spectrum for compound 3 in $CDCl_3$**



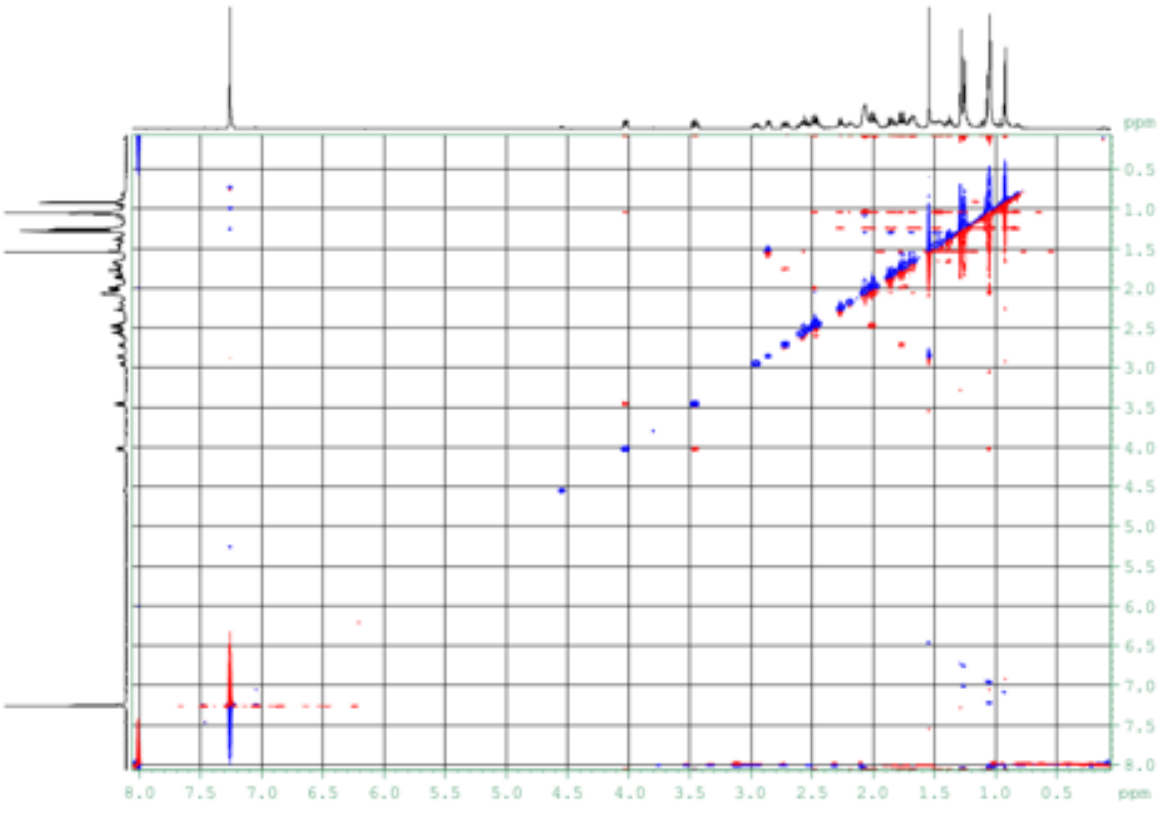

**Spectrum S.3.3.6: HSQCDEPT spectrum for compound 3 in $CDCl_3$**



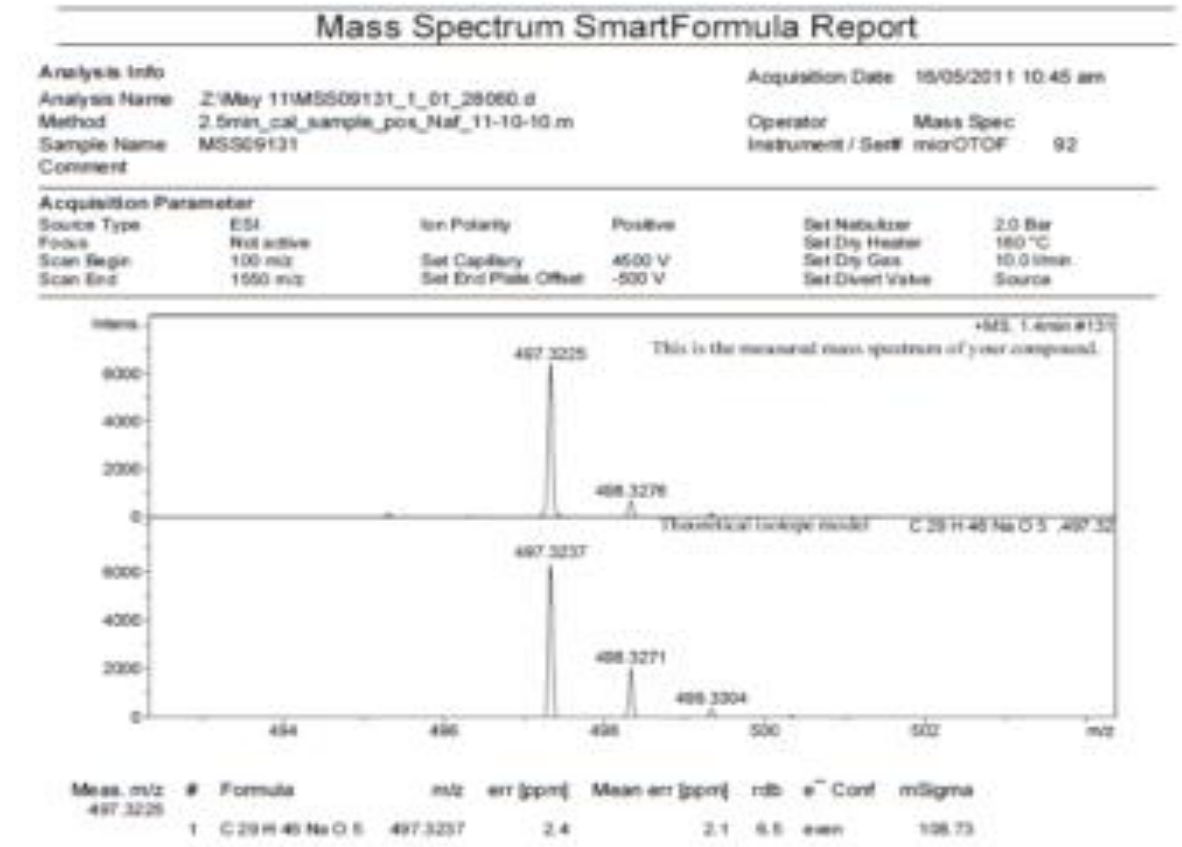

**Spectrum S.3.3.7: HMBC spectrum for compound 3 in $CDCl_3$**



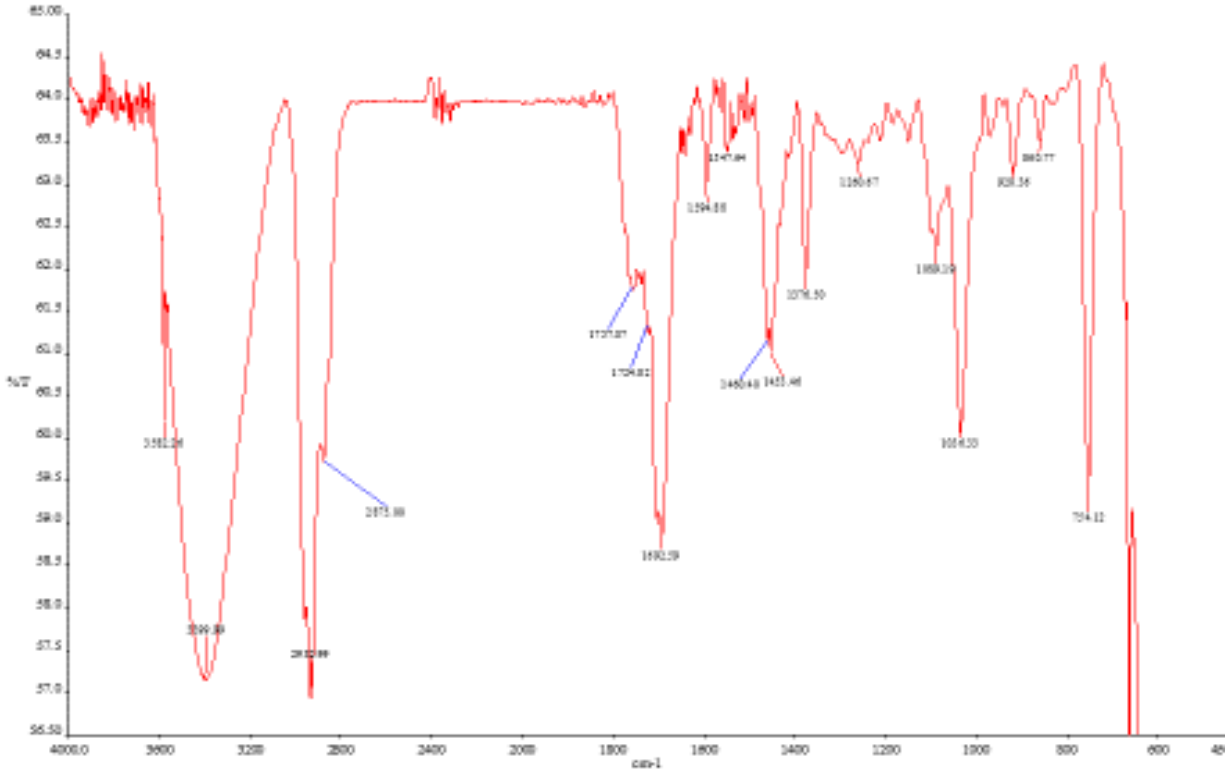

**Spectrum S.3.3.8: COSY spectrum for compound 3 in $CDCl_3$**



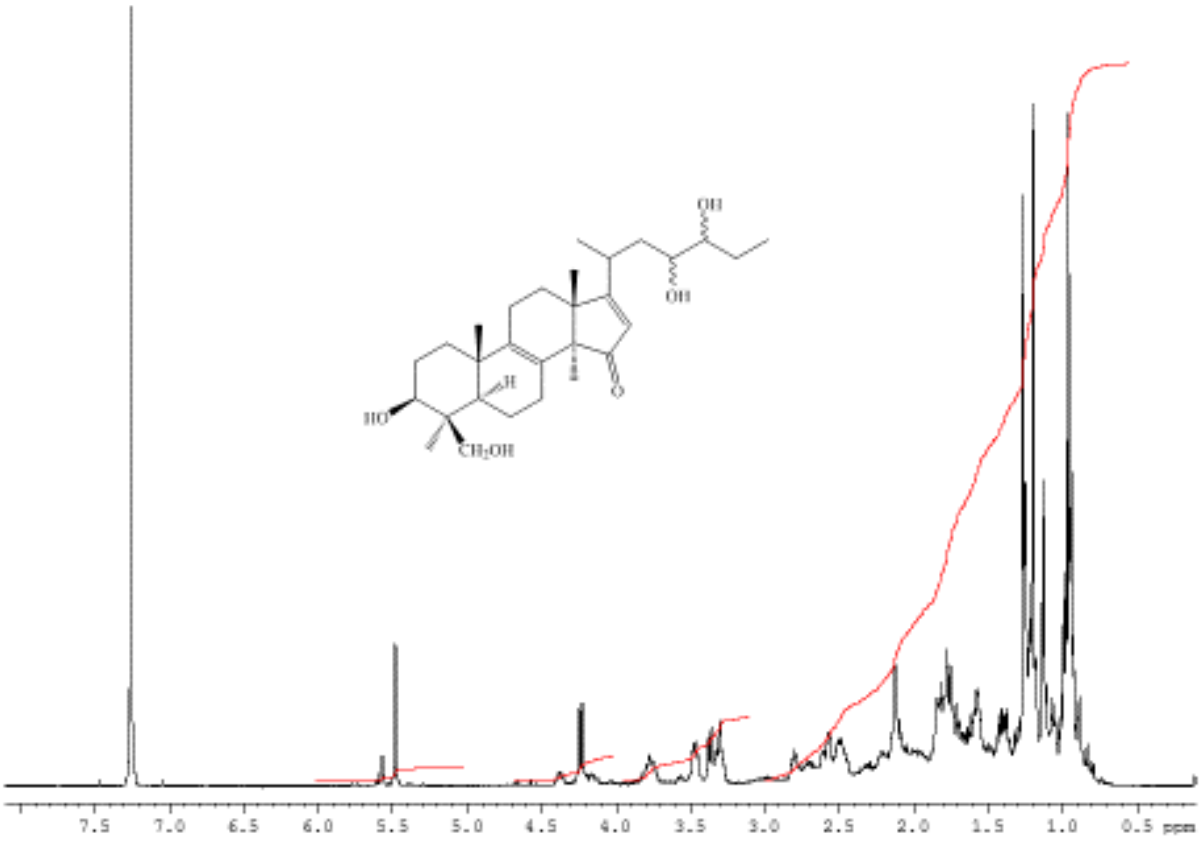

**Spectrum S.3.3.9: NOESY spectrum for compound 3 in $CDCl_3$**



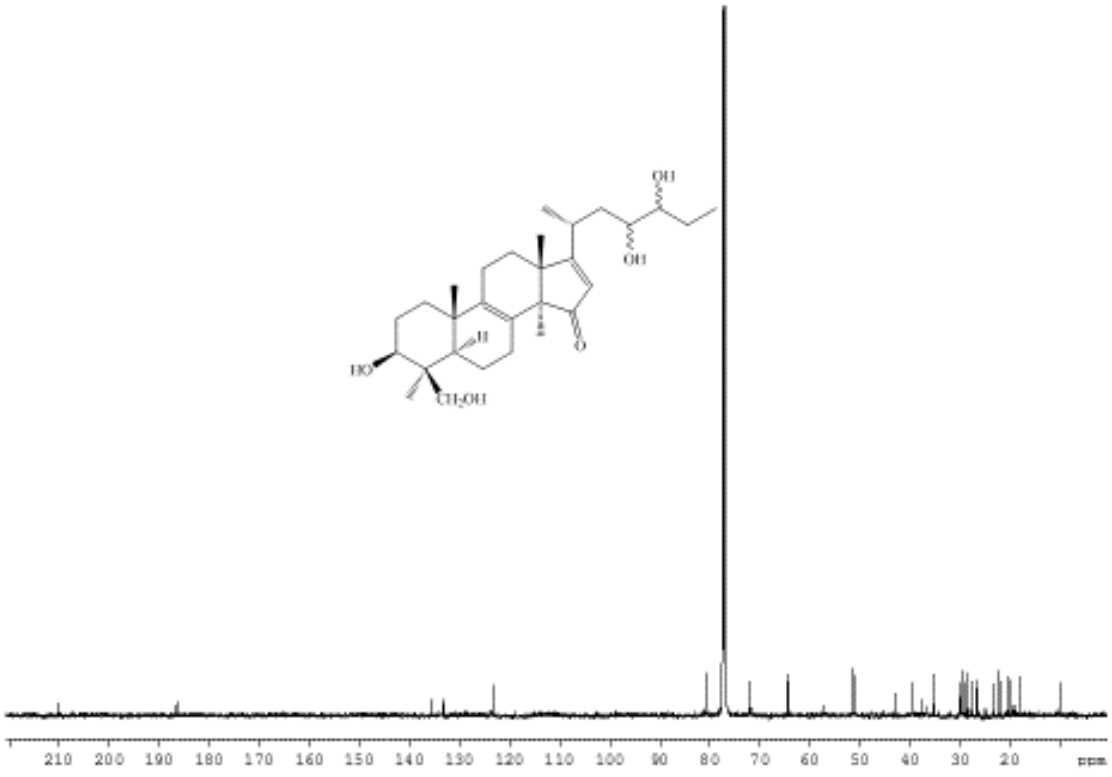

**Spectrum S.3.4.1: Mass spectrum for compound 4**



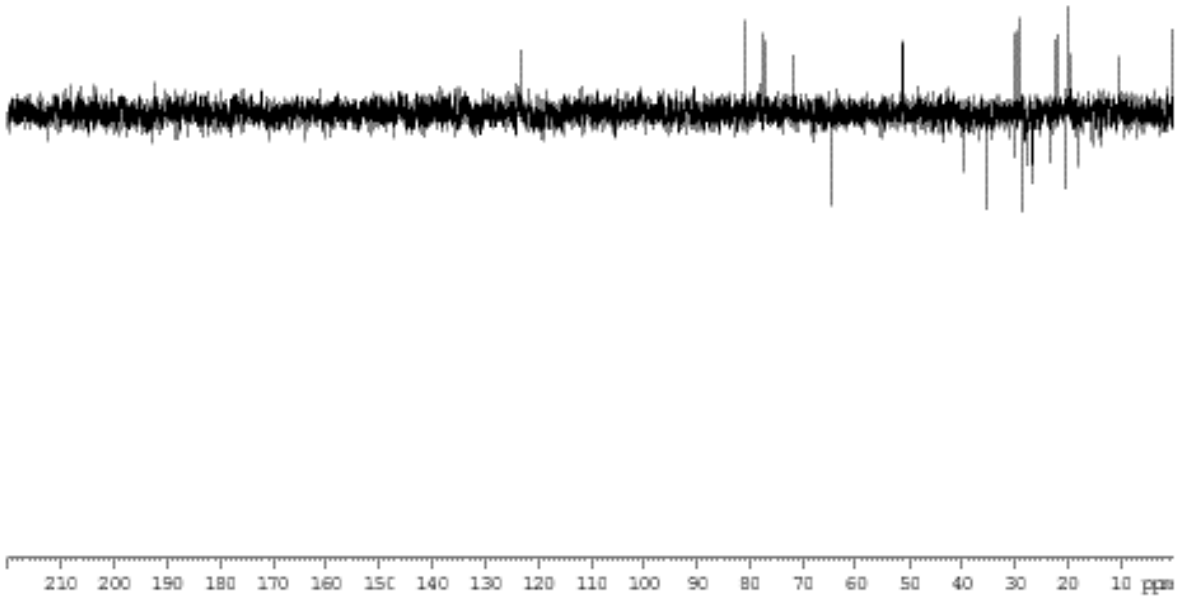

**Spectrum S.3.4.2: FTIR spectrum for compound 4**



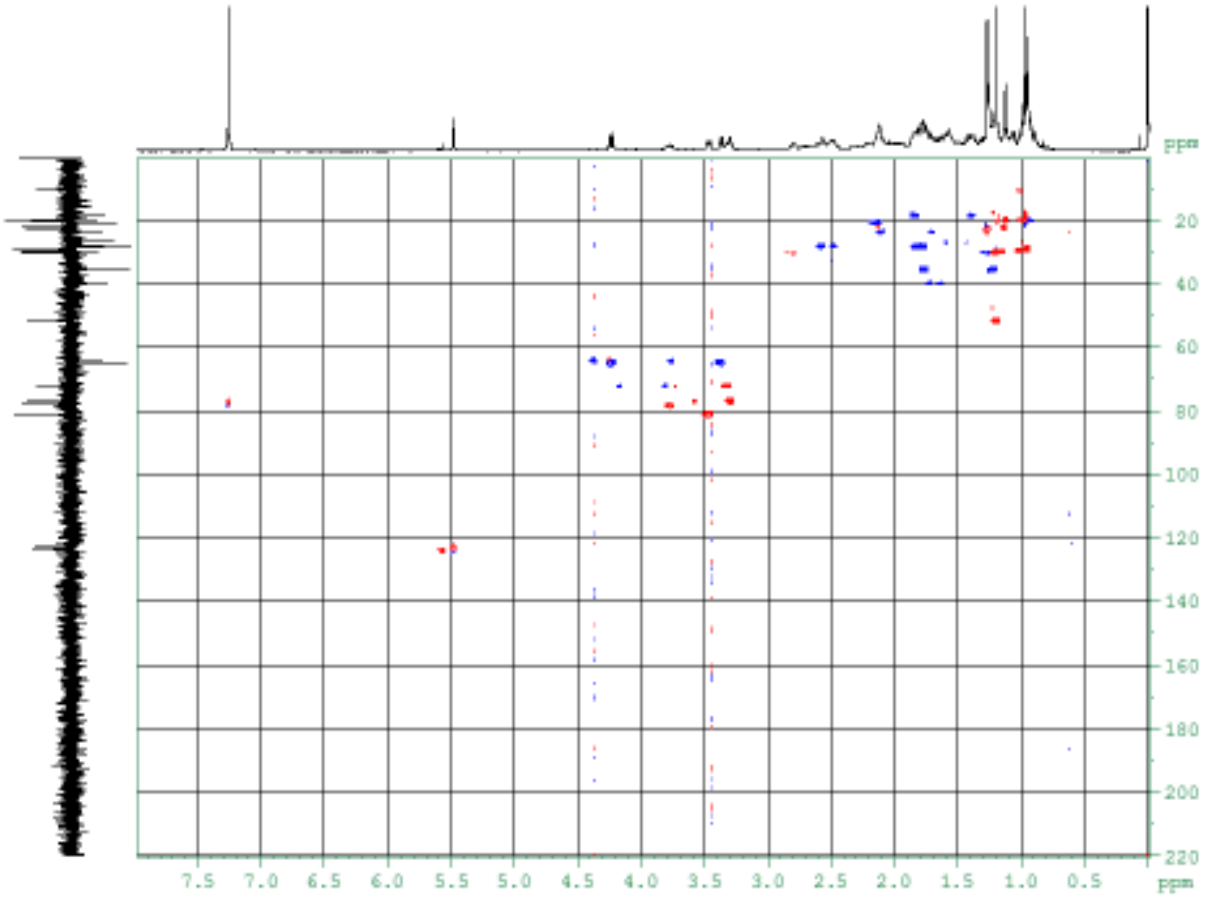

**Spectrum S.3.4.3: $^{1}H$ NMR spectrum for compound 4 in $CDCl_3$**



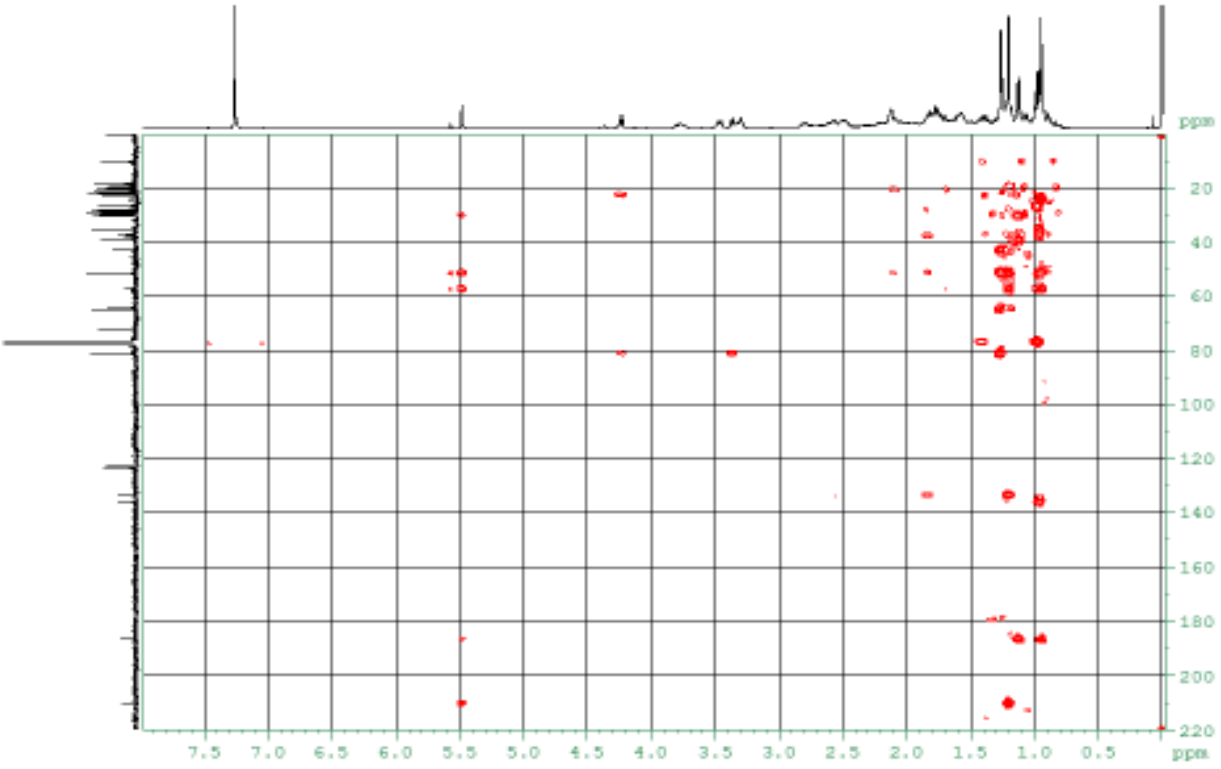

**Spectrum S.3.4.3: $^{13}C$ NMR spectrum for compound 4 in $CDCl_3$**



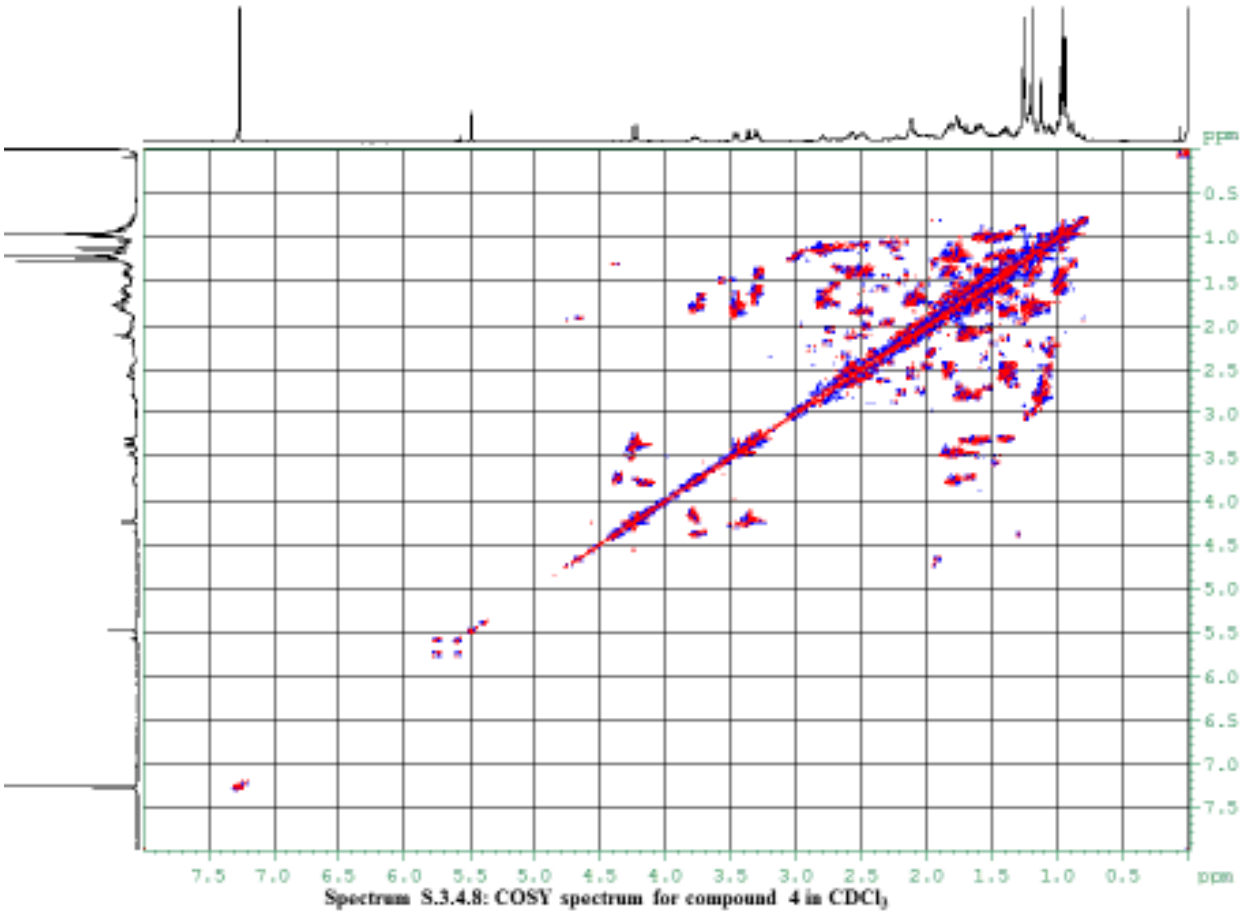

**Spectrum S.3.4.4: DEPT spectrum for compound 4 in $CDCl_3$**



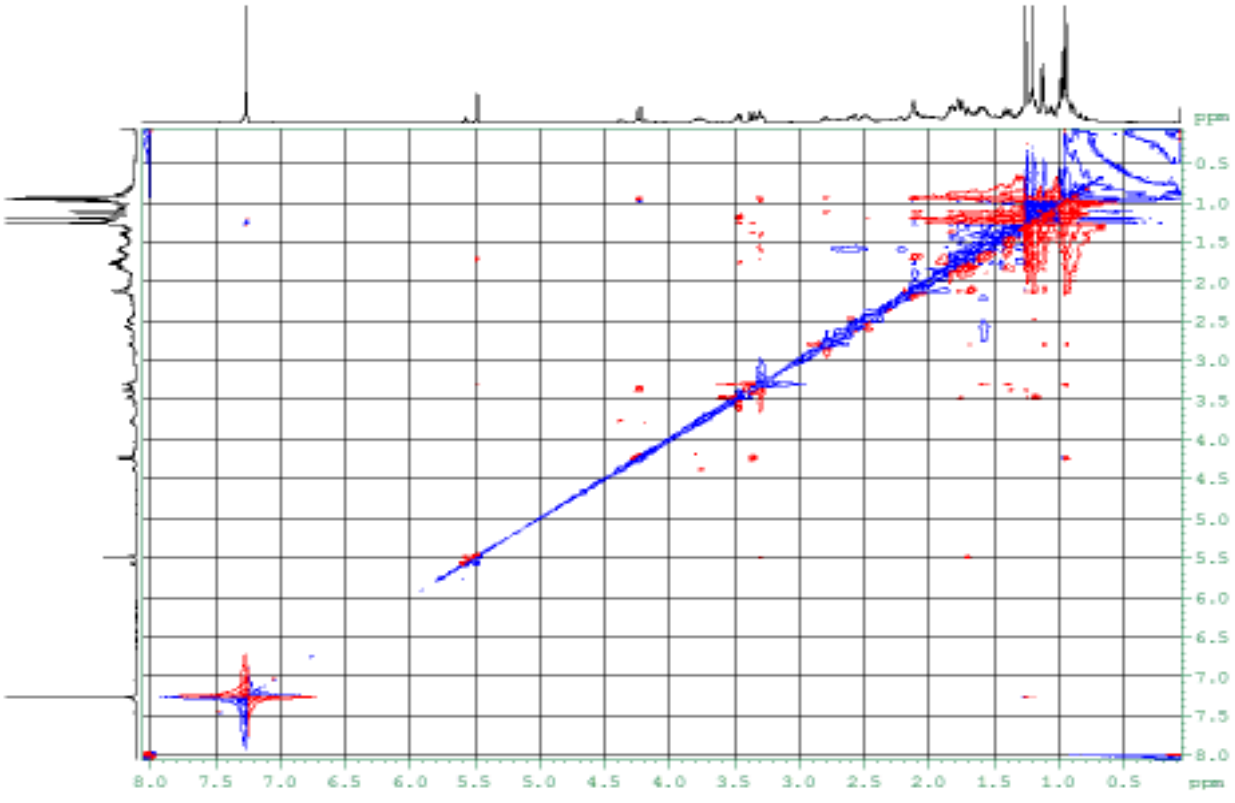

**Spectrum S.3.4.6: HSQCDEPT spectrum for compound 4 in $CDCl_3$**



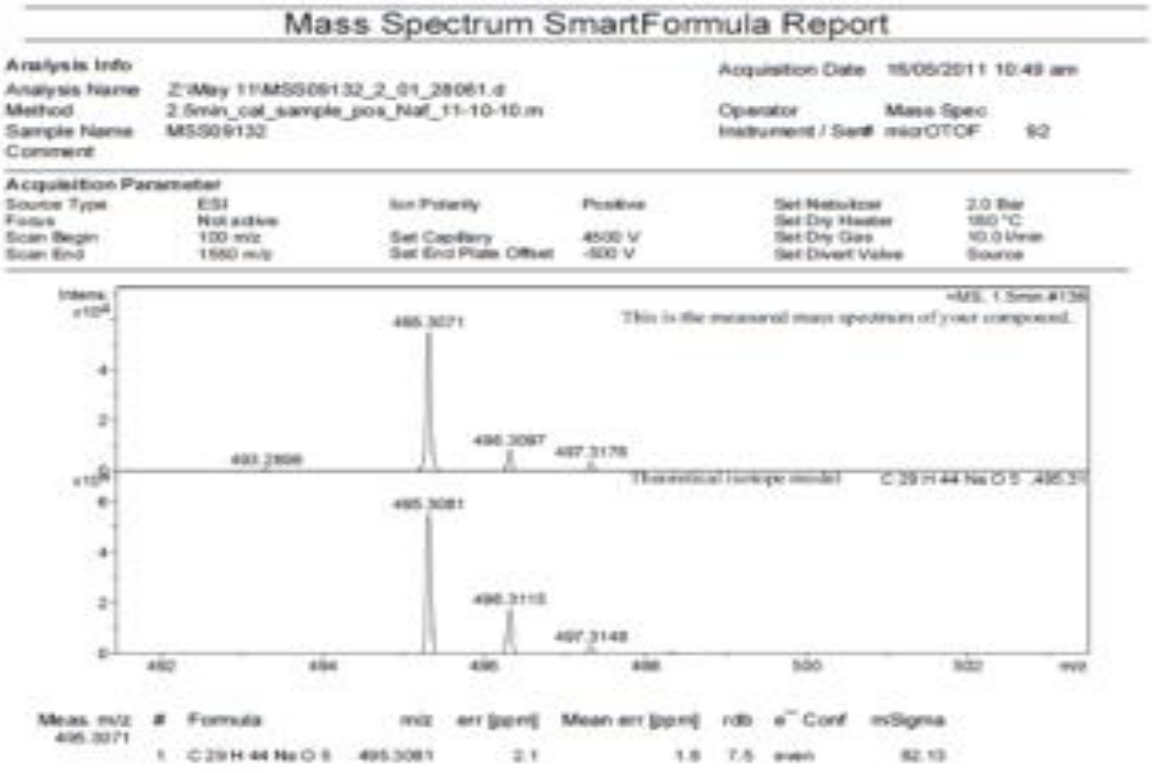

**Spectrum S.3.4.7: HMBC spectrum for compound 4 in $CDCl_3$**



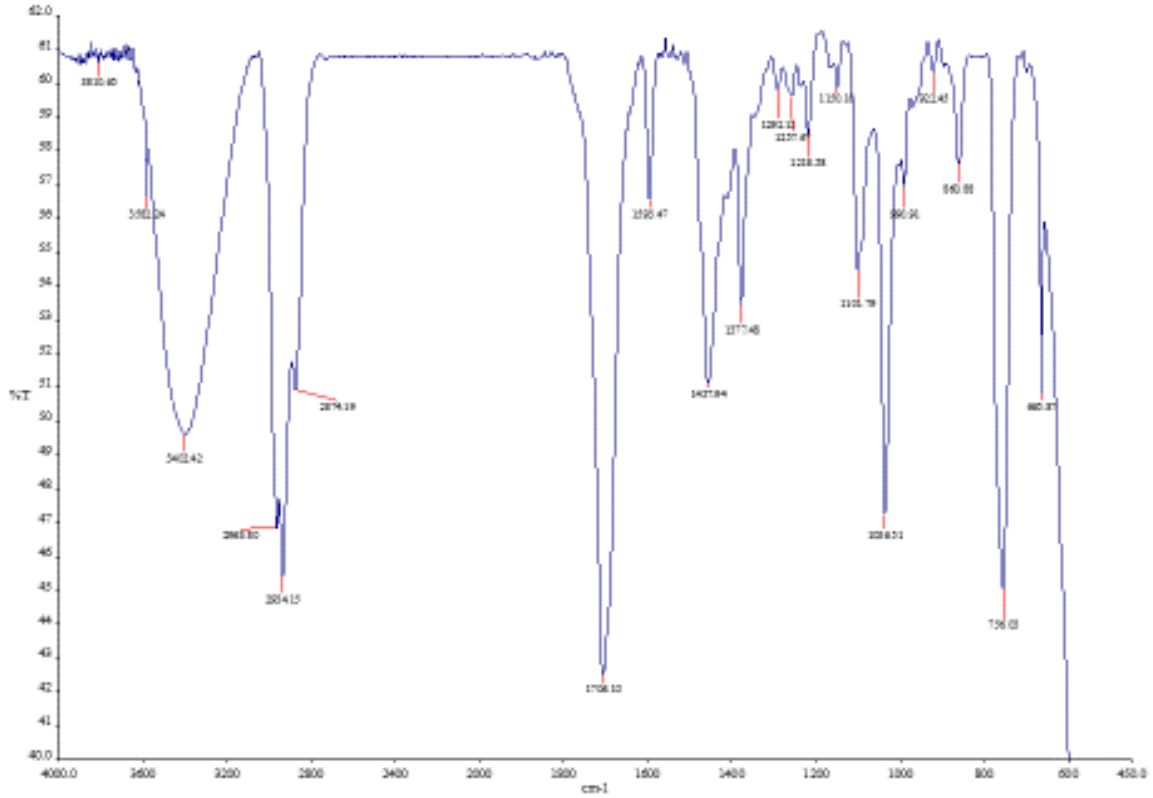

**Spectrum S.3.4.8: COSY spectrum for compound 4 in $CDCl_3$**



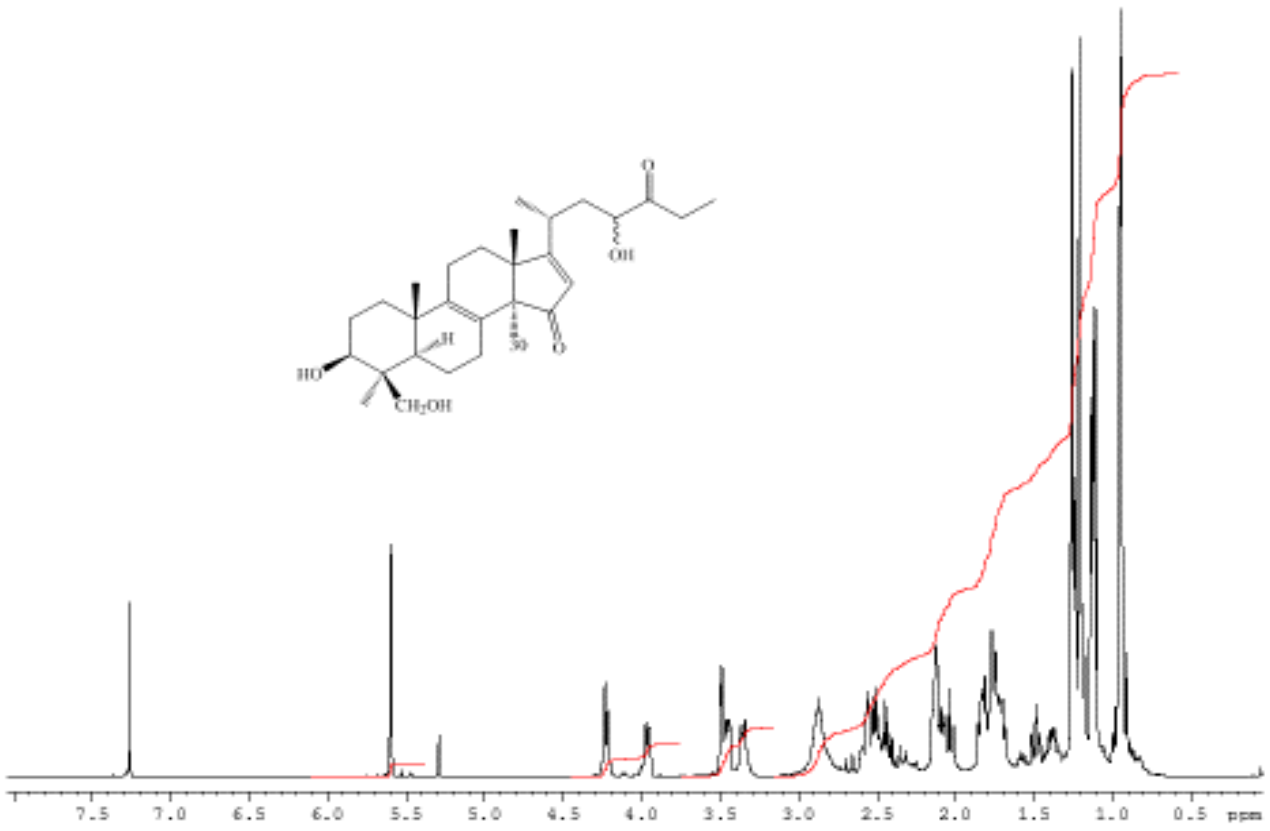

**Spectrum S.3.4.9: NOESY spectrum for compound 4 in $CDCl_3$**



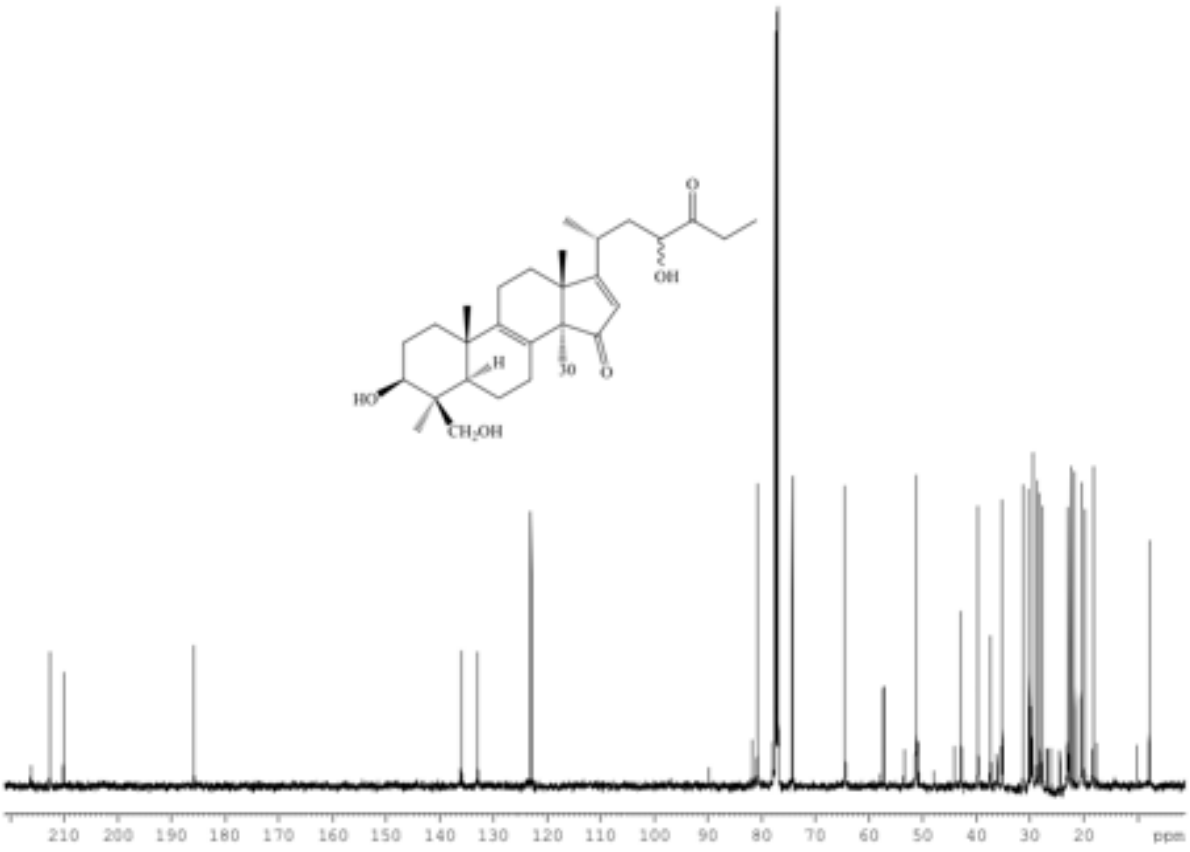

**Spectrum S.3.5.1: Mass spectrum for compound 5**



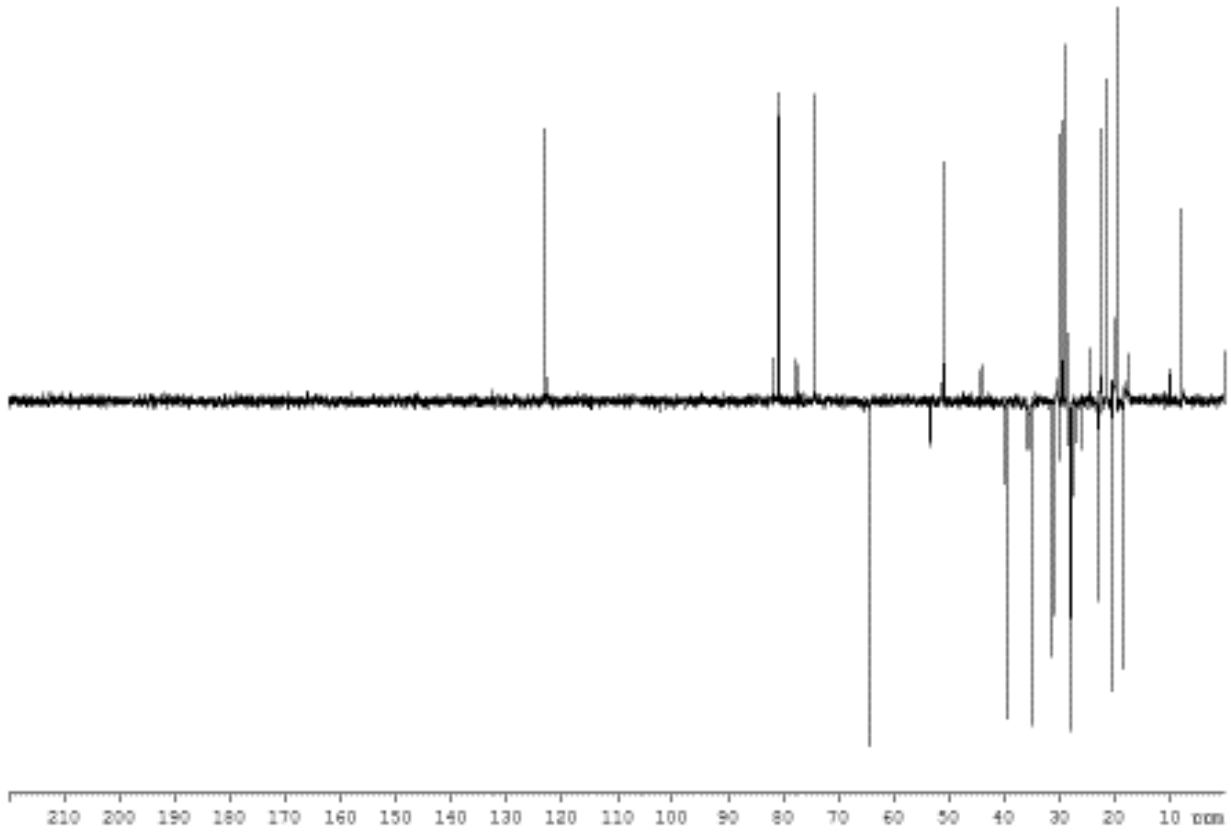

**Spectrum S.3.5.2: FTIR spectrum for compound 5**



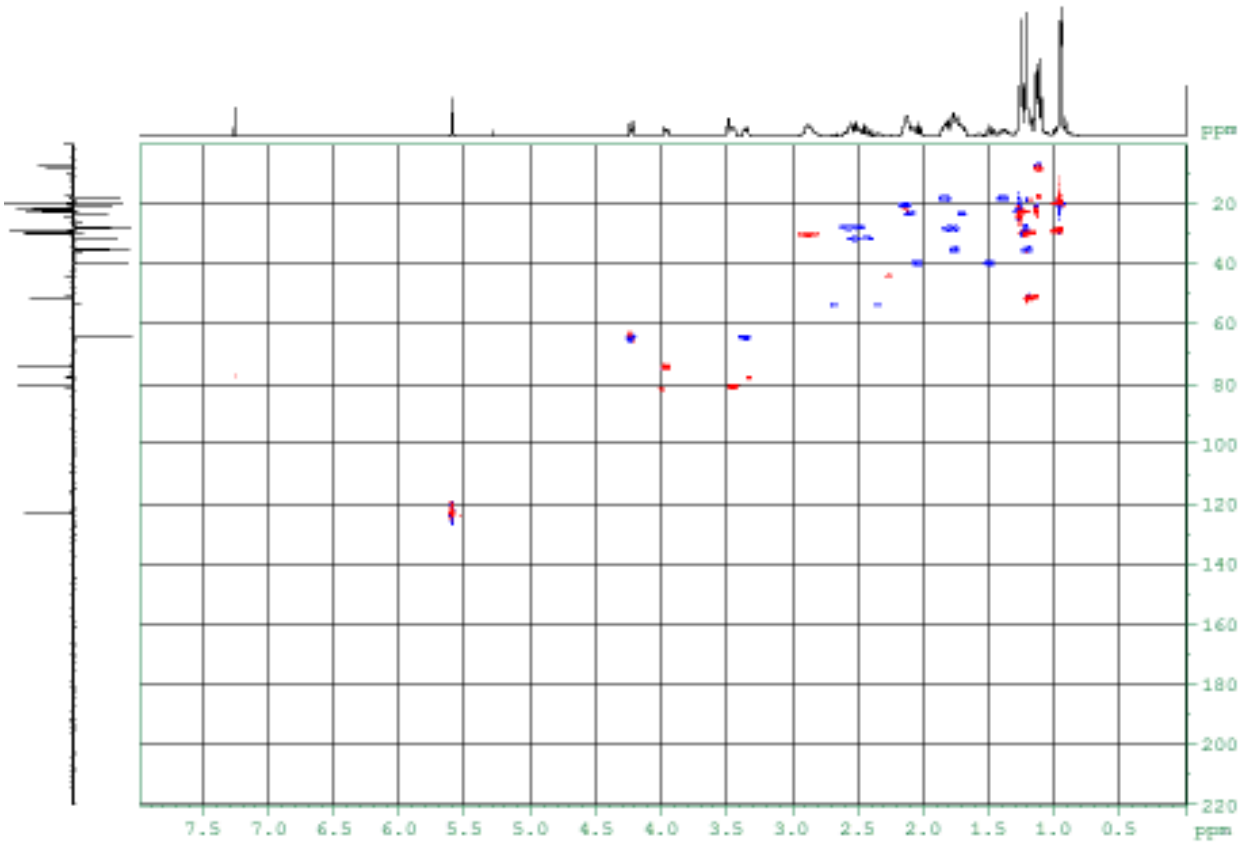

**Spectrum S.3.5_3: $^1$H NMR spectrum for compound 5 in $CDCl_3$**



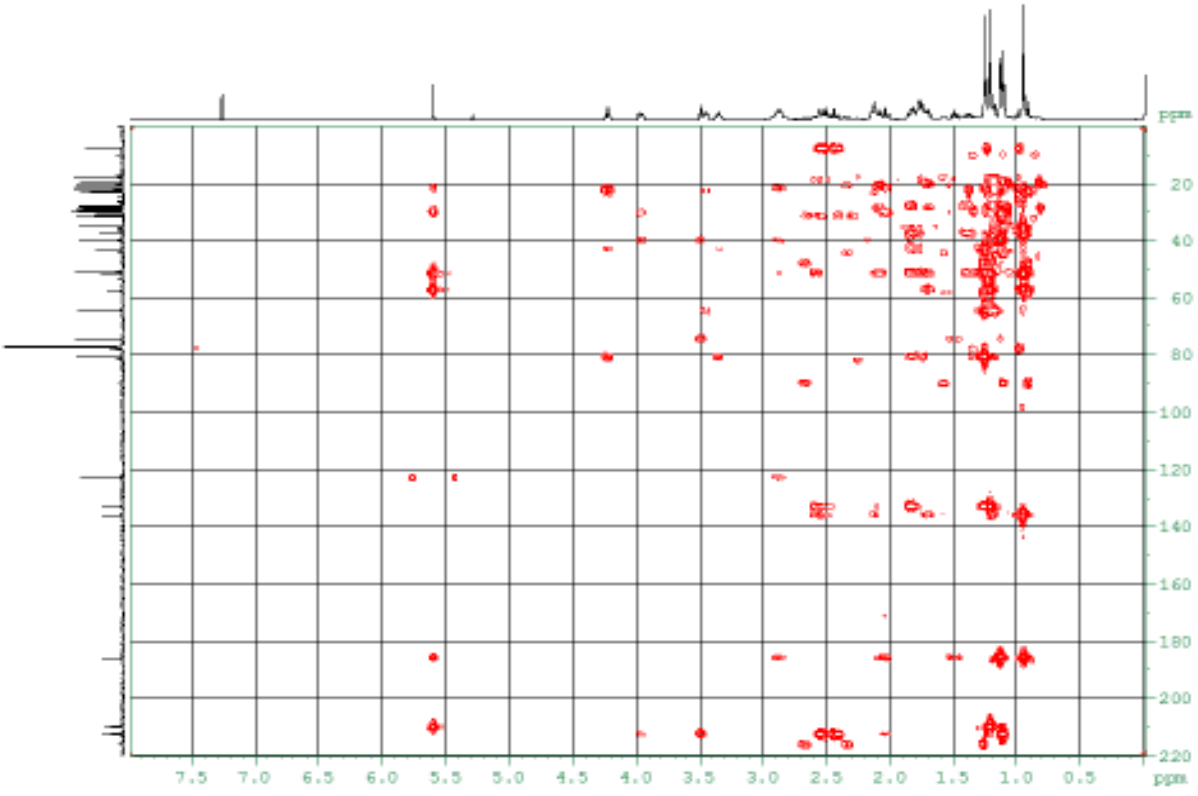

**Spectrum S.3.5.4: $^{13}C$ NMR spectrum for compound 5 in $CDCl_3$**



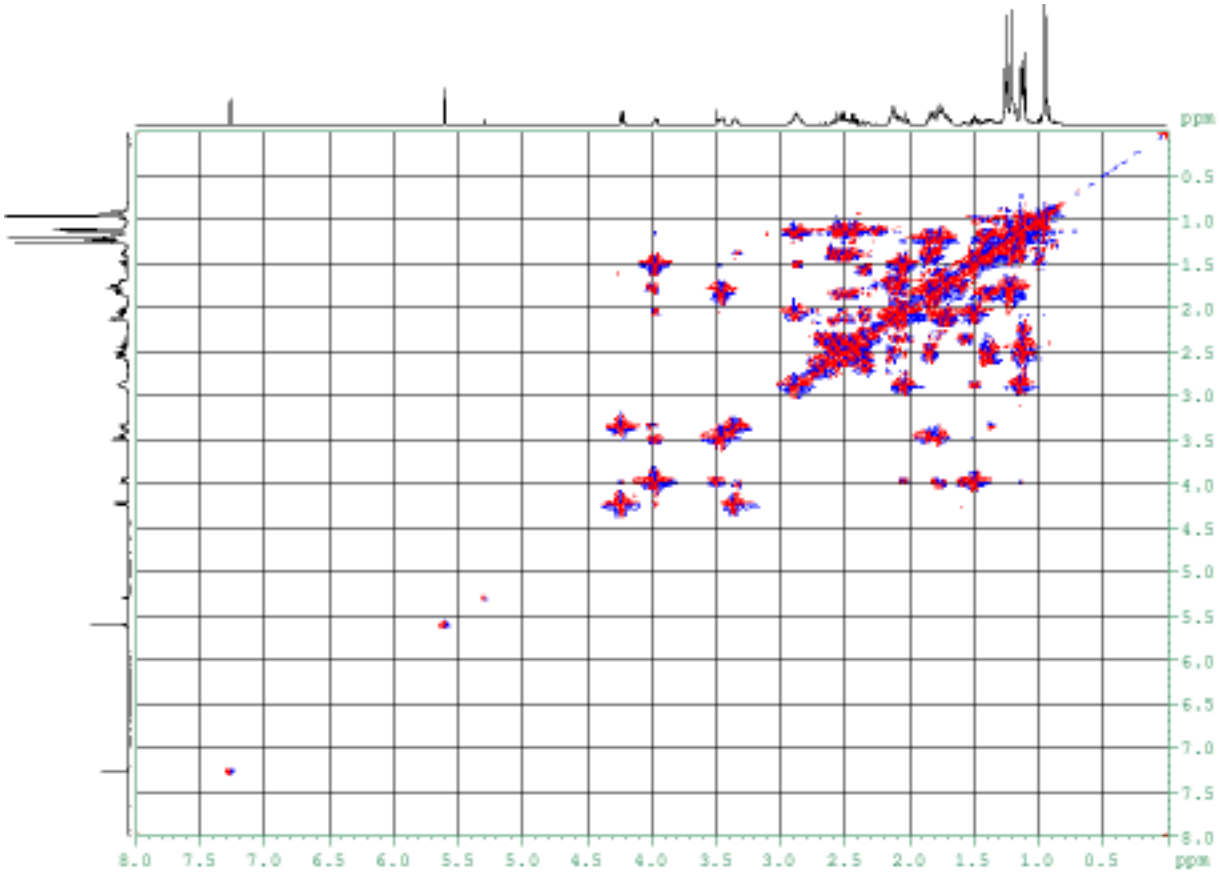

**Spectrum S.3.5.5: DEPT spectrum for compound 5 in $CDCl_3$**



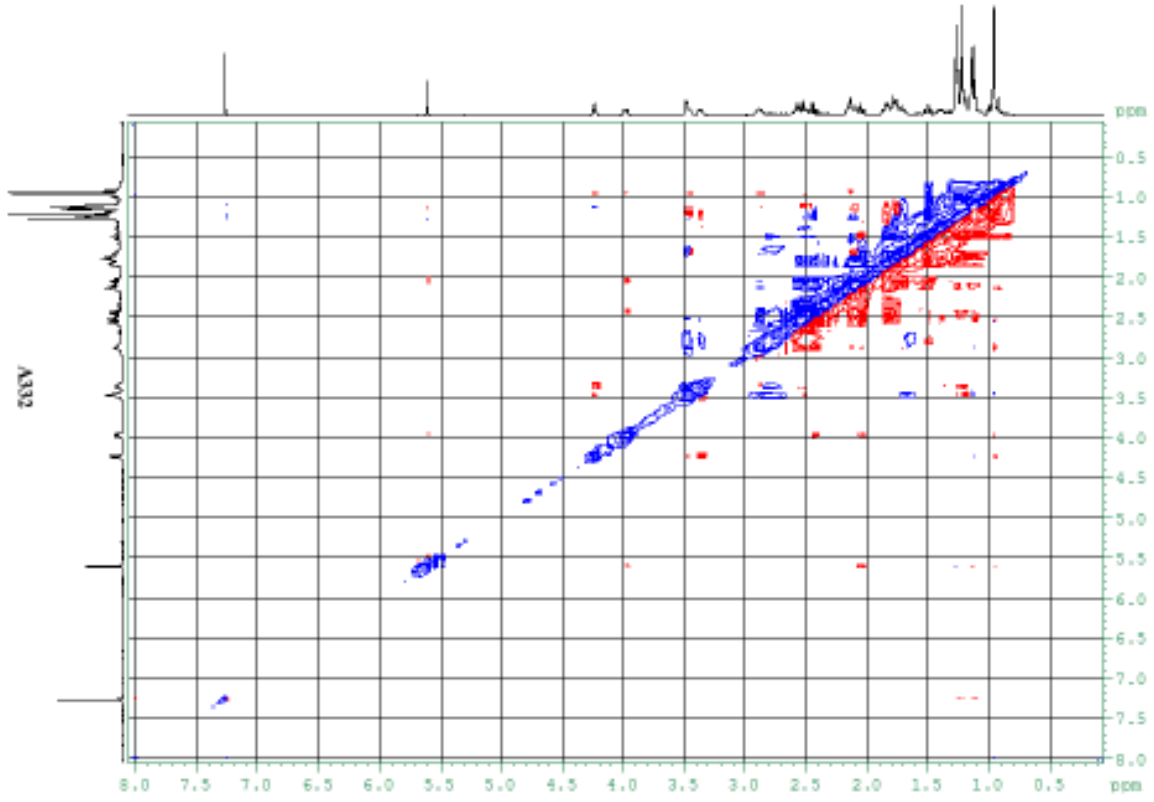

**Spectrum S.3.5_6: HSQCDEPT spectrum for compound 5 in $CDCl_3$**



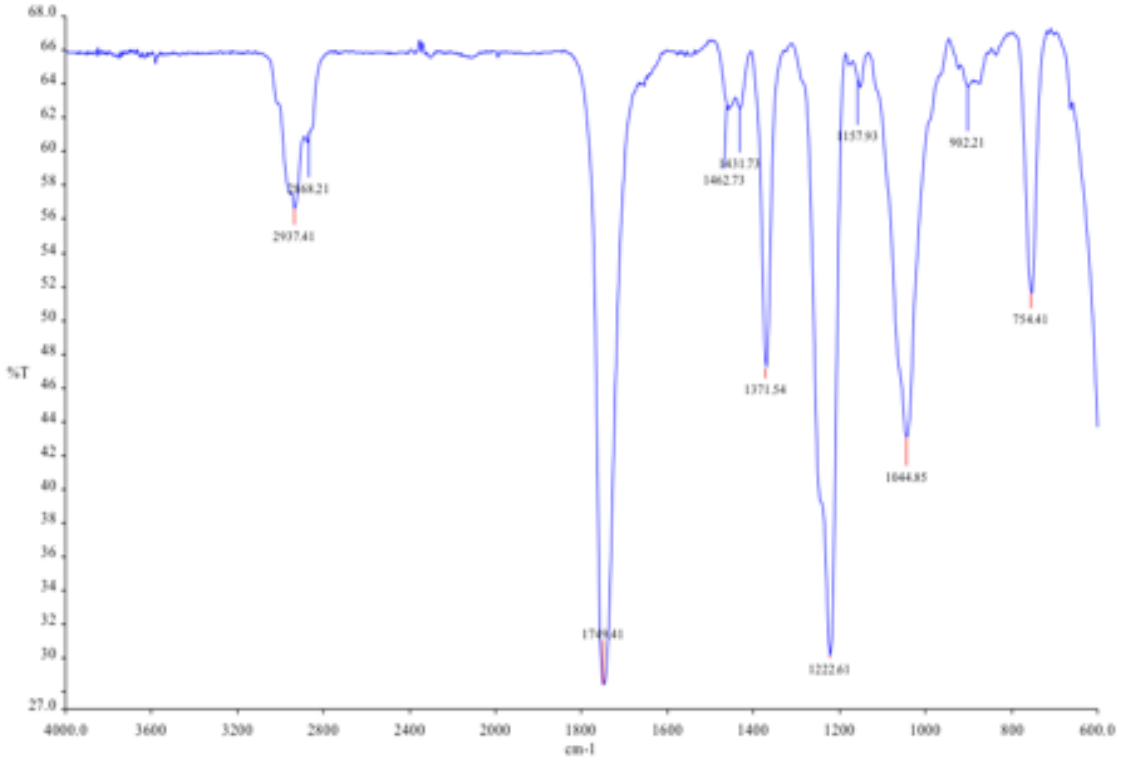

