Four new taraxastane-type triterpenoic acids from *Cirsium setosum*

Electronic Supplementary Information

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Single Mass Analysis (displaying only valid results)

Tolerance = 10.0 PPM / DBE: min = -2.0, max = 40.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

32 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-500 H: 0-1000 O: 0-4

EI

XJ-59-HREI 19 (1.014) Cn (Cen,5, 30.00, Ht); Sm (Mn, 2x3.00); Cm (18:19)

Autospec

14-May-2014

Voltage EI+

997

Minimum:

-2.0

Maximum:

5.0

10.0

40.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

Formula

472.3516

472.3553

-3.7

-7.8

7.0

0.7

C30 H48 O4

The HRESIMS Spectrum of Compound 1

日期: 星期四 2月 27 16:46:44 2014 (GMT+08:00) Sample Name: XJ - 59

(显微镜透射法 FT- IR Microscope Transmission)

扫描次数: 100

傅里叶变换显微镜红外(FT-IR Microscope): Centaurus

分辨率: 8.000

美国热电公司(Thermo)傅里叶变换红外光谱仪:Nicolet 5700

The IR Spectrum of Compound 1

The ^{13}C NMR Spectrum of Compound 1

The DEPT-135 °Spectrum of Compound 1

The DEPT-90 °Spectrum of Compound 1

The ^1H - ^1H COSY Spectrum of Compound 1

The HSQC Spectrum of Compound 1

The HMBC Spectrum of ompound 1

Current Data Parameters
NAME 20131031-XJ-59
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131102
Time 13.44
INSTRUM spect
PROBHD 5 mm F400 MQ/
PULPROG roesyphpp_2
TD 2648
SOLVENT Pyr
NS 32
DS 32
SWH 5000.000 Hz
FIDRES 2.441406 Hz
AQ 0.2048500 sec
RG 203
SW 100.000 usec
DE 6.50 usec
TE 298.0 K
D0 0.00008637 sec
D1 0.97378558 sec
D11 0.03000000 sec
D12 0.00002000 sec
IM0 0.00019995 sec
L4 1000
P15 200000.00 usec

===== CHANNEL f1 =====
NUC1 1H
P1 15.10 usec
P17 2500.00 usec
P25 100.00 usec
PLW1 12.38799953 W
PLW10 4.17840004 W
PLW27 1.12979996 W
SFO1 500.0625003 MHz

F1 - Acquisition parameters
TD 128
SFO1 500.0625 MHz
FIDRES 39.067421 Hz
SW 10.000 ppm
F2MODE States-TFPI

F2 - Processing parameters
SI 1024
SF 500.0599953 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 States-TFPI
SF 500.0599954 MHz
WDW
SSB 2
LB 0 Hz
GB 0

BRUKER AV-III-500 ROESY-NMR XJ-59 IN PYR 2013.10.31
ROESYPHSW Pyr E:\\ shangxiaoya 39

The ROESY Spectrum of Compound 1

Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 10.0 PPM / DBE: min = -2.0, max = 40.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

30 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-500 H: 0-1000 O: 0-4

EI

XJ-64-HREI 20 (1.067) Cn (Cen,5, 30.00, Ht); Sm (Mn, 2x3.00); Cm (20)

Autospec

14-May-2014

Voltage EI+

2.95e3

Minimum:

-2.0

Maximum:

5.0

10.0

40.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
470.3359	470.3396	-3.7	-7.9	8.0	1.6	C30 H46 O4

The HRESIMS Spectrum of Compound 2

日期: 星期四 2月 27 15:16:39 2014 (GMT+08:00) Sample Name: XJ - 64

(显微镜透射法 FT- IR Microscope Transmission)

扫描次数: 100

傅里叶变换显微镜红外(FT-IR Microscope): Centaur μ s

分辨率: 8.000

美国热电公司(Thermo)傅里叶变换红外光谱仪: Nicolet 5700

The IR Spectrum of Compound 2

S15

The ^1H NMR Spectrum of Compound 2

The ^{13}C NMR Spectrum of Compound 2

The DEPT-135 °Spectrum of Compound 2

The DEPT-90 °Spectrum of Compound 2

The HSQC Spectrum of Compound 2

The HMBC Spectrum of ompound 2

BRUKER AV-III-500 ROESY-NMR XJ-64 IN PYR 2013.10.31
ROESYPHSW Pyr E:\\ shangxiaoya 39

```
Current Data Parameters
NAME      20131031-XJ-64
EXPNO     7
PROCNO    1

F2 - Acquisition Parameters
Date_     20131102
Time      19.47
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   roesyphpp.2
TD        2048
SOLVENT   Pyr
NS         4
DS        32
SWH        4166.667 Hz
FIDRES     2.034505 Hz
AQ         0.2458100 sec
RG         18
CW         120.000 usec
DE         6.50 usec
TE         298.1 K
DD         0.00010639 sec
D1         1.95494401 sec
D11        0.03000000 sec
D12        0.00002000 sec
IM0        0.00024000 sec
L4         1000
P15        200000.00 usec

===== CHANNEL f1 =====
NUC1       1H
P1         15.10 usec
P17        2500.00 usec
P25        100.00 usec
PLW1       12.38799953 W
PLW10      4.17840004 W
PLW27      1.12979996 W
SFO1       500.0620032 MHz

F1 - Acquisition parameters
TD         256
SFO1       500.062 MHz
FIDRES     16.276041 Hz
SW         0.332 ppm
F2MODE     States-TPPI
F2 - Processing parameters
SI         1024
SF         500.0600143 MHz
WDW        QSINE
SSB        2
LB         0 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         500.0600164 MHz
WDW        SSB
SSB        2
LB         0 Hz
GB         0
```

