

Cytotoxic C₄₇-Polyacetylene Carboxylic Acids from a Marine Sponge *Petrosia* sp.

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Figure S1. ¹H NMR spectrum of petroformynic acid B (**3**) in CD₃OD.

Figure S2. ¹³C NMR spectrum of petroformynic acid B (**3**) in CD₃OD.

Figure S3. COSY spectrum of petroformynic acid B (**3**) in CD₃OD.

Figure S4. HSQC spectrum of petroformynic acid B (**3**) in CD₃OD.

Figure S5. HMBC spectrum of petroformynic acid B (**3**) in CD₃OD.

Figure S6. Tandem FAB mass spectrum of petroformynic acid B (**3**) [Precursor, (M+Na)⁺].

Figure S7. Tandem FAB mass spectrum of petroformynic acid B (**3**) [Precursor, (M+2Na-H)⁺].

Figure S8. Tandem FAB mass spectrum of petroformynic acid B (**3**) [Precursor, (M-H)⁻].

Figure S9. ¹H NMR spectrum of petroformynic acid C (**4**) in CD₃OD.

Figure S10. ¹³C NMR spectrum of petroformynic acid C (**4**) in CD₃OD.

Figure S11. COSY spectrum of petroformynic acid C (**4**) in CD₃OD.

Figure S12. HSQC spectrum of petroformynic acid C (**4**) in CD₃OD.

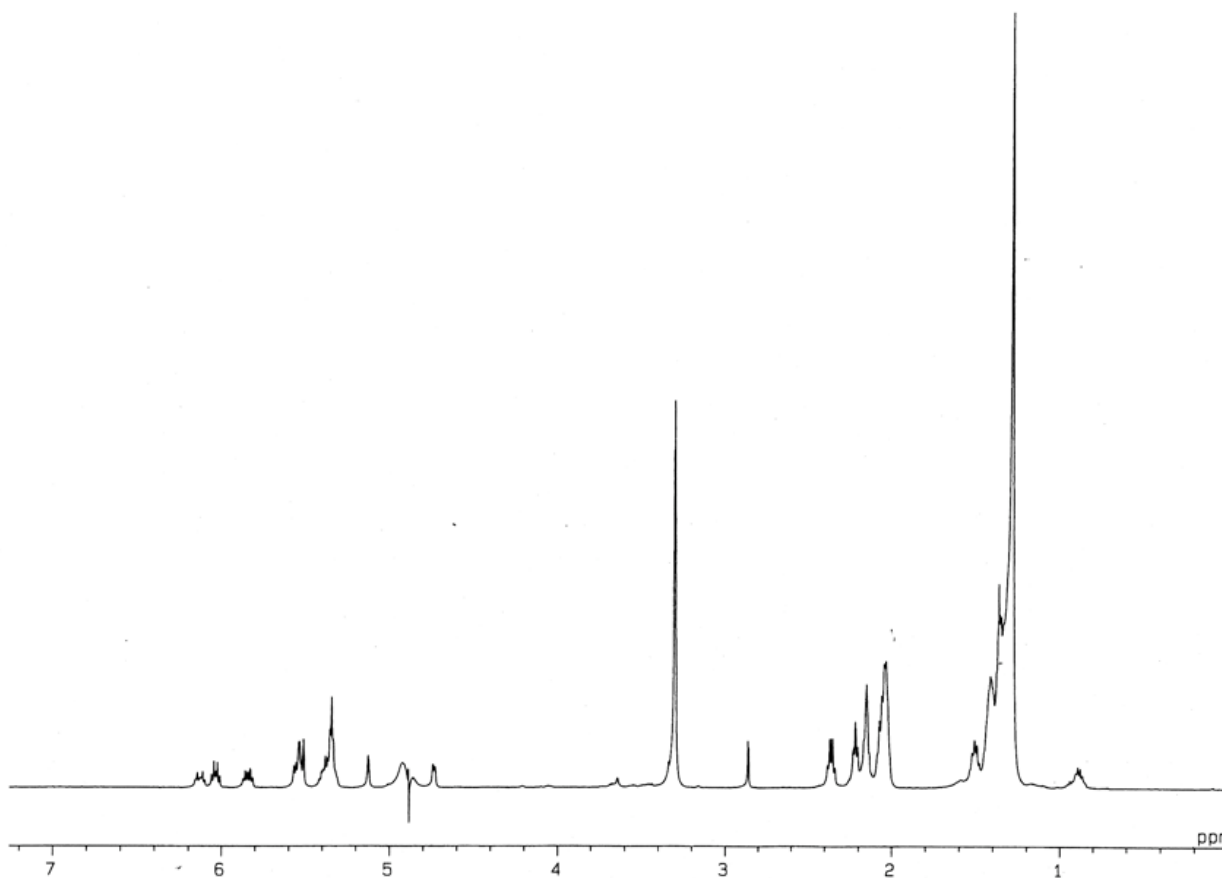
Figure S13. HMBC spectrum of petroformynic acid C (**4**) in CD₃OD.

Figure S14. Tandem FAB mass spectrum of petroformynic acid C (**4**) [Precursor, (M+Na)⁺].

Figure S15. Tandem FAB mass spectrum of petroformynic acid C (**4**) [Precursor, (M+2Na-H)⁺].

Figure S16. Tandem FAB mass spectrum of petroformynic acid C (**4**) [Precursor, (M-H)⁻].

S110KA-16-4-CD30D-1H



13-NOV-2003 18: 52: 37.05

DFILE : MDA0: [ALPHA] ALPHA
SFIL : S110KA-16-4-CD30D-1H

COMNT : S110KA-16-4-CD30D-1H
EXMOD : SINGL
IRMOD : HMG
POINT : 16384
FREQ : 5000.00 Hz
SCANS : 16
DUMMY : 2
ACQTM : 3.2768 sec
PD : 1.4000 sec
RGAIN : 18

PW1 : 5.70 usec

OBNUC : ^1H
OBFRQ : 500.00 MHz
OBSET : 162300.00 Hz

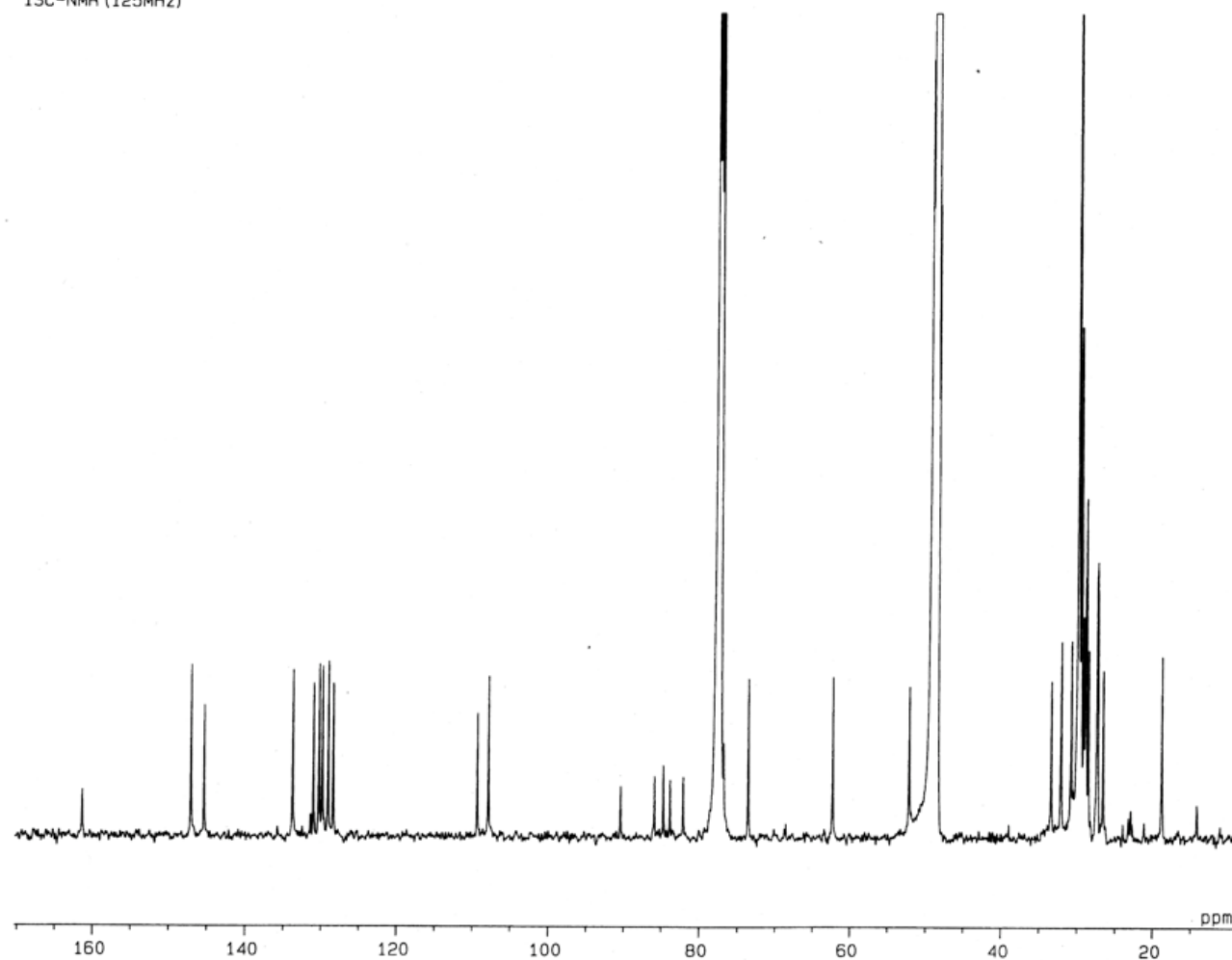
IRNUC : ^1H
IRFRQ : 500.00 MHz
IRSET : 162361.04 Hz
IRATN : 160
IRRPW : 50.0 usec
IRBP1 : 10
IRBP2 : 2
IRRNS : 0