**Spectrum S.3.5.7: HMBC spectrum for compound 5 in $CDCl_3$**



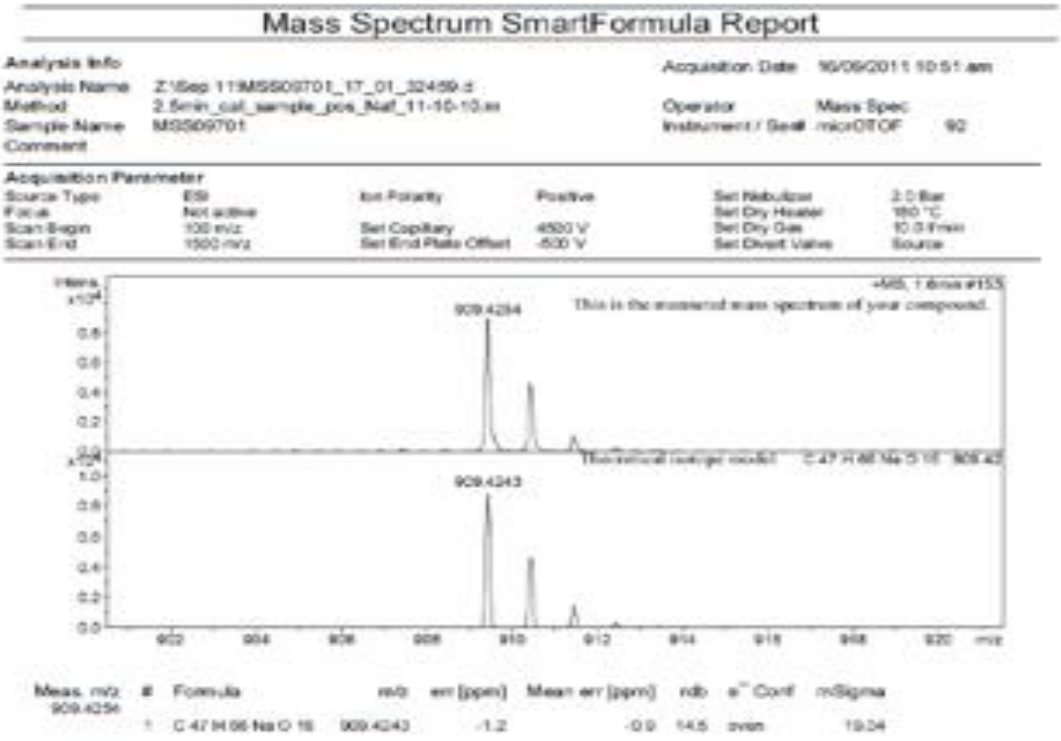

**Spectrum S.3.5_8: COSY spectrum for compound 5 in $CDCl_3$**



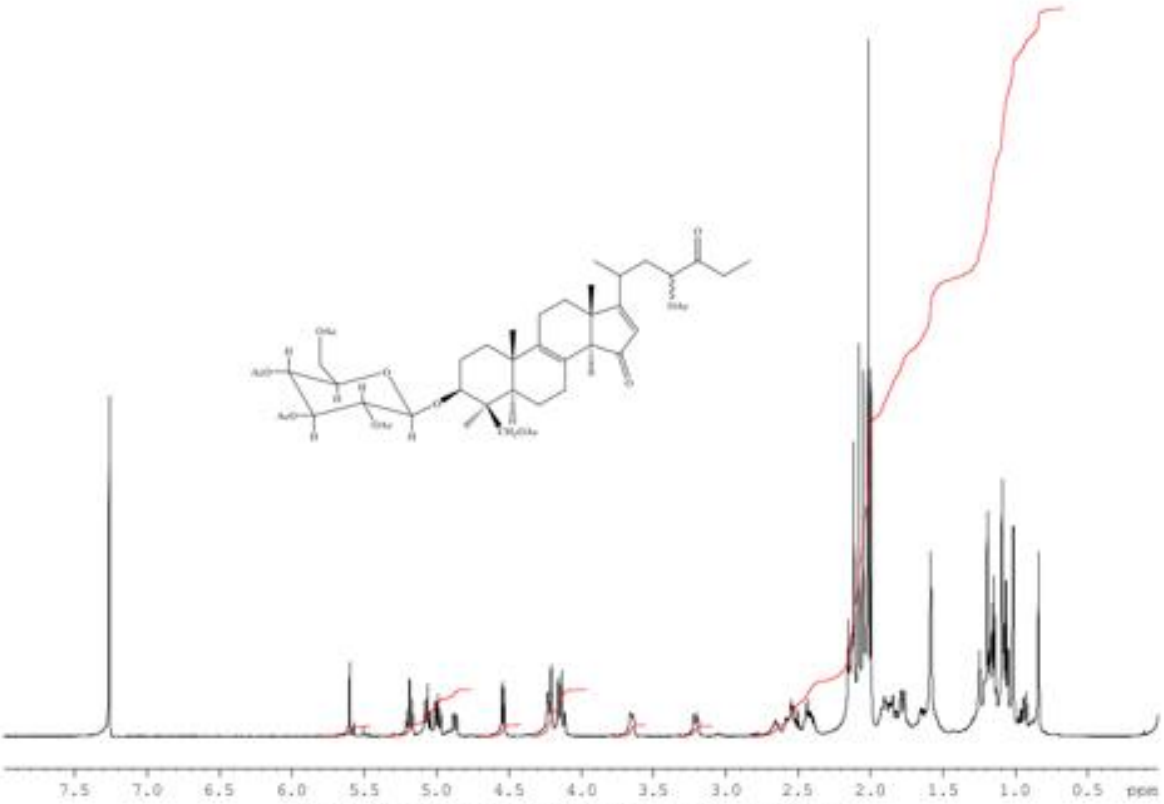

**Spectrum S.3.5.9: NOESY spectrum for compound 5 in $CDCl_3$**



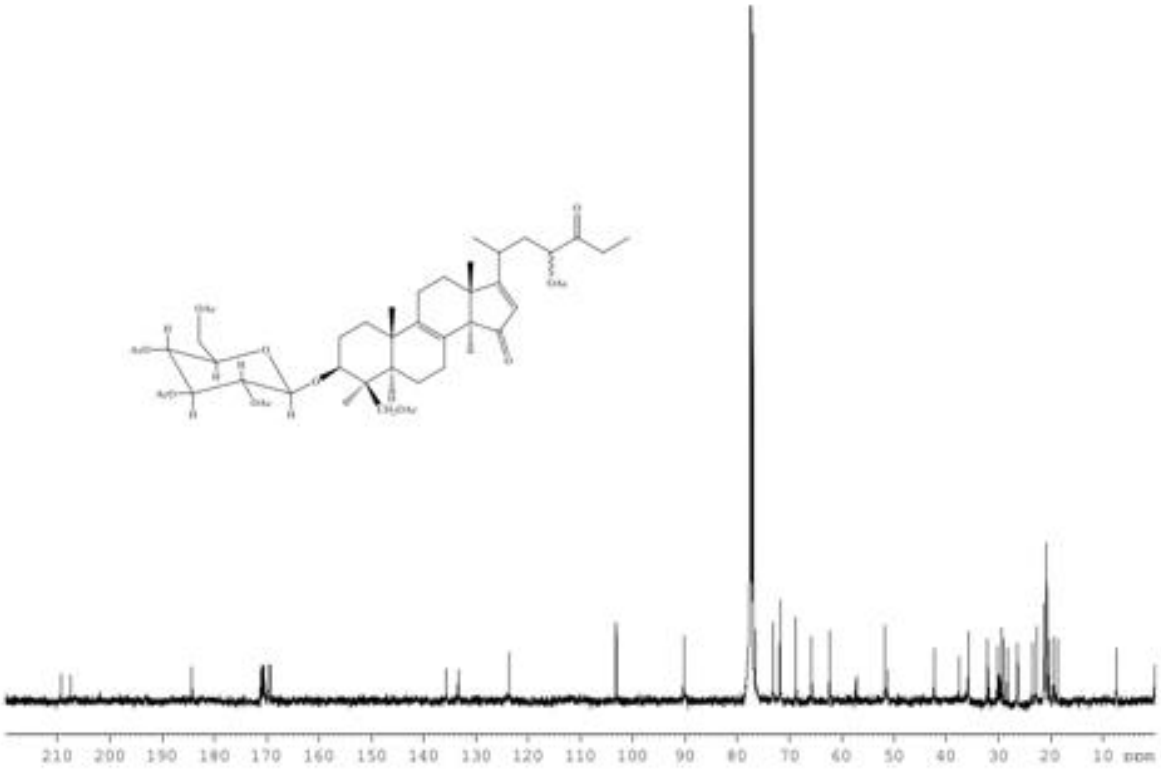

**Spectrum S3.6.2: FTIR spectrum for compound 6Ac**



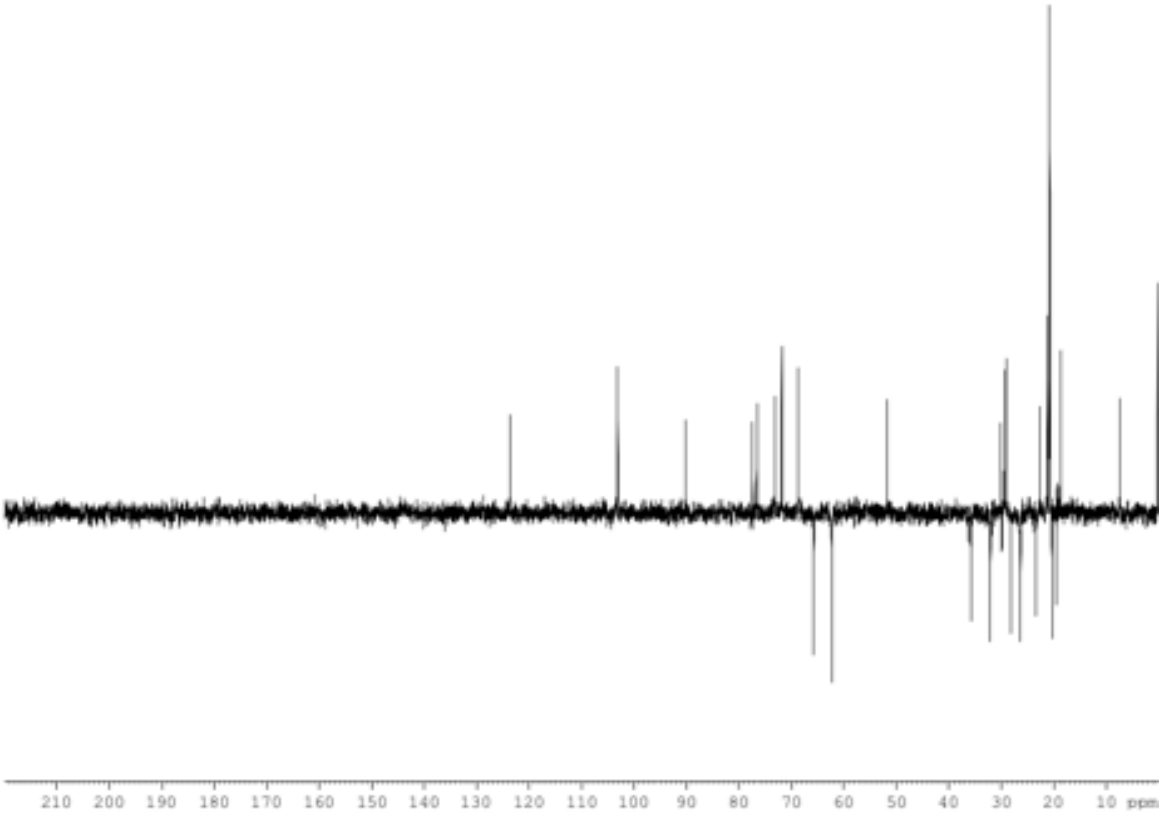

**Spectrum S3.6.1: Mass spectrum for compound 6Ac**



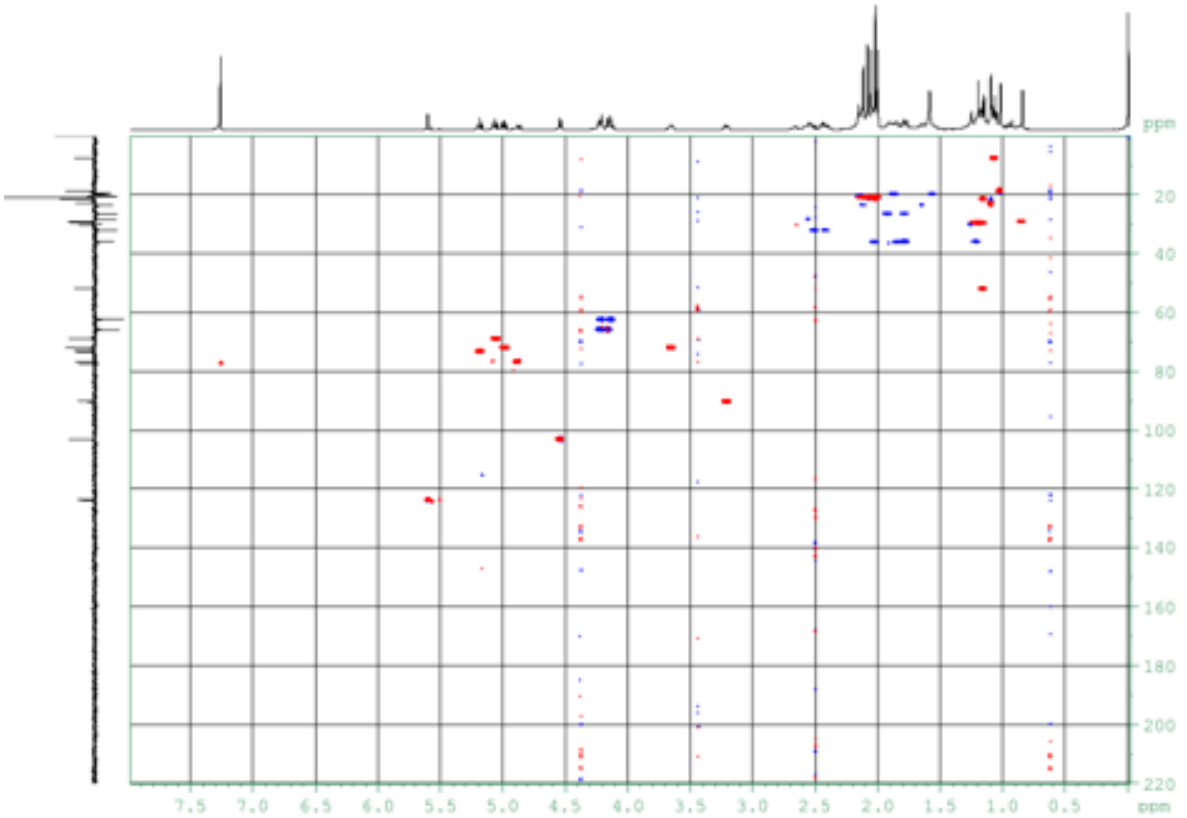

**Spectrum S3.6.3: $^{1}H$ NMR spectrum for compound 6Ac in $CDCl_3$**



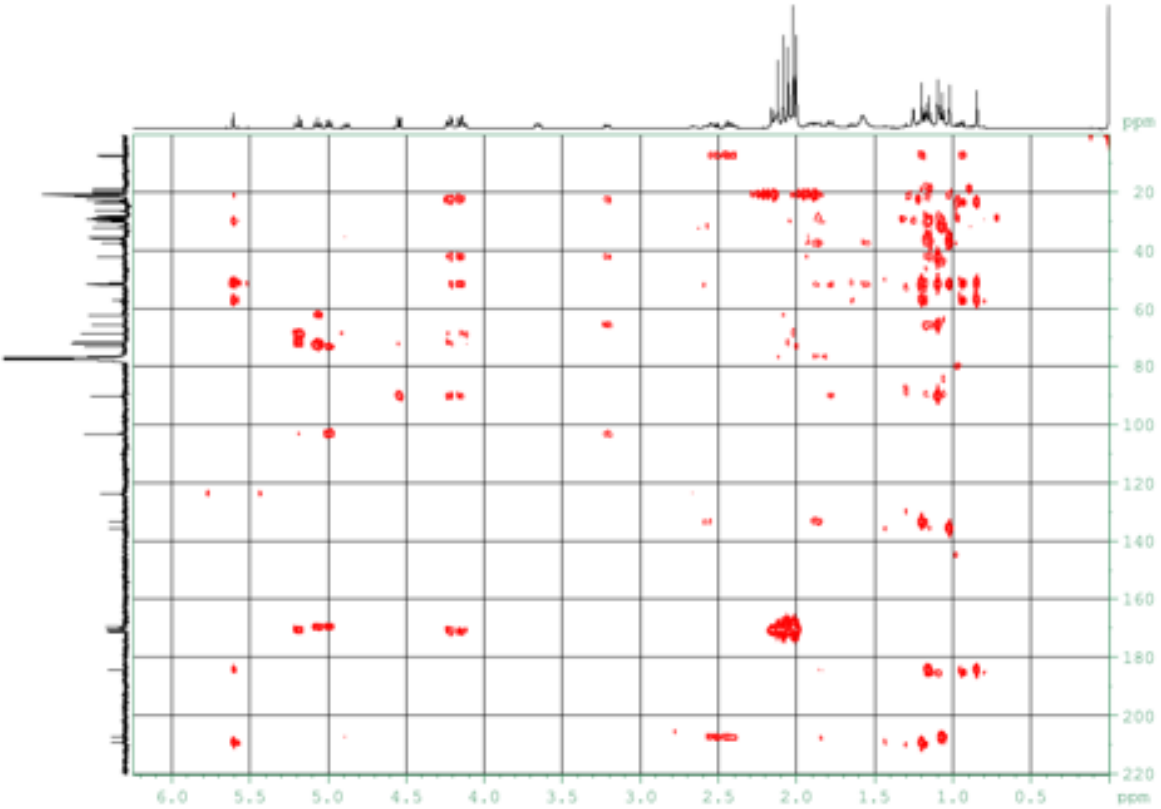

**Spectrum S3.6.4: $^{13}C$ NMR spectrum for compound EB 17 acetate in $CDCl_3$**



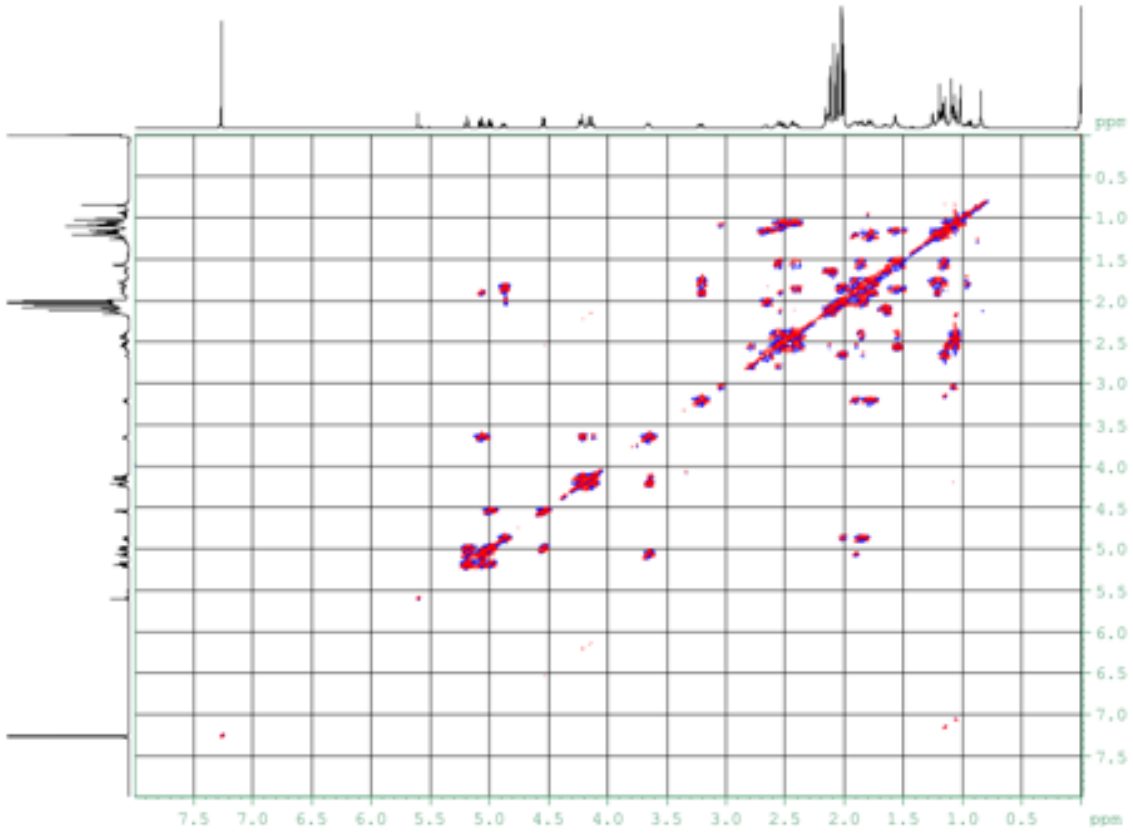

**Spectrum S3.6.5: DEPT spectrum for compound 6Ac in $CDCl_3$**



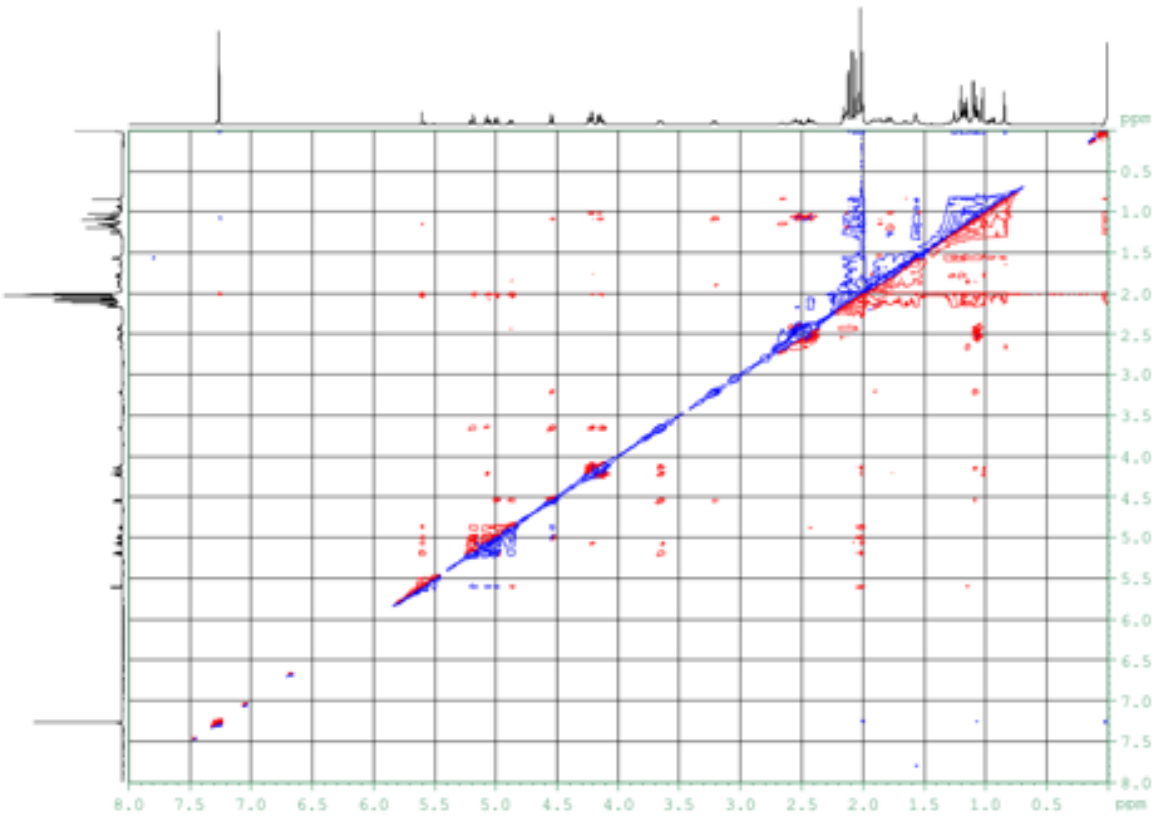

**Spectrum S3.6.6: HSQCDEPT spectrum for compound 6Ac in $CDCl_3$**




**Spectrum S3.6.7: HMBC spectrum for compound 6Ac in $CDCl_3$**




**Spectrum S3.6.8: COSY spectrum for compound 6Ac in $CDCl_3$**




**Spectrum S3.6.9: NOESY spectrum for compound 6Ac in $CDCl_3$**

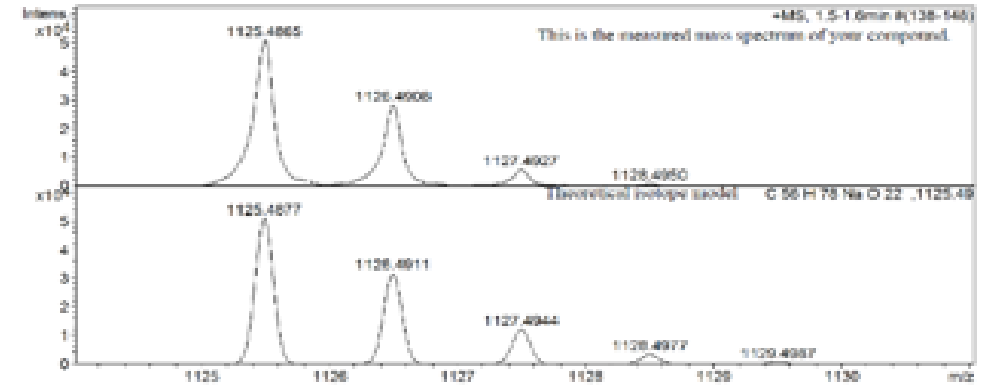
## Mass Spectrum SmartFormula Report

**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | \\Utof\Data\Jun 12\MSS 109881b_84_01_44082.d | Acquisition Date | 25/06/2012 17:08:44 |
| Method | 2.5min_cal_sample_pos_Naf_Mid_mass.m | Operator | Mass Spec |
| Sample Name | MSS 109881b | Instrument / Ser# | micrOTOF 92 |
| Comment | | | |

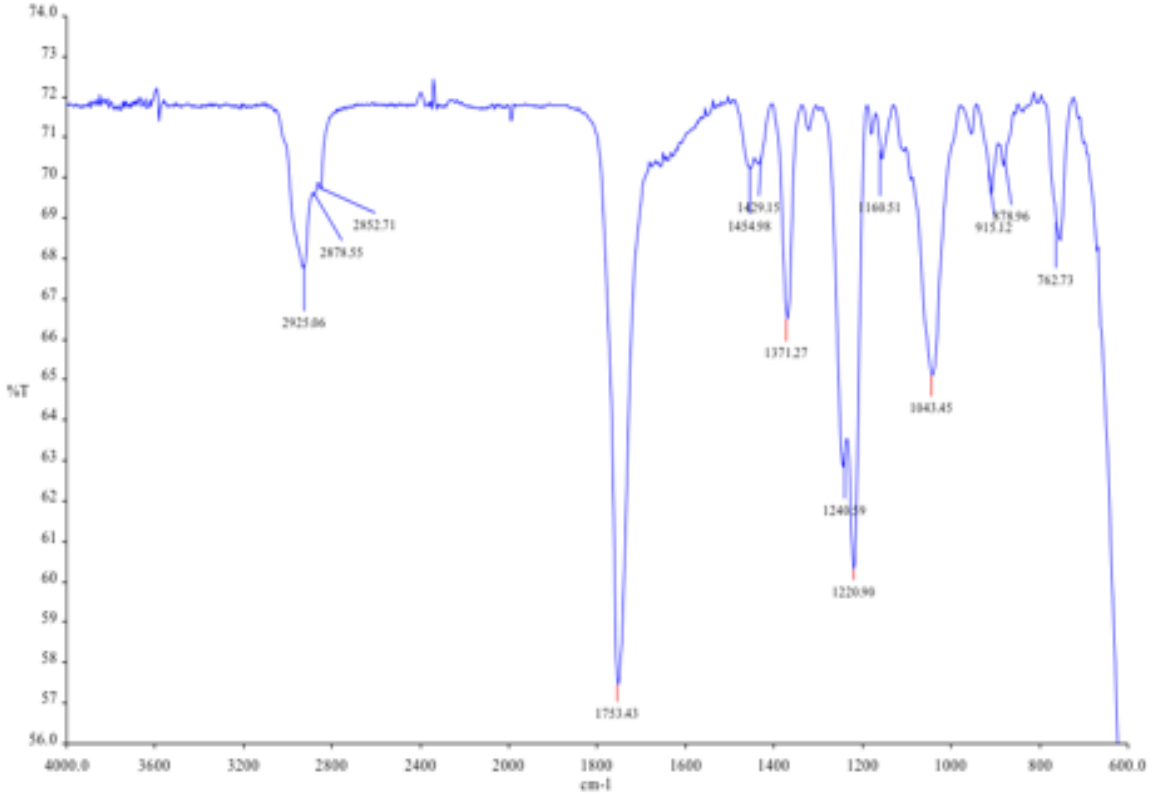
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 2.0 Bar |
| Focus | Not active | | | Set Dry Heater | 180 °C |
| Scan Begin | 100 m/z | Set Capillary | 4500 V | Set Dry Gas | 10.0 l/min |
| Scan End | 1500 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Source |

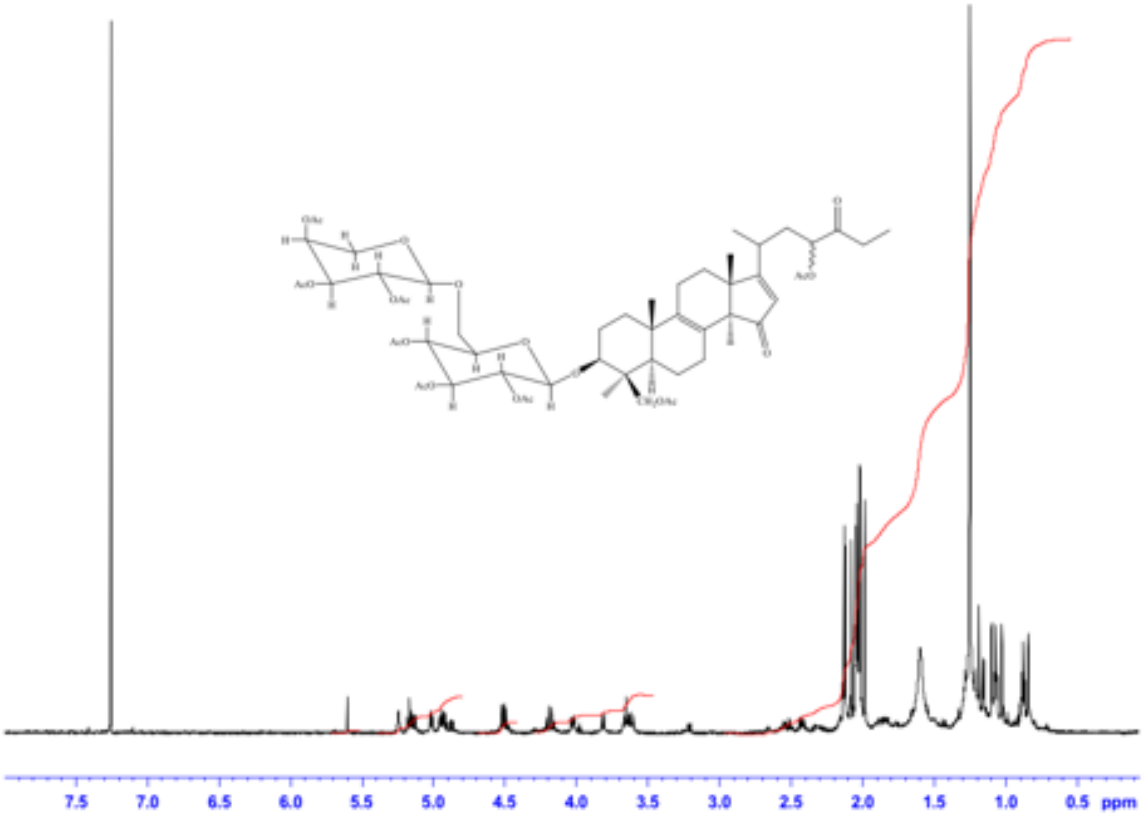



| Meas. m/z | # | Formula | m/z | err [ppm] | Mean err [ppm] | rdb | e⁻ Conf | mSigma |
|---|---|---|---|---|---|---|---|---|
| 1125.4865 | | | | | | | | |
| | 1 | C 56 H 78 Na O 22 | 1125.4877 | 1.0 | 0.8 | 17.5 | even | 67.14 |

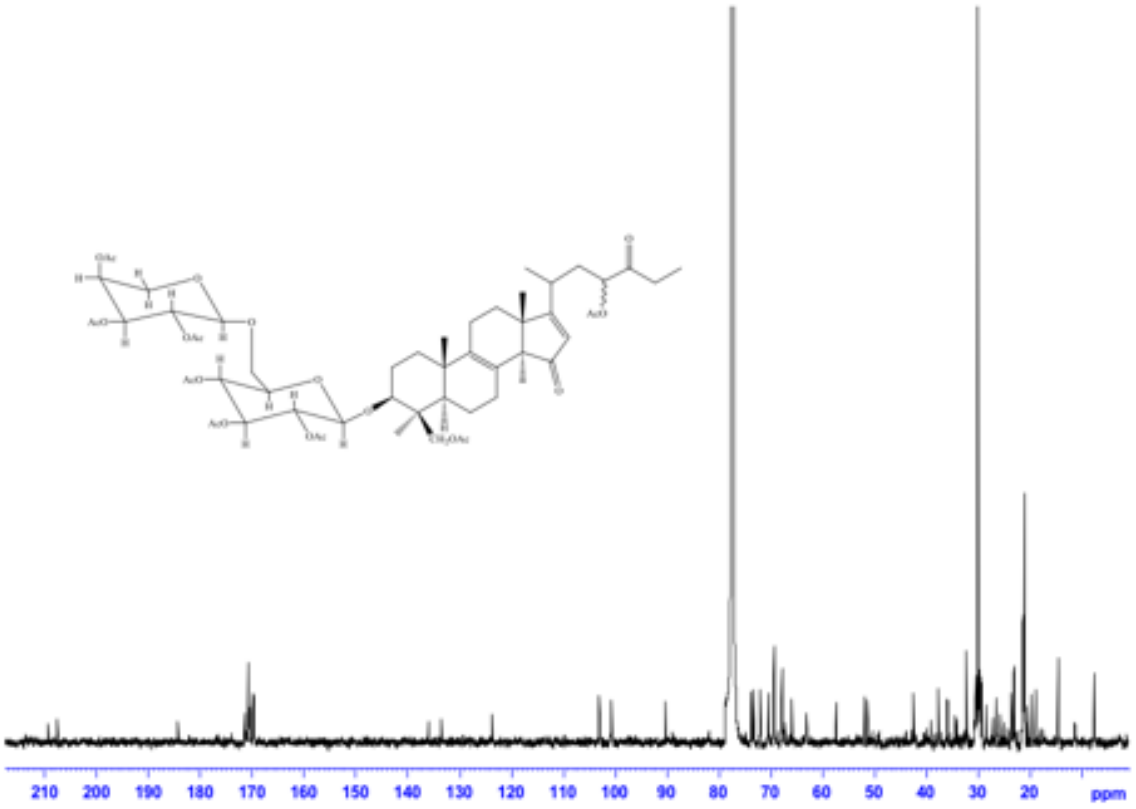
**Spectrum S3.7.1: Mass spectrum for compound 7Ac**



