# 3D-Printing of Capsule Devices as Compartmentalization Tools for Supported Reagents in the Search of Antiproliferative Isatins

## Supplementary Material

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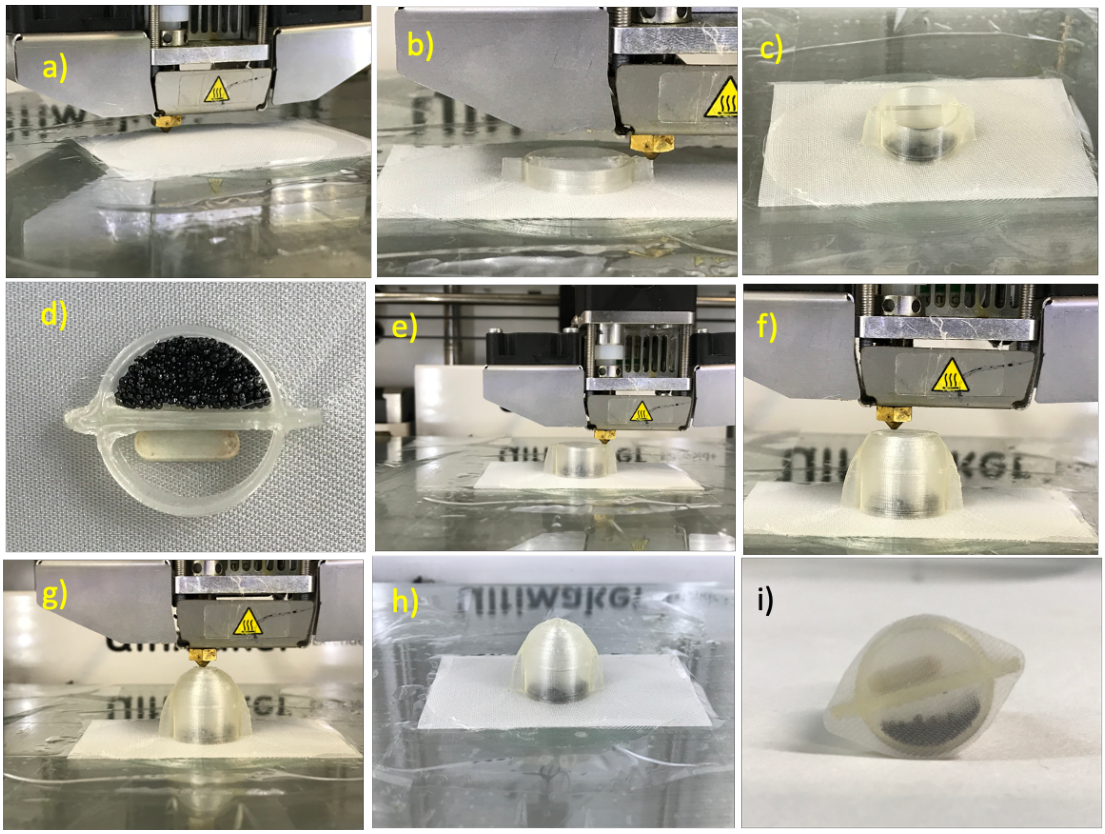
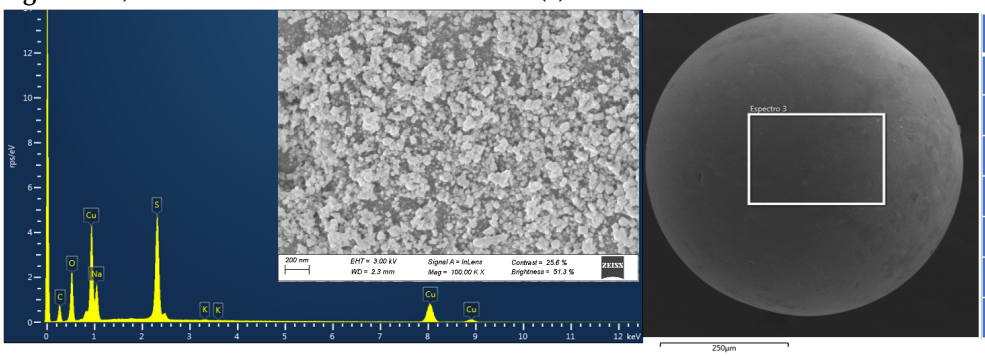


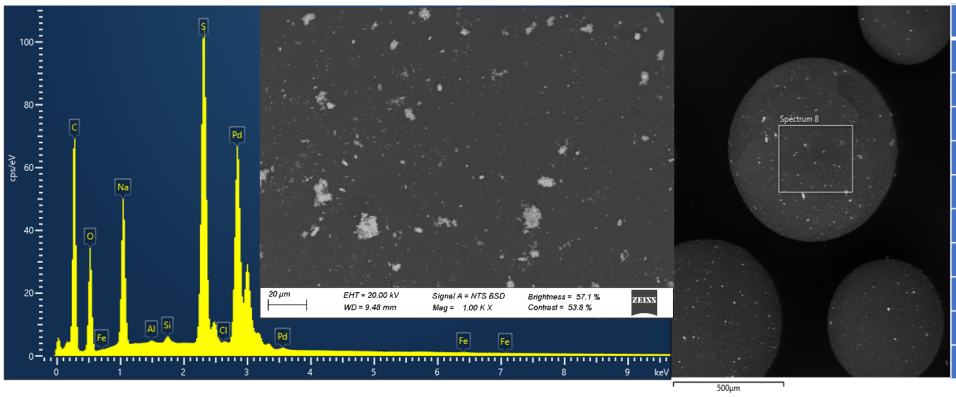

**Figure S1**. a) Porous polypropylene membrane placed at the base of the platform just before the start of the 3D-printing process. b) image at 5 min. c) moment of pause in the 3D-printing and loading of the catalytic material and the magnetic stirrer. d) Parietal view of the capsule at the time of paused loading. e, f) resumption of 3D-printing. g) final moment of 3D-printing (23 min). h) finished capsule. i) View of the porous area after cutting the excess membrane part with scissors.