The ROESY Spectrum of Compound 2

The HRESIMS Spectrum of Compound 3

Elemental Composition Report

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

4 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:	80.00			-1.5	
Maximum:	100.00			50.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE
470.3403	100.00	470.3396	0.7	1.5	8.0
					Score
					1
					Formula
					C30 H46 O4

日期: 星期四 2月 27 17:17:09 2014 (GMT+08:00) Sample Name: XJ - 67

(显微镜透射法 FT- IR Microscope Transmission)

扫描次数: 100

傅里叶变换显微镜红外(FT-IR Microscope): Centaurus

分辨率: 8.000

美国热电公司(Thermo)傅里叶变换红外光谱仪:Nicolet 5700

The IR Spectrum of Compound 3

S25

The ^1H NMR Spectrum of Compound 3

The ^{13}C NMR Spectrum of Compound 3

The DEPT-135 °Spectrum of Compound 3

The DEPT-90 °Spectrum of Compound 3

The ^1H - ^1H COSY Spectrum of Compound 3

The HSQC Spectrum of Compound 3

The HMBC Spectrum of ompound 3

BRUKER AV-III-500 ROESY-NMR XJ-67 IN Pyr 2014.04.09
ROESYPHSW Pyr E:\\ shangxiaoya 46

Current Data Parameters
NAME 20140409-XJ-67
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140410
Time 0.49
INSTRUM spect
PROBHD 5 mm F400 HG/
PULPROG roesyphpp.2
TD 2648
SOLVENT Pyr
NS 32
DS 32
SWH 5319.149 Hz
FIDRES 2.597241 Hz
AQ 0.1925620 sec
RG 28.5
DW 94.000 usec
DE 8.00 usec
TE 298.1 K
D0 0.00008039 sec
D1 1.01228797 sec
D11 0.03000000 sec
D12 0.00002000 sec
IN0 0.00018000 sec
L4 1000
P15 200000.00 usec

----- CHANNEL f1 -----
NUC1 1H
P1 15.10 usec
P17 2500.00 usec
P25 100.00 usec
PLW1 12.38799953 W
PLW10 4.17840004 W
PLW27 1.12979996 W
SFO1 500.0627964 MHz

F1 - Acquisition parameters
TD 256
SFO1 500.0628 MHz
FIDRES 20.777925 Hz
SW 10.637 ppm
FMODE States-TFPI

F2 - Processing parameters
SI 1024
SF 500.0599951 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 States-TFPI
SF 500.0599951 MHz
WDW
SSB 2
LB 0 Hz
GB 0

The ROESY Spectrum of Compound 3

Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 10.0 PPM / DBE: min = -2.0, max = 40.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

32 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-500 H: 0-1000 O: 0-4

EI

XJ-61-HREI 26 (1.388) Cn (Cen,5, 30.00, Ht); Sm (Mn, 2x3.00); Cm (25:27)

Autospec

14-May-2014

Voltage EI+

471

Minimum:

-2.0

Maximum:

5.0

10.0

40.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
472.3542	472.3553	-1.1	-2.3	7.0	66.1	C30 H48 O4

The HRESIMS Spectrum of Compound 4

日期: 星期四 2月 27 16:29:06 2014 (GMT+08:00) Sample Name: XJ - 61

(显微镜透射法 FT-IR Microscope Transmission)

扫描次数: 100

傅里叶变换显微镜红外(FT-IR Microscope): Centaurus

分辨率: 8.000

美国热电公司(Thermo)傅里叶变换红外光谱仪:Nicolet 5700

The IR Spectrum of Compound 4

The ^1H NMR Spectrum of Compound 4

The ^{13}C NMR Spectrum of Compound 4

The DEPT-135 °Spectrum of Compound 4

The DEPT-90 °Spectrum of Compound 4

The ^1H - ^1H COSY Spectrum of Compound 4

The HSQC Spectrum of Compound 4

The HMBC Spectrum of ompound 4

BRUKER AV-III-500 ROESY-NMR XJ-61 IN C5D5N 2013.07.07
ROESYPHSW Pyr E:\\ shangxiaoya 51

Current Data Parameters
NAME 20130707-XJ-61
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130707
Time 23.46
INSTRUM spect
PROBHD 5 mm PABBO HD/
PULPROG roesyphpp.2
TD 2648
SOLVENT Pyr
NS 32
DS 32
SWH 4807.692 Hz
FIDRES 2.347506 Hz
AQ 0.2130420 sec
RG 64
CW 104.000 usec
DE 6.50 usec
TE 298.1 K
D0 0.00009039 sec
D1 0.99180782 sec
D11 0.03000000 sec
D12 0.00002000 sec
IM0 0.00020000 sec
L4 1000
F15 200000.00 usec

===== CHANNEL f1 =====
NUC1 1H
P1 15.10 usec
P17 2500.00 usec
P25 100.00 usec
PLW1 12.38799953 W
PLW10 4.17840004 W
PLW27 1.12979996 W
SFO1 500.0623472 MHz

F1 - Acquisition parameters
TD 256
SFO1 500.0623 MHz
FIDRES 18.780048 Hz
SW 9.614 ppm
FPMODE States-TPPI

F2 - Processing parameters
SI 1024
SF 500.0600011 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 500.0599985 MHz
WDW
SSB 2
LB 0 Hz
GB 0

The ROESY Spectrum of Compound 4