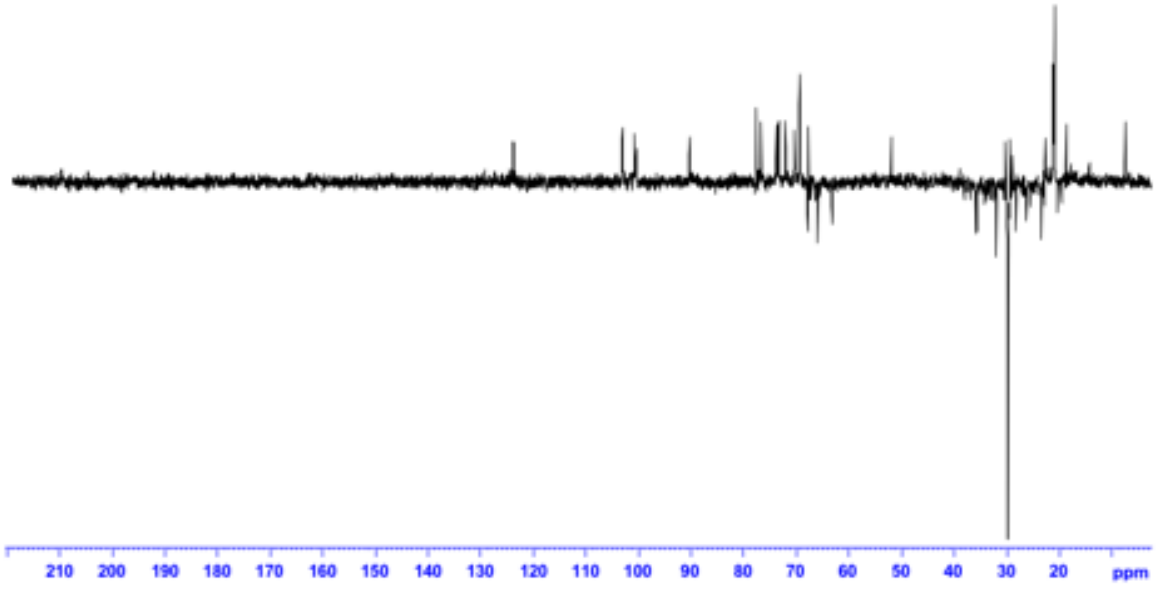

**Spectrum S.3.7.2: FTIR spectrum for compound 7Ac**



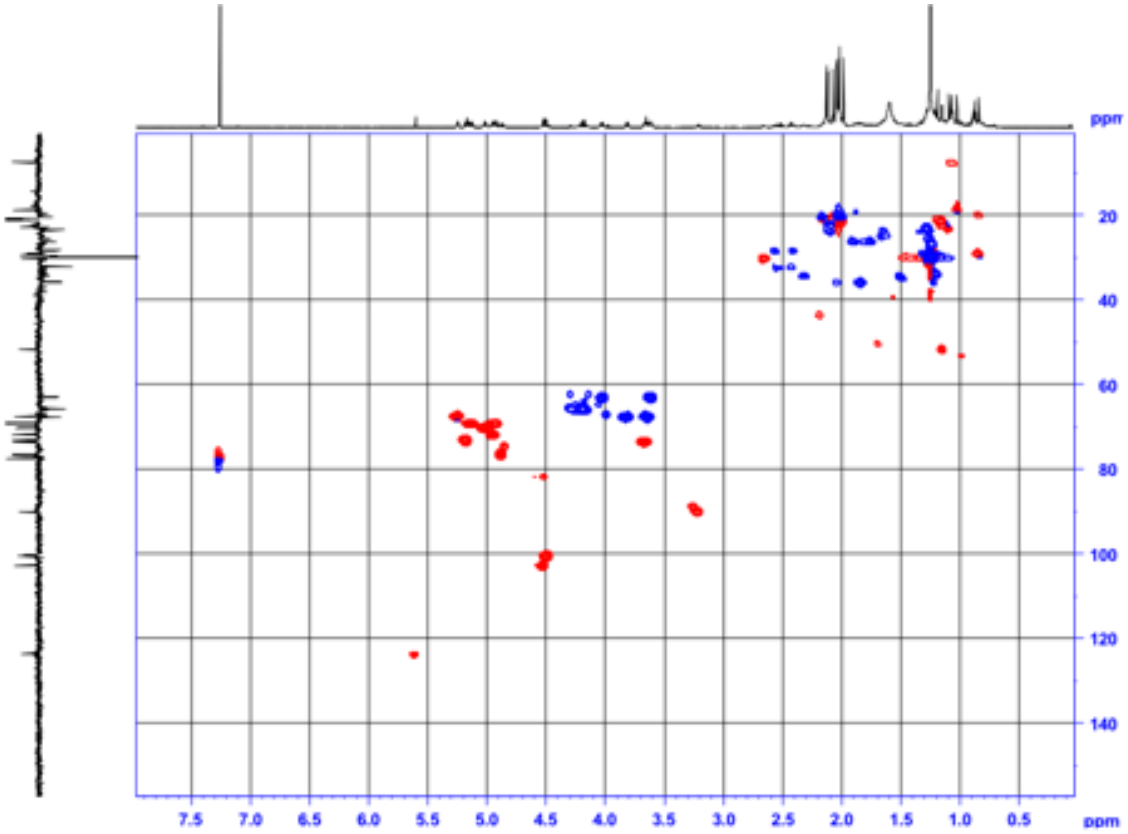

**Spectrum S.3.7.3: $^1H$ NMR spectrum for compound 7Ac in $CDCl_3$**



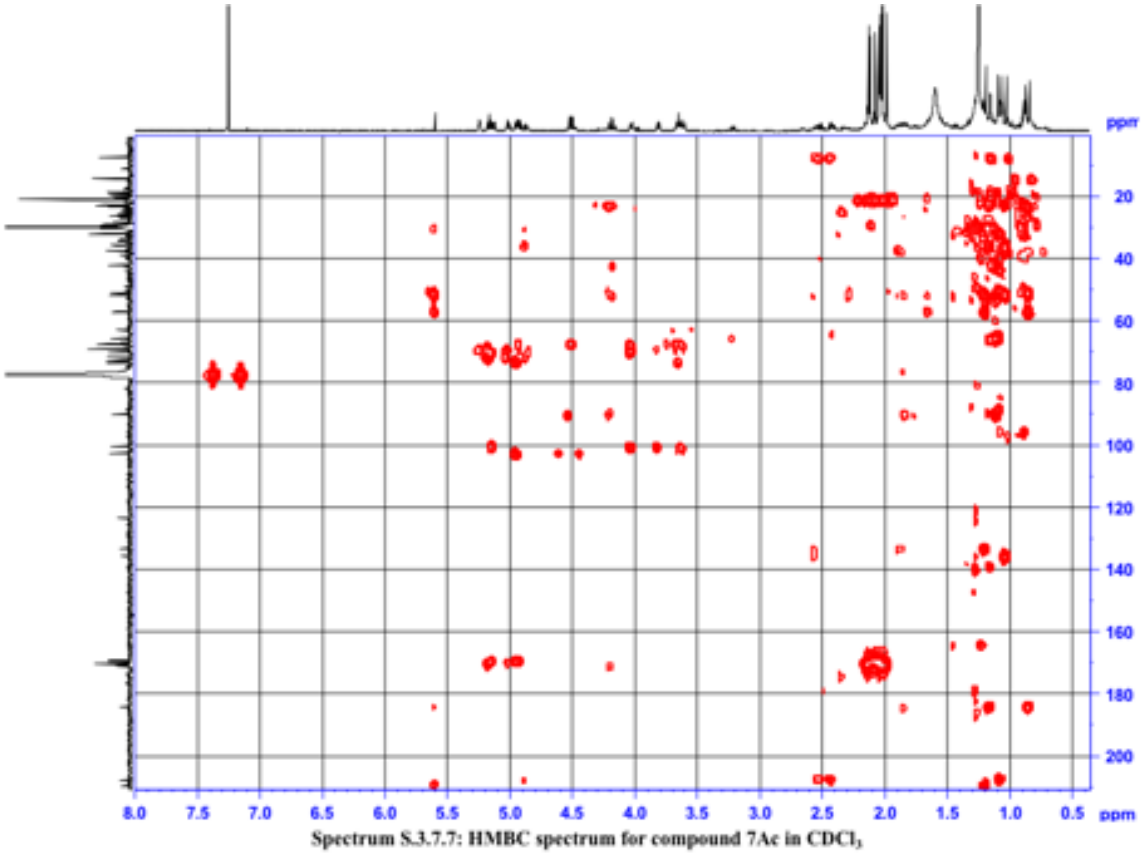

Spectrum S.3.7.4: $^{13}C$ NMR spectrum for compound 7Ac in $CDCl_3$



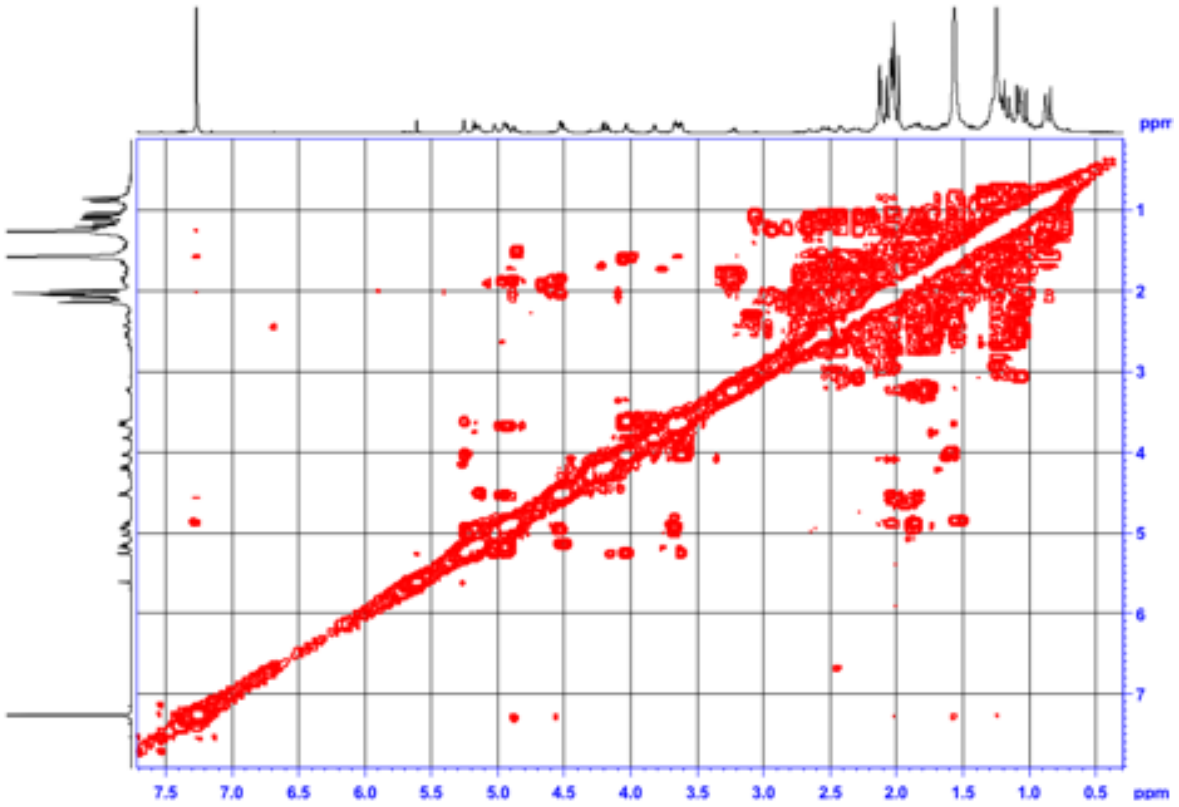

Spectrum S.3.7.5: DEPT spectrum for compound 7Ac in $CDCl_3$



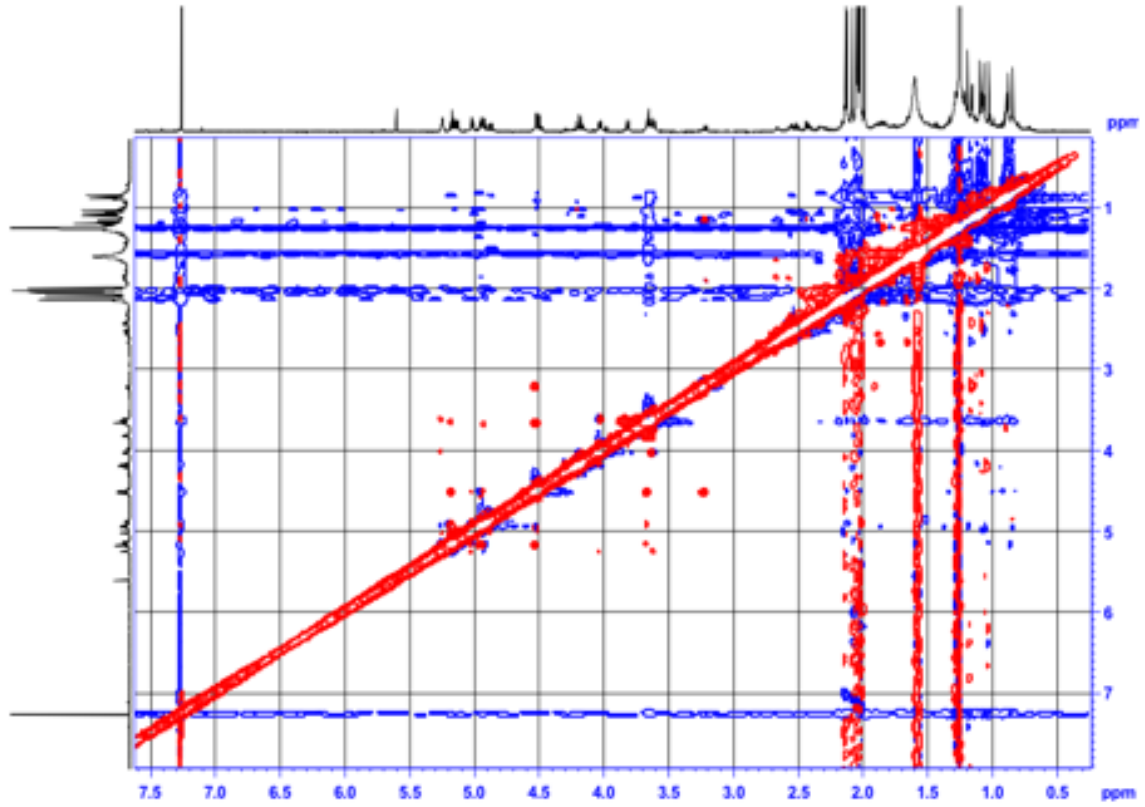

**Spectrum S.3.7.6: HSQCDEPT spectrum for compound 7Ac in $CDCl_3$**



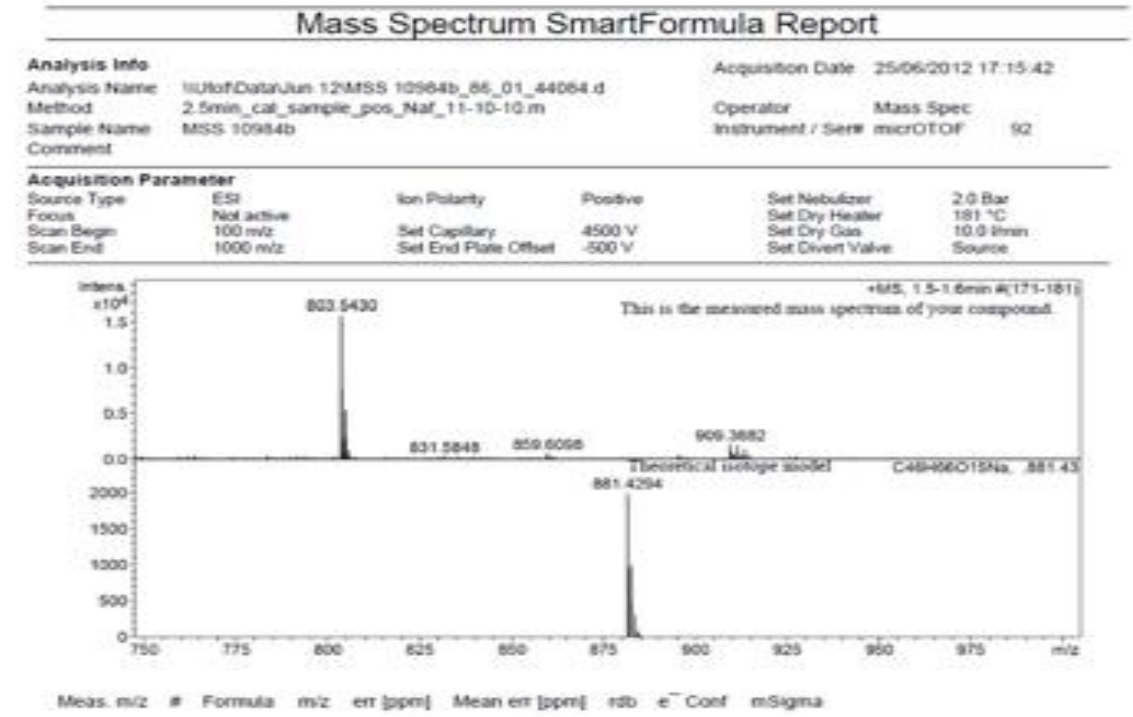

**Spectrum S.3.7.7: HMBC spectrum for compound 7Ac in $CDCl_3$**



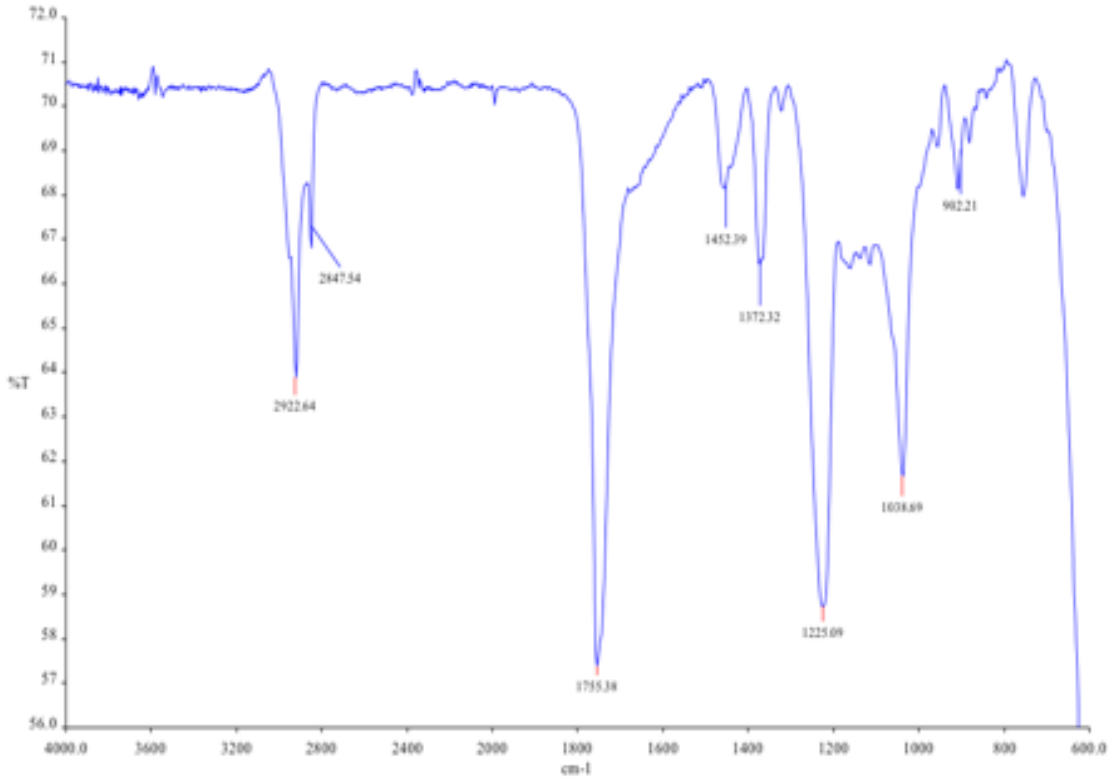

**Spectrum S.3.7.8: COSY spectrum for compound 7Ac in $CDCl_3$**



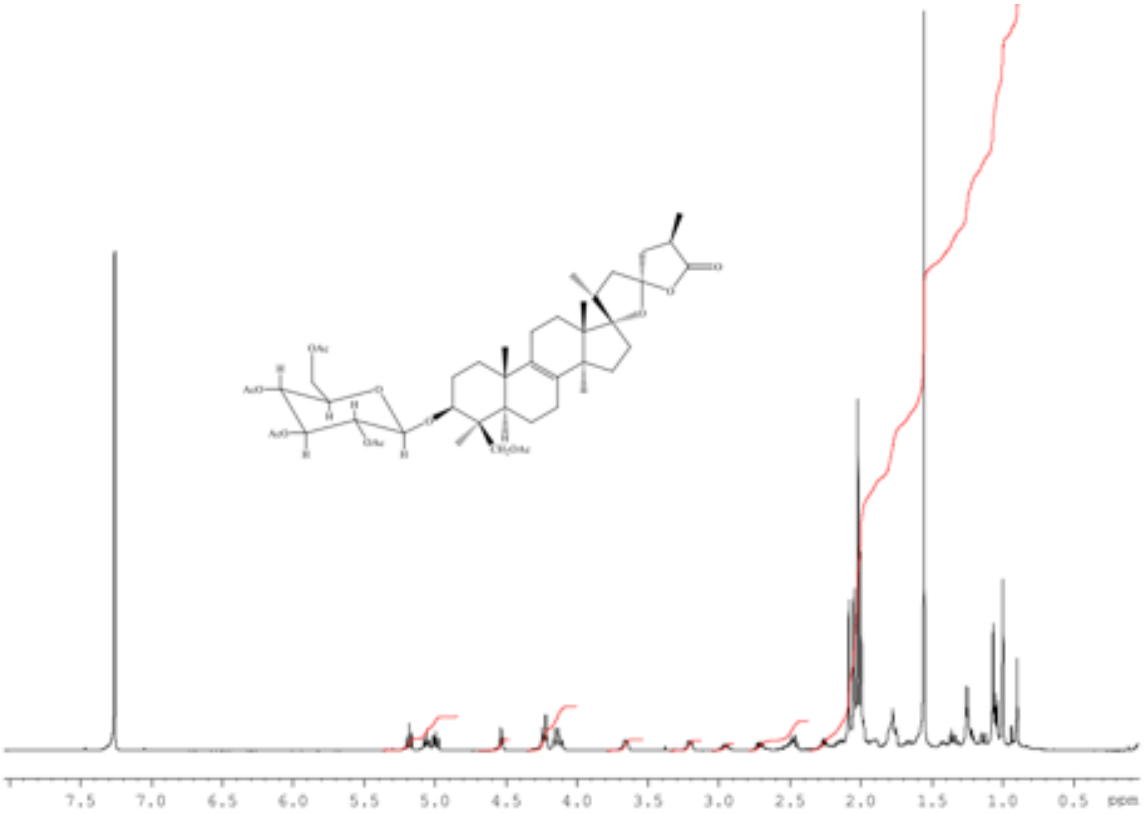

**Spectrum S.3.7.9: NOESY spectrum for compound 7Ac in $CDCl_3$**



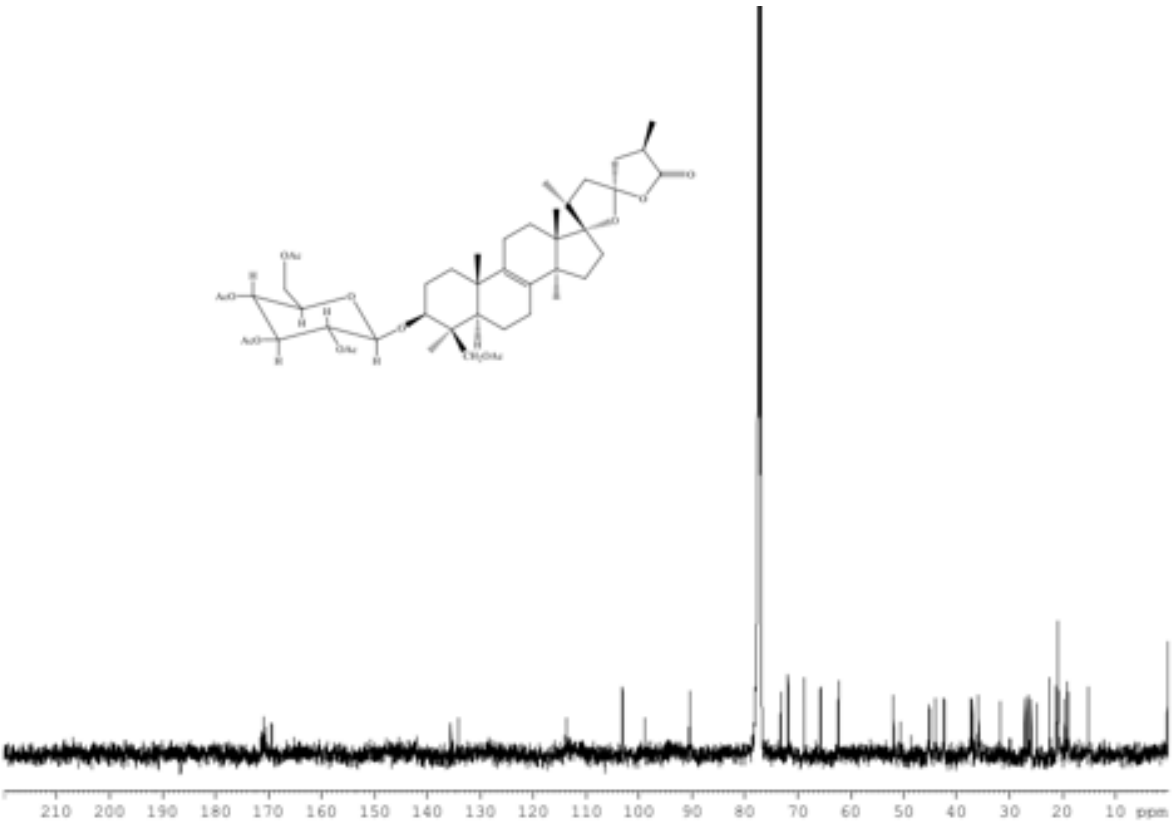

**Spectrum S3.8.1: Mass spectrum for compound 8Ac**



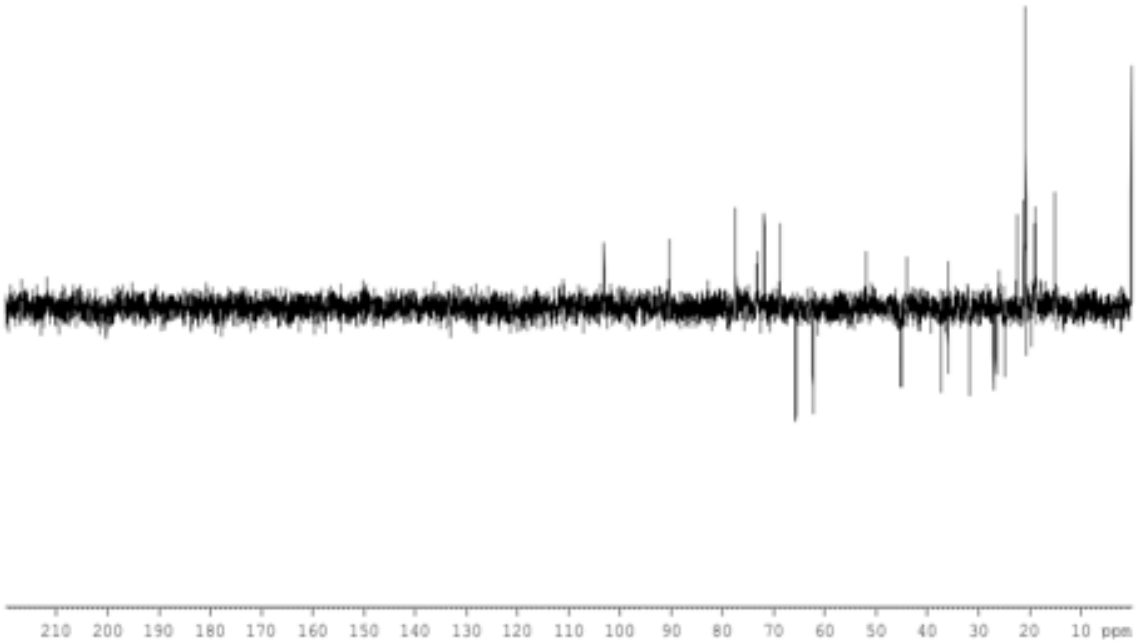

**Spectrum S3.8.2: FTIR spectrum for compound 8A**



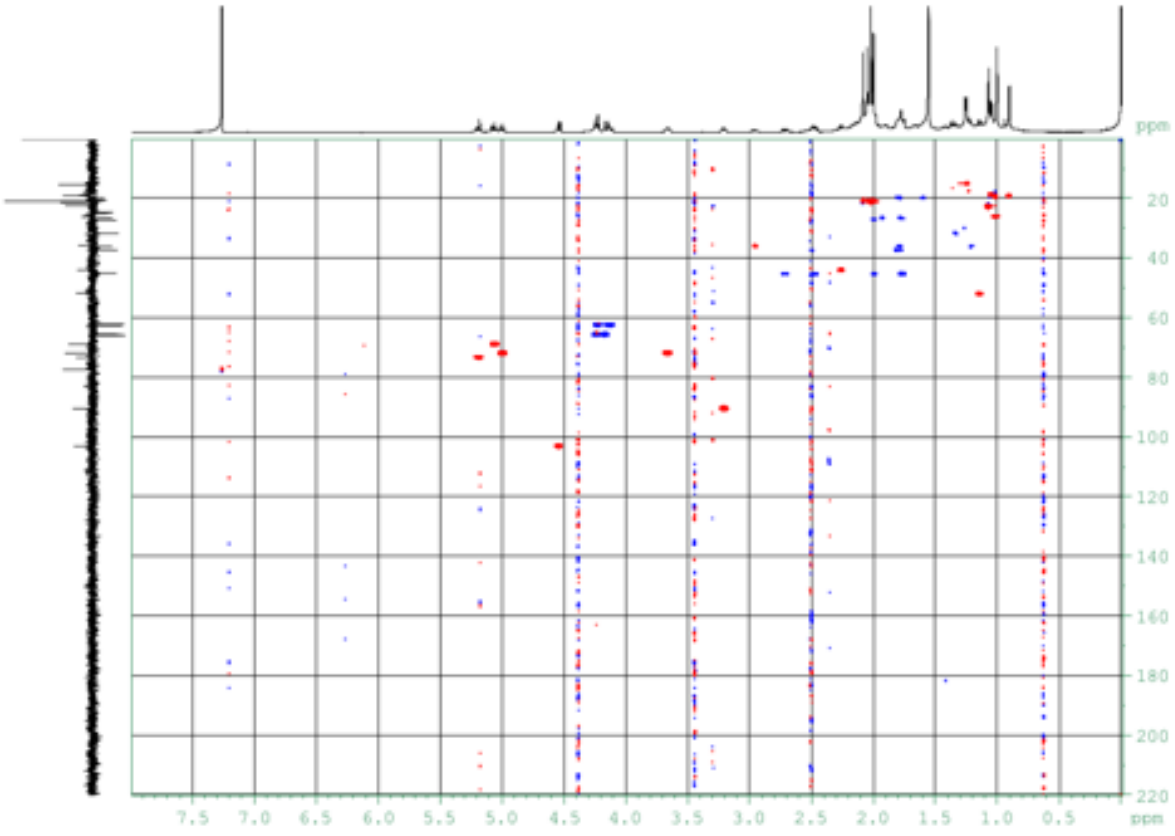

**Spectrum S3.8.3: $^{1}$H NMR spectrum for compound 8Ac in $CDCl_3$**



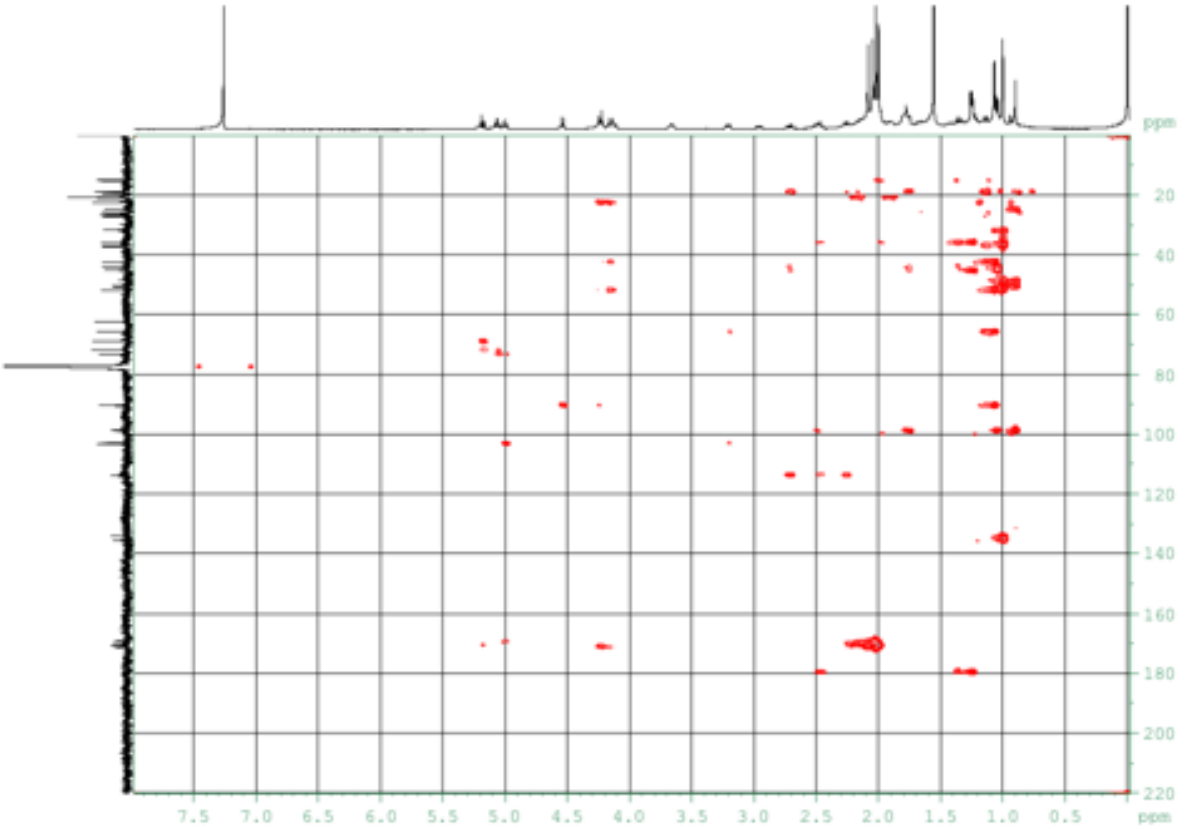

**Spectrum S3.8.4: $^{13}$C NMR spectrum for compound 8Ac in $CDCl_3$**



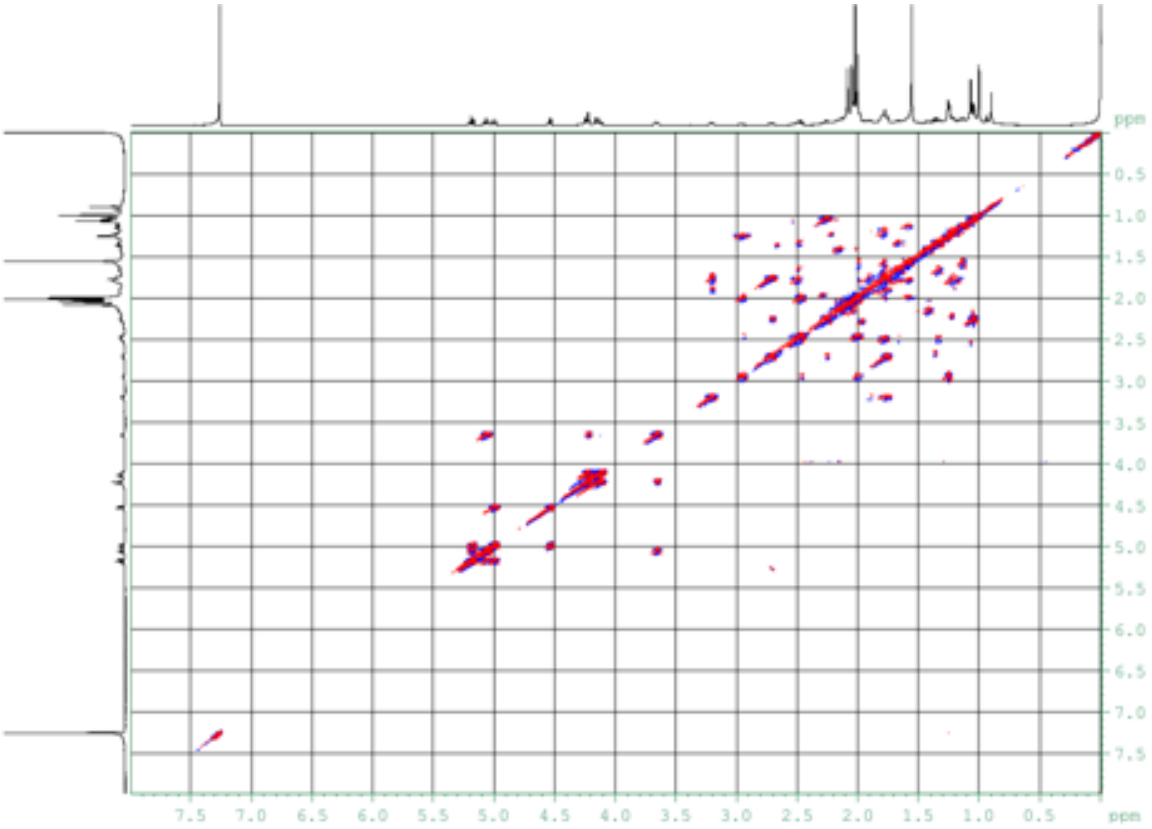

**Spectrum S3.8.5: DEPT spectrum for compound 8Ac in $CDCl_3$**



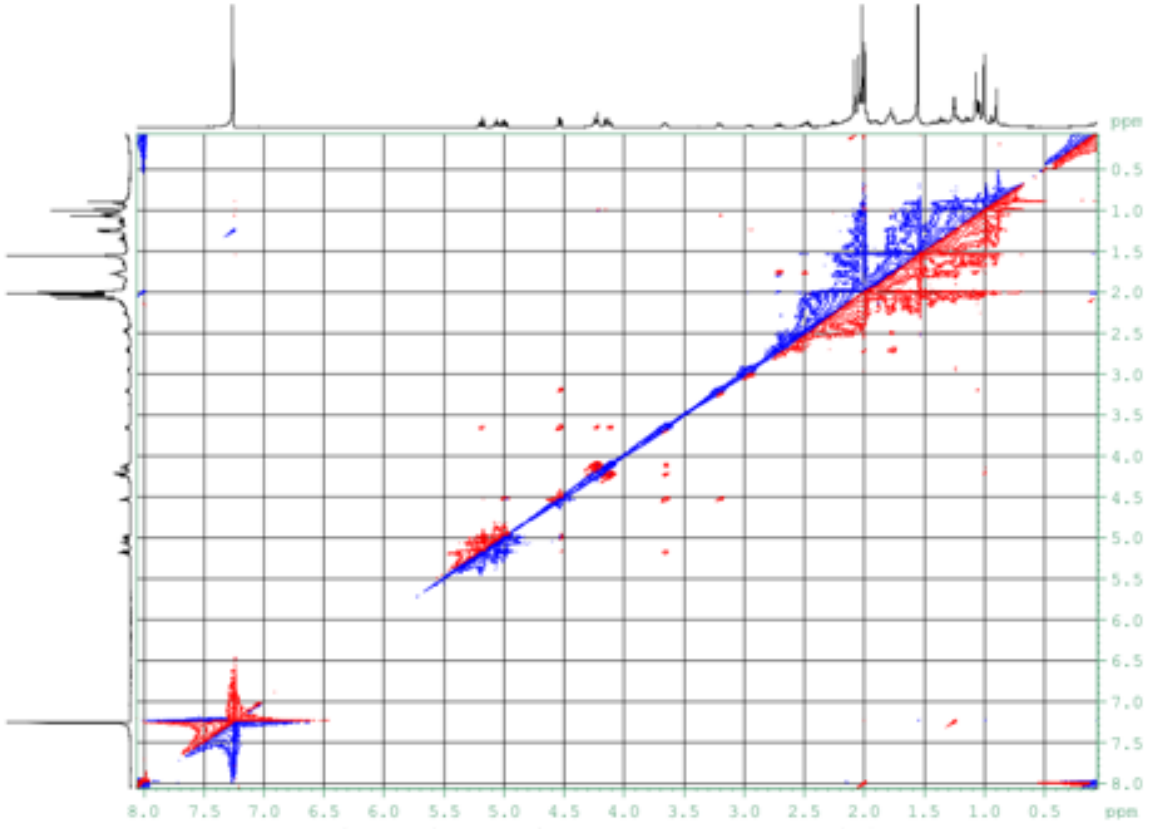

**Spectrum S3.8.6: HSQCDEPT spectrum for compound 8Ac in $CDCl_3$**



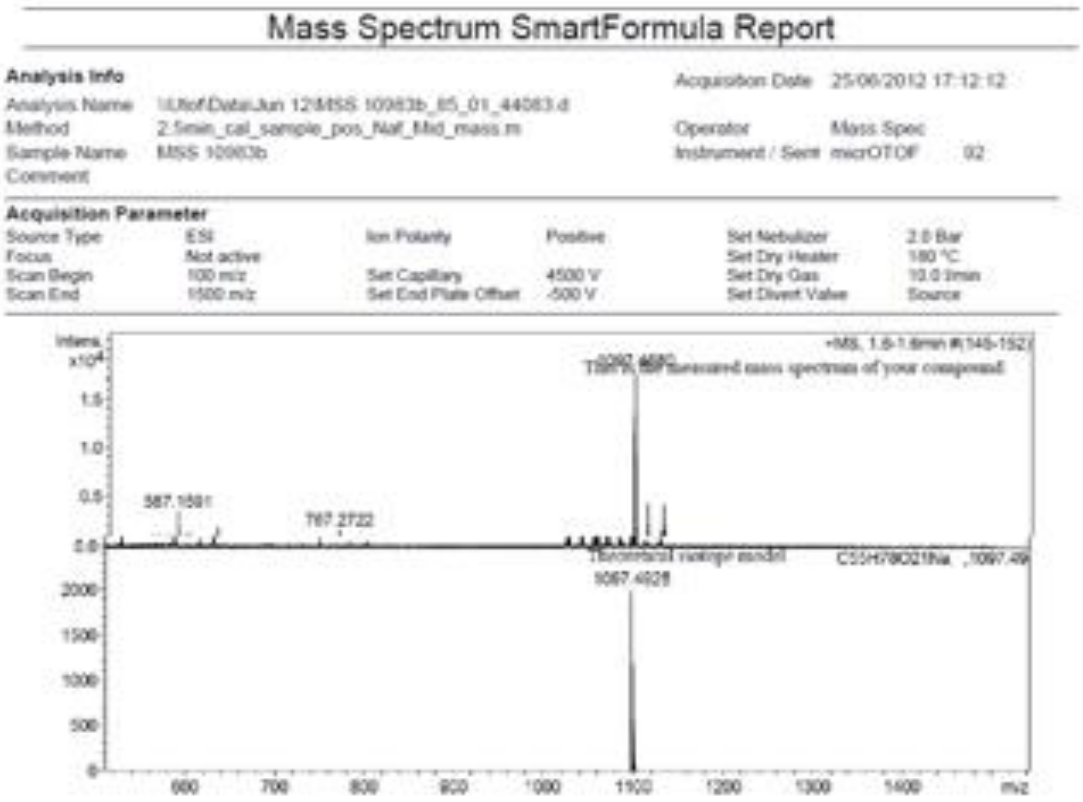

**Spectrum S3.8.7: HMBC spectrum for compound 8Ac in $CDCl_3$**



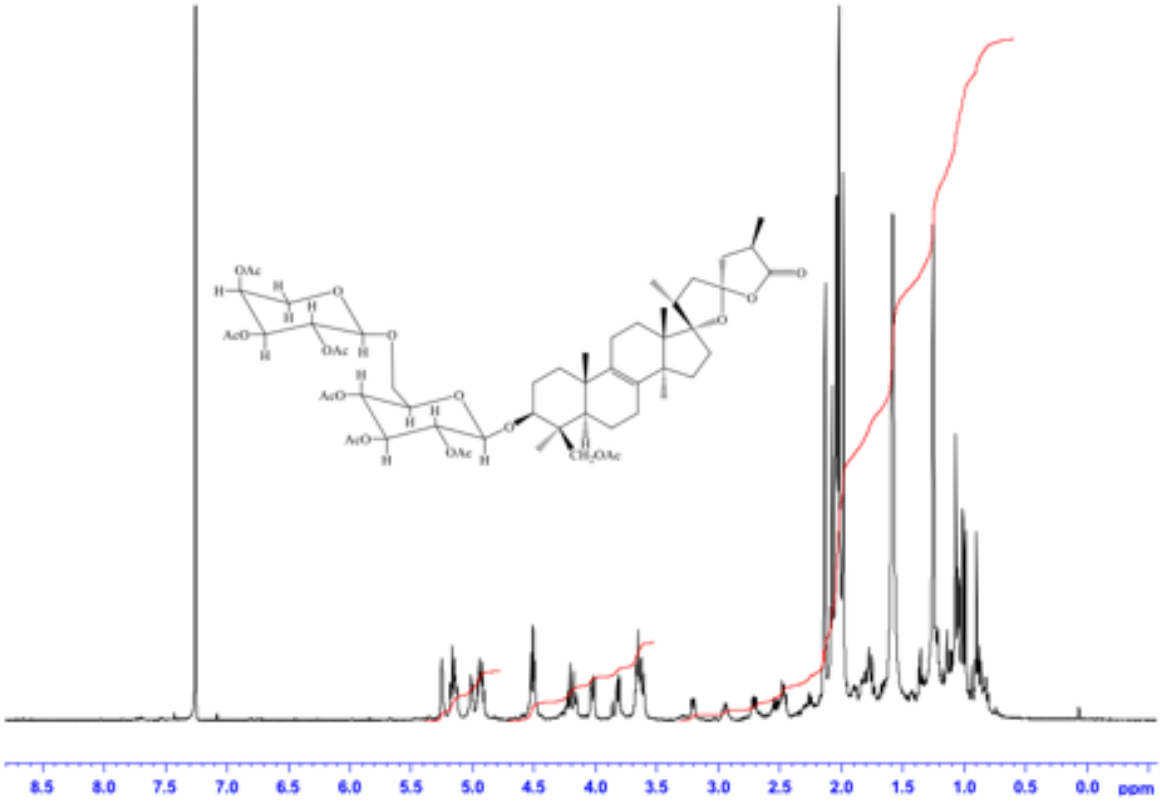