**Figure S2 a) EDX and FESEM data of IRA-120-Cu(0)**



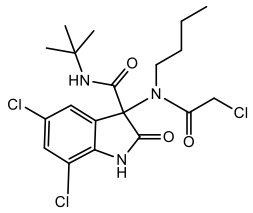

| Element | Weight (%) |
|---|---|
| C | 26.15 |
| O | 28.33 |
| Na | 8.30 |
| S | 15.34 |
| K | 0.14 |
| Cu | 21.74 |
| Total | 100.00 |

**Figure S2 b) EDX and SEM data of IRA-120-Pd(0)**




| Element | Weight % |
|---|---|
| C | 40.38 |
| O | 18.65 |
| Na | 7.12 |
| Al | 0.07 |
| Si | 0.19 |
| S | 11.11 |
| Cl | 0.11 |
| Fe | 0.08 |
| Pd | 22.29 |
| Total | 100.00 |

3. RMN spectra of the related compounds

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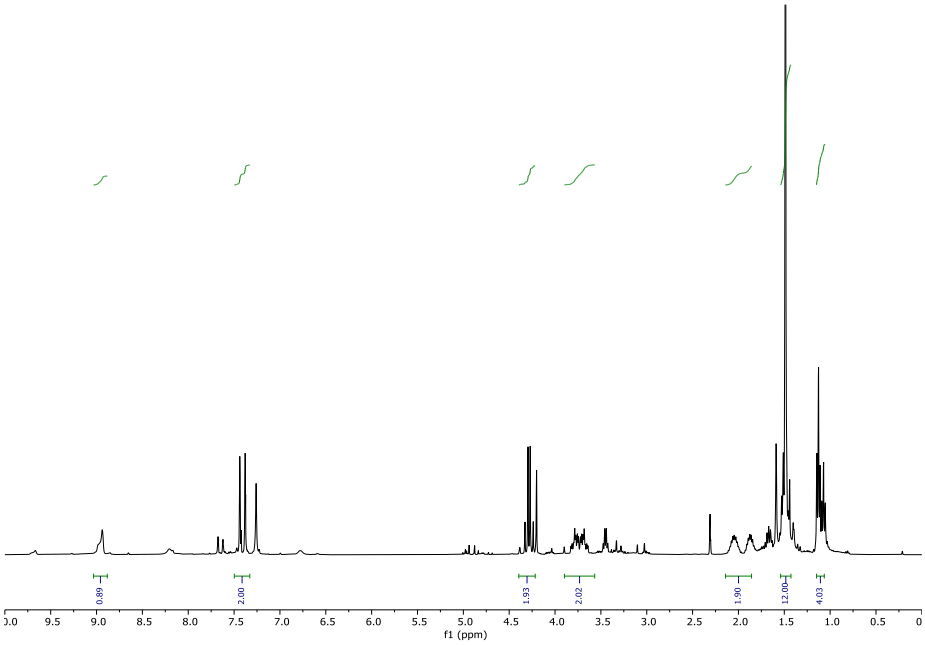


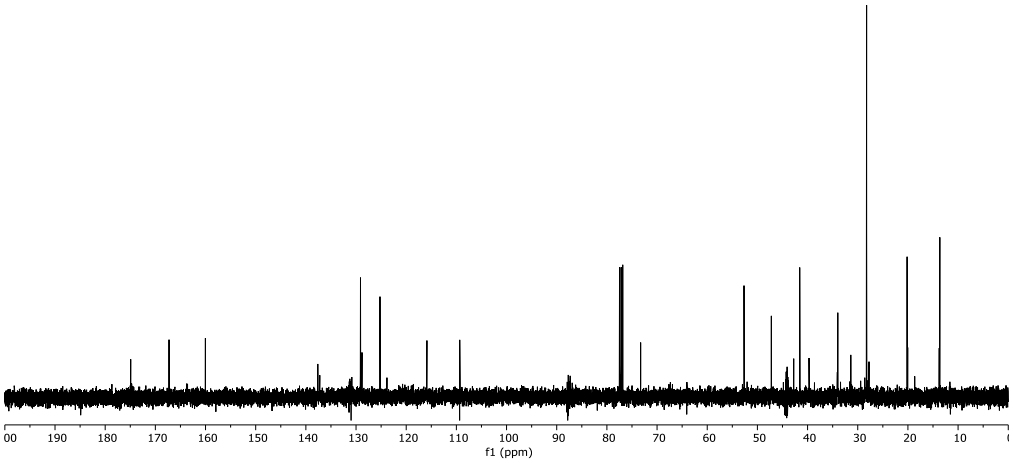

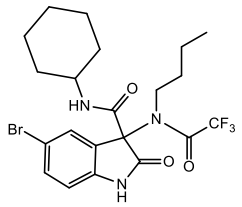


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