ADBIT : 16
CTEMP : -27.5 c
CSPED : 0 Hz
SLVNT : CD_3OD

RESOL : 0.31 Hz
BF : 0.10 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 3.30 ppm
XE : 3648.38 Hz
XS : 574.19 Hz
operator

Figure S1. ^1H NMR spectrum of petroformynic acid B (3) in CD_3OD

¹³C-NMR (125MHz)



28-DEC-2003 11:04:19.34

DFILE : MDA0: [ALPHA] ALPHA
SFIL : S120KA-16-4-C4M1-13C-2

COMNT : 13C-NMR (125MHz)
EXMOD : SINGL
IRMOD : BCM
POINT : 16384
FREQU : 30303.03 Hz
SCANS : 33421
DUMMY : 2
ACQTM : 0.5407 sec
PD : 2.0000 sec
RGAIN : 23

PW1 : 4.70 usec

OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 129800.00 Hz

IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162300.00 Hz
IRATN : 160
IRRPW : 50.0 usec
IRBP1 : 25
IRBP2 : 4
IRRNS : 0

ADBIT : 16
CTEMP : -204.8 c
CSPED : 10 Hz
SLVNT : CD3OD

RESOL : 1.85 Hz
BF : 10.00 Hz
T1 : 0.00 %
T2 : 0.00 %
T3 : 90.00 %
T4 : 100.00 %
REFVL : 49.00 ppm
XE : 20319.16 Hz
XS : 3273.70 Hz
operator