**Spectrum S3.8.8: COSY spectrum for compound 8Ac in $CDCl_3$**



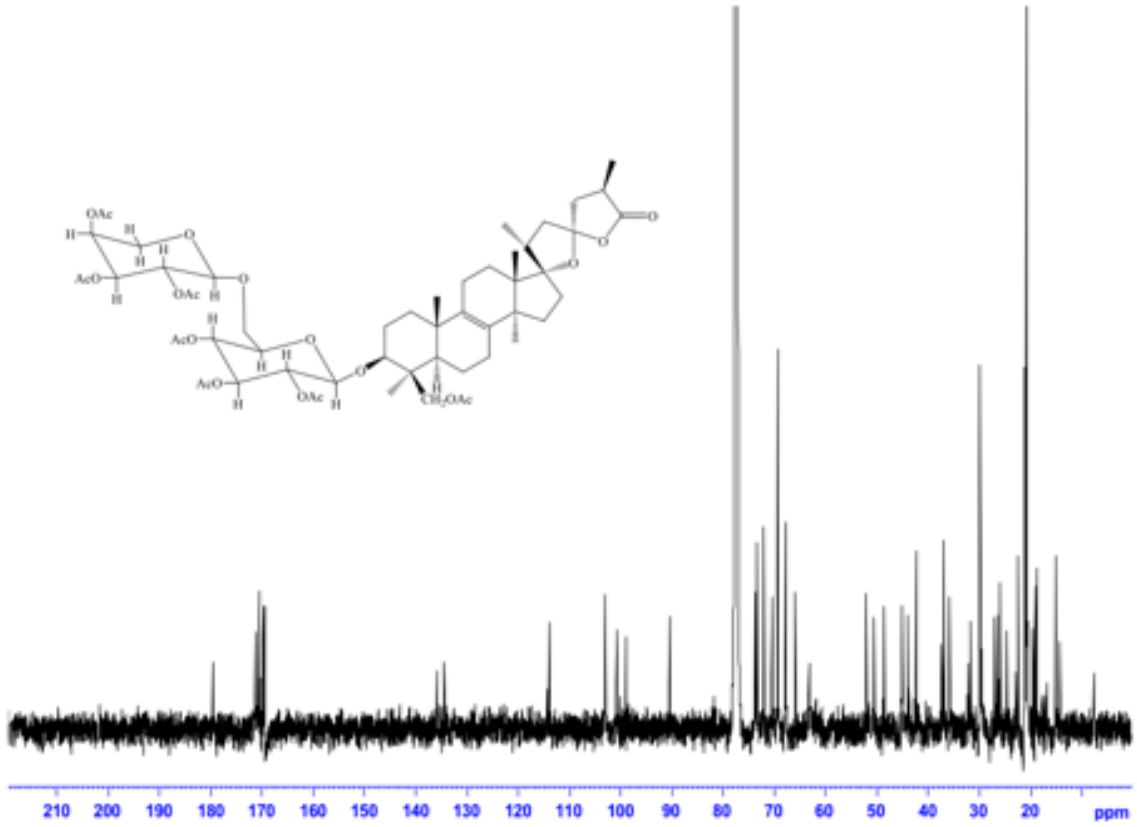

**Spectrum S3.8.9: NOESY spectrum for compound 8Ac in $CDCl_3$**



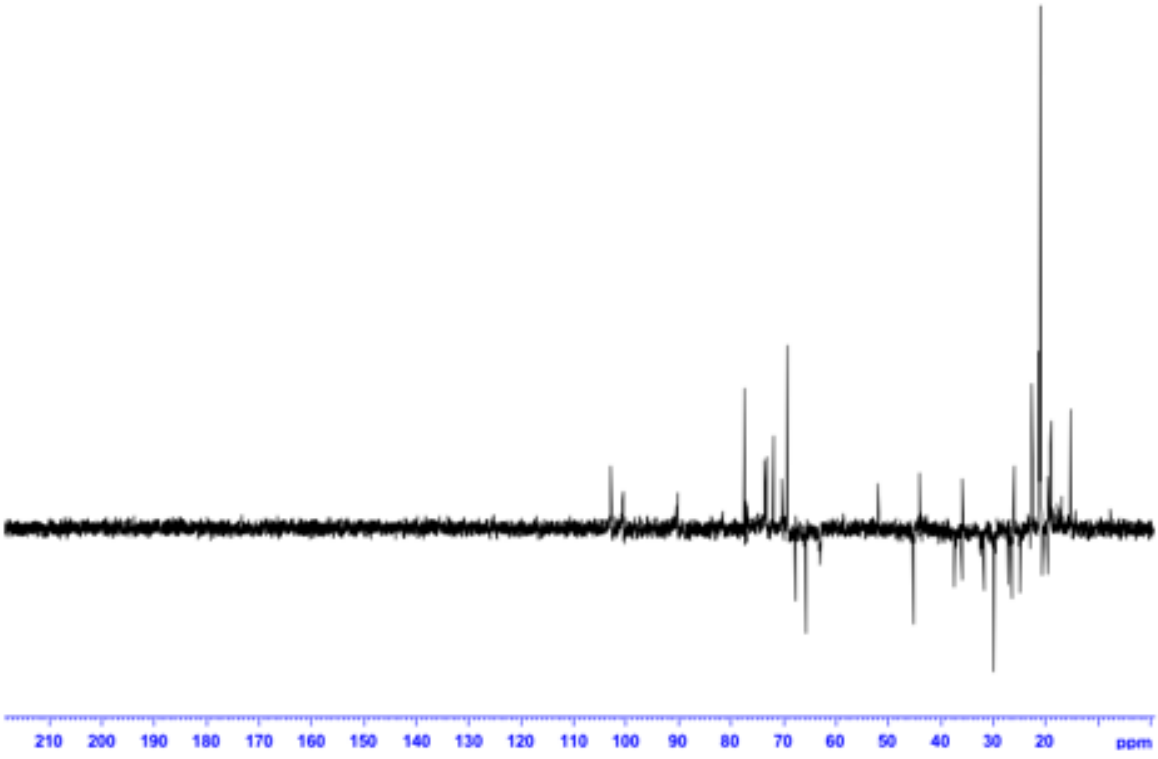

**Spectrum S3.9.1: Mass spectrum for compound 9Ac**



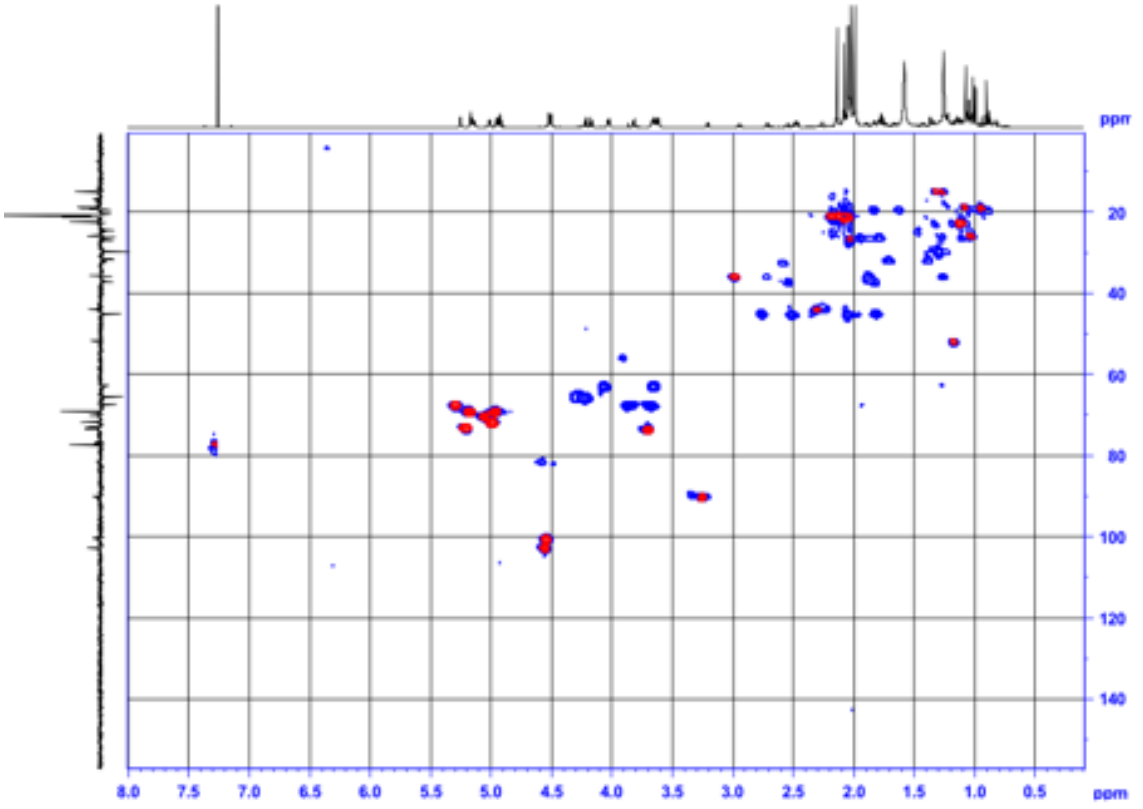

**Spectrum S3.9.3: $^1$H NMR spectrum for compound 9Ac in $CDCl_3$**



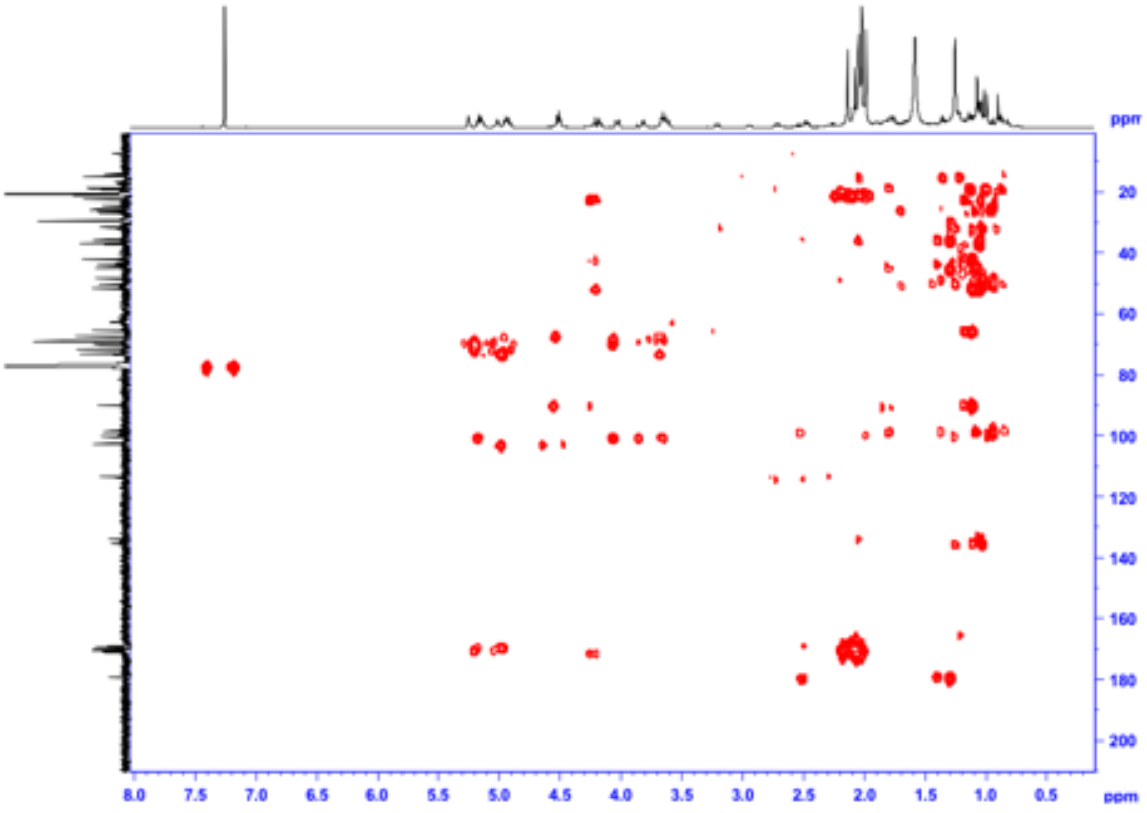

**Spectrum S3.9.4: $^{13}C$ NMR spectrum for compound 9Ac in $CDCl_3$**



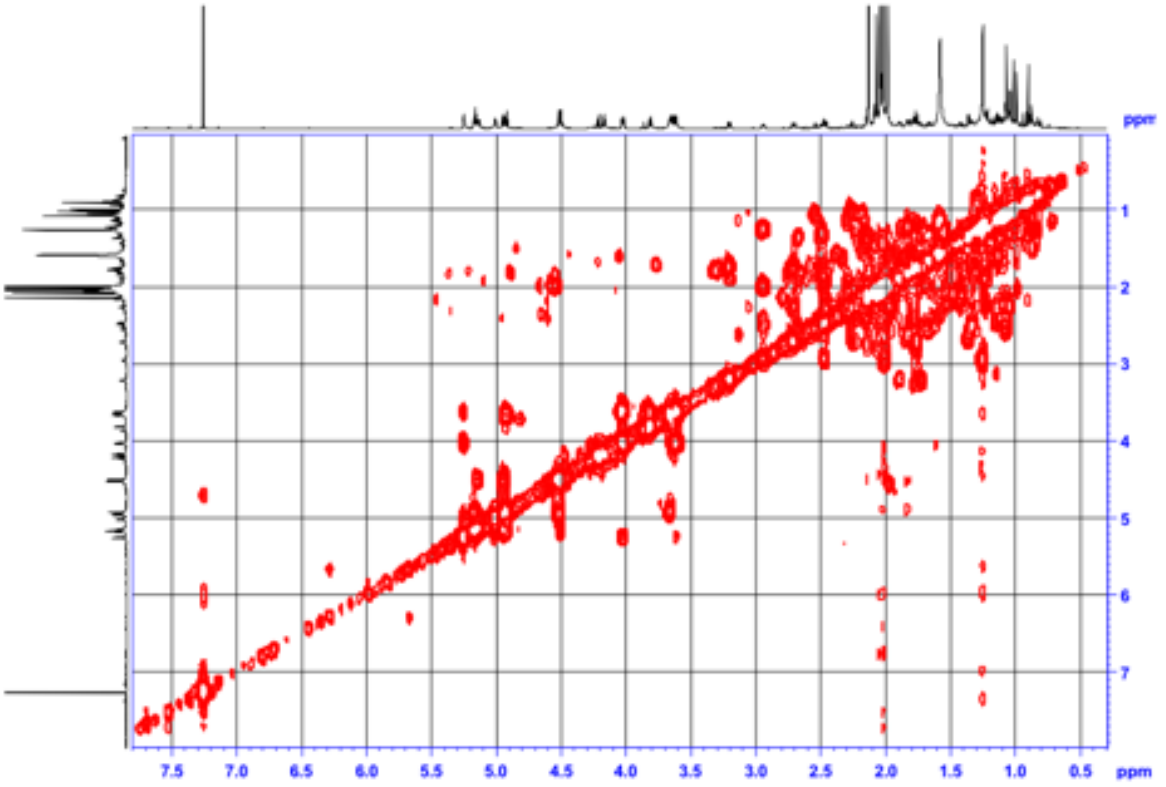

**Spectrum S3.9.5: DEPT spectrum for compound 9Ac in $CDCl_3$**



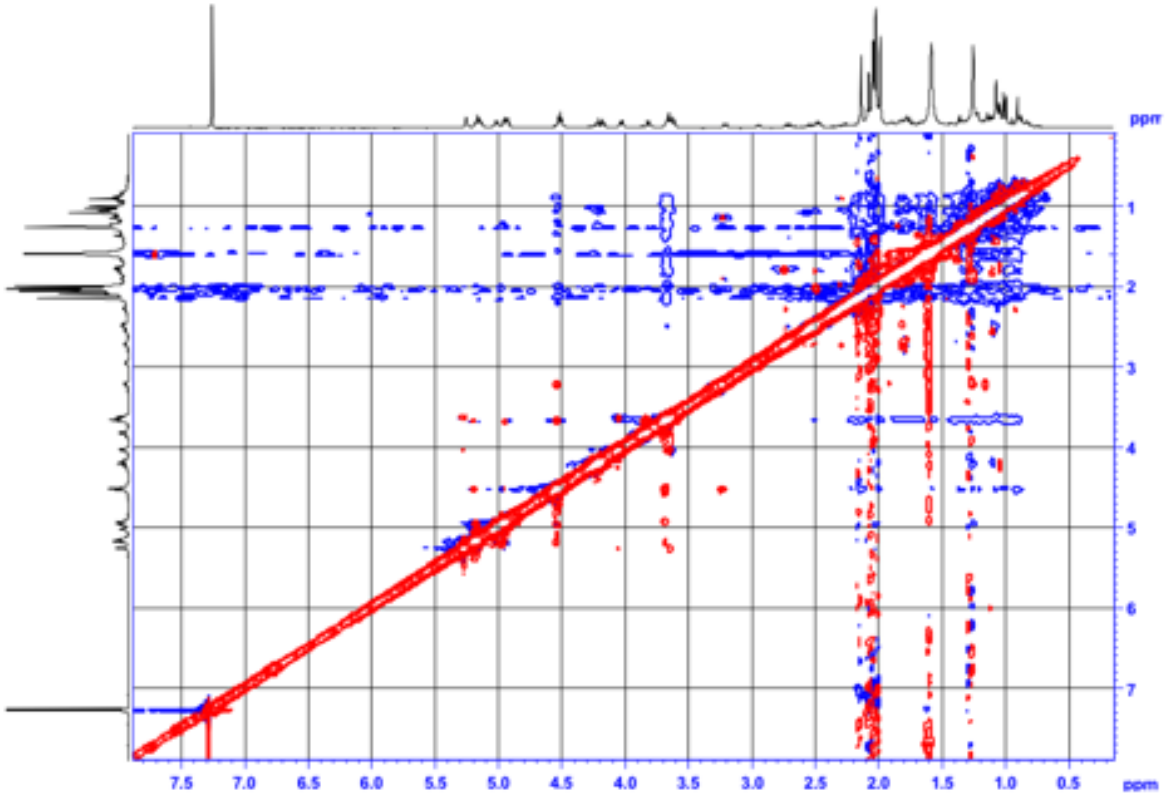

**Spectrum S3.9.6: HSQCDEPT spectrum for compound 9Ac in $CDCl_3$**



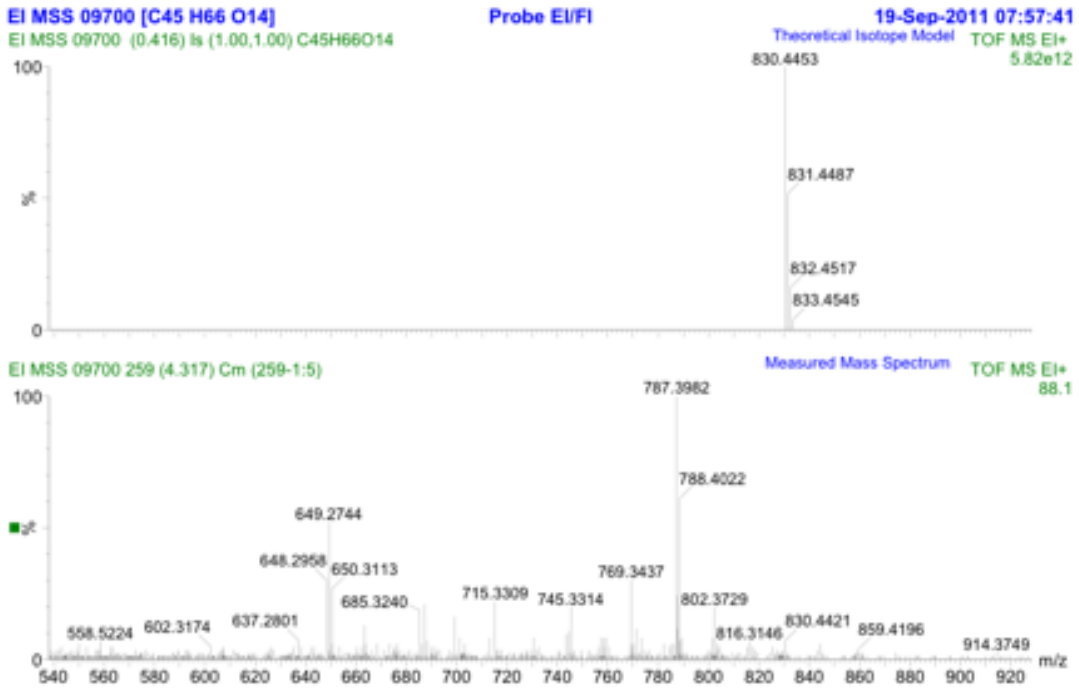

**Spectrum S3.9.7: HMBC spectrum for compound 9Ac in $CDCl_3$**



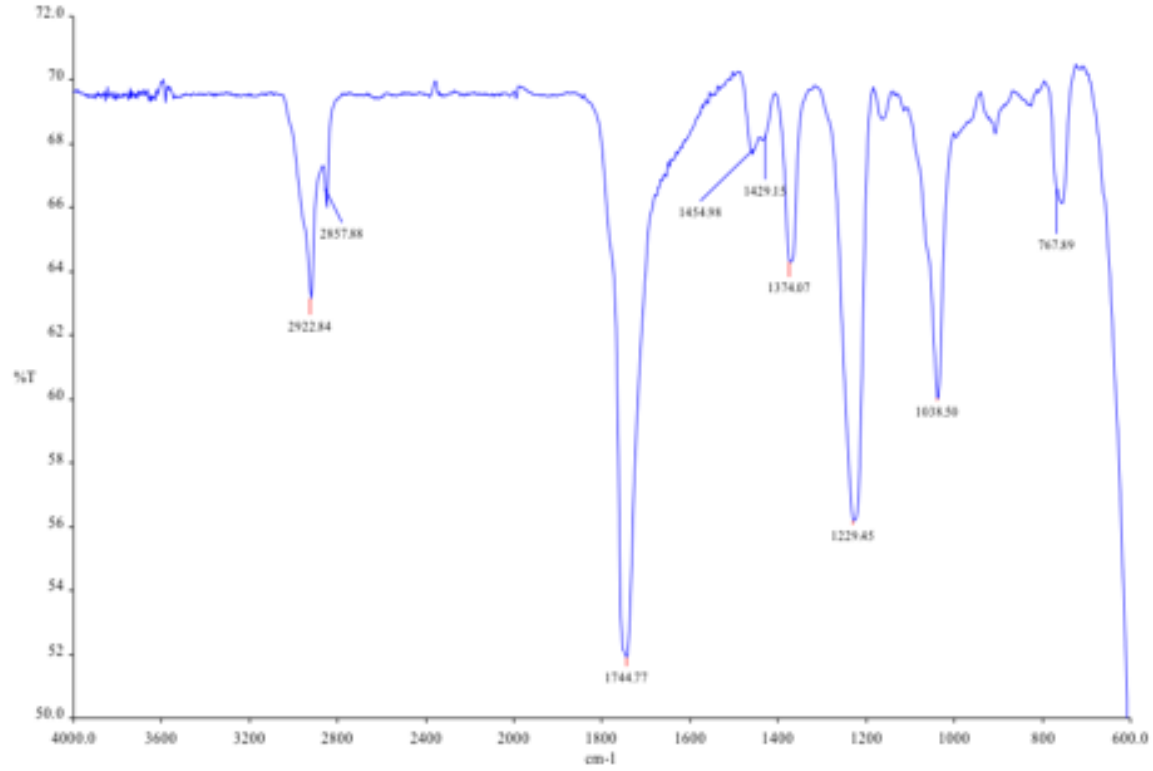

**Spectrum S3.9.8: COSY spectrum for compound 9Ac in $CDCl_3$**



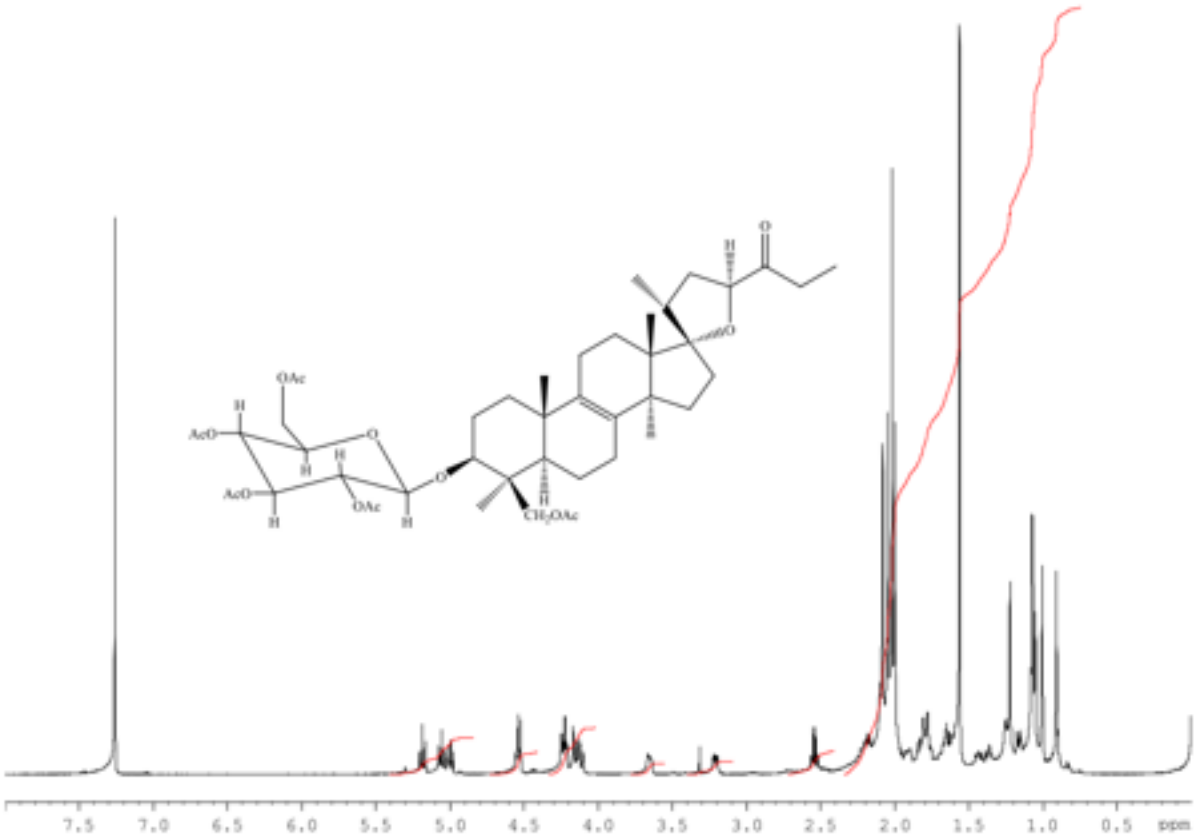

**Spectrum S3.9.9: NOESY spectrum for compound 9Ac in $CDCl_3$**



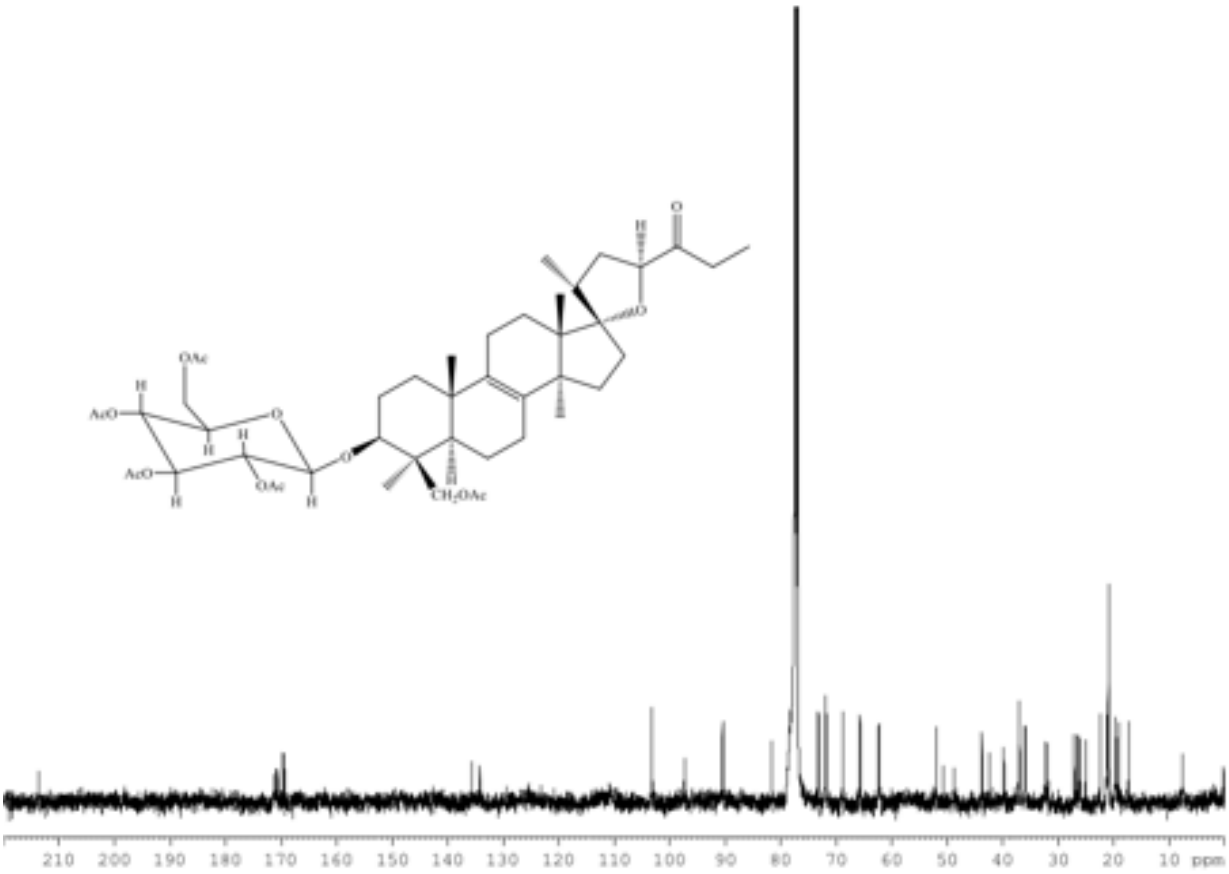

**Spectrum S3.10.1: Mass spectrum for compound 10Ac**



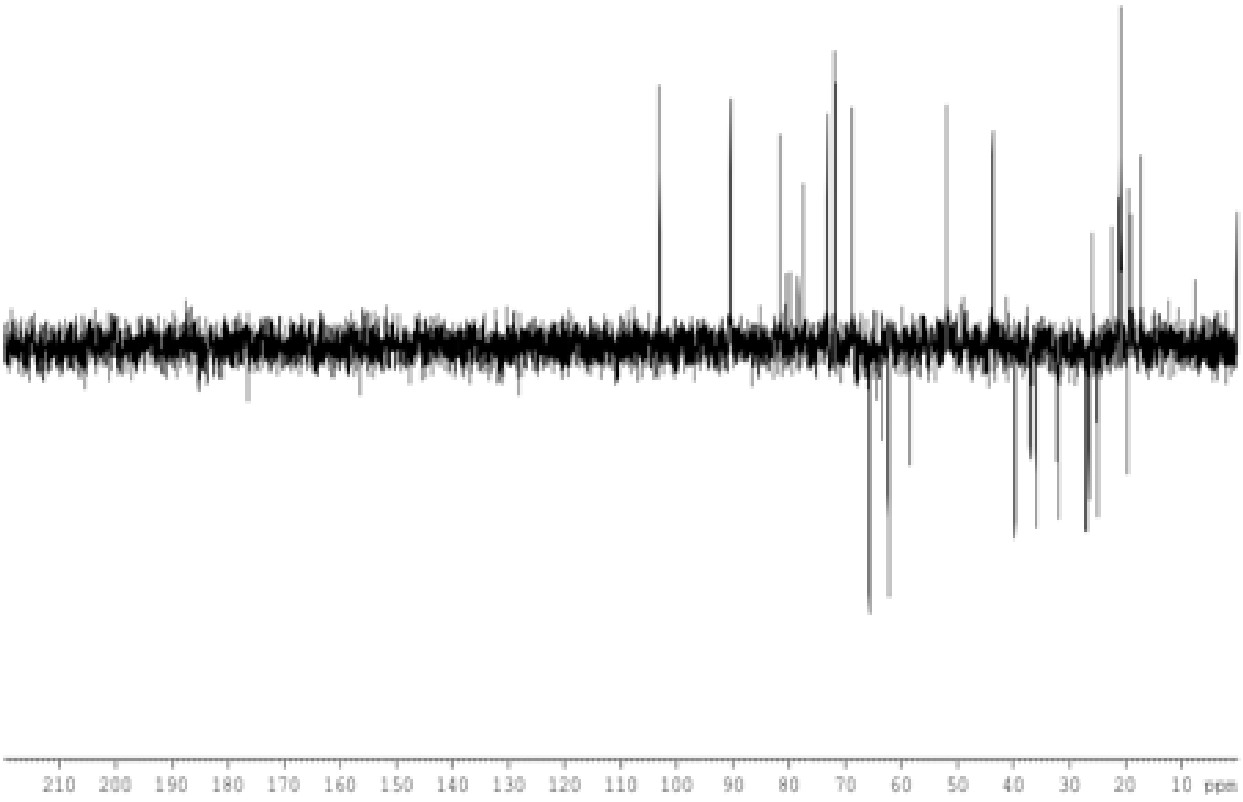

**Spectrum S3.10.2: FTIR spectrum for compound 10Ac**



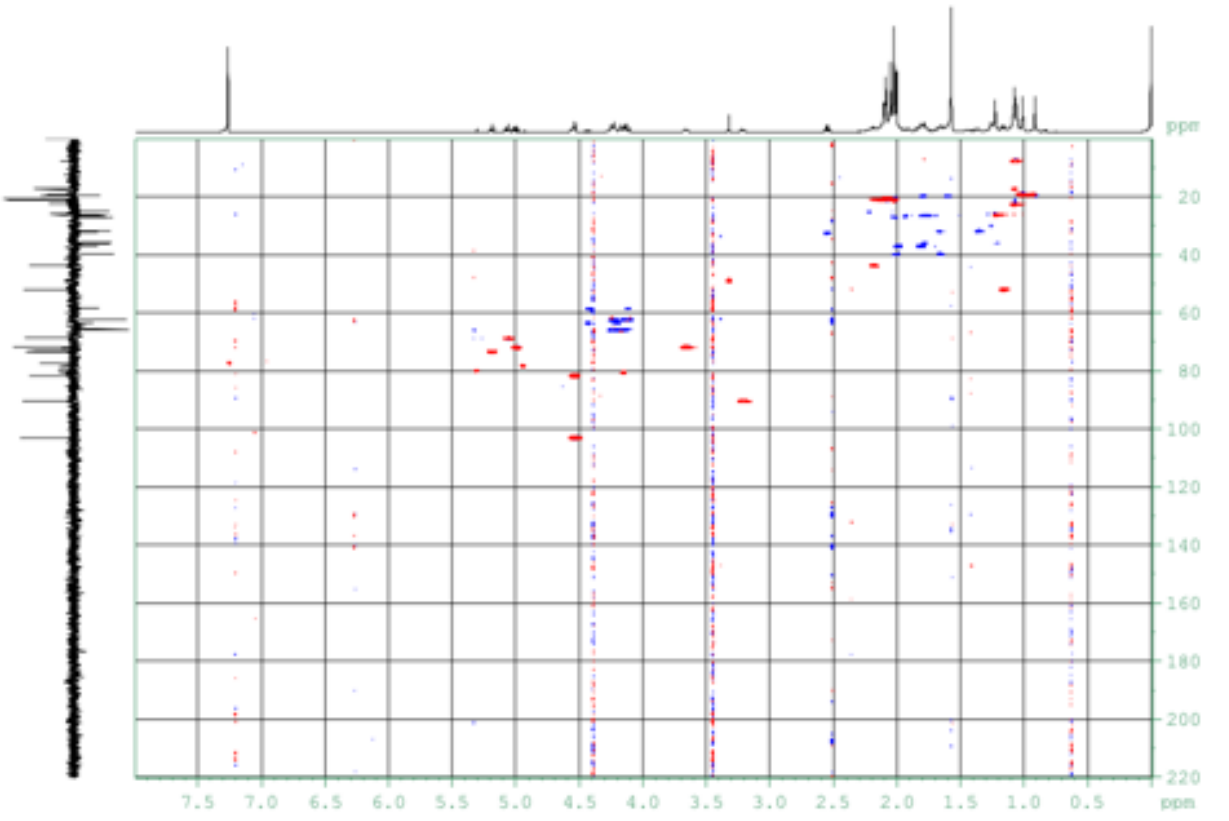

**Spectrum S3.10.3: $^{1}H$ NMR spectrum for compound 10Ac in $CDCl_3$**



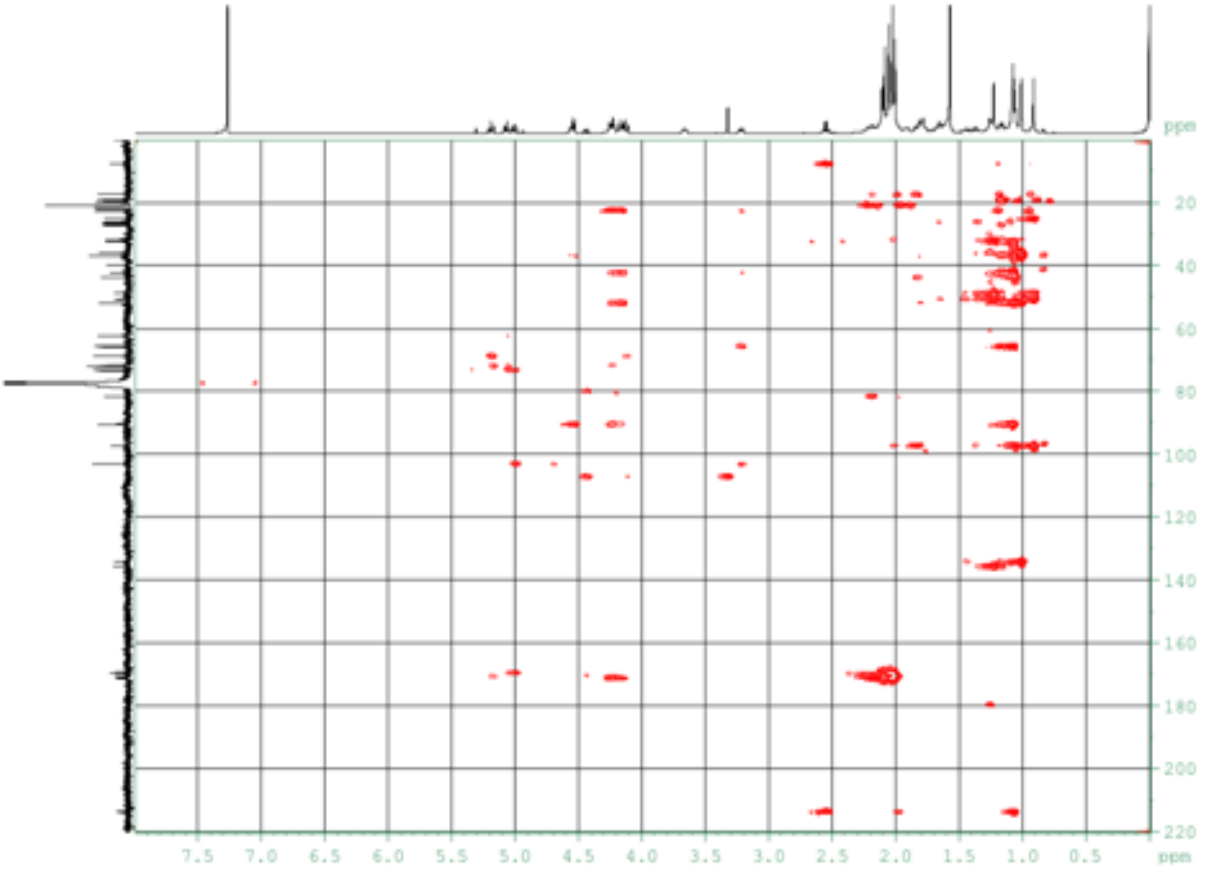

**Spectrum S3.10.4: $^{13}C$ NMR spectrum for compound 10Ac in $CDCl_3$**



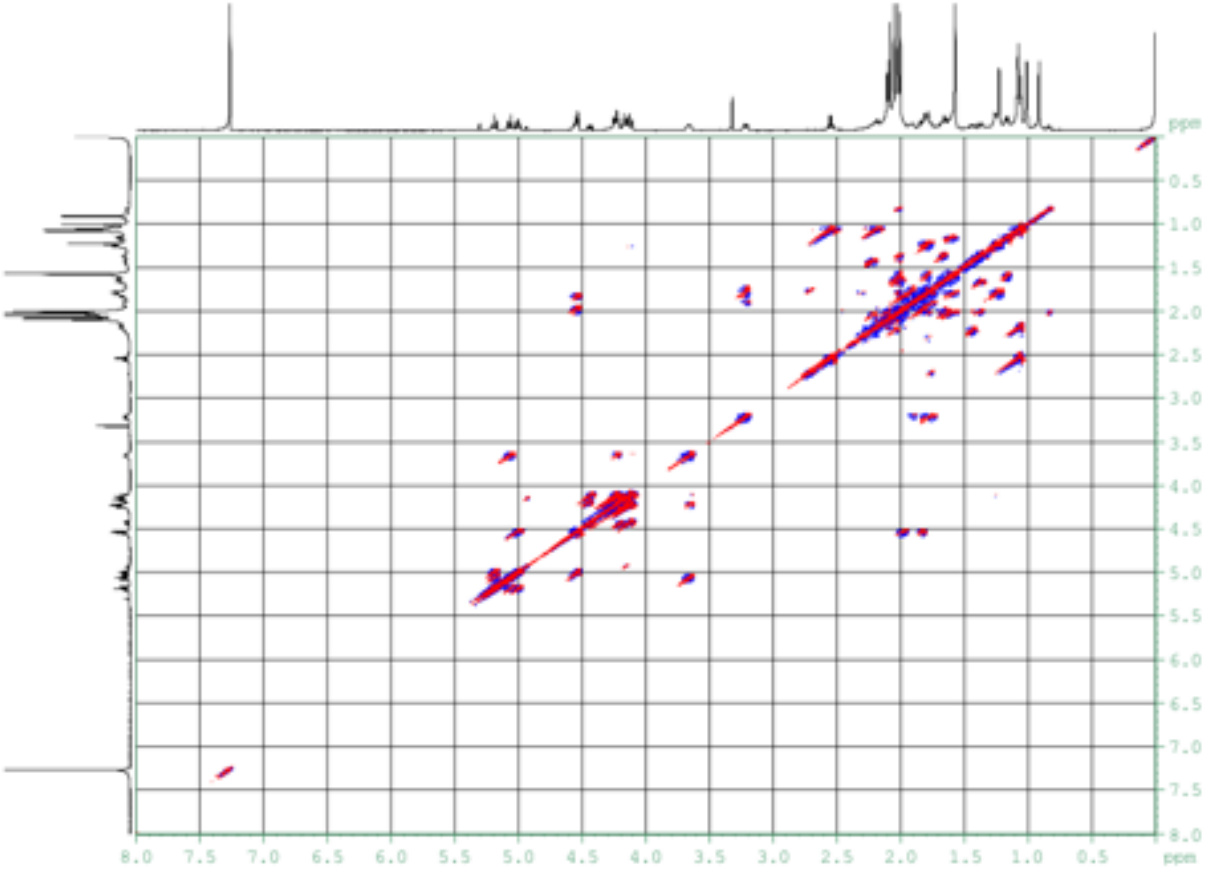

**Spectrum S3.10.5: DEPT spectrum for compound 10Ac in $CDCl_3$**



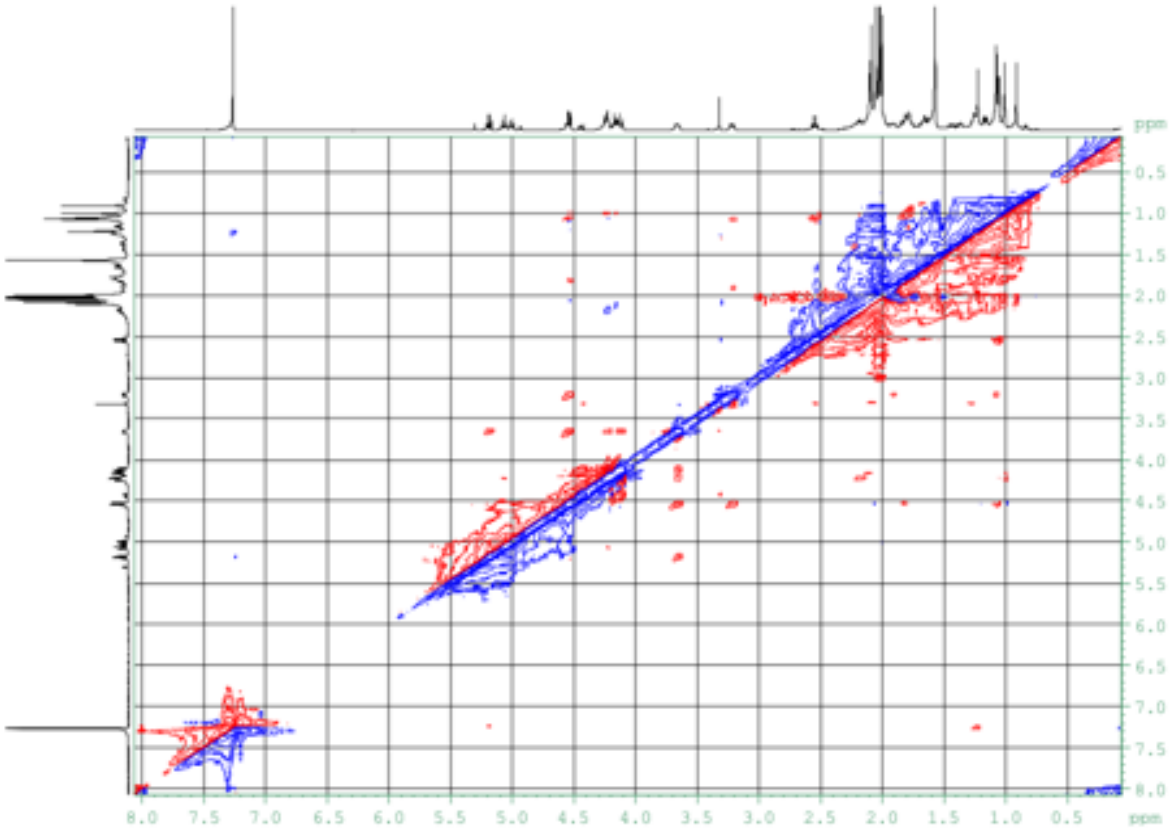

**Spectrum S3.10.6: HSQCDEPT spectrum for compound 10Ac in $CDCl_3$**



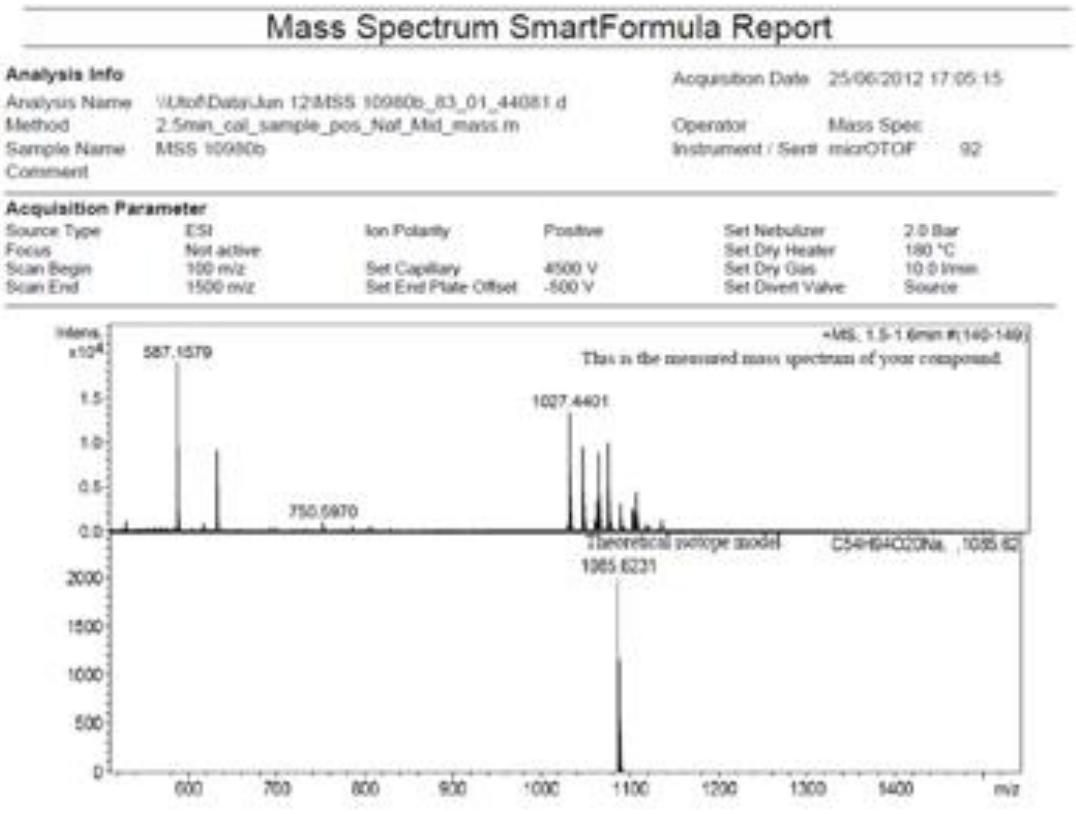

**Spectrum S3.10.7: HMBC spectrum for compound 10Ac in $CDCl_3$**



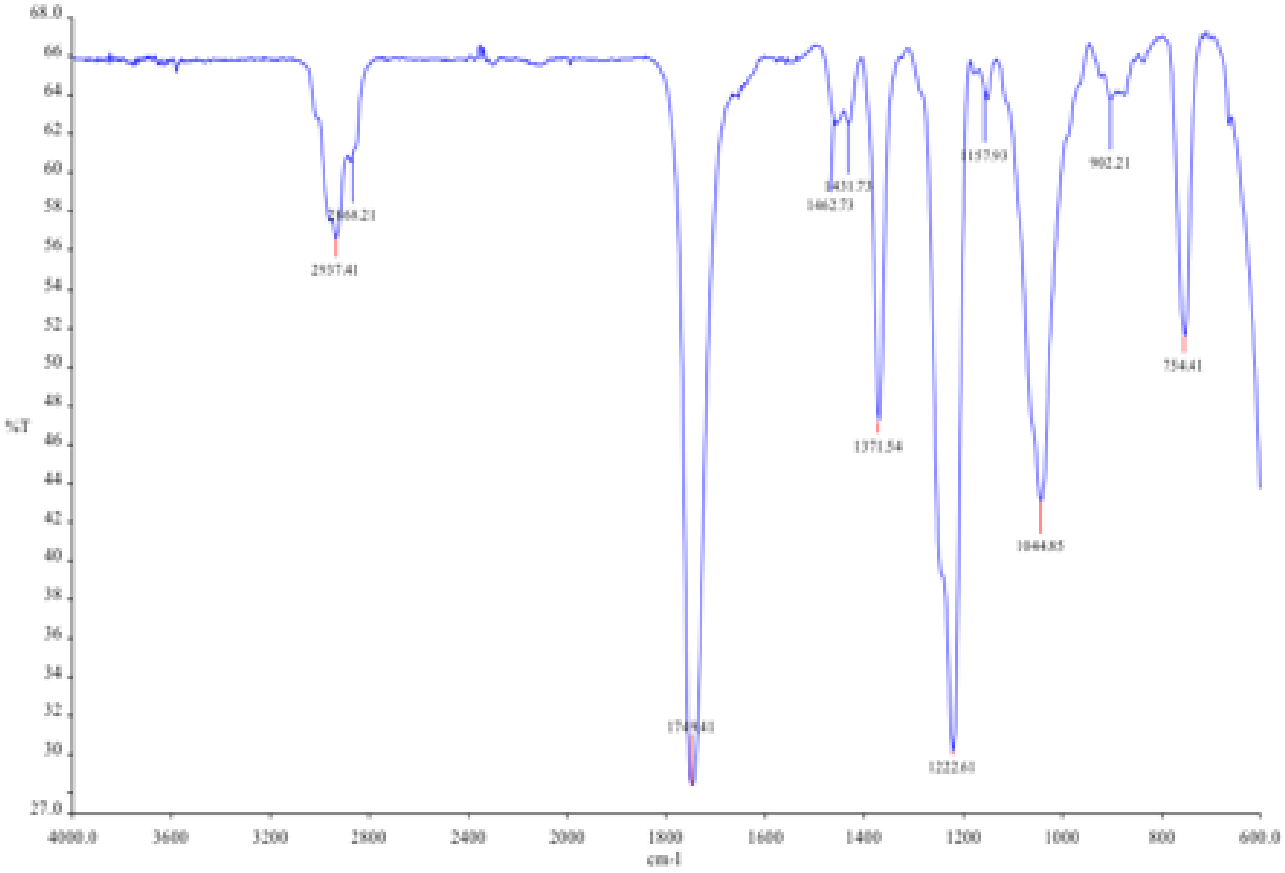

**Spectrum S3.10.8: COSY spectrum for compound 10Ac in $CDCl_3$**



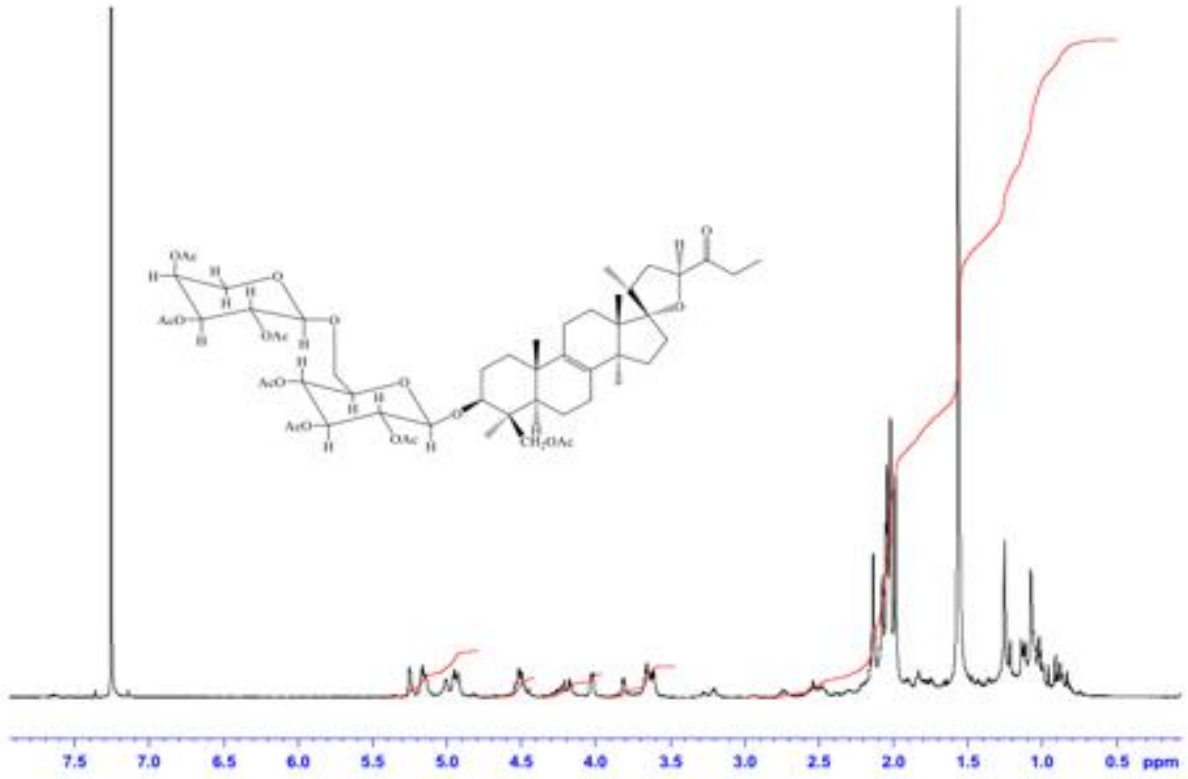

**Spectrum S3.10.9: NOESY spectrum for compound 10Ac in $CDCl_3$**



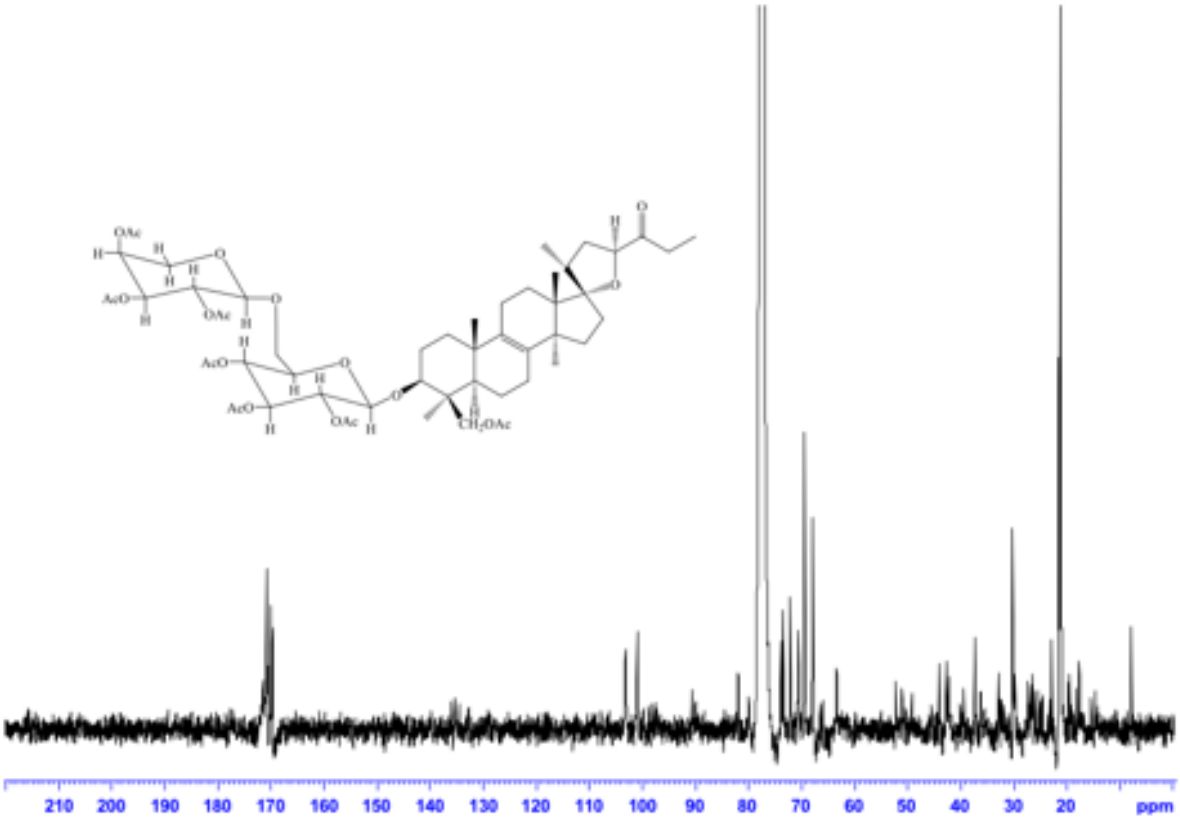

**Spectrum S.3.11.1: Mass spectrum for compound 11Ac**



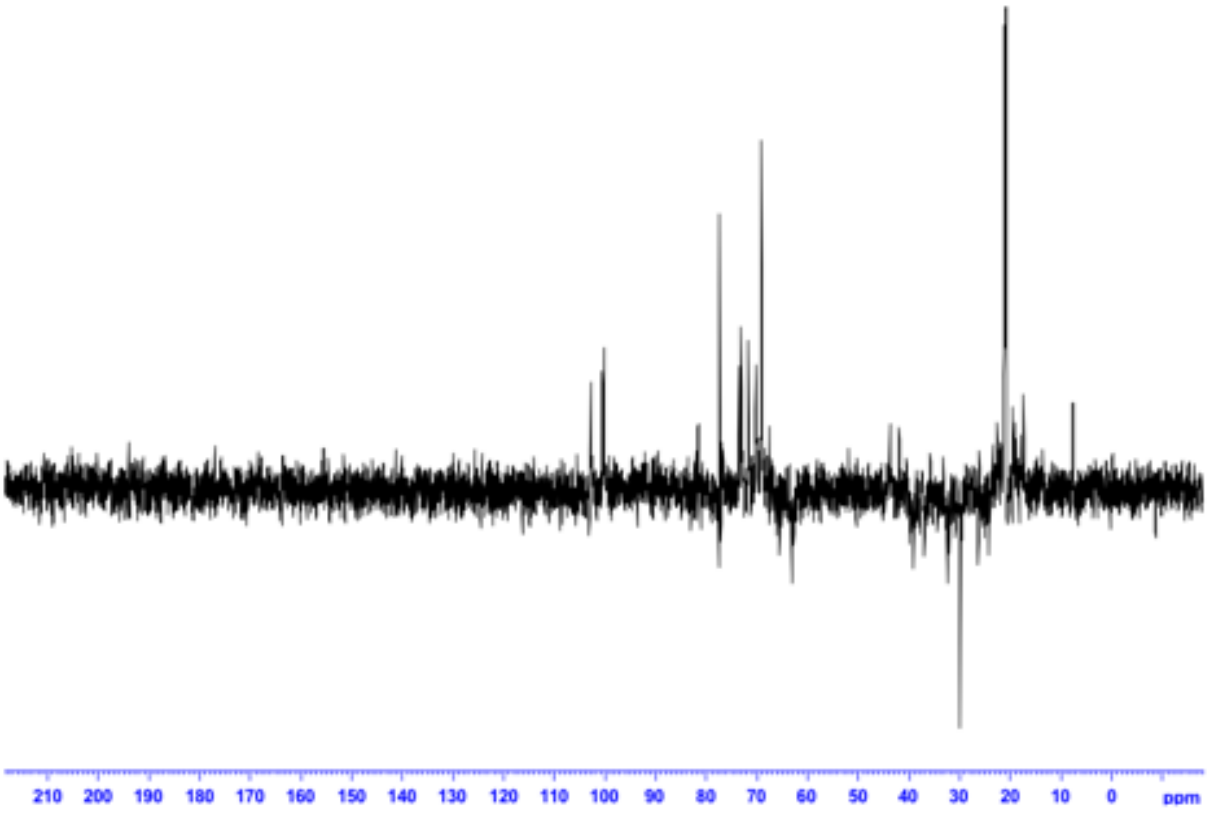

**Spectrum S3.11.2: FTIR spectrum for compound 11Ac**



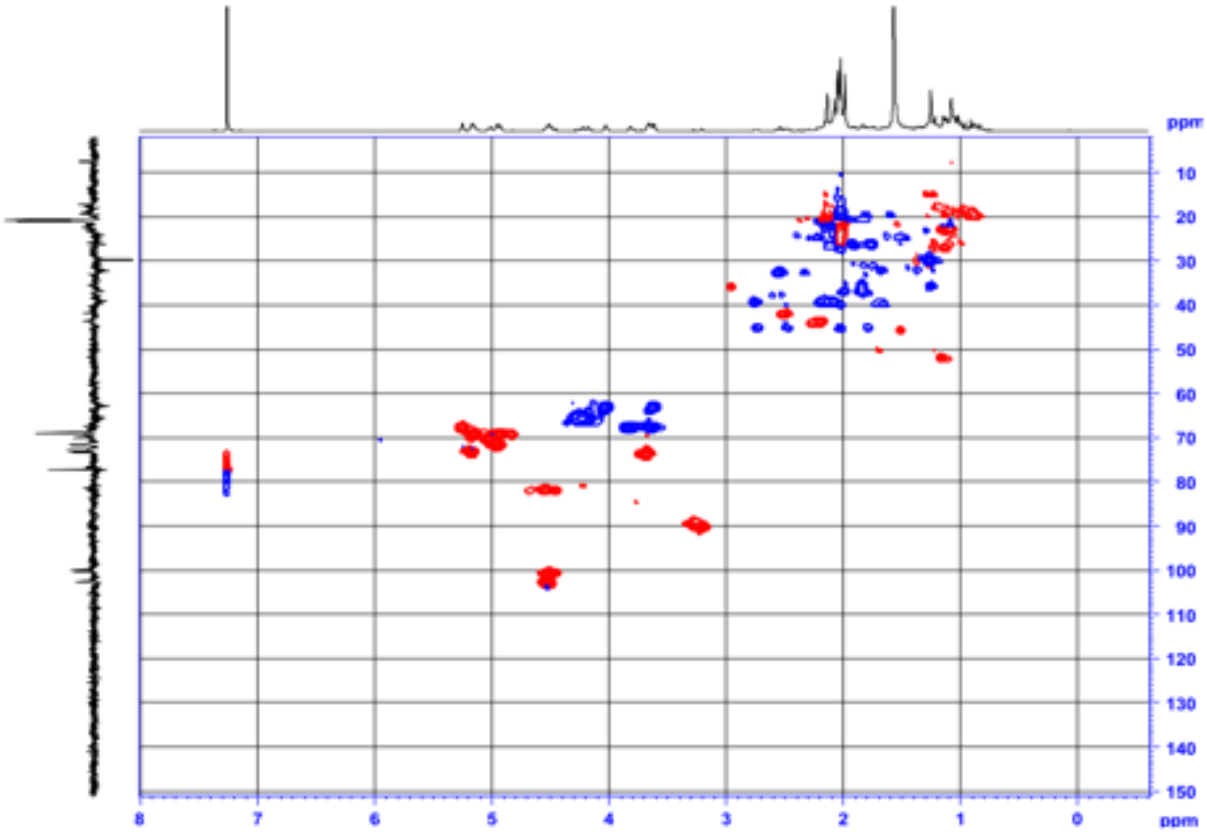

**Spectrum S3.11.3: $^1$H NMR spectrum for compound 11Ac in $CDCl_3$**



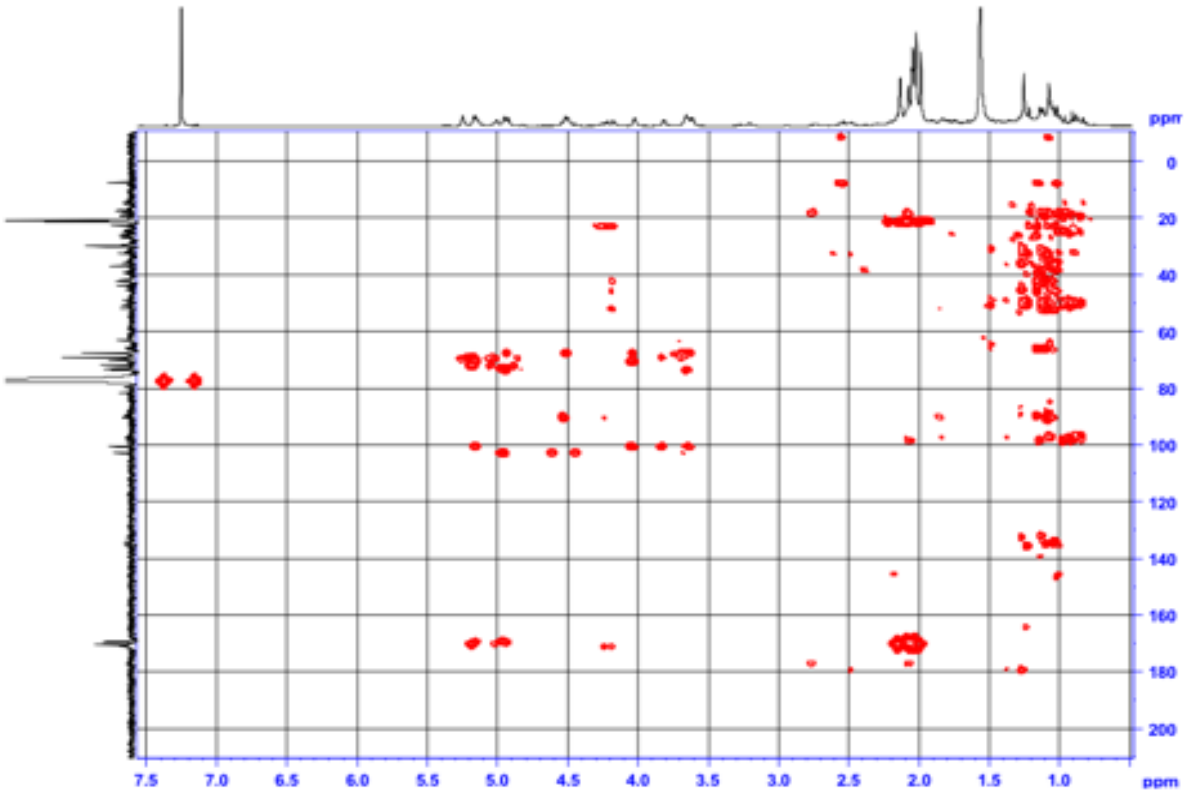

**Spectrum S.3.11.4: $^{13}C$ NMR spectrum for compound 11Ac in $CDCl_3$**



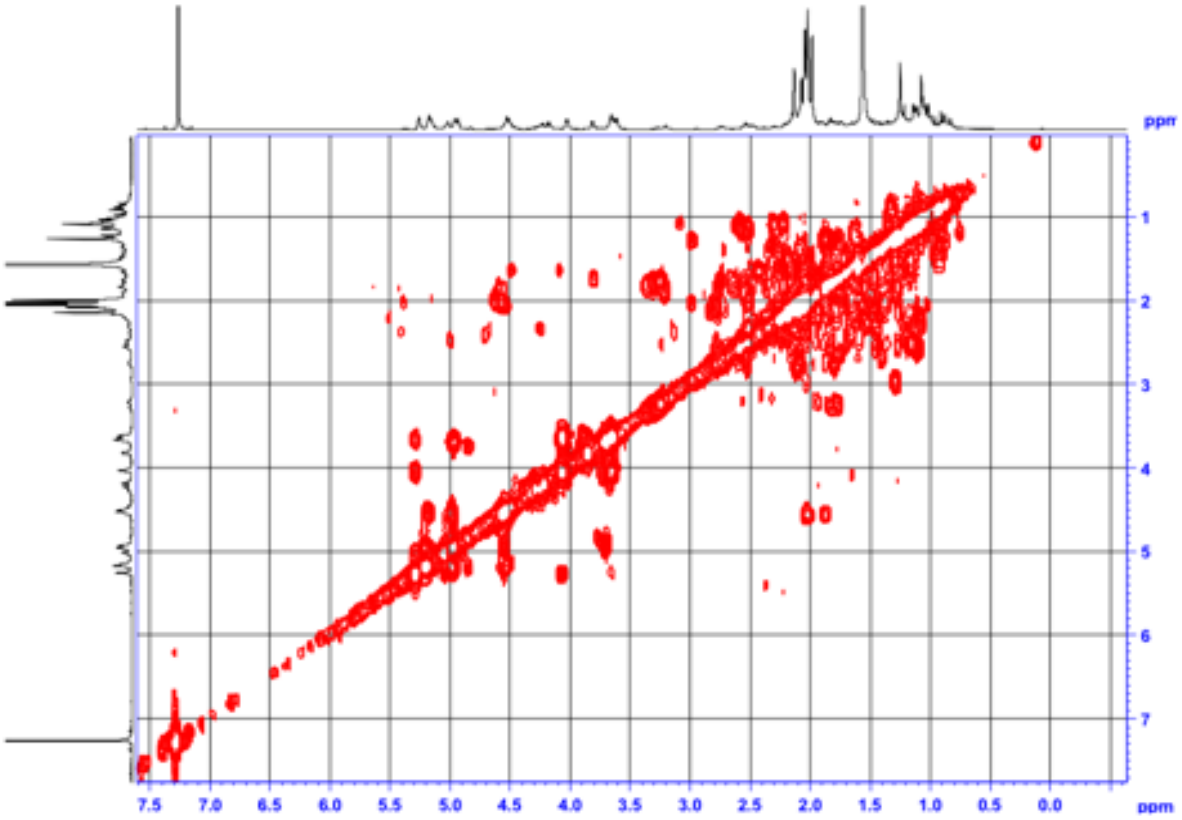

**Spectrum S.3.11.5: DEPT spectrum for compound 11Ac in $CDCl_3$**



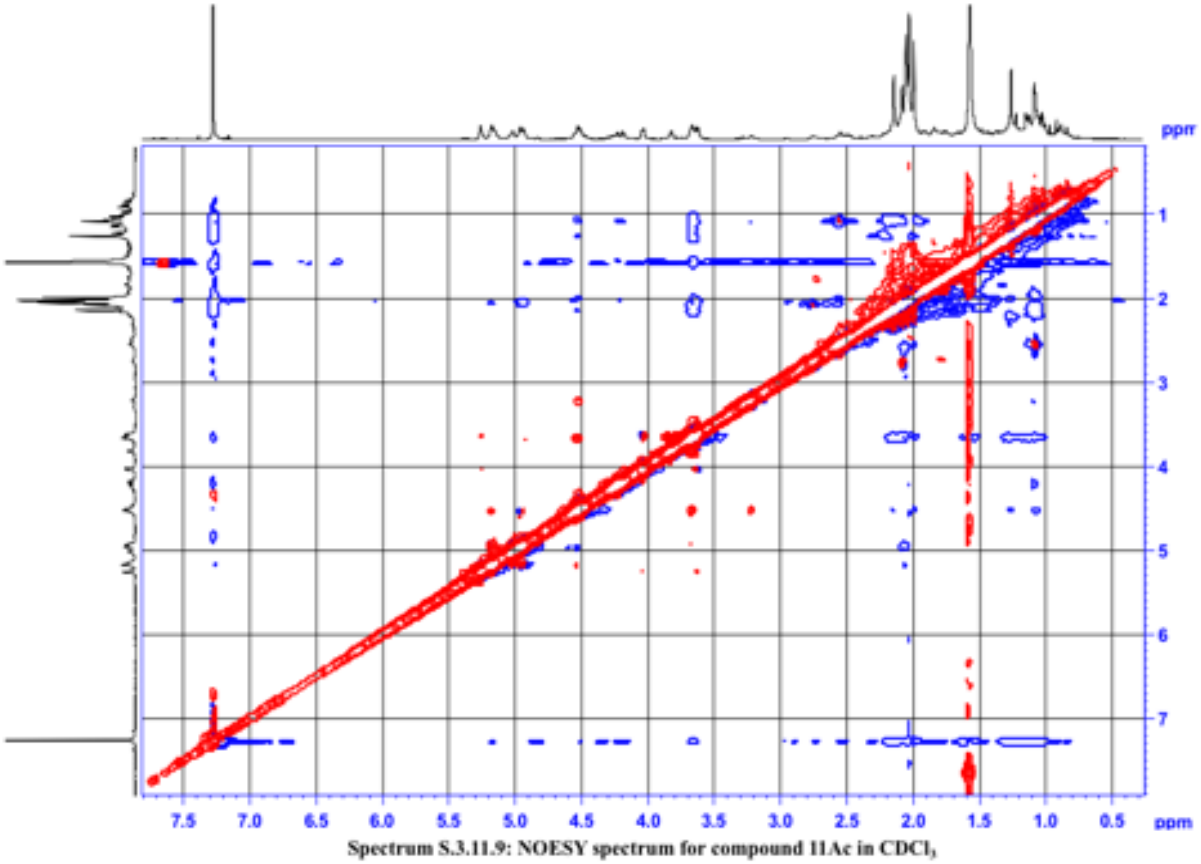

**Spectrum S.3.11.6: HSQCDEPT spectrum for compound 11Ac in $CDCl_3$**



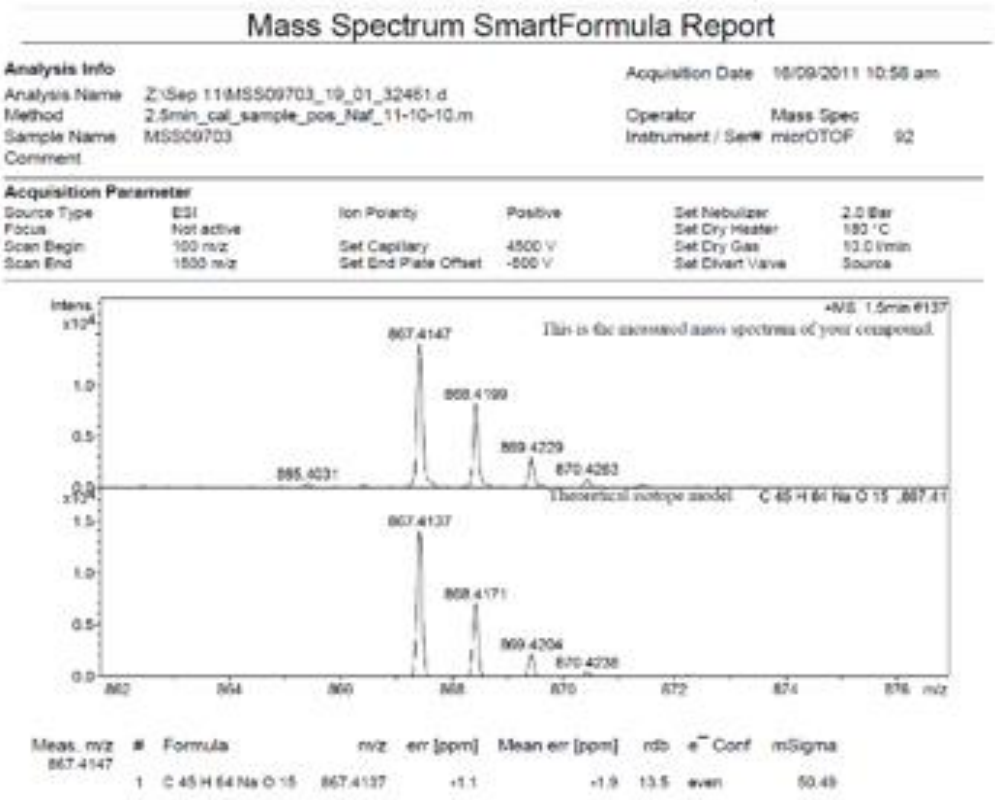

**Spectrum S.3.11.7: HMBC spectrum for compound 11Ac in $CDCl_3$**



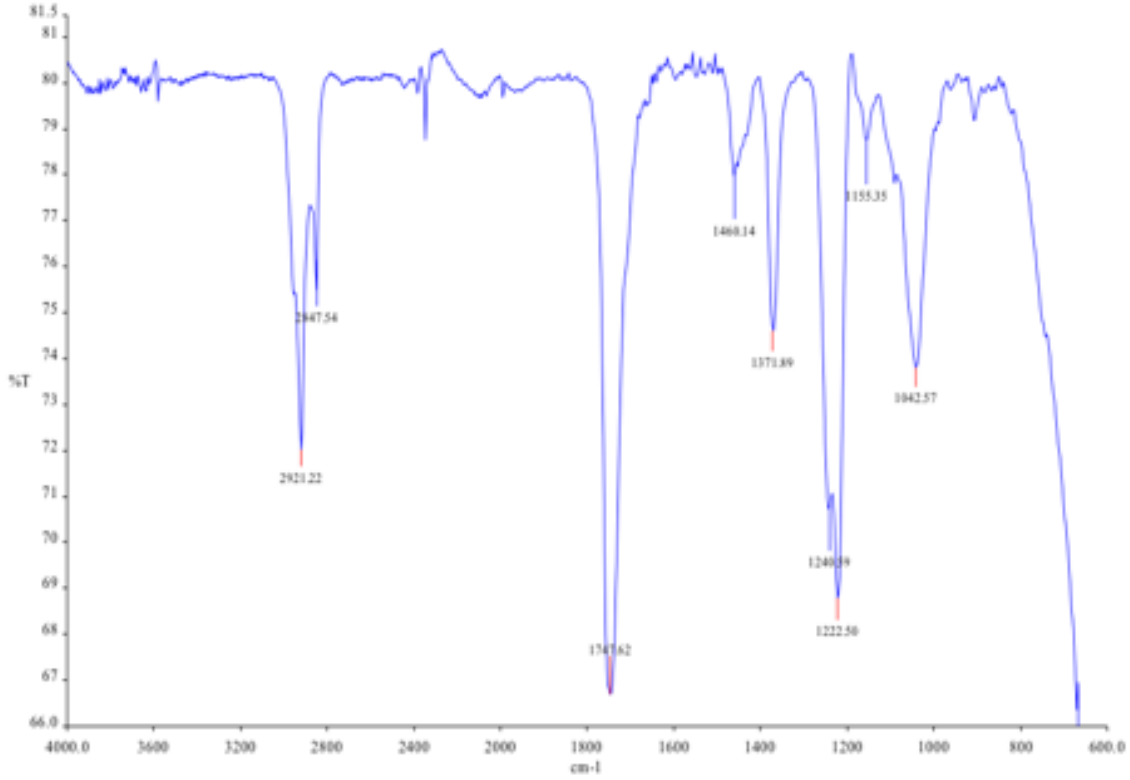

**Spectrum S.3.11.8: COSY spectrum for compound 11Ac in $CDCl_3$**



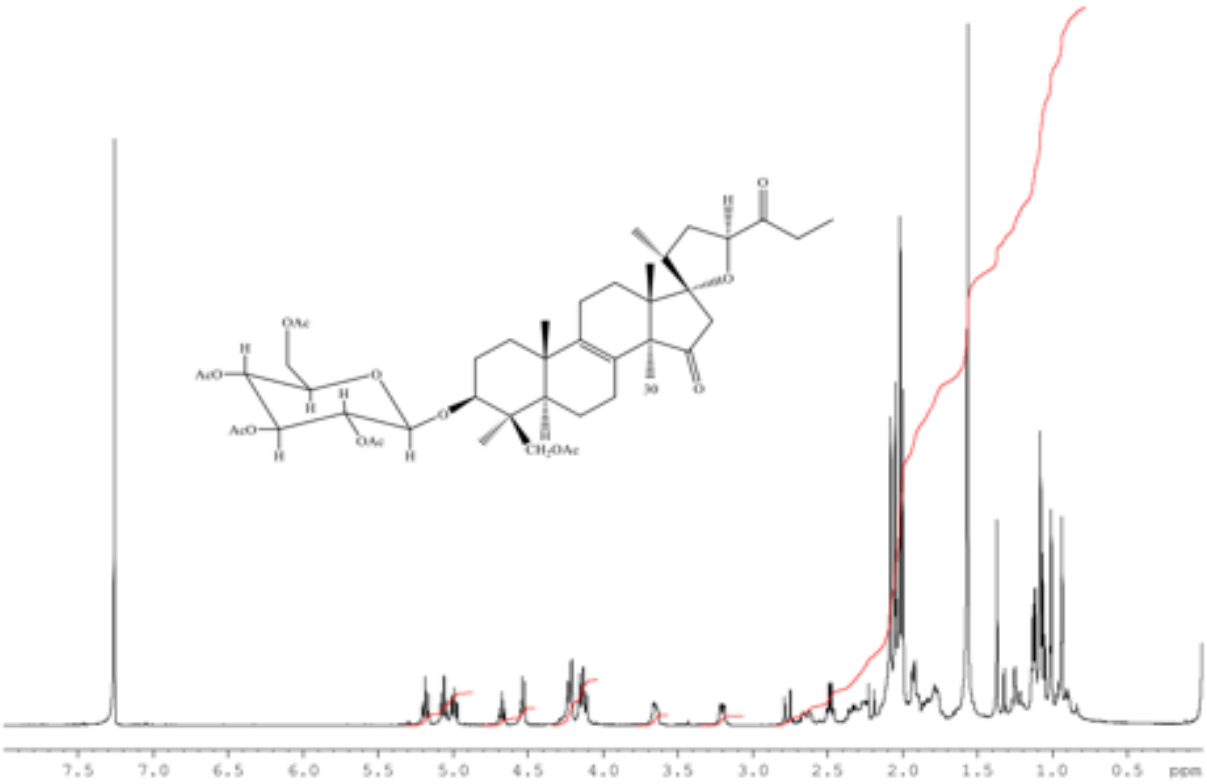

**Spectrum S.3.11.9: NOESY spectrum for compound 11Ac in $CDCl_3$**



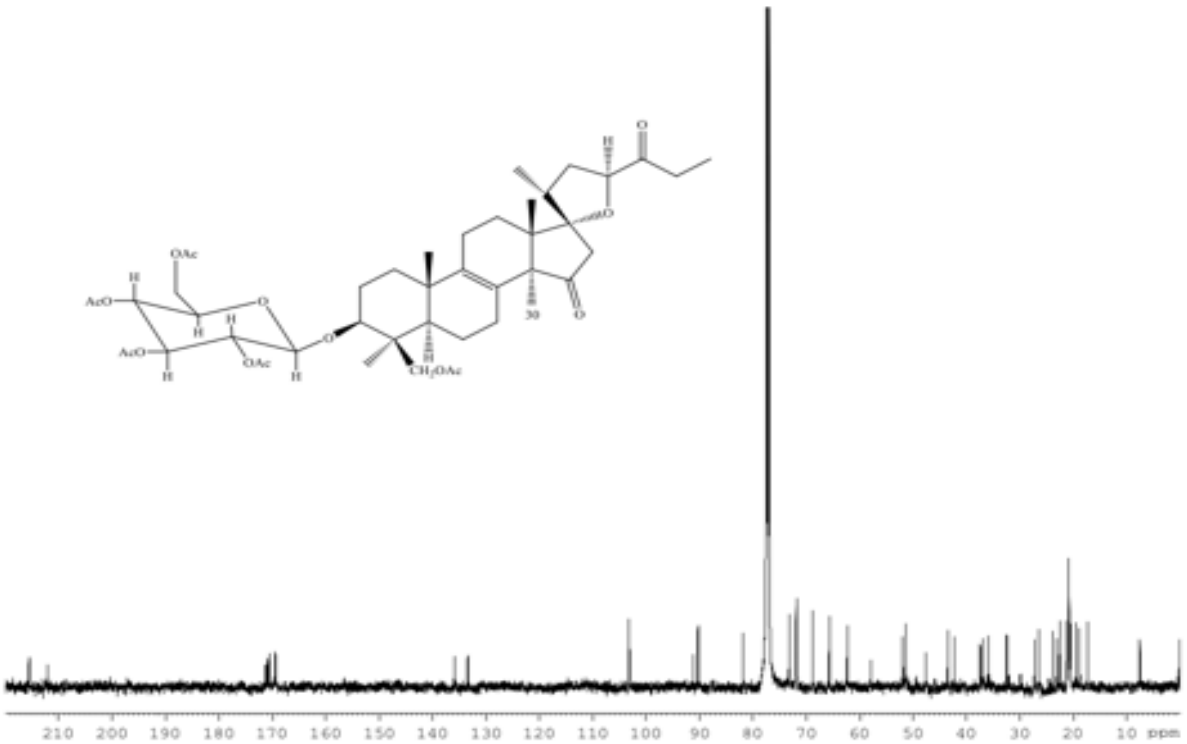

**Spectrum S.3.12.1: Mass spectrum for compound 12Ac**



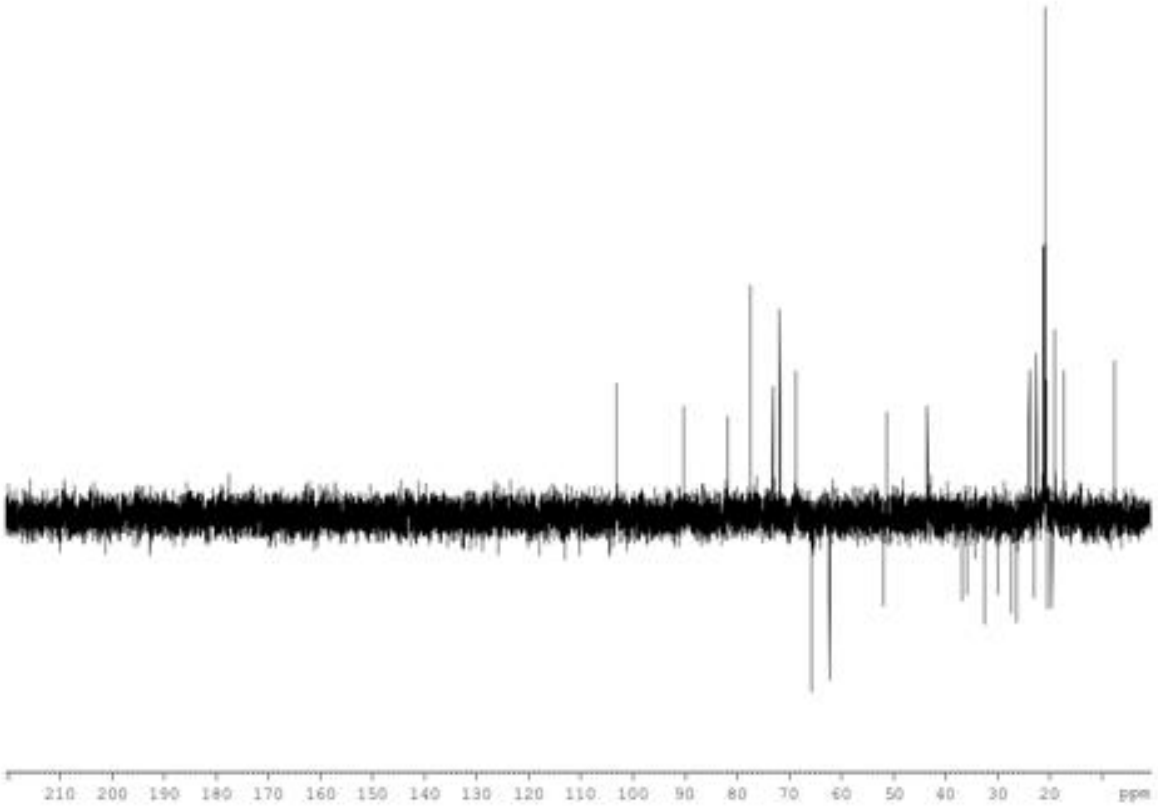

**Spectrum S.3.12.2: FTIR spectrum for compound 12Ac**



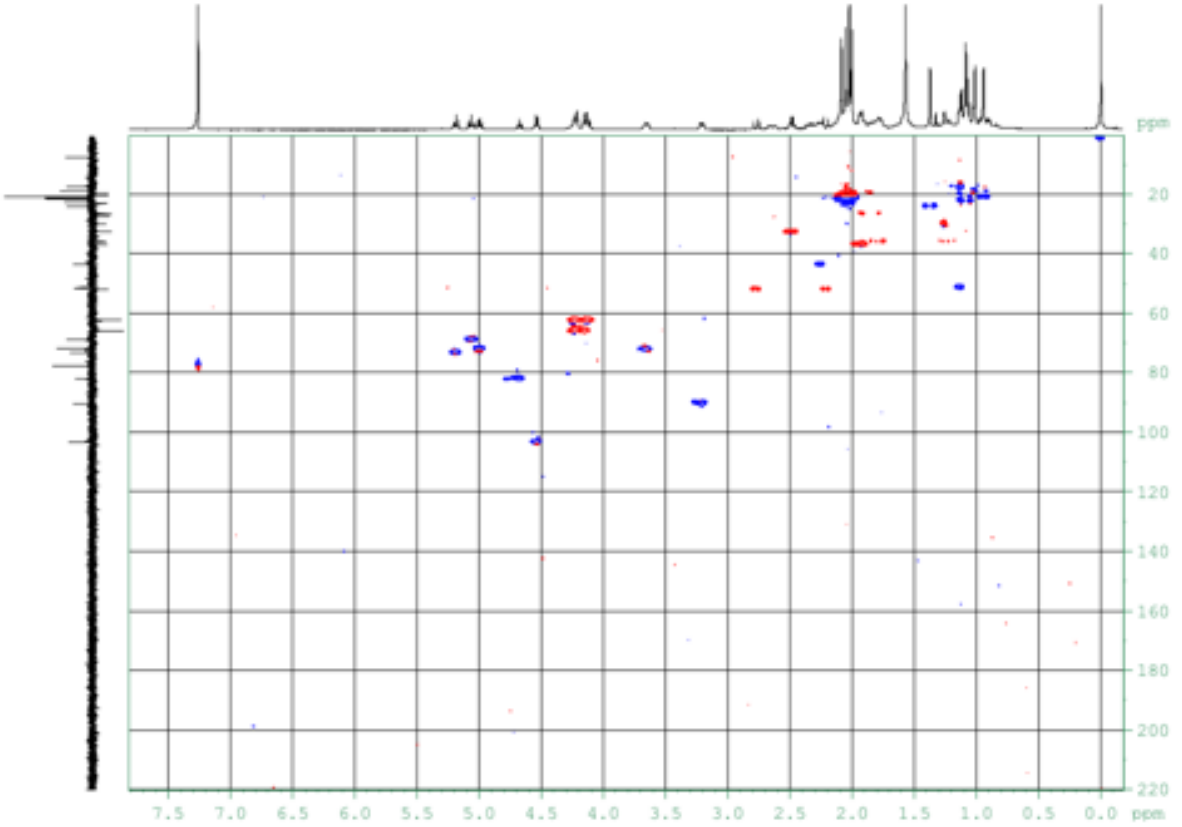

**Spectrum S3.12.3: $^1$H NMR spectrum for compound 12Ac in $CDCl_3$**



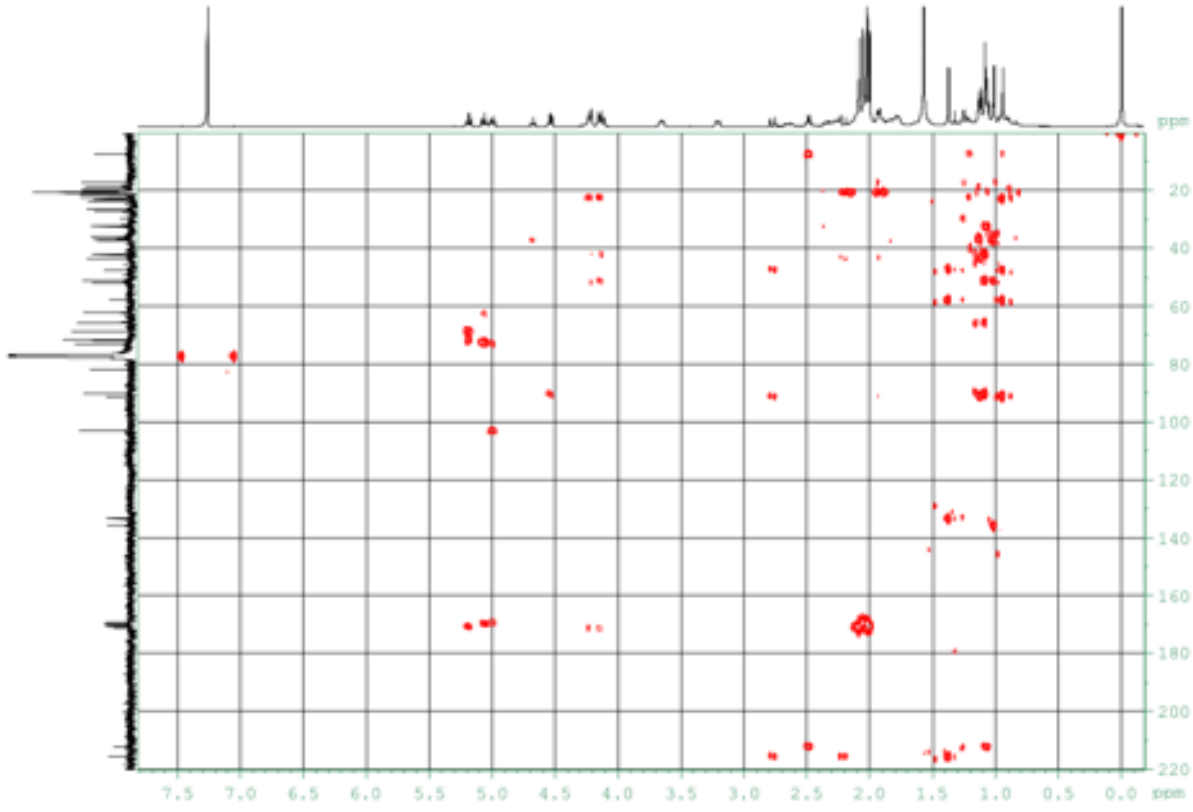