Figure S2. ¹³C NMR spectrum of petroformynic acid B (**3**) in CD₃OD

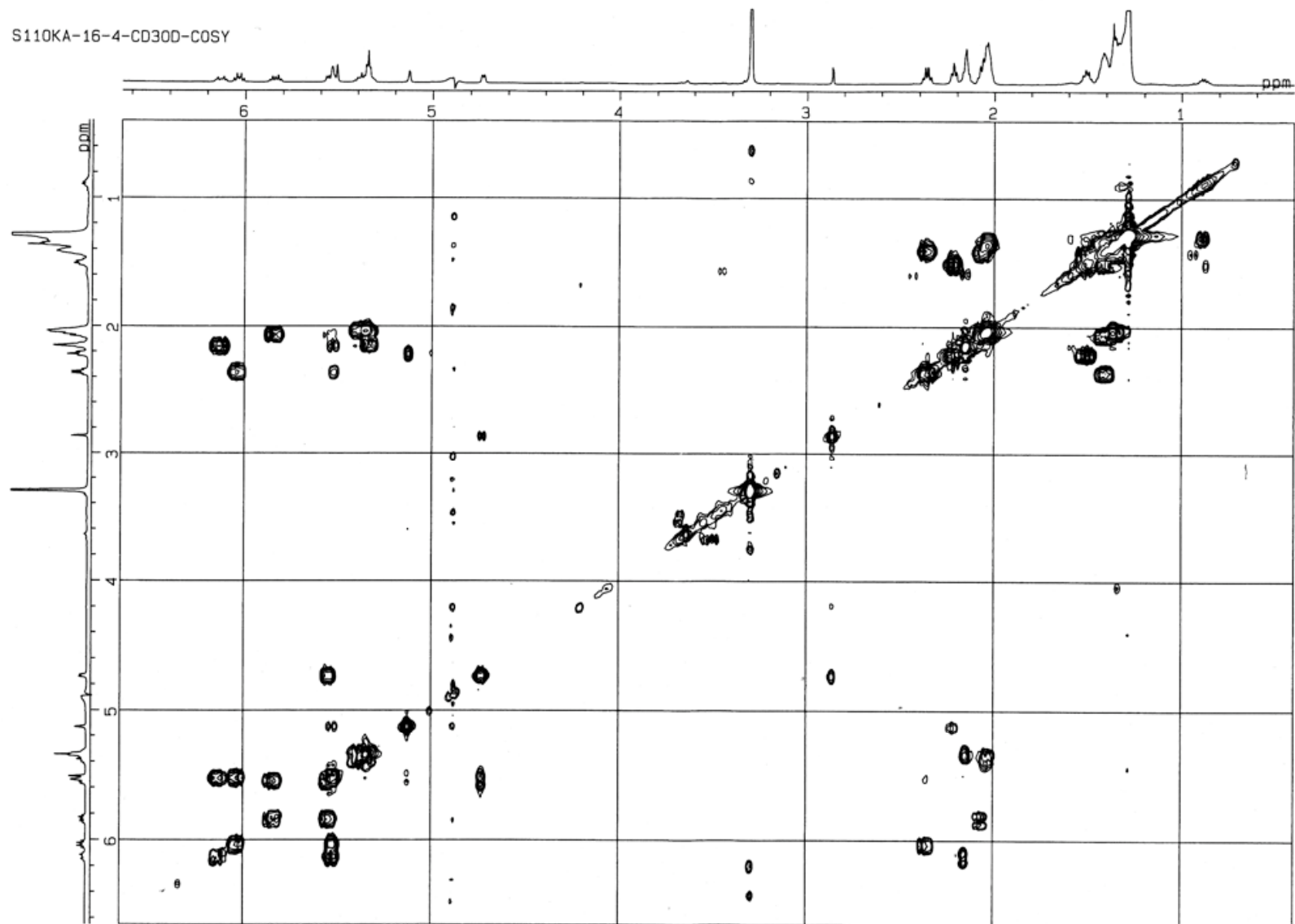


Figure S3. COSY spectrum of petrof ormynic acid B (**3**) in CD₃OD

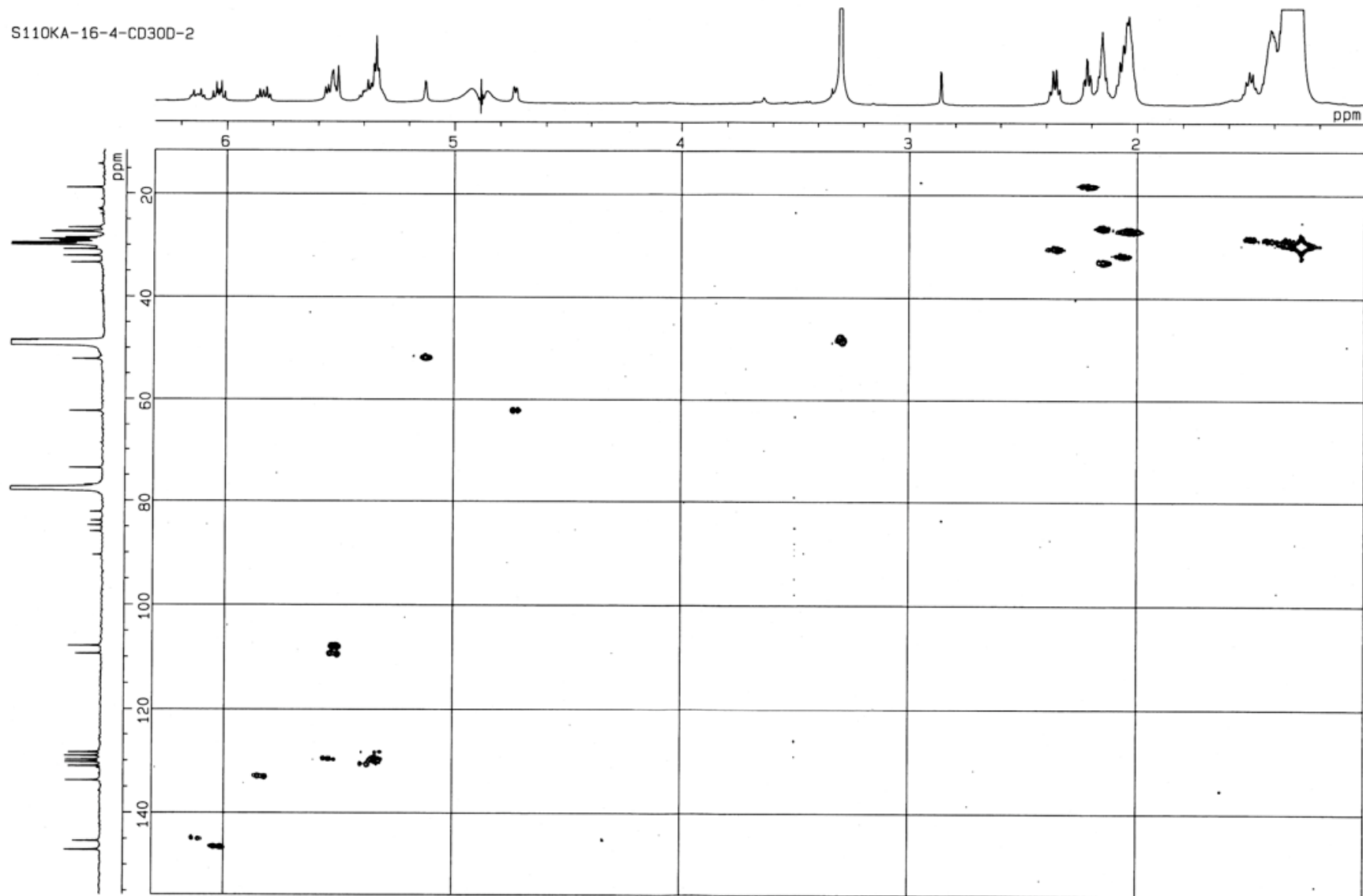
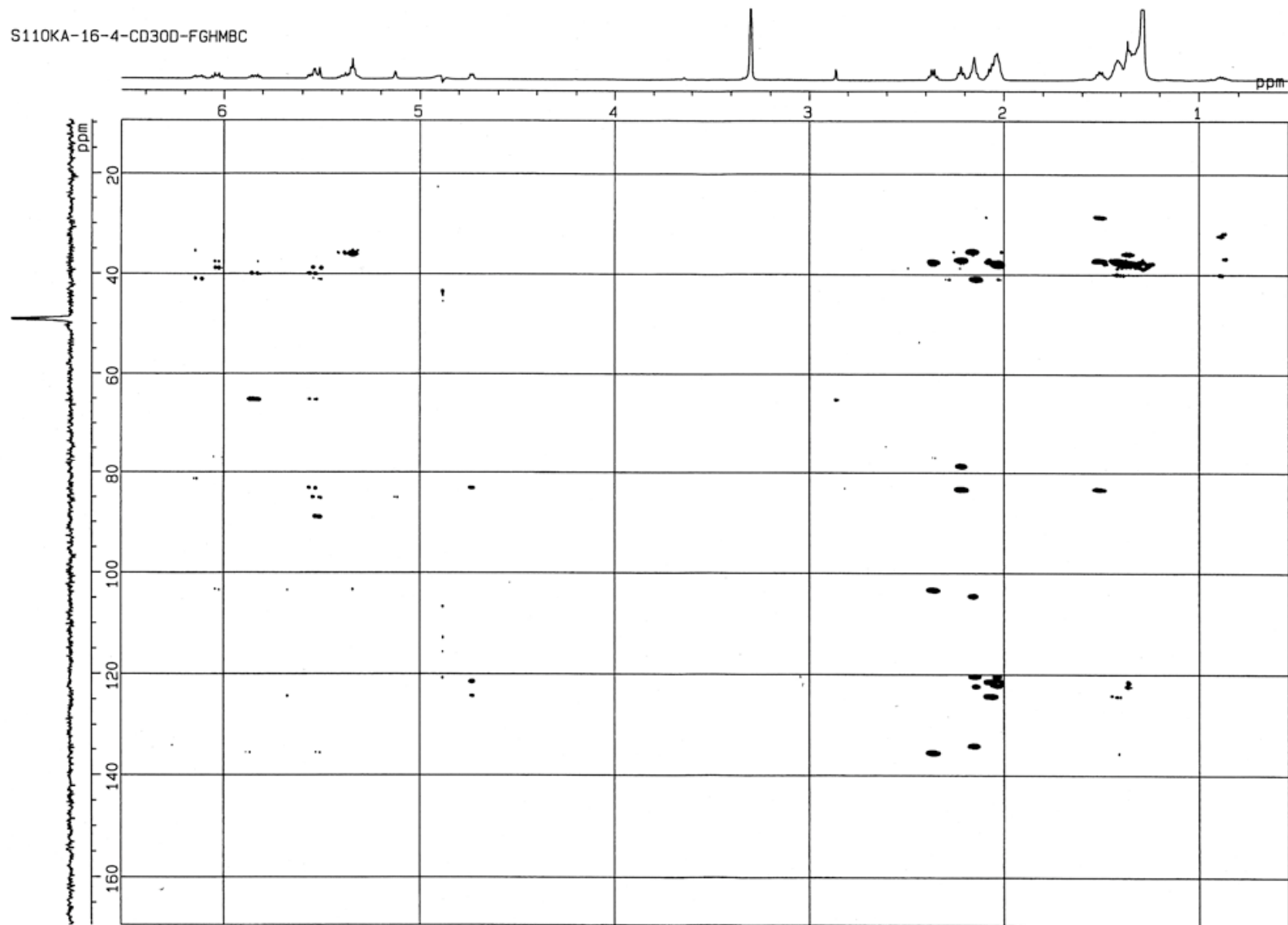


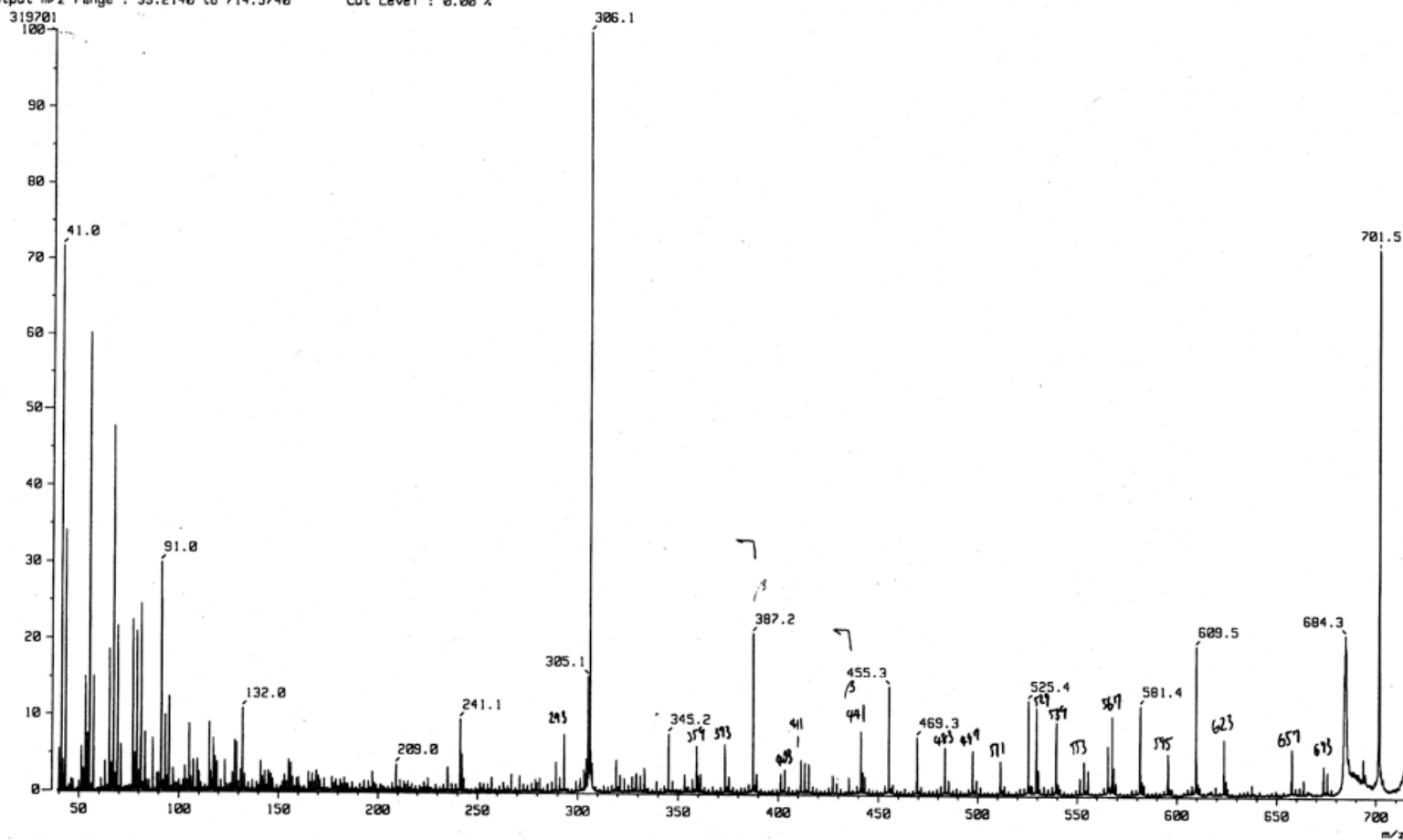
Figure S4. HSQC spectrum of petroformynic acid B (**3**) in CD₃OD