**Spectrum S3.12.4: $^{13}C$ NMR spectrum for compound 12Ac in $CDCl_3$**



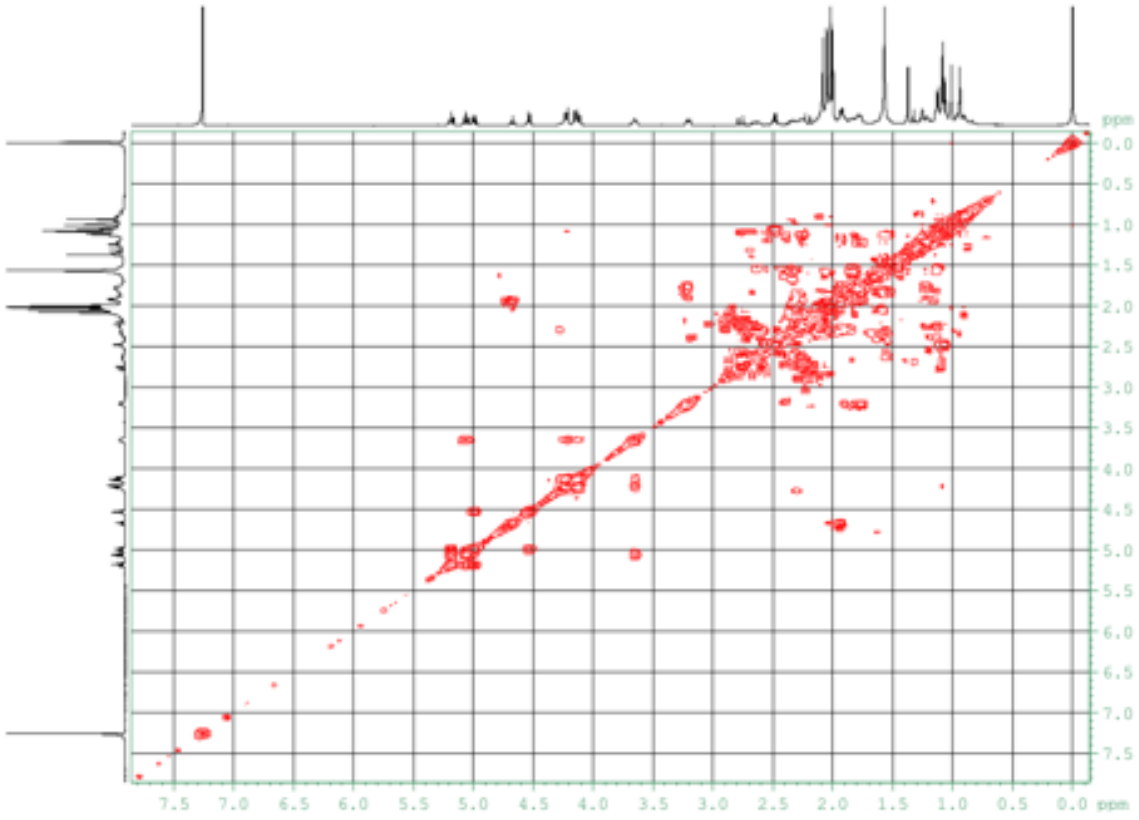

**Spectrum S3.12.5: DEPT spectrum for compound 12Ac in $CDCl_3$**



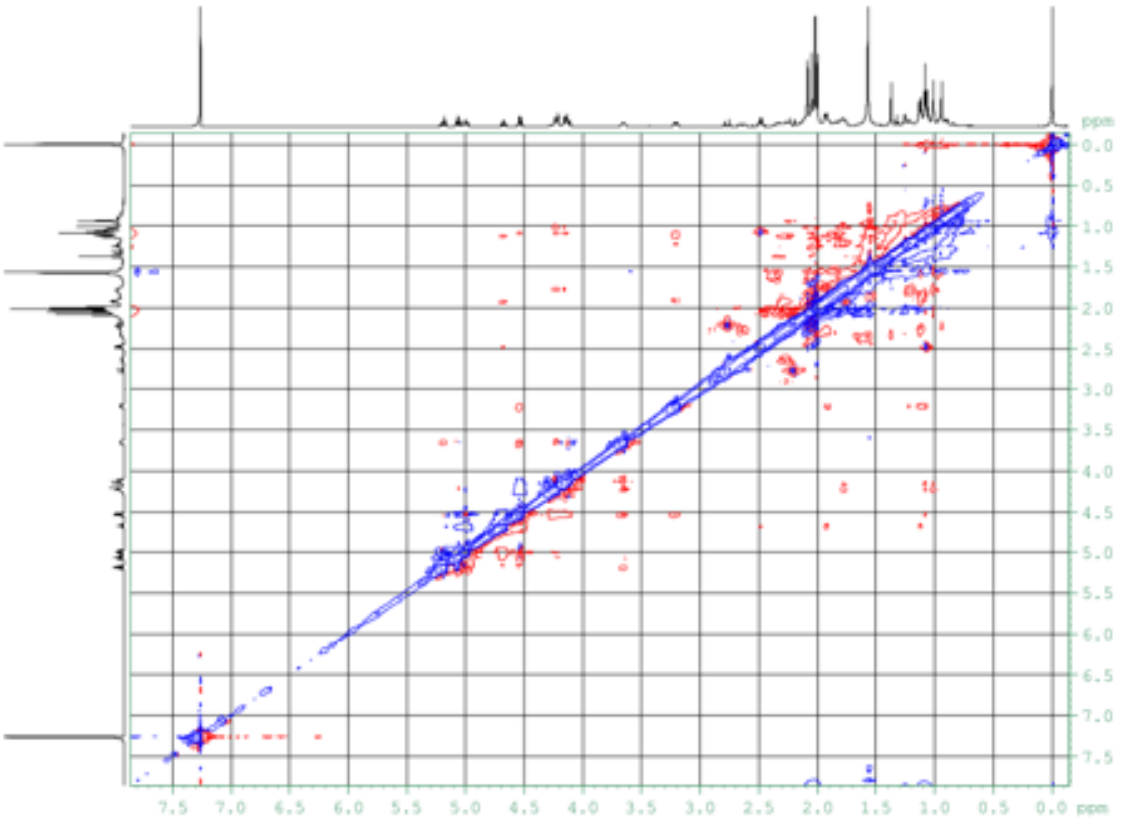

**Spectrum S3.12.6: HSQCDEPT spectrum for compound 12Ac in $CDCl_3$**



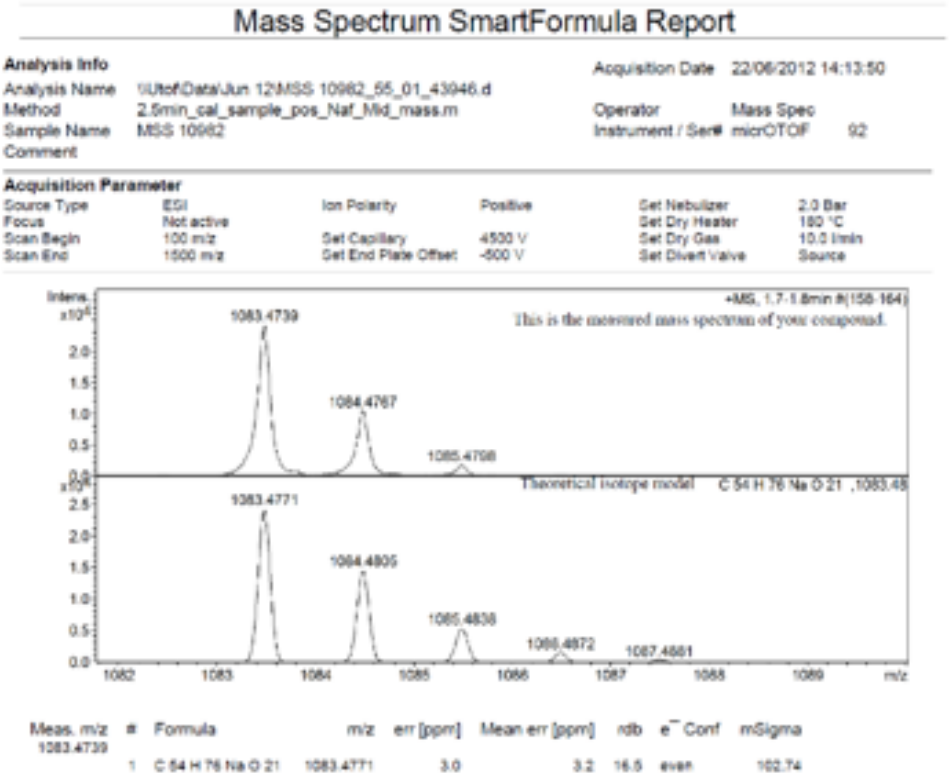

**Spectrum S3.12.7: HMBC spectrum for compound 12Ac in $CDCl_3$**



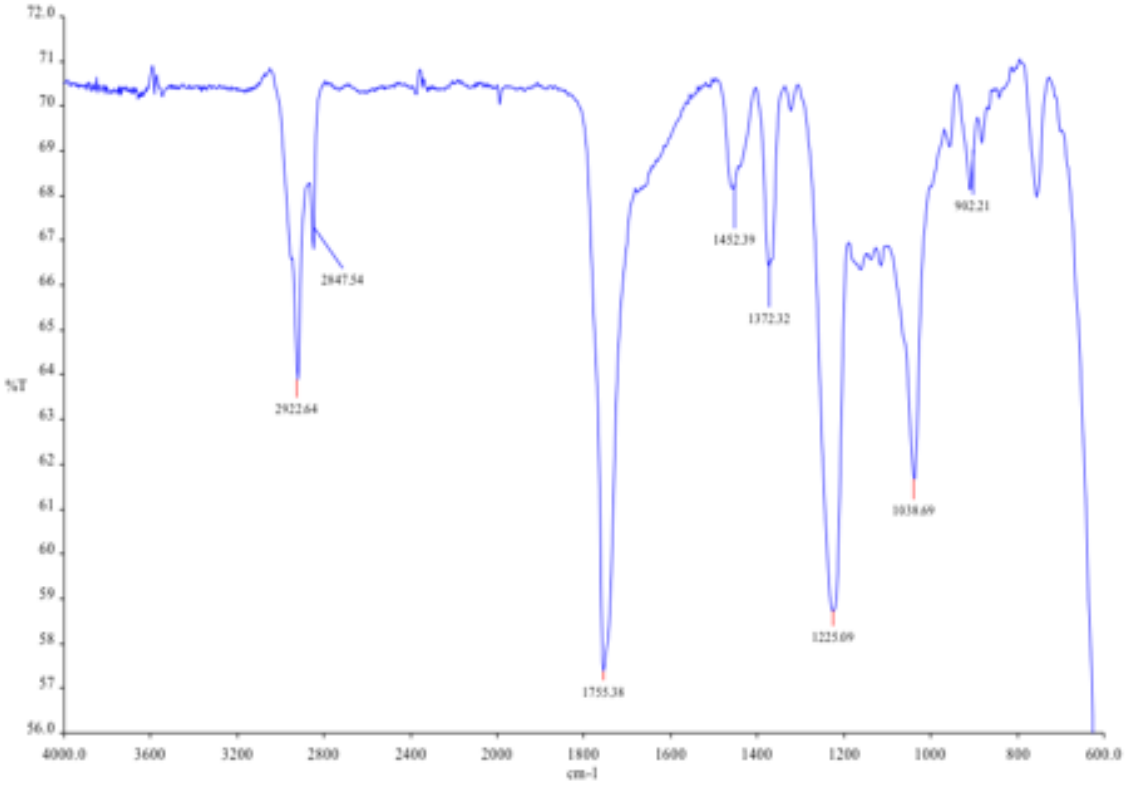

**Spectrum S3.12.8: COSY spectrum for compound 12Ac in $CDCl_3$**



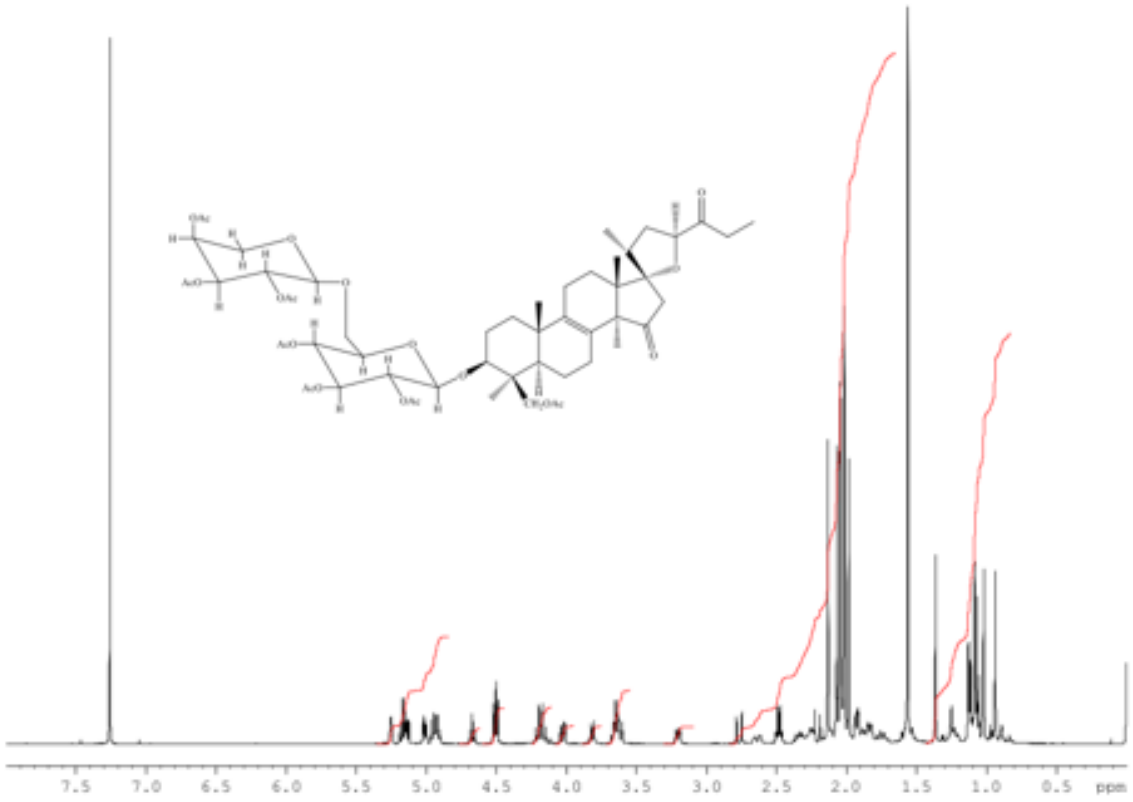

**Spectrum S3.12.9: NOESY spectrum for compound 12Ac in $CDCl_3$**



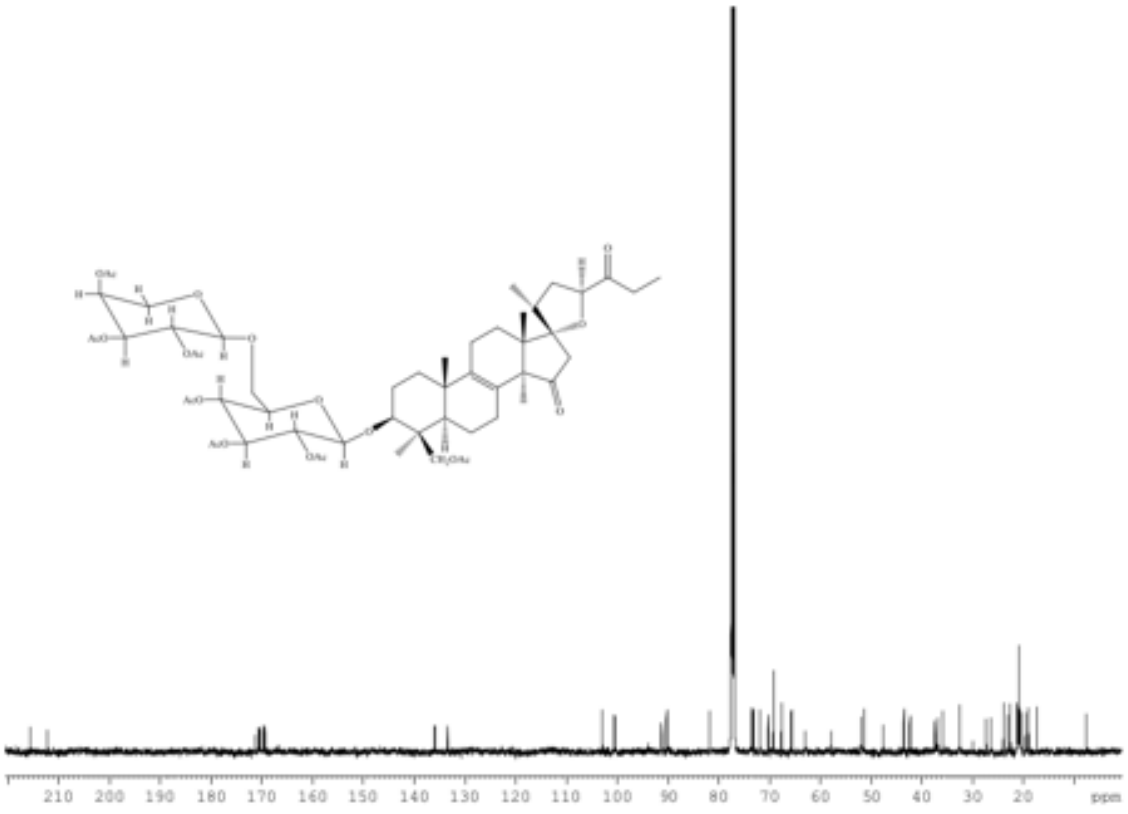

**Spectrum S3.13.1: Mass spectrum for compound 13Ac**



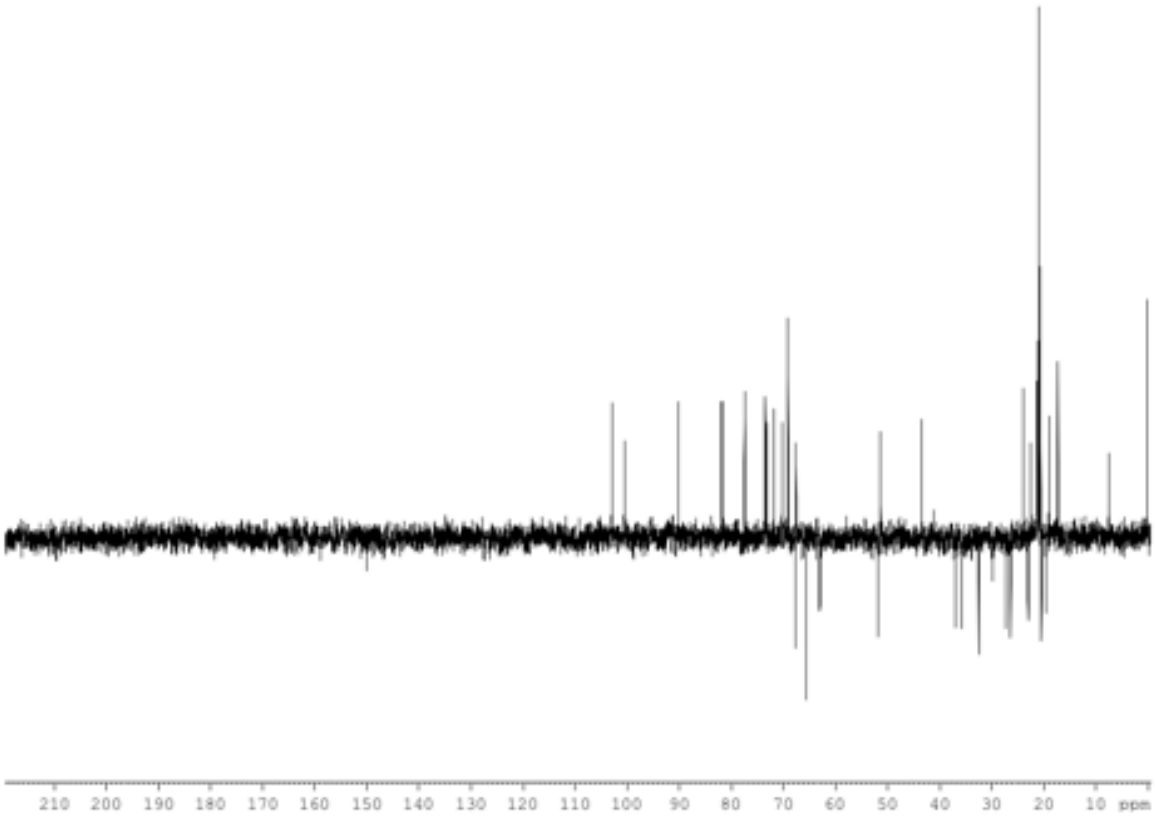

**Spectrum S3.13.2: FTIR spectrum for compound 13Ac**



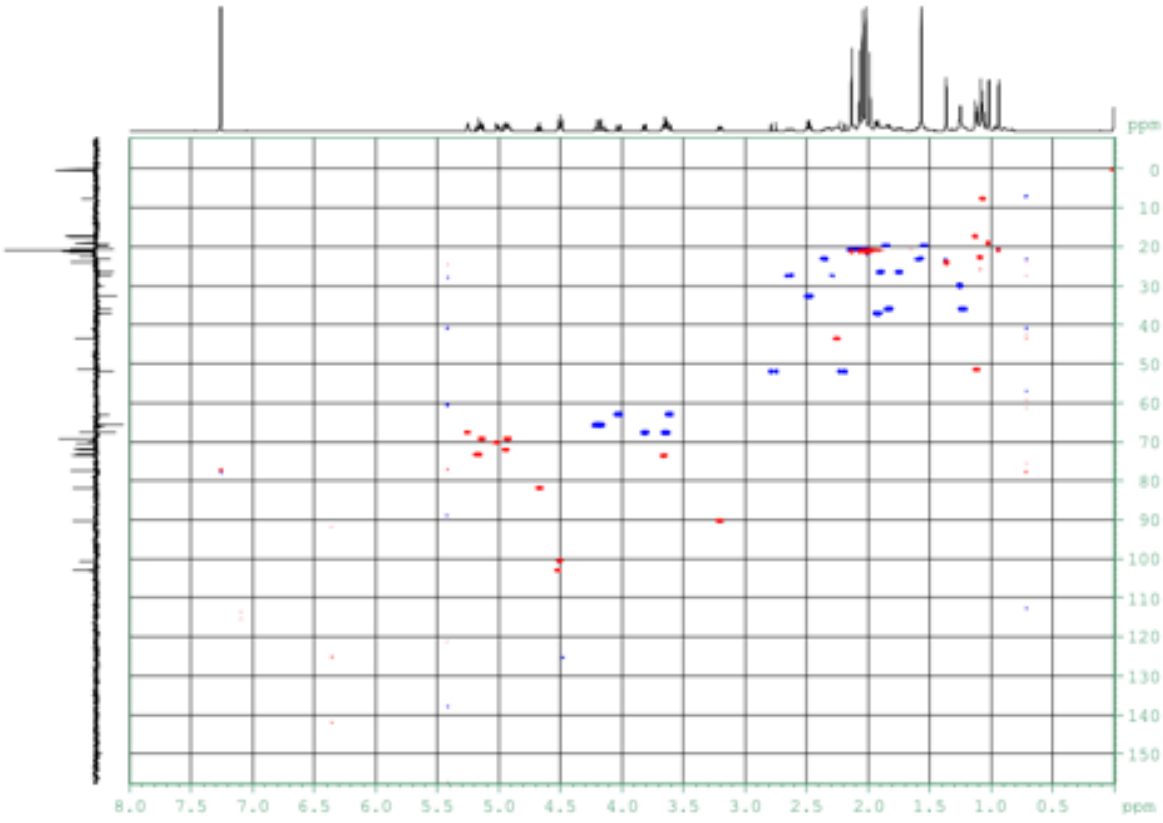

**Spectrum S3.13.3: $^{1}H$ NMR spectrum for compound 13Ac in $CDCl_3$**



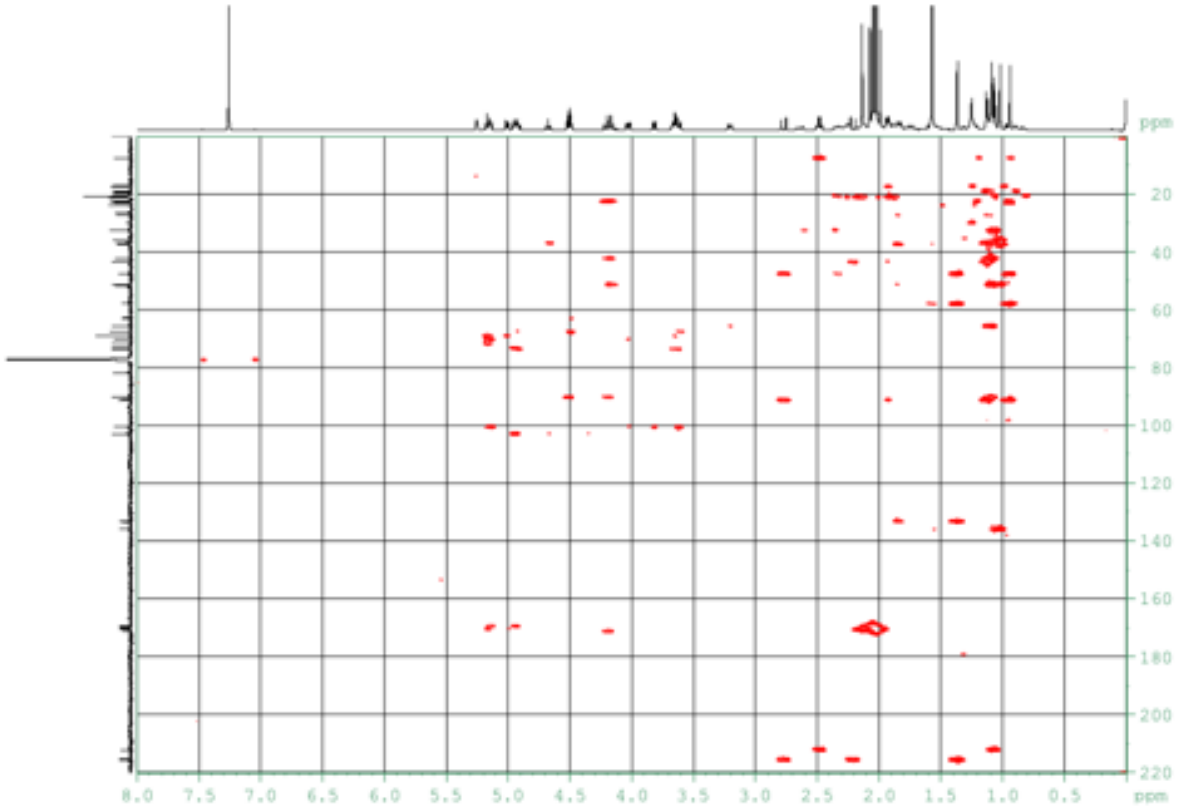

**Spectrum S3.13.4: $^{13}C$ NMR spectrum for compound 13Ac in $CDCl_3$**



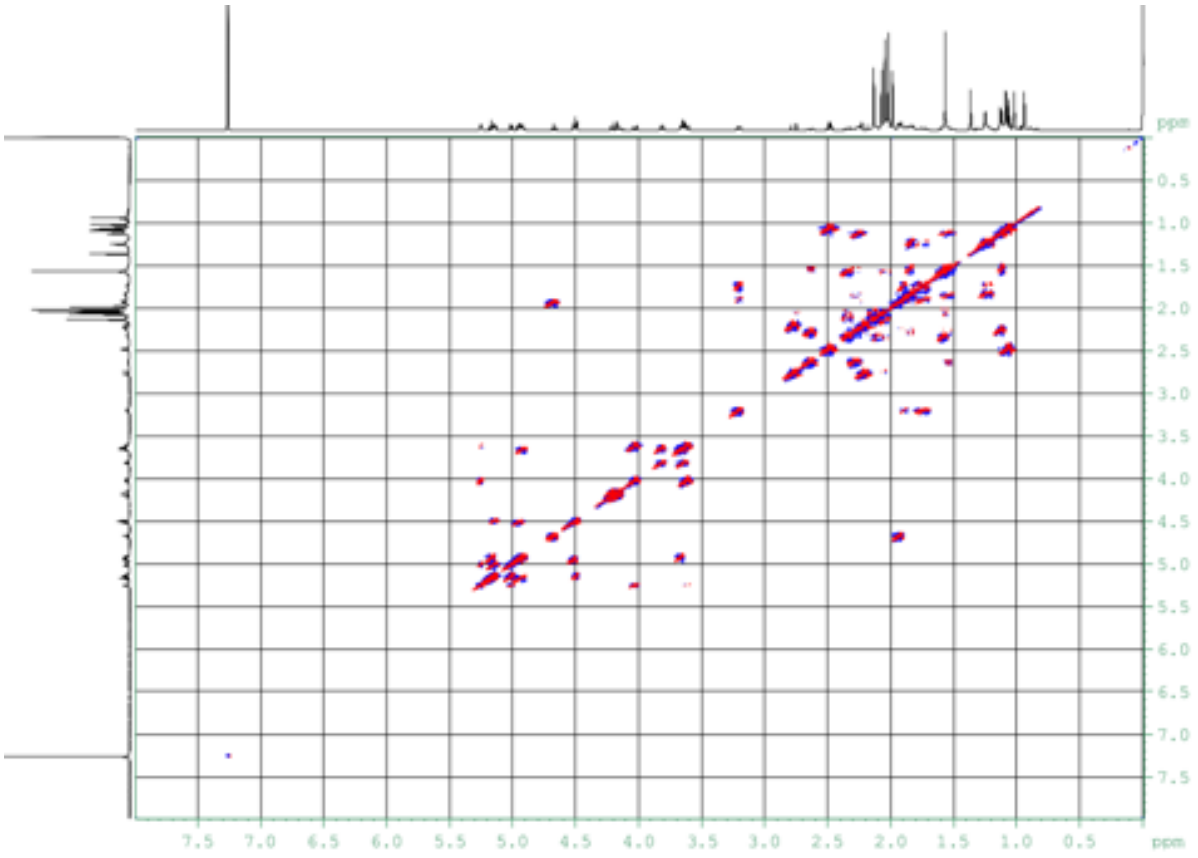

**Spectrum S3.13.5: DEPT spectrum for compound 13Ac in $CDCl_3$**



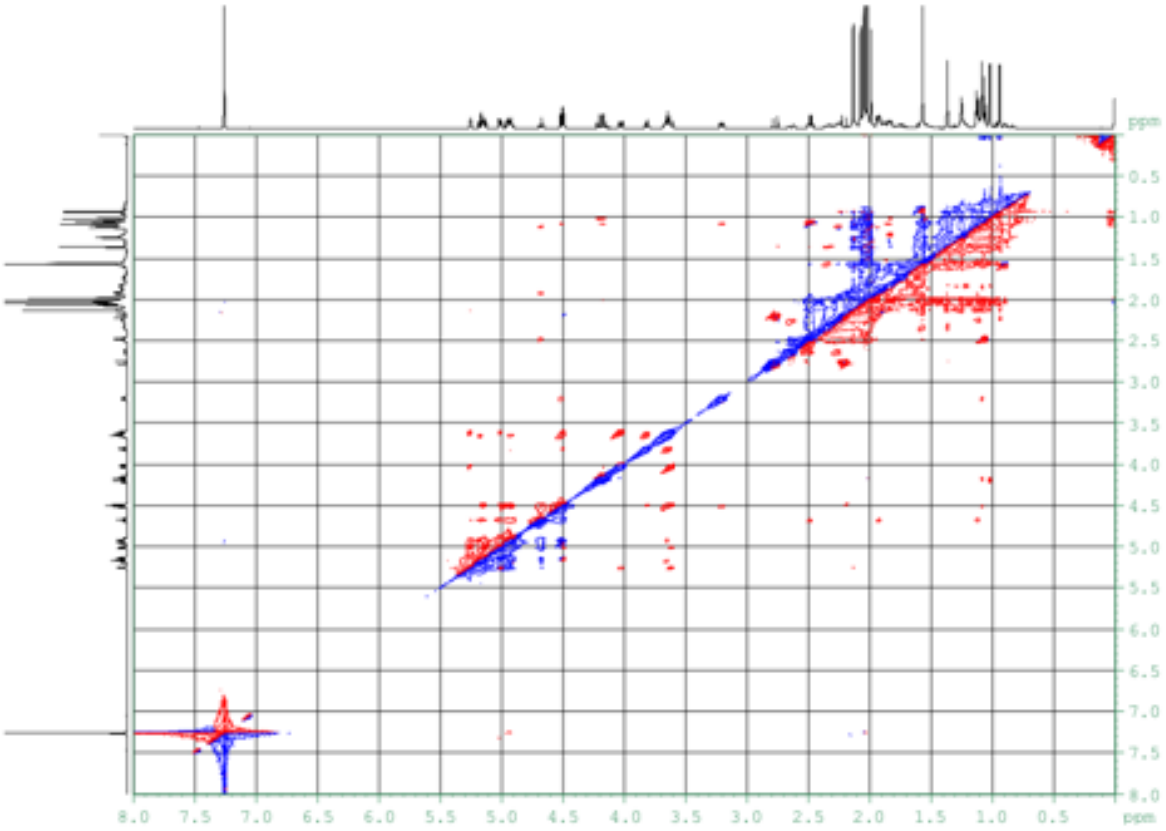

**Spectrum S3.13.6: HSQCDEPT spectrum for compound 13Ac in $CDCl_3$**



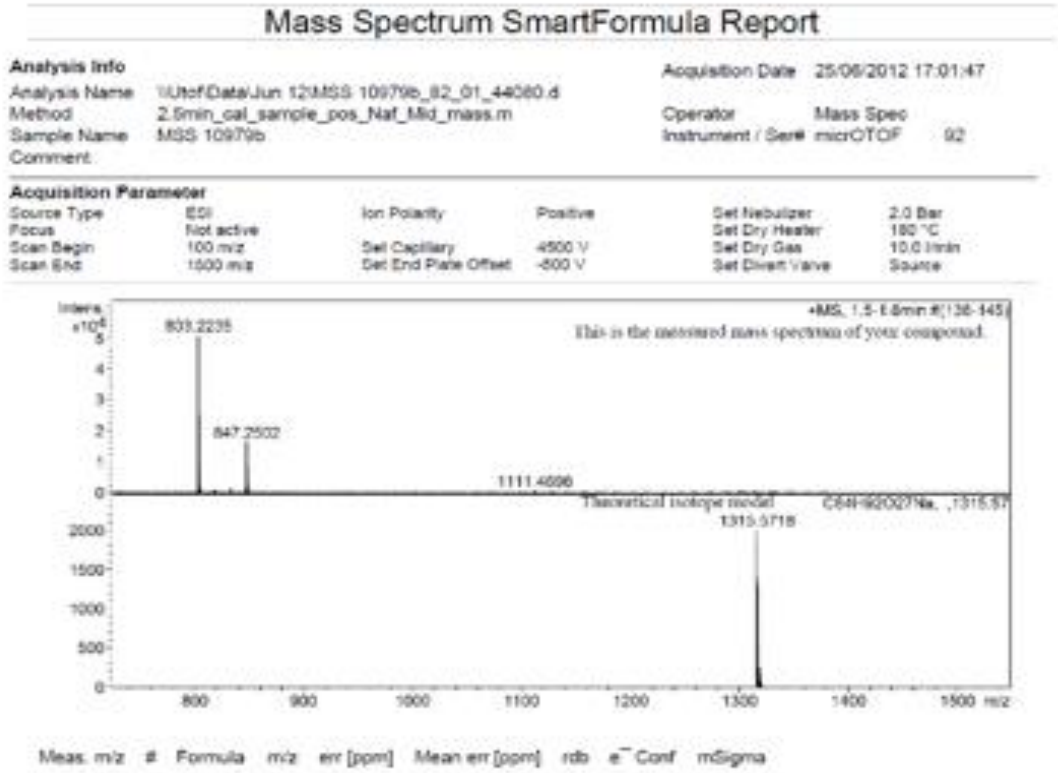

**Spectrum S3.13.7: HMBC spectrum for compound 13Ac in $CDCl_3$**



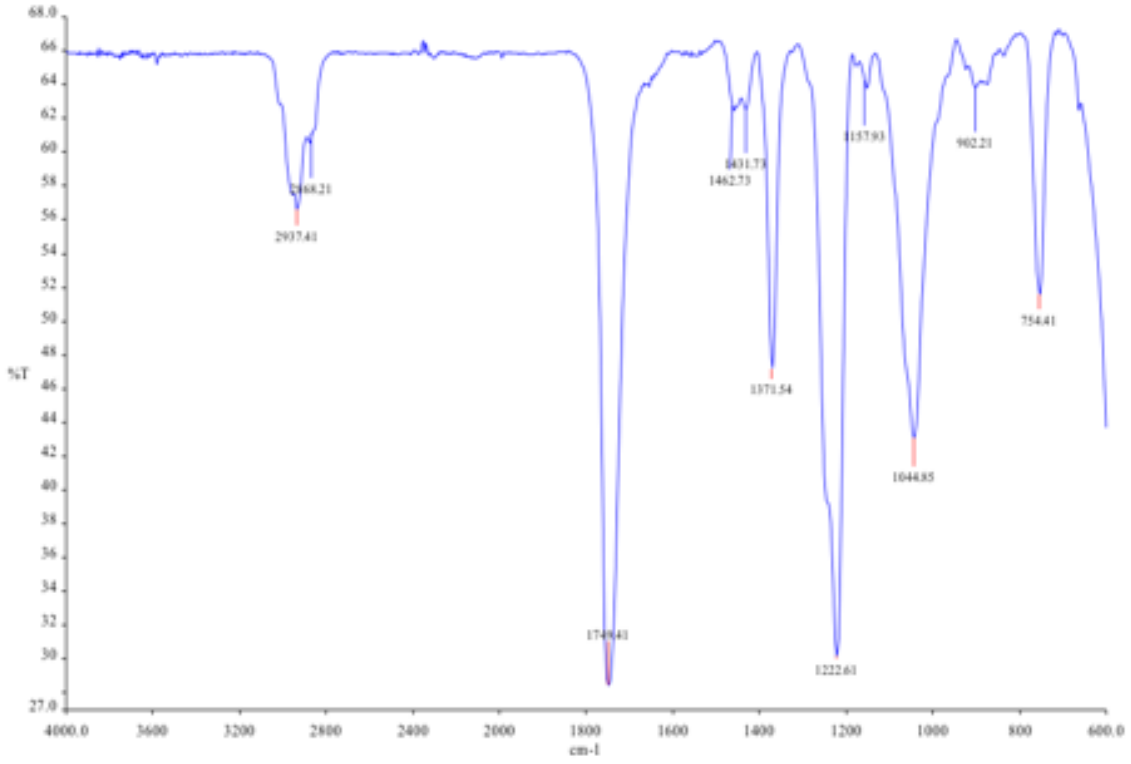

**Spectrum S3.13.8: COSY spectrum for compound 13Ac in $CDCl_3$**



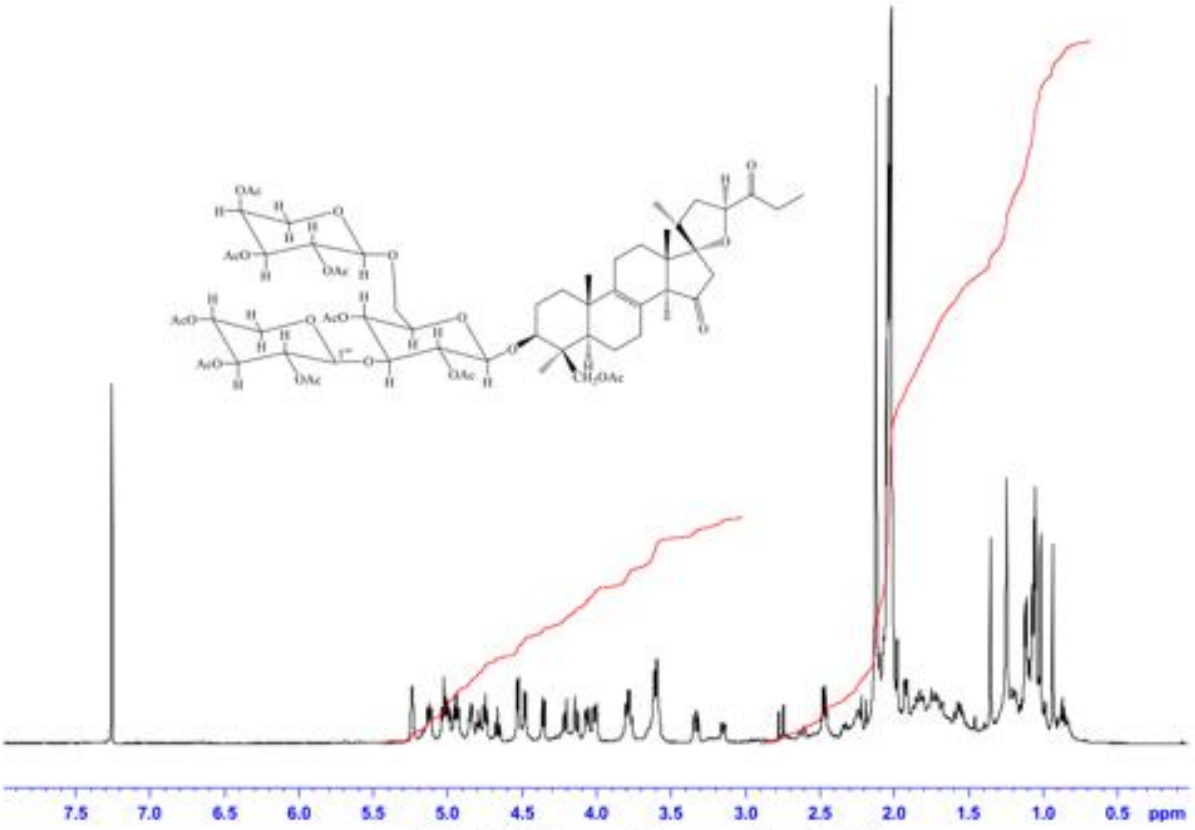

**Spectrum S3.13.9: NOESY spectrum for compound 13Ac in $CDCl_3$**



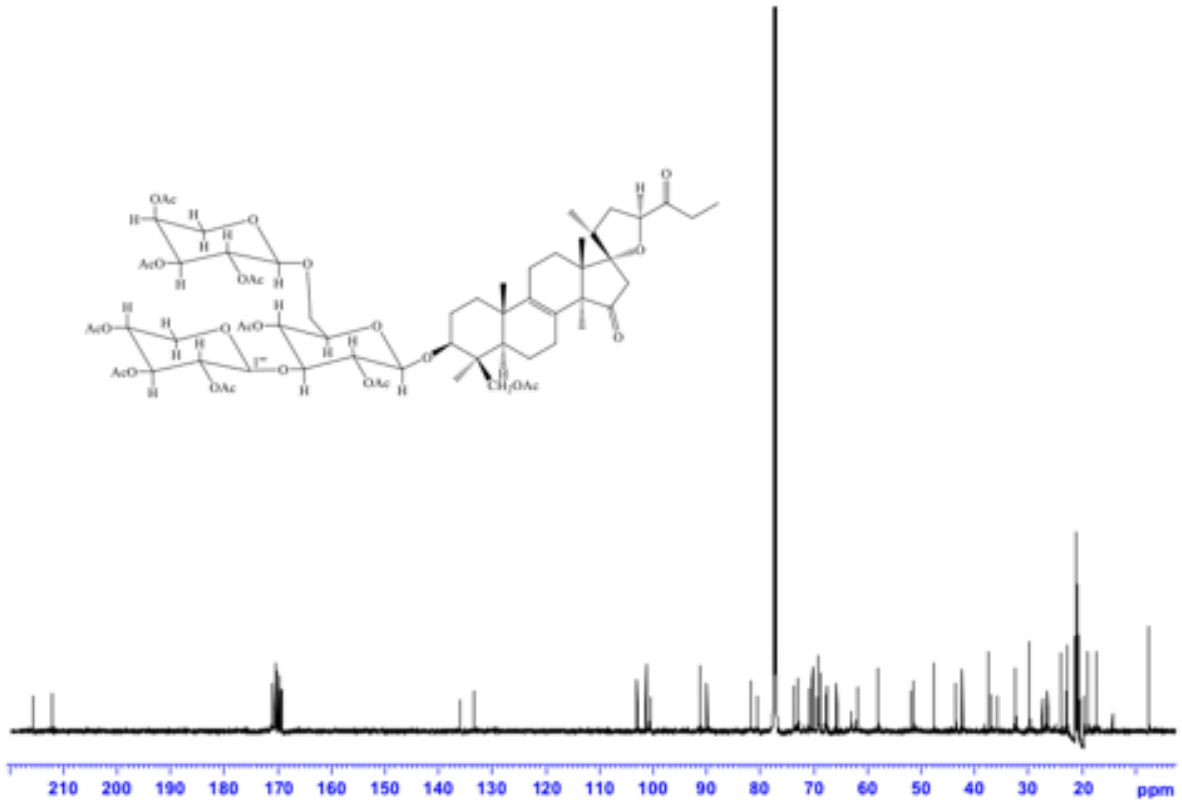

**Spectrum S3.14.1: Mass spectrum for compound 14Ac**



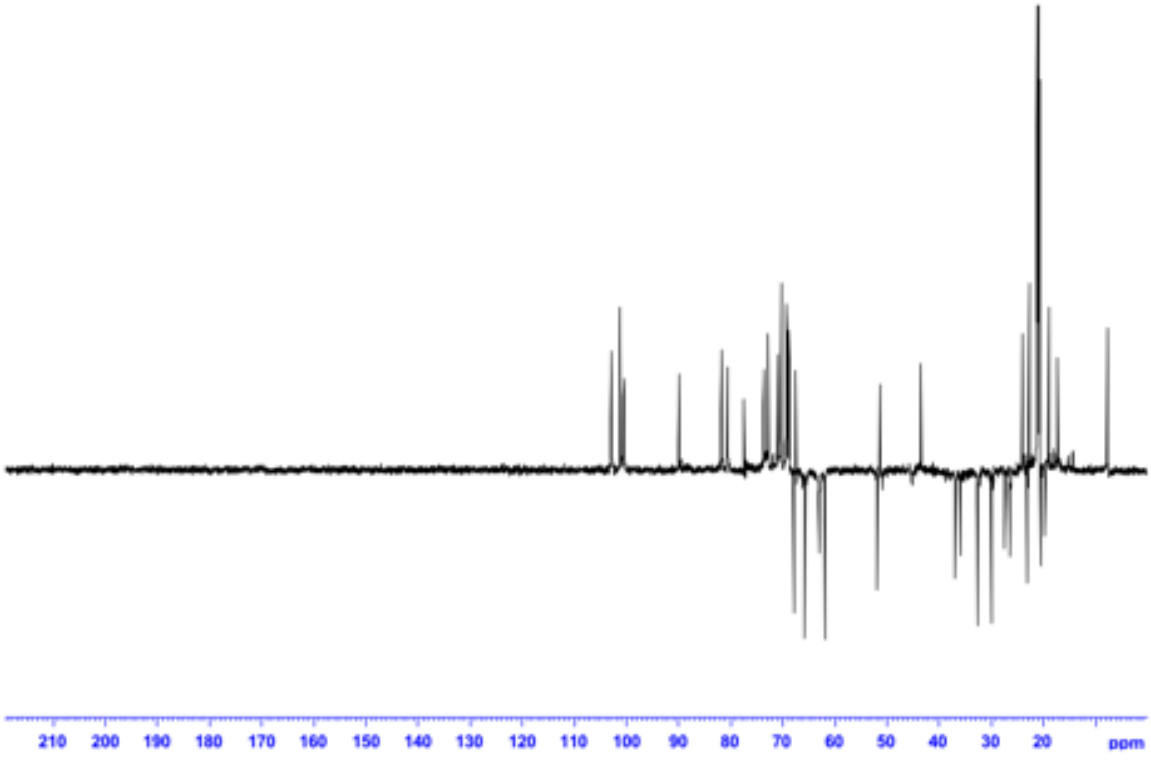

**Spectrum S3.14.2: FTIR spectrum for compound 14Ac**



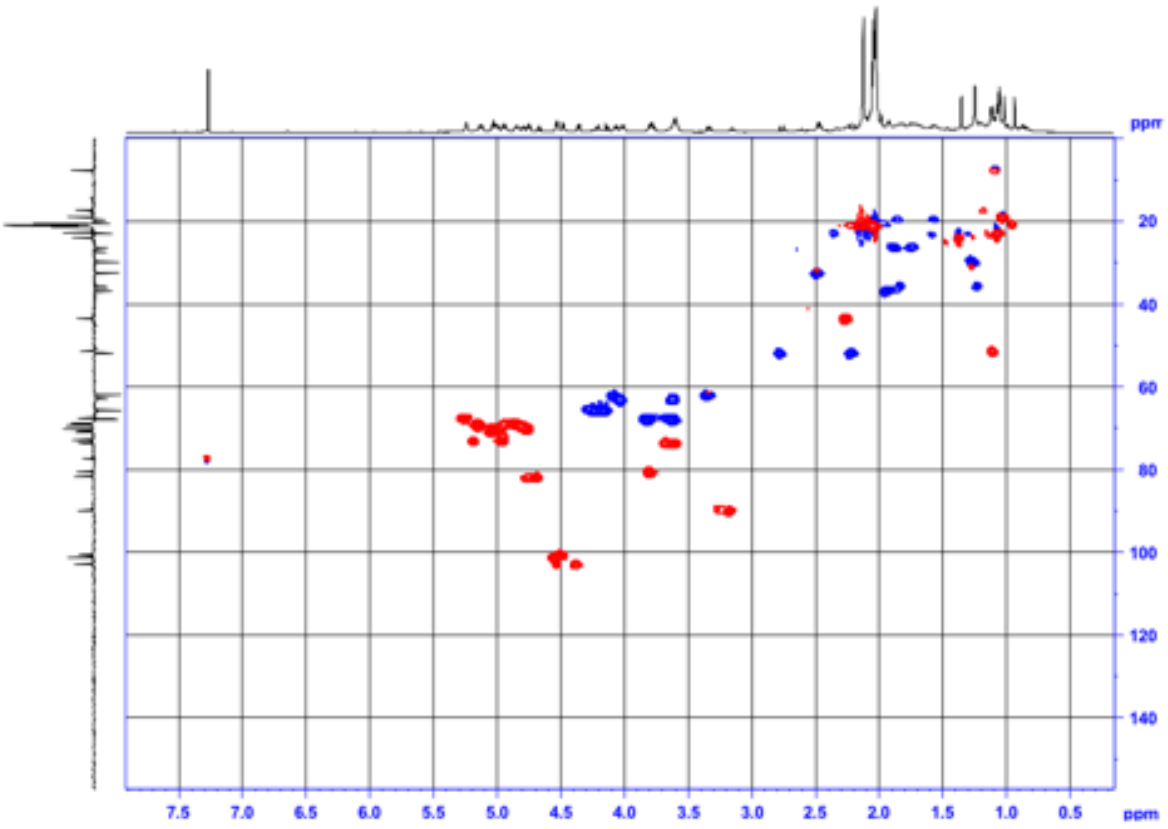

**Spectrum S3.14.3: $^1$H NMR spectrum for compound 14Ac in $CDCl_3$**



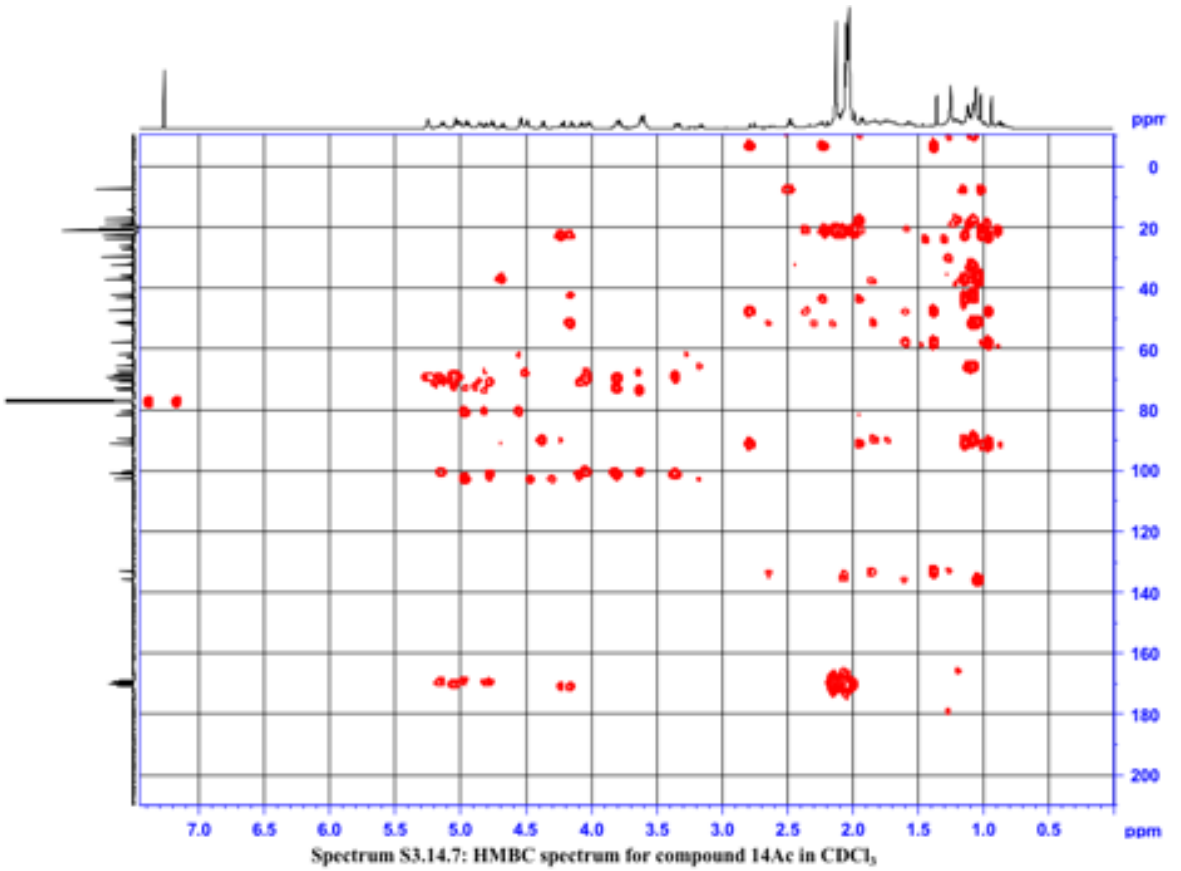

**Spectrum S3.14.4: $^{13}C$ NMR spectrum for compound 14Ac in $CDCl_3$**



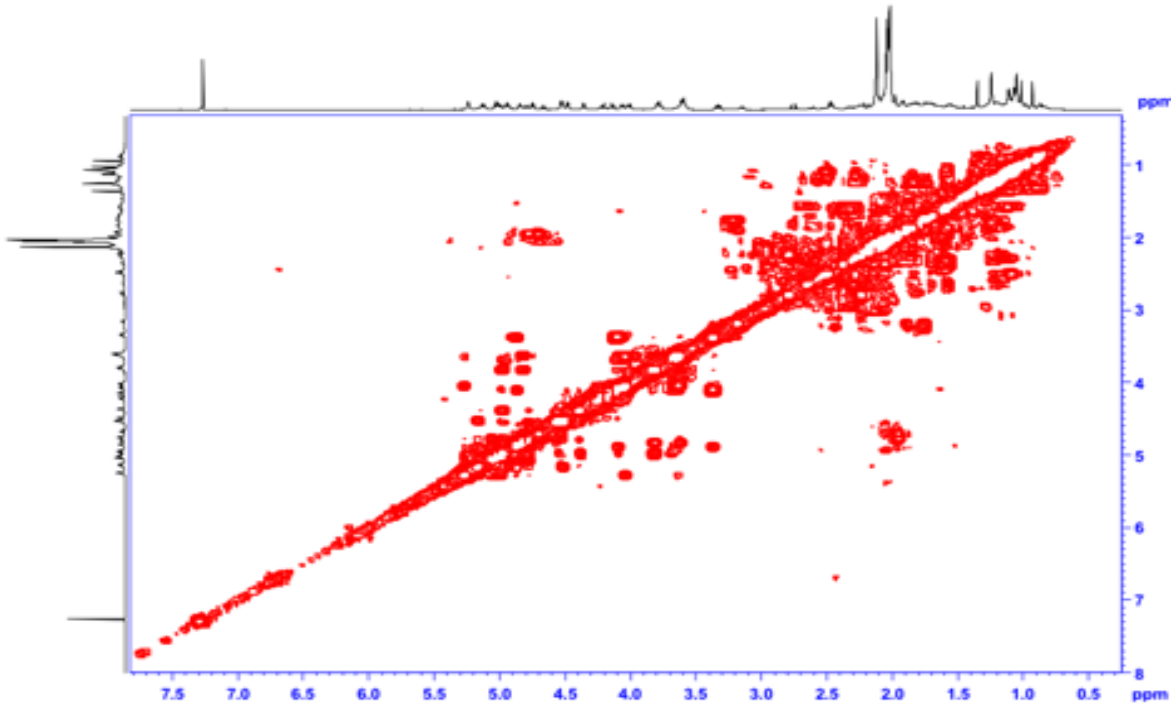

**Spectrum S3.14.5: DEPT spectrum for compound 14Ac in $CDCl_3$**



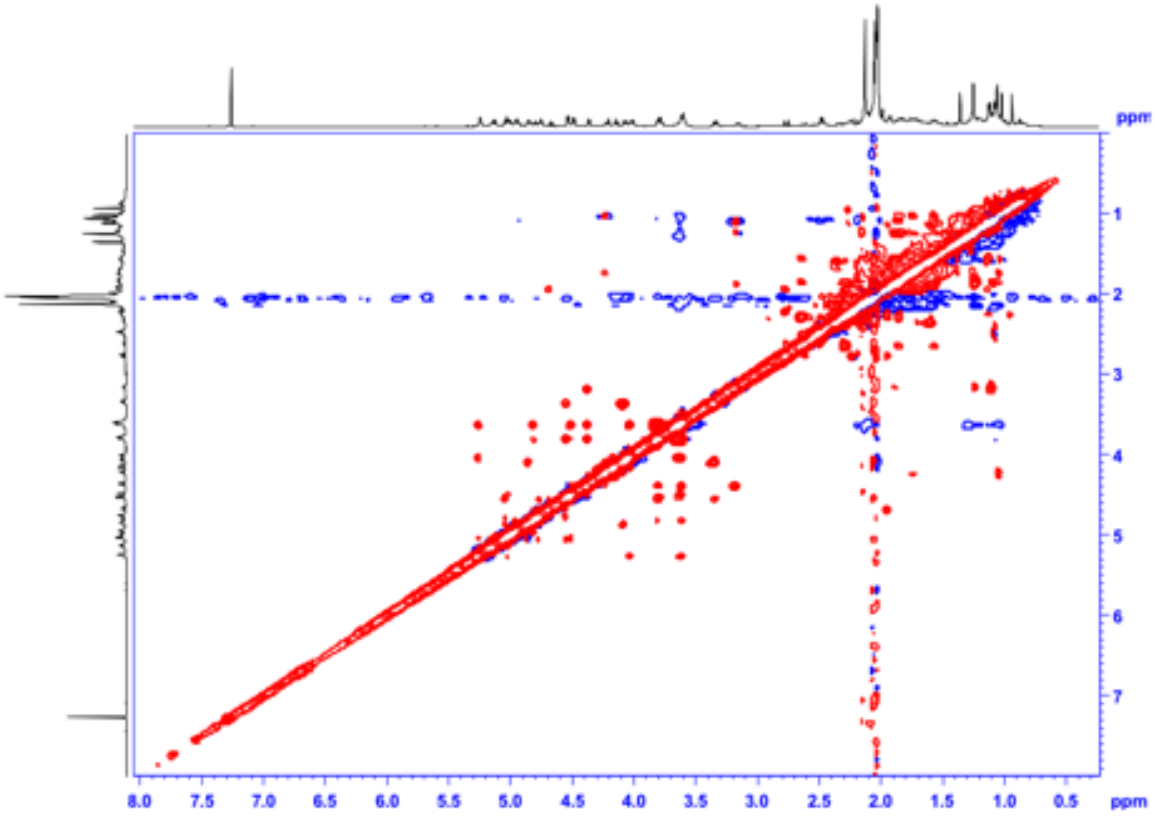

Spectrum S3.14.6: HSQCDEPT spectrum for compound 14Ac in $CDCl_3$




Spectrum S3.14.7: HMBC spectrum for compound 14Ac in $CDCl_3$




**Spectrum S3.14.8: COSY spectrum for compound 14Ac in $CDCl_3$**




**Spectrum S3.14.9: NOESY spectrum for compound 14Ac in $CDCl_3$**

## S.5 NCI 59 CELL SCREENING DATA FOR COMPOUNDS

**The following compounds were submitted to the NCI-59 Panel:**

5,7-dihydroxy-8-methoxy-3-(4'-methoxybenzyl)-4-chromanone (**EB1**) , eucosterol (**EB4**), (23*S*)-17α,23-epoxy-3β,28,29-trihydrox-27-norlanost-8-en-24-one (**EB5**), **2Ac**, **3**, **4** and **5**. Structures are provided in S.1

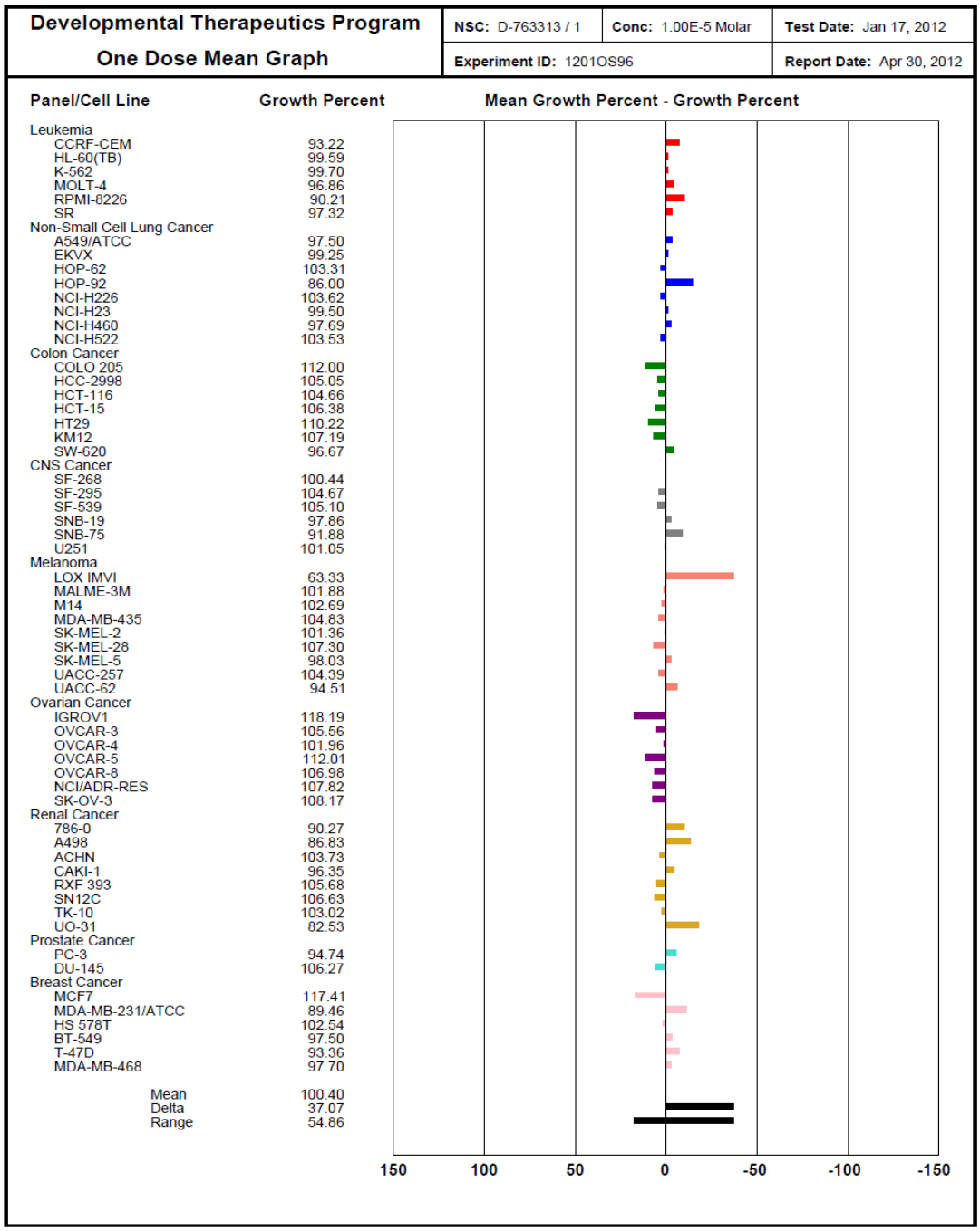



**Figure S.4.1: Single dose screen report for compound EB1**

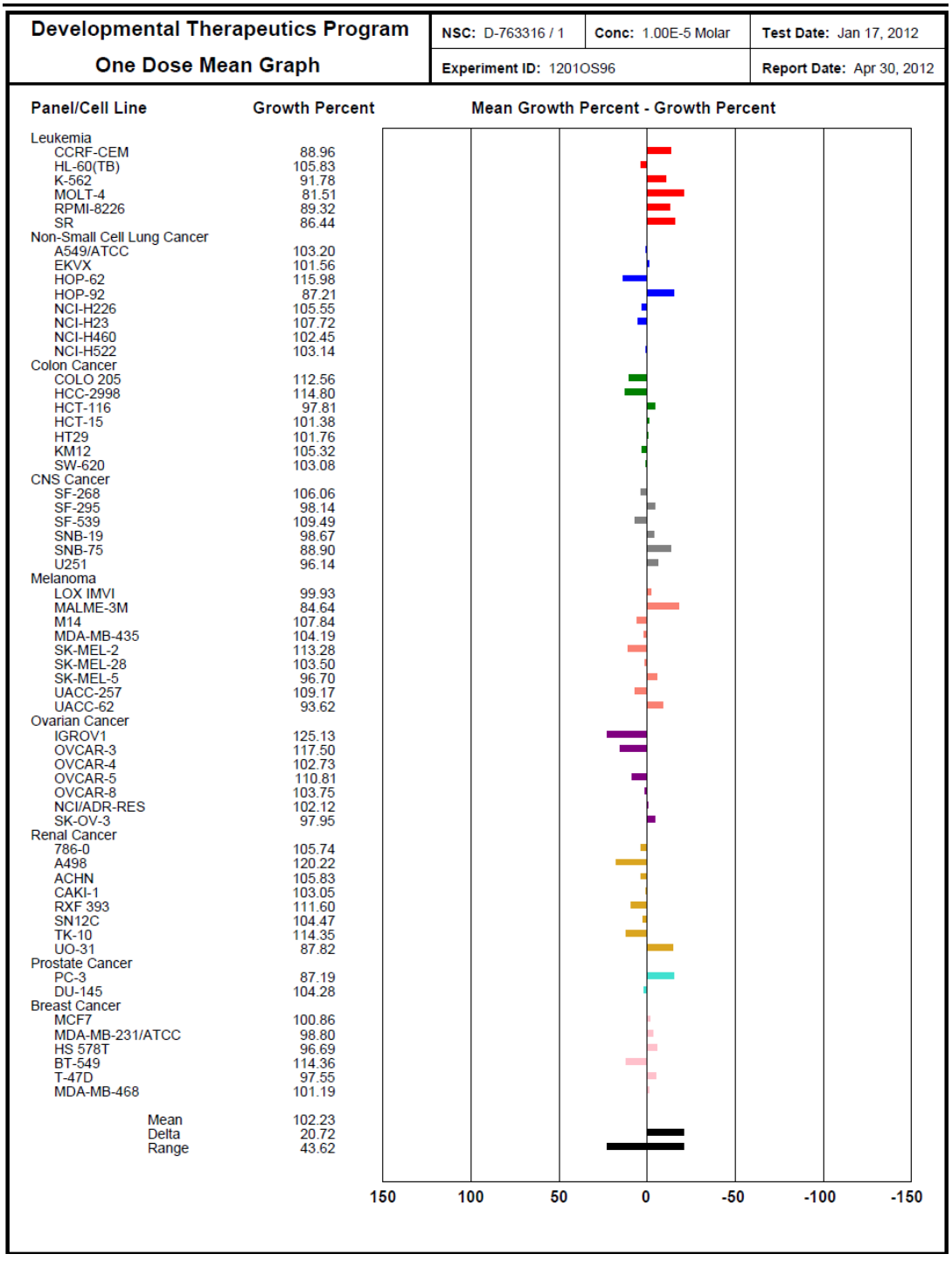



**Figure S.4.2: Single dose screen report for compound EB4**

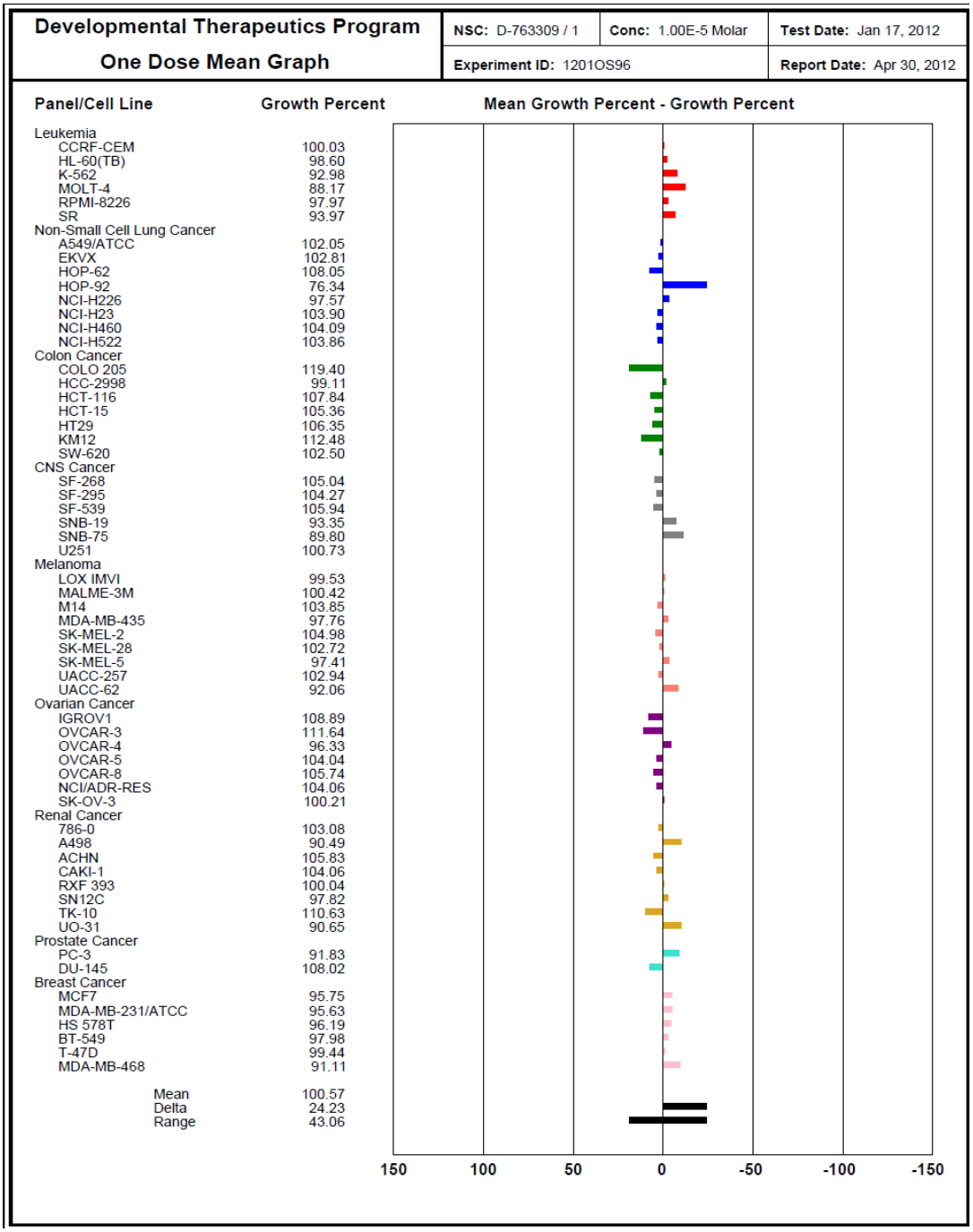



**Figure S.4.3: Single dose screen report for compound EB5**

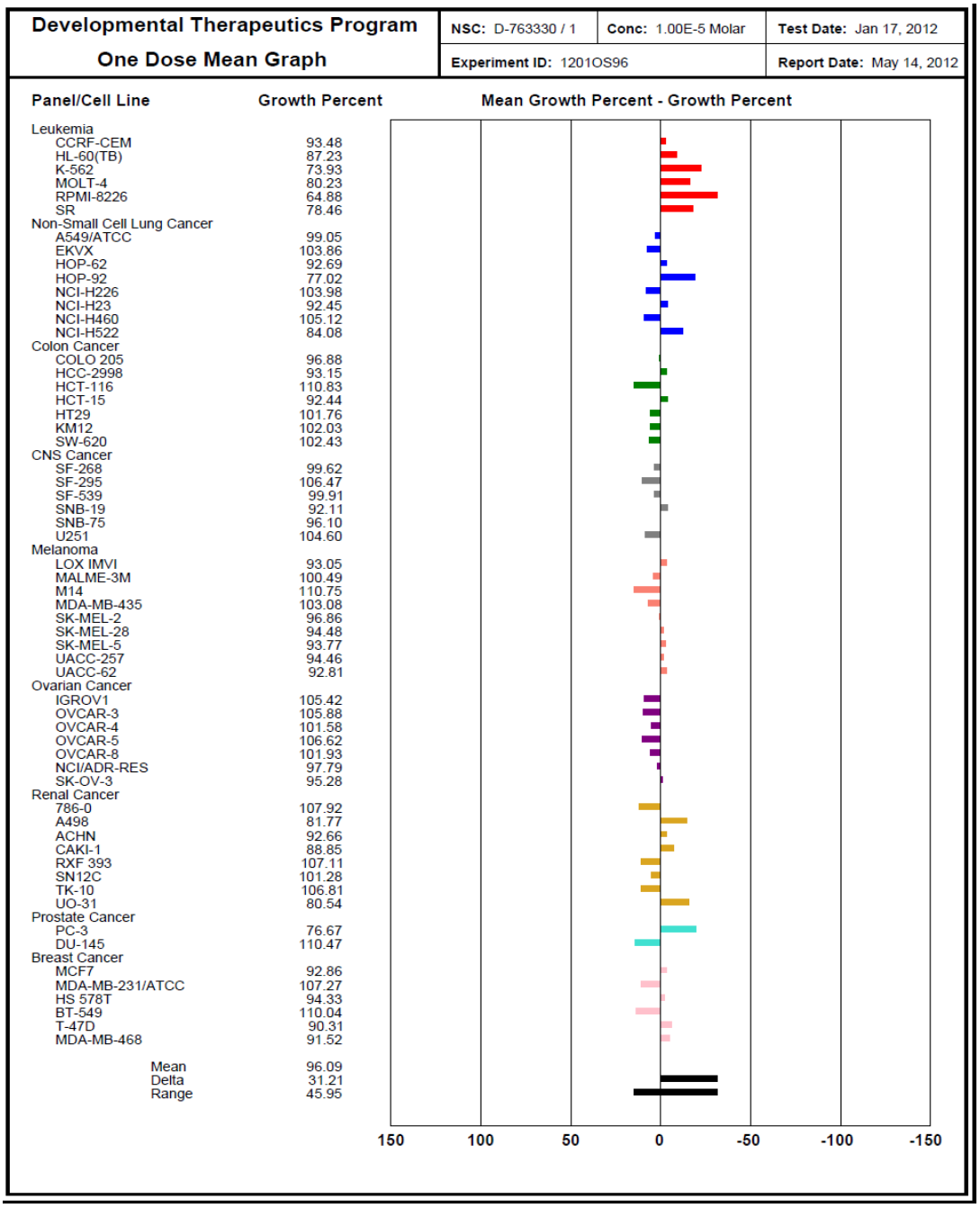



**Figure S.4.4: Single dose screen report for compound 2Ac**

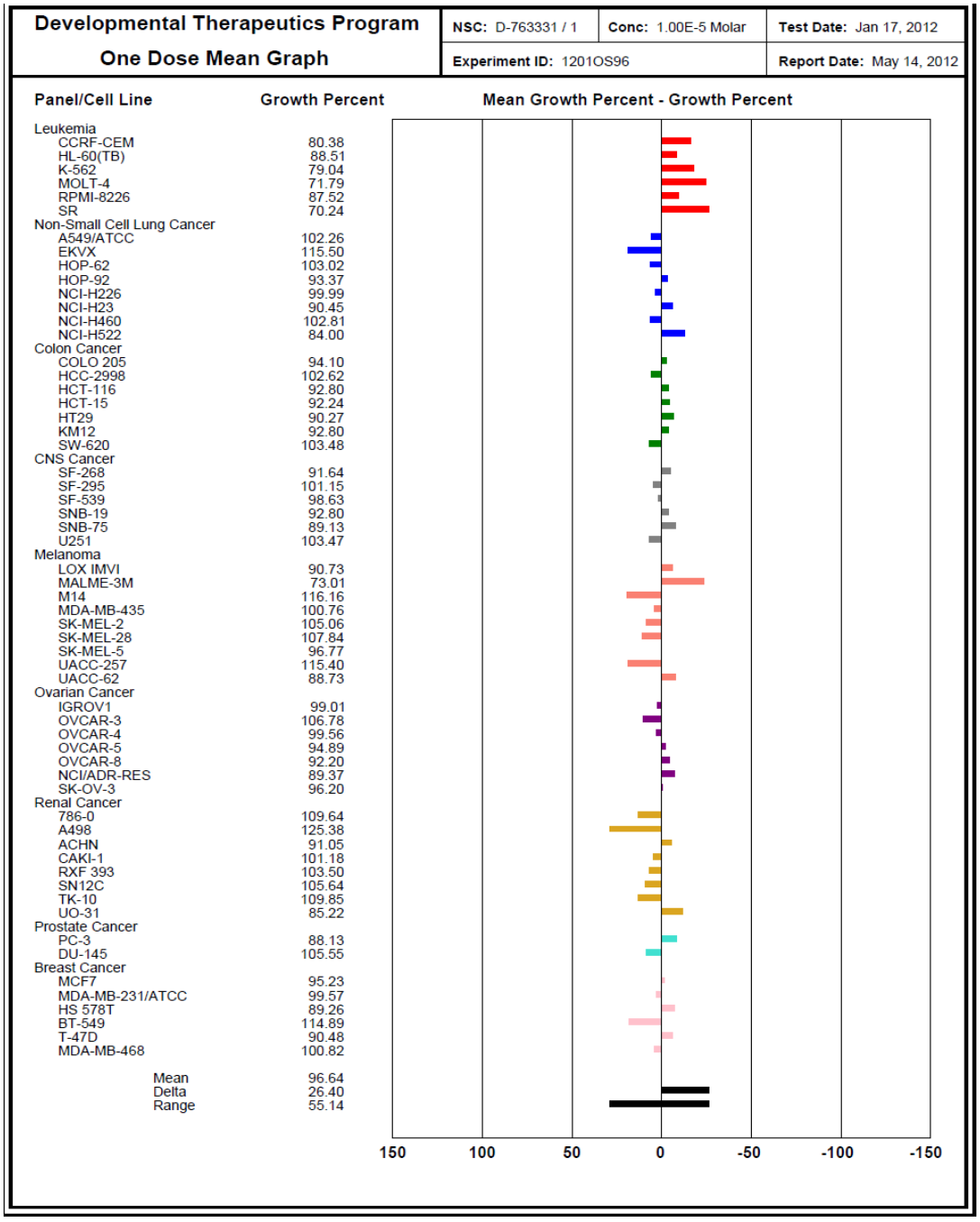



**Figure S.4.5: Single dose screen report for compound 3**

**Developmental Therapeutics Program**

**One Dose Mean Graph**

| NSC: D-763328 / 1 | Conc: 1.00E-5 Molar | Test Date: Jan 17, 2012 |