. *Figure S5.* HMBC spectrum of petroformynic acid B (**3**) in CD_3OD

[Mass Spectrum]
 Date : 16-Jul-92 14:45
 Sample:
 Note : Array Detector Data
 Inlet : Direct Ion Mode : FFB+
 Spectrum Type : Product (m/z 714.57, 2nd FFR, FC: 0.00kV)
 RT : 0.00 min Scan# : 1
 BP : m/z 306.1001 Int. : 30.49
 Output m/z range : 39.2140 to 714.5740 Cut Level : 0.00 %

<A16-4>
 $(M+Na)^+$ 719 $\xrightarrow{CID}$



. **Figure S6.** Tandem FAB mass spectrum of petroformynic acid B (**3**) [Precursor, $(M+Na)^+$].

[Mass Spectrum]
 Date : 16-Jul-92 14:45
 Sample:
 Note : Array Detector Data
 Inlet : Direct Ion Mode : FAB+
 Spectrum Type : Product (m/z 735.59, 2nd FFR, FC: 0.00kV)
 RT : 0.00 min Scan# : 1
 BP : m/z 70.9425 Int. : 24.81
 Output m/z range : 40.3674 to 735.5874 Cut Level : 0.00 %

<A16-4>

(M+2Na-H)⁺

741
 (1D)

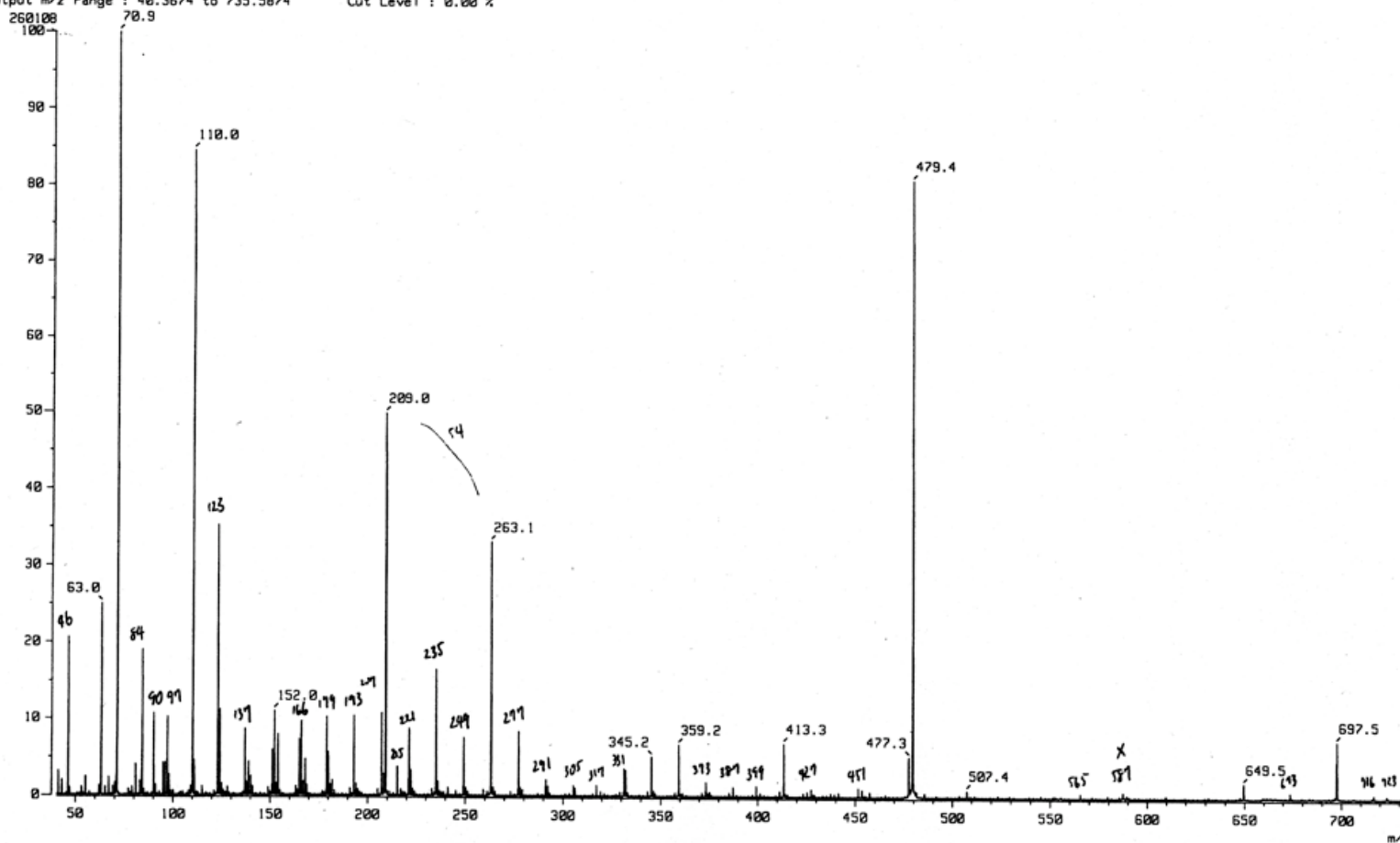


Figure S7. Tandem FAB mass spectrum of petroformynic acid B (**3**) [Precursor, (M+2Na-H)⁺].

[Mass Spectrum]
 Date : 16-Jul-92 14:45
 Sample:
 Note : Array Detector Data
 Inlet : Direct Ion Mode : FAB+
 Spectrum Type : Product (m/z 691.37, 2nd FFR, FC: 0.00kV)
 RT : 0.00 min Scan# : 1
 BP : m/z 191.0067 Int. : 22.26
 Output m/z range : 41.1711 to 691.3711 Cut Level : 0.00 %

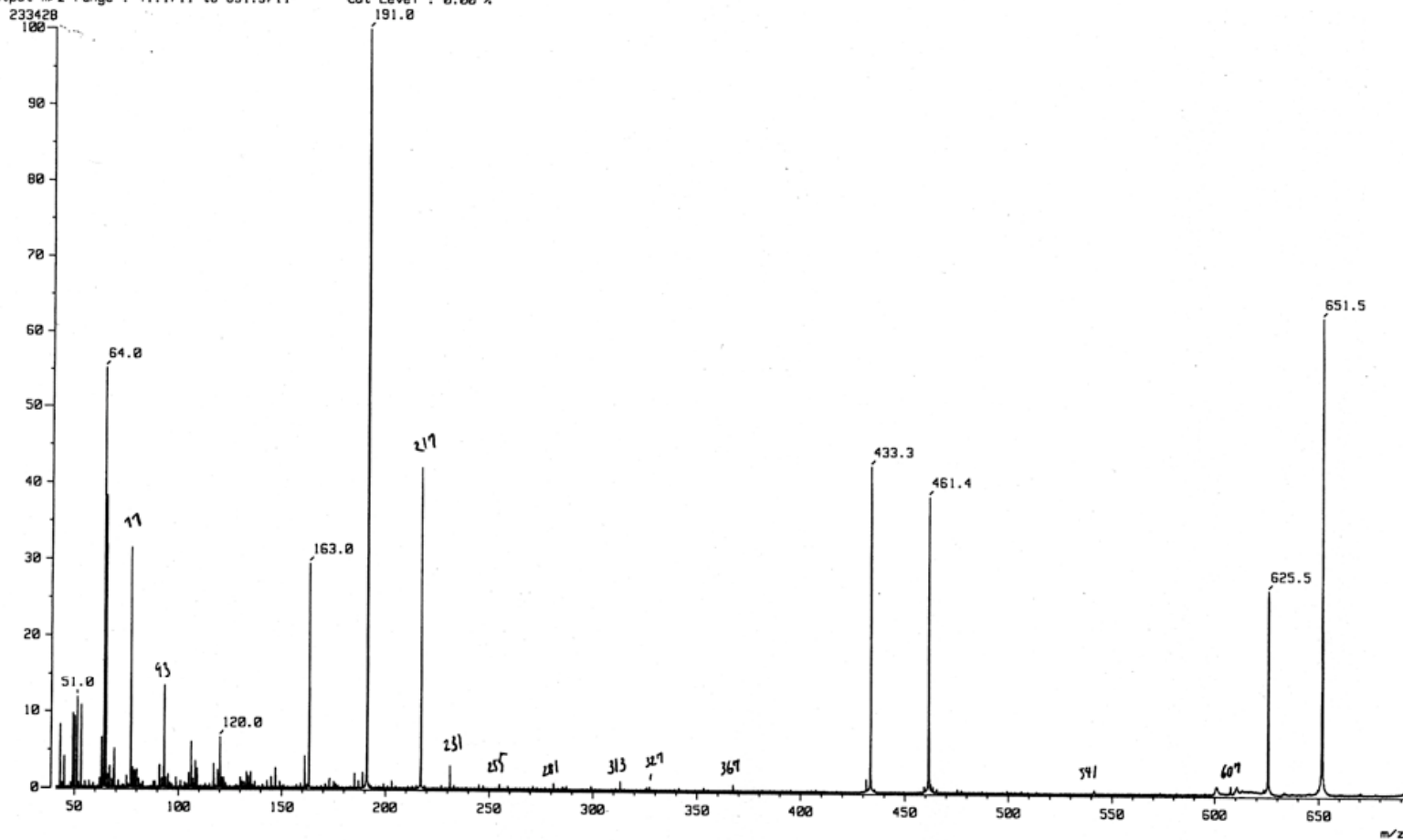


Figure S8. Tandem FAB mass spectrum of petroformynic acid B (**3**) [Precursor, (M-H)⁻].

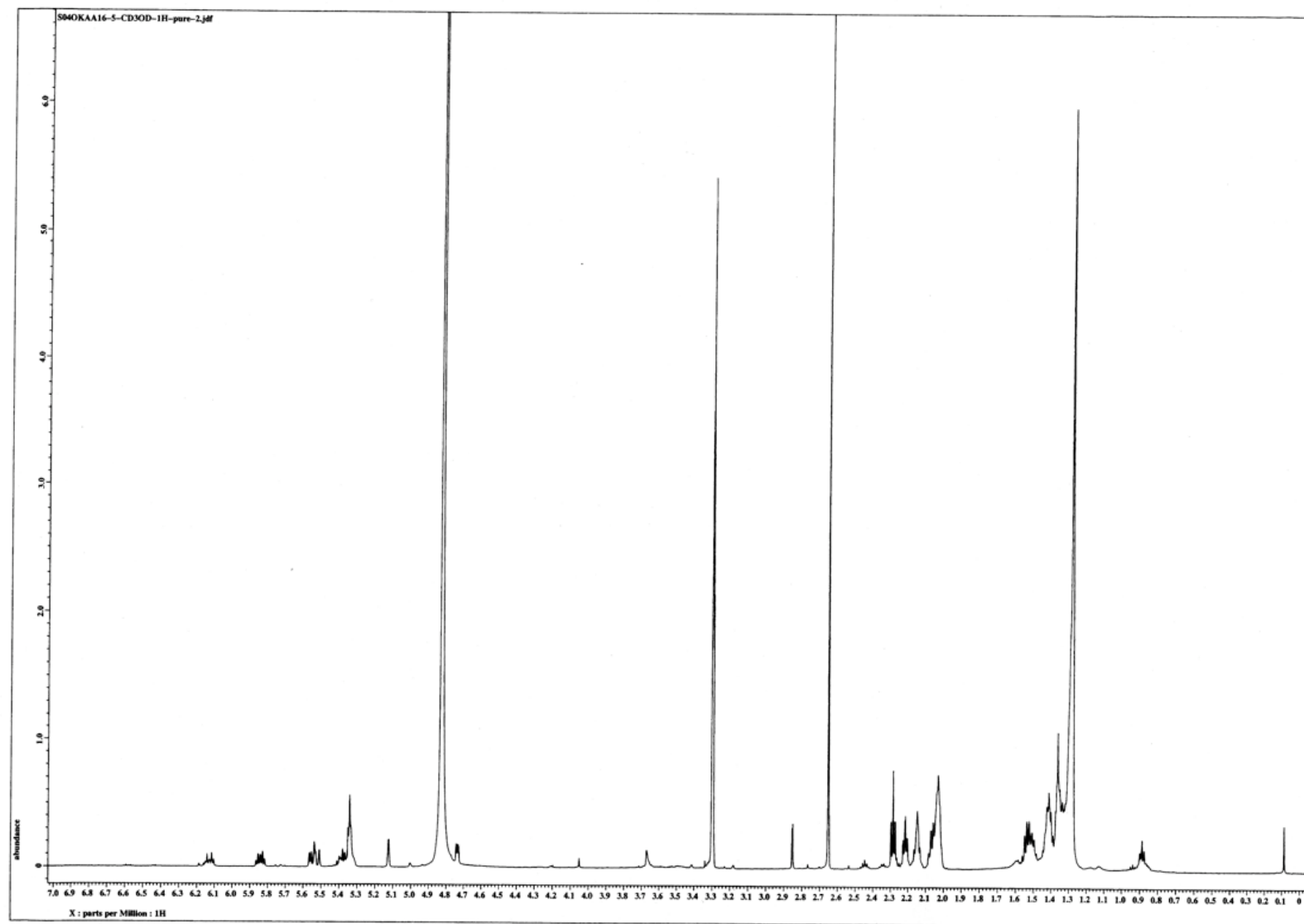


Figure S9. ^1H NMR spectrum of petroformynic acid C (4) in CD_3OD .