|---|---|---|
| Experiment ID: 1201OS96 | | Report Date: May 14, 2012 |

| Panel/Cell Line | Growth Percent |
|---|---|
| Leukemia | |
| CCRF-CEM | 92.33 |
| HL-60(TB) | 98.35 |
| K-562 | 80.64 |
| MOLT-4 | 84.72 |
| RPMI-8226 | 90.17 |
| SR | 81.00 |
| Non-Small Cell Lung Cancer | |
| A549/ATCC | 108.14 |
| EKVX | 110.73 |
| HOP-62 | 106.18 |
| HOP-92 | 85.11 |
| NCI-H226 | 107.67 |
| NCI-H23 | 93.21 |
| NCI-H460 | 103.39 |
| NCI-H522 | 27.45 |
| Colon Cancer | |
| COLO 205 | 94.09 |
| HCC-2998 | 100.22 |
| HCT-116 | 106.78 |
| HCT-15 | 97.65 |
| HT29 | 96.29 |
| KM12 | 98.11 |
| SW-620 | 103.34 |
| CNS Cancer | |
| SF-268 | 103.99 |
| SF-295 | 112.39 |
| SF-539 | 103.97 |
| SNB-19 | 101.29 |
| SNB-75 | 95.31 |
| U251 | 94.05 |
| Melanoma | |
| LOX IMVI | 72.65 |
| MALME-3M | 89.71 |
| M14 | 118.78 |
| MDA-MB-435 | 105.64 |
| SK-MEL-2 | 105.88 |
| SK-MEL-28 | 108.64 |
| SK-MEL-5 | 97.02 |
| UACC-257 | 108.43 |
| UACC-62 | 95.94 |
| Ovarian Cancer | |
| IGROV1 | 107.12 |
| OVCAR-3 | 103.47 |
| OVCAR-4 | 104.45 |
| OVCAR-5 | 106.55 |
| OVCAR-8 | 102.80 |
| NCI/ADR-RES | 92.71 |
| SK-OV-3 | 93.07 |
| Renal Cancer | |
| 786-0 | 112.94 |
| A498 | 105.48 |
| ACHN | 105.81 |
| CAKI-1 | 98.76 |
| RXF 393 | 111.08 |
| SN12C | 90.18 |
| TK-10 | 108.97 |
| UO-31 | 84.45 |
| Prostate Cancer | |
| PC-3 | 95.36 |
| DU-145 | 99.59 |
| Breast Cancer | |
| MCF7 | 96.33 |
| MDA-MB-231/ATCC | 101.99 |
| HS 578T | 93.43 |
| BT-549 | 120.78 |
| T-47D | 91.48 |
| MDA-MB-468 | 107.10 |
| Mean | 98.53 |
| Delta | 71.08 |
| Range | 93.33 |

Mean Growth Percent - Growth Percent

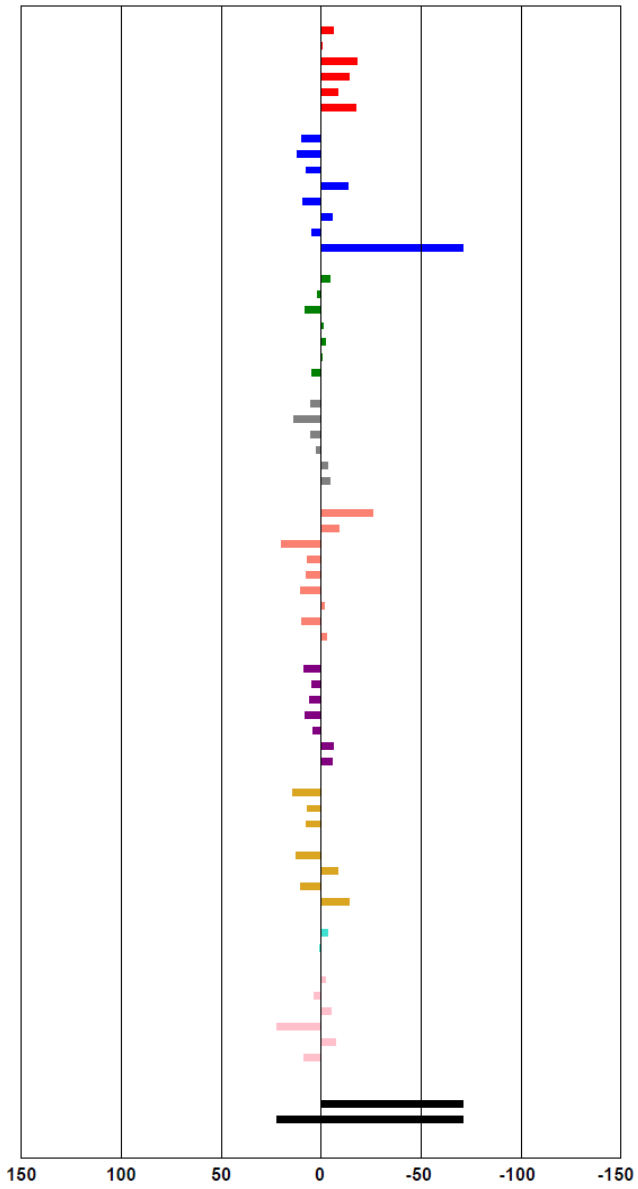



**Figure S.4.6: Single dose screen report for compound 5**