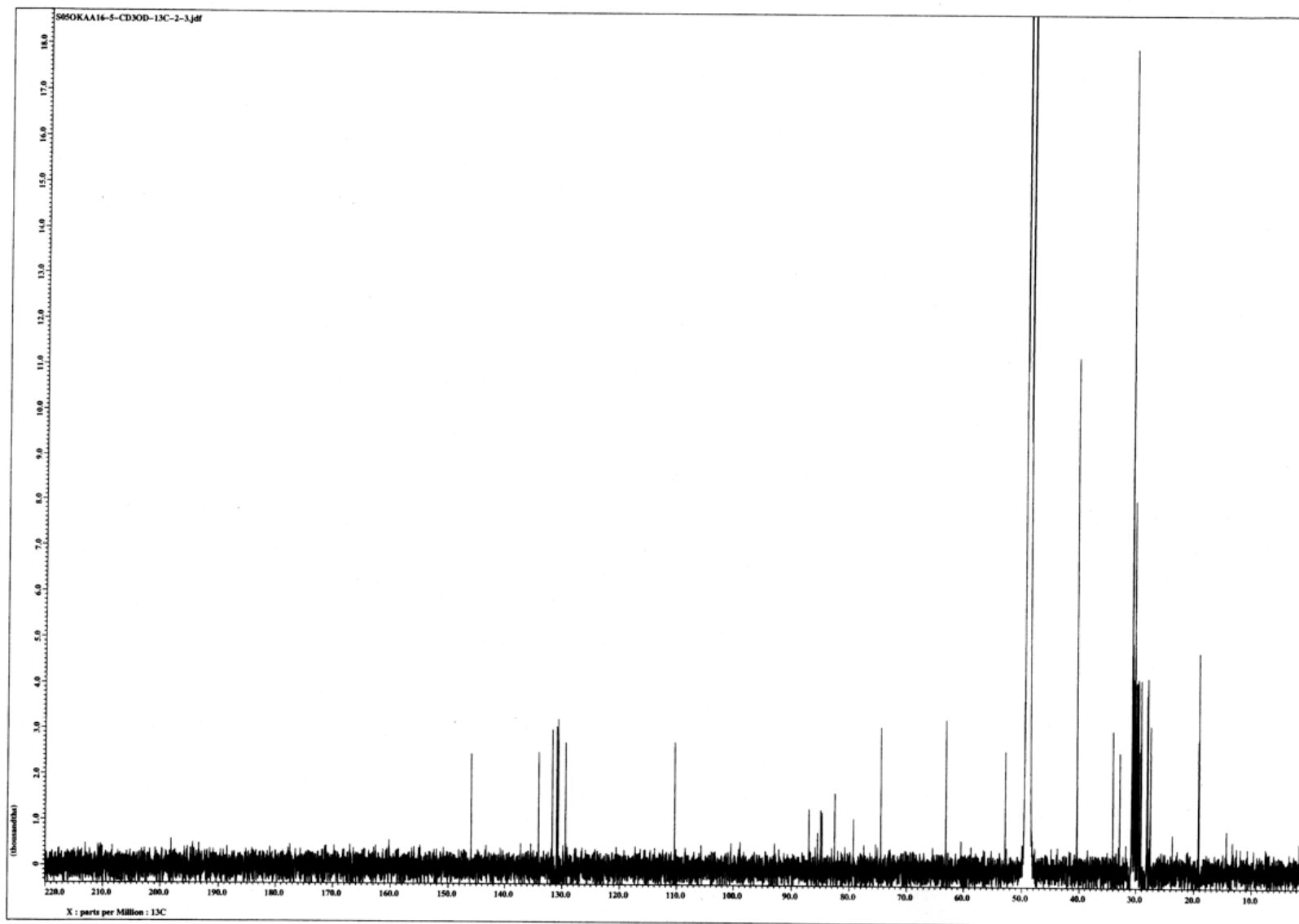


Figure S10. ^{13}C NMR spectrum of petroformynic acid C (4) in CD_3OD

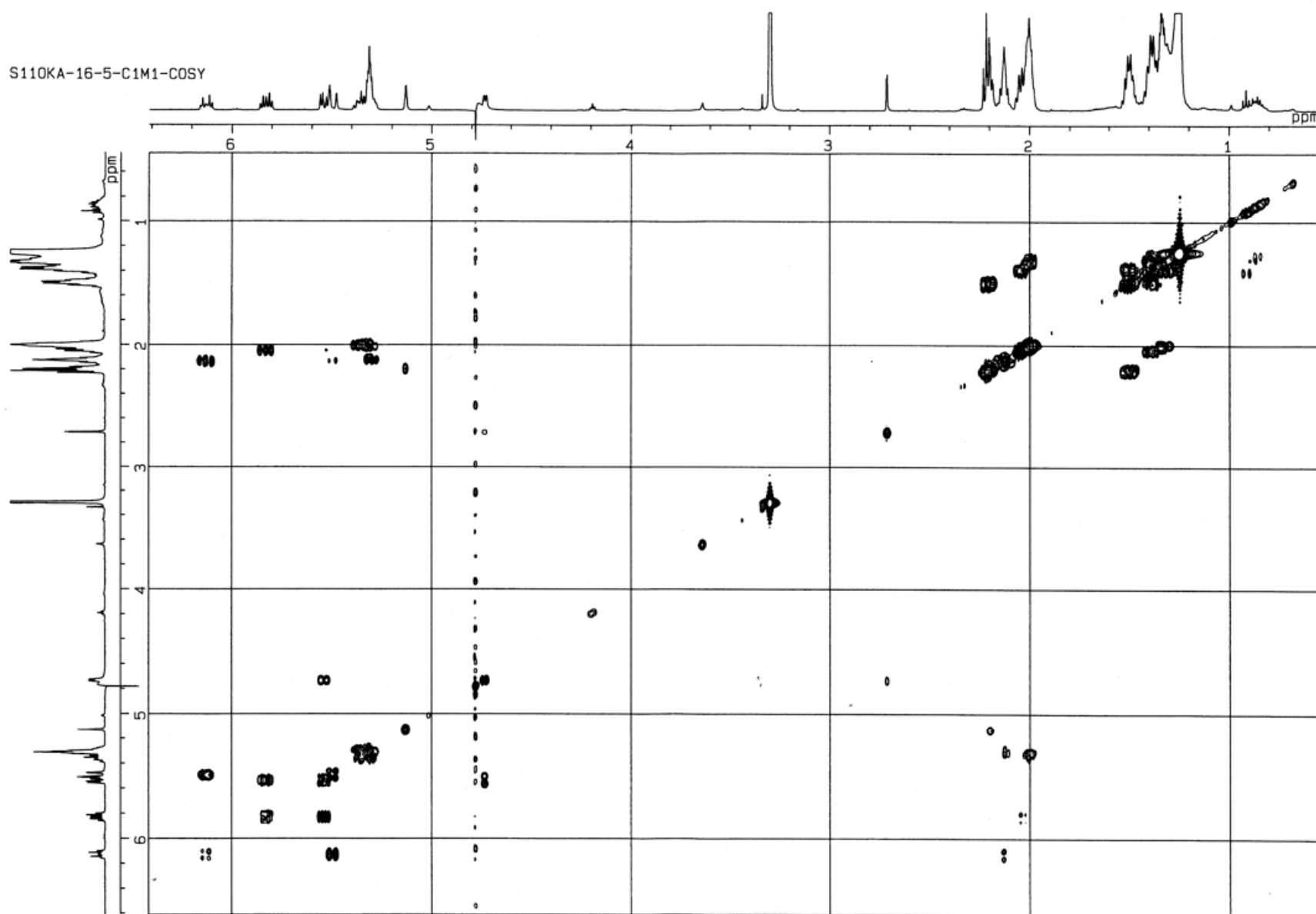


Figure S11. COSY spectrum of petroformynic acid C (**4**) in CD₃OD.

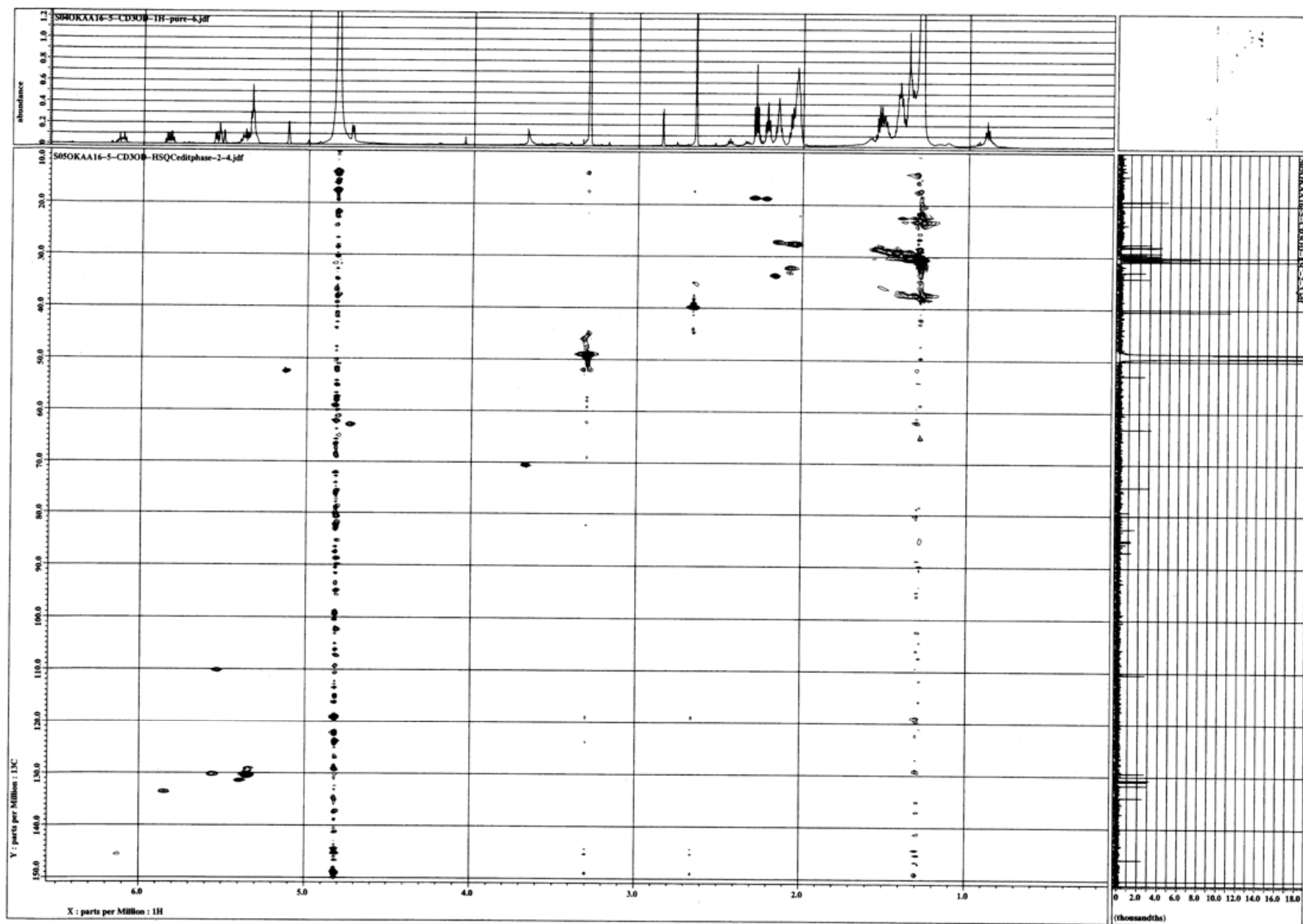


Figure S12. HSQC spectrum of petroformynic acid C (**4**) in CD₃OD.

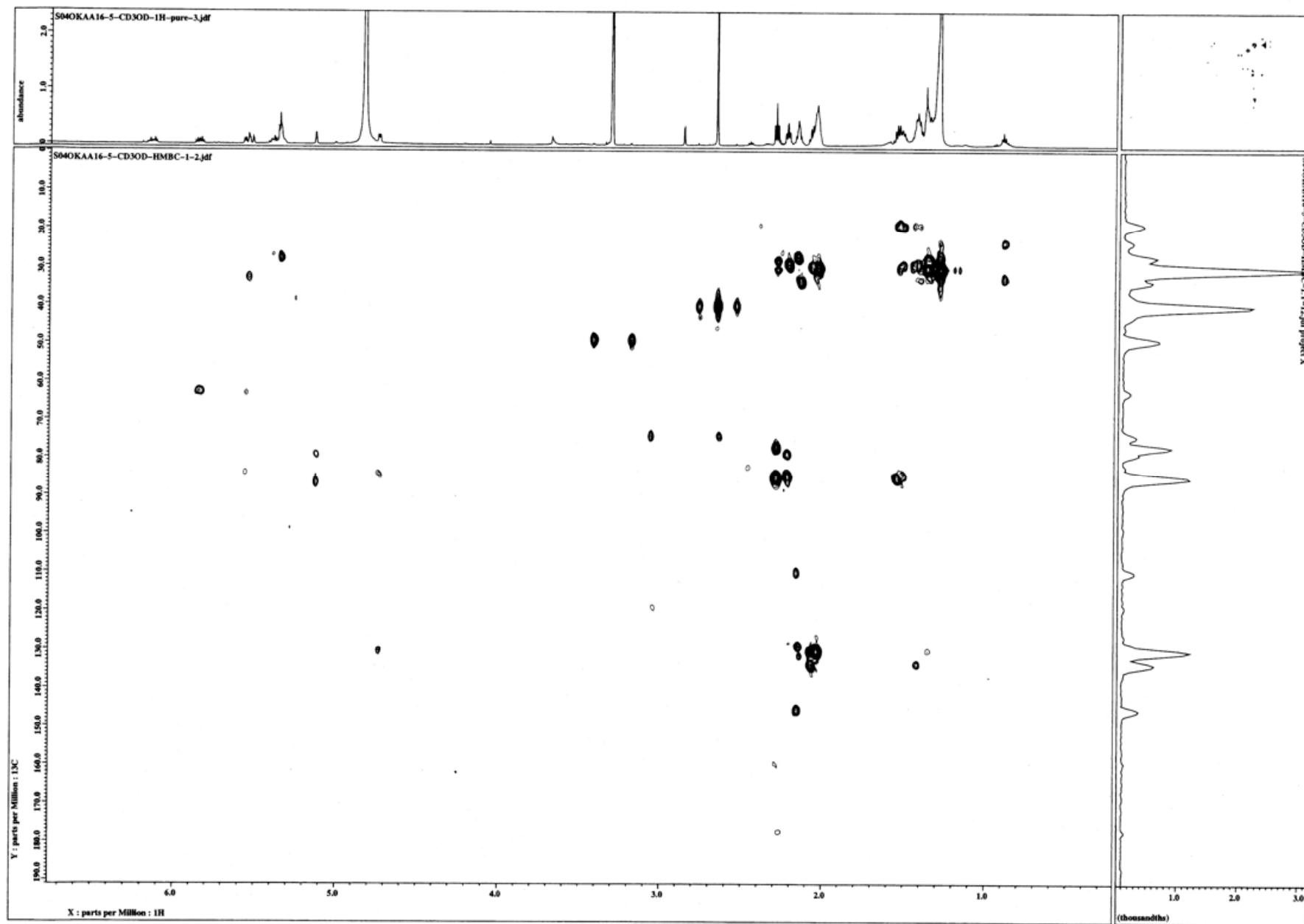


Figure S13. HMBC spectrum of petroformynic acid C (4) in CD₃OD.

[Mass Spectrum]
 Data : 721PB Date : 16-Jul-92 14:45
 Sample:
 Note : Array Detector Data
 Inlet : Direct Ion Mode : FAP+
 Spectrum Type : Product (m/z 714.57, 2nd FFR, FC: 0.00kV)
 RT : 0.00 min Scan# : 1
 BP : m/z 306.0951 Int. : 30.17
 Output m/z range : 39.2140 to 714.5740 Cut Level : 0.00 %

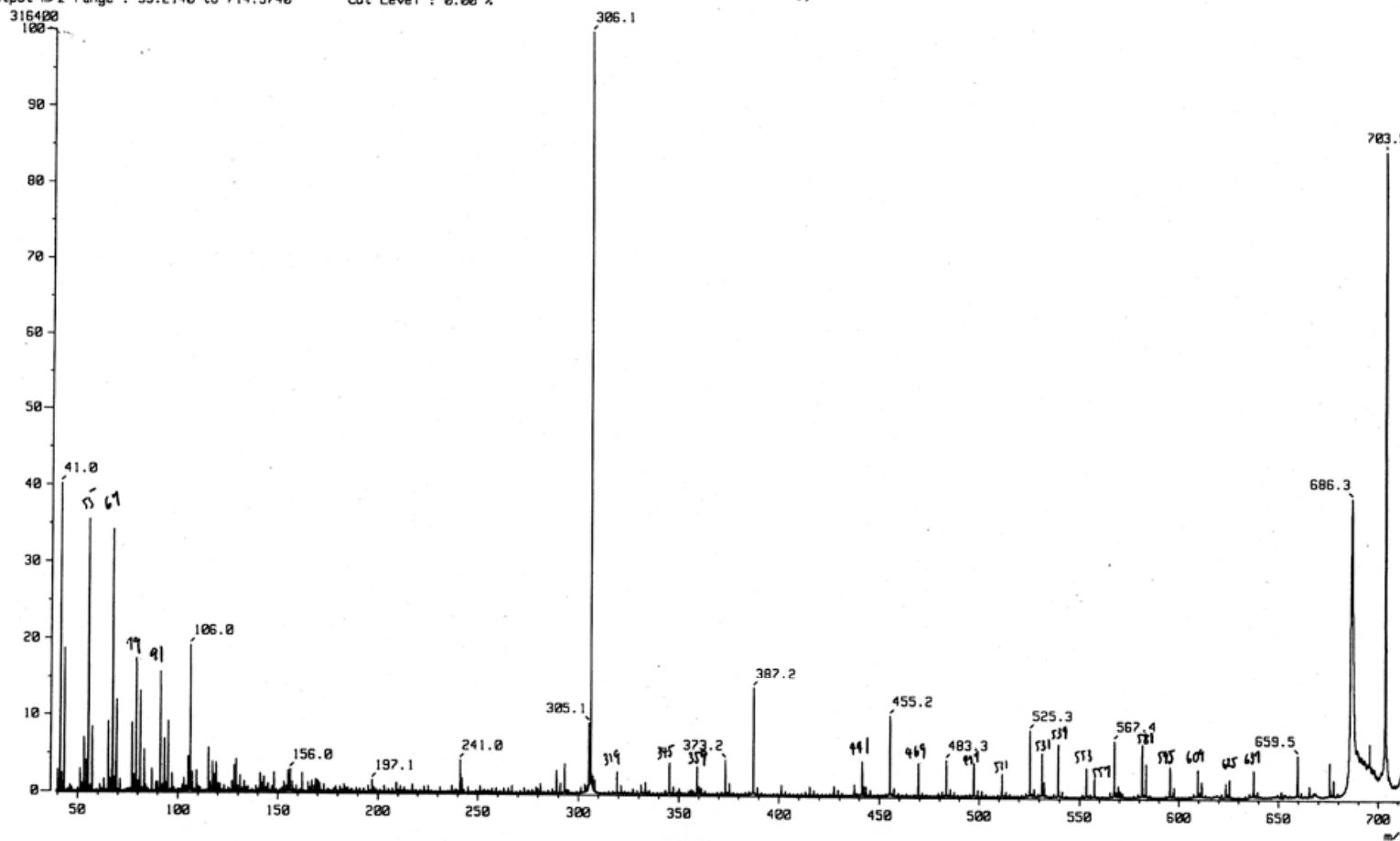


Figure S14. Tandem FAB mass spectrum of petroformynic acid C (4) [Precursor, (M+Na)⁺].

[Mass Spectrum]
 Date : 16-Jul-92 14:45
 Sample :
 Note : Array Detector Data
 Inlet : Direct Ion Mode : FAB+
 Spectrum Type : Product (m/z 735.59, 2nd FFR, FC: 0.00kV)
 RT : 0.00 min Scan# : 1
 BP : m/z 70.9400 Int. : 24.81
 Output m/z range : 40.3674 to 735.5874 Cut Level : 0.00 %

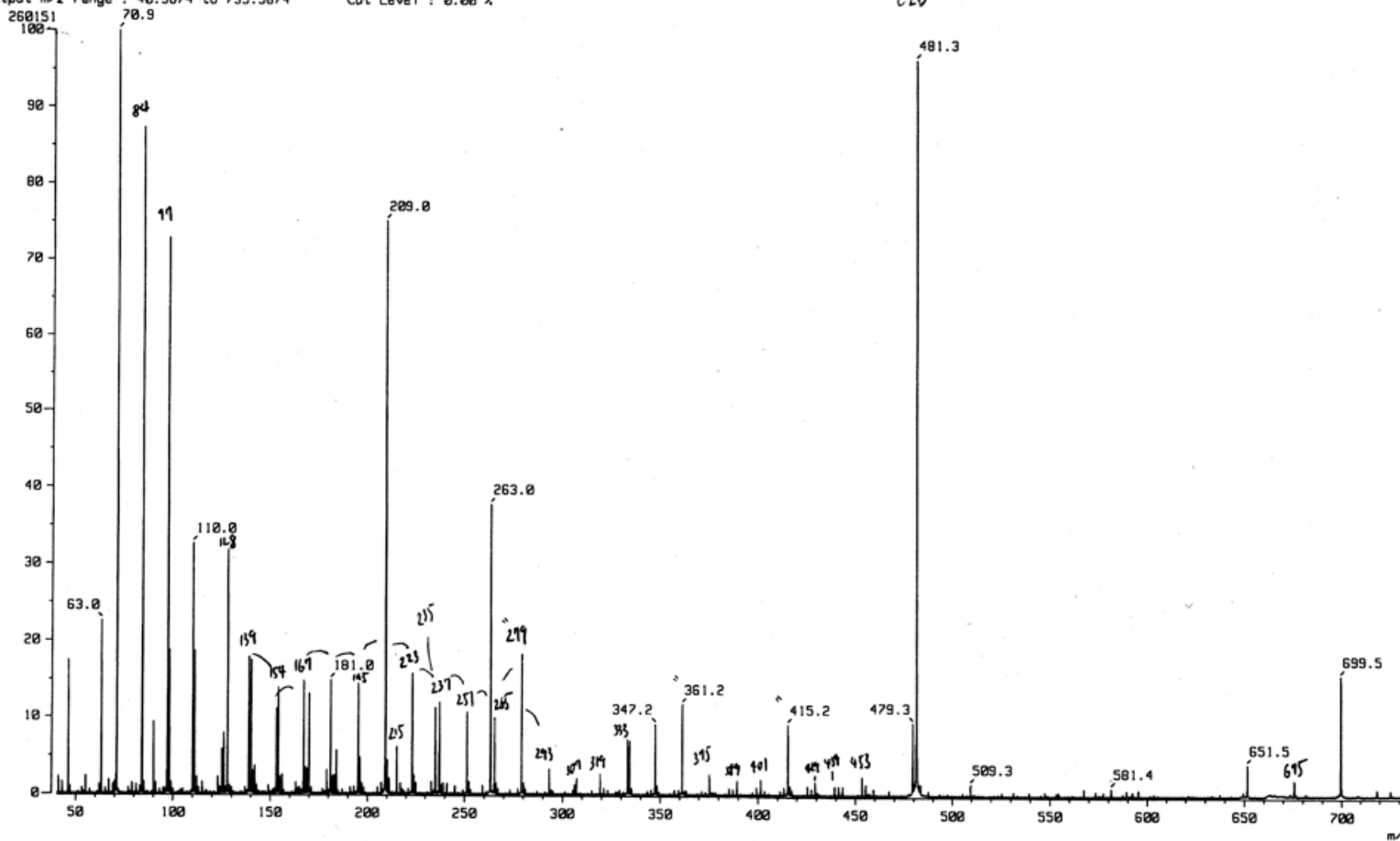


Figure S15. Tandem FAB mass spectrum of petroformynic acid C (4) [Precursor, (M+2Na-H)⁺].

[Mass Spectrum]
Data : 165NA Date : 16-Jul-92 14:45
Sample:
Note : Array Detector Data
Inlet : Direct Ion Mode : FAB+
Spectrum Type : Product (m/z 691.37, 2nd FFR, FC: 0.00kV)
RT : 0.00 min Scan# : 1
BP : m/z 191.0242 Int. : 22.57
Output m/z range : 41.1711 to 691.3711 Cut Level : 0.00 %

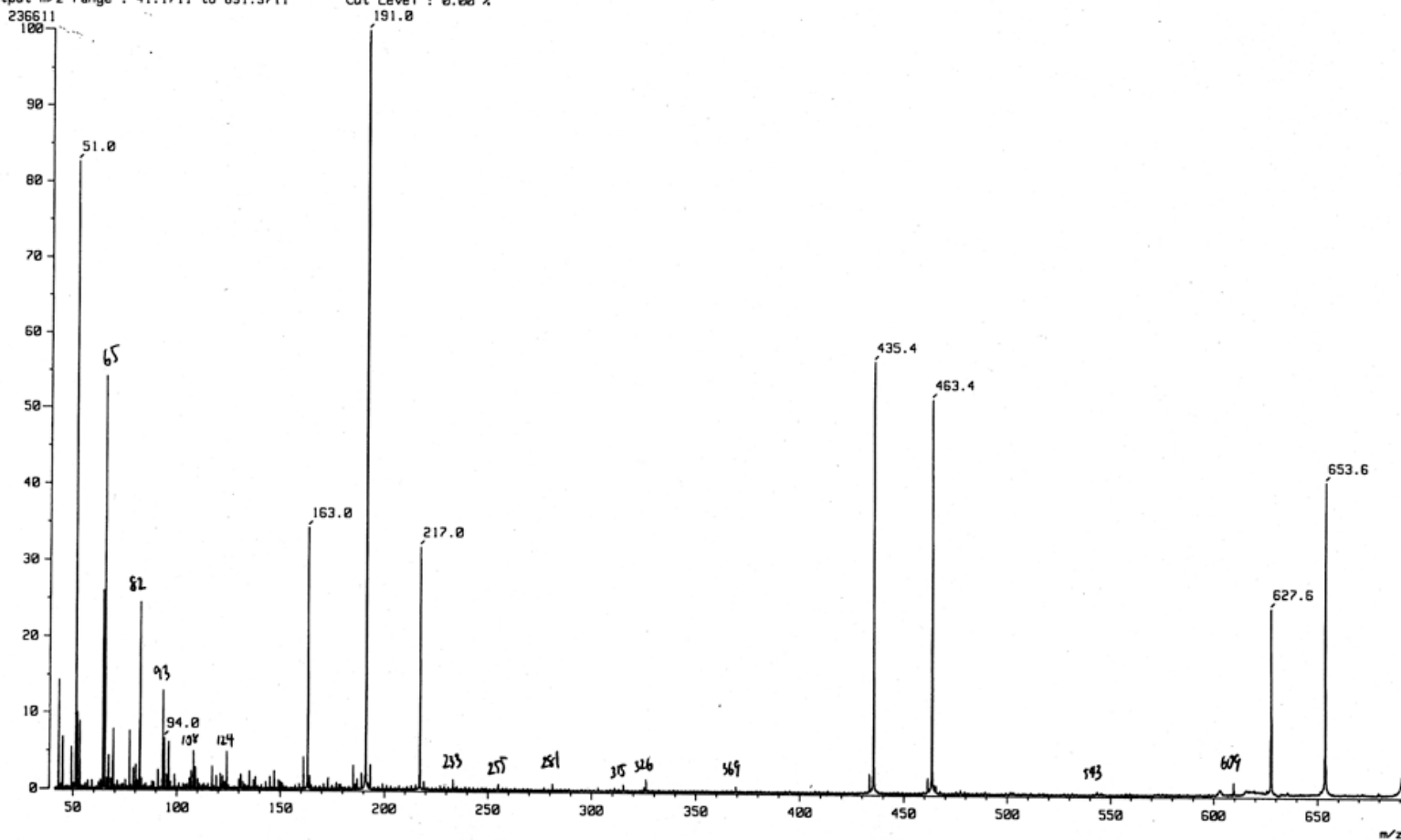


Figure S16. Tandem FAB mass spectrum of petroformynic acid C (4) [Precursor, (M-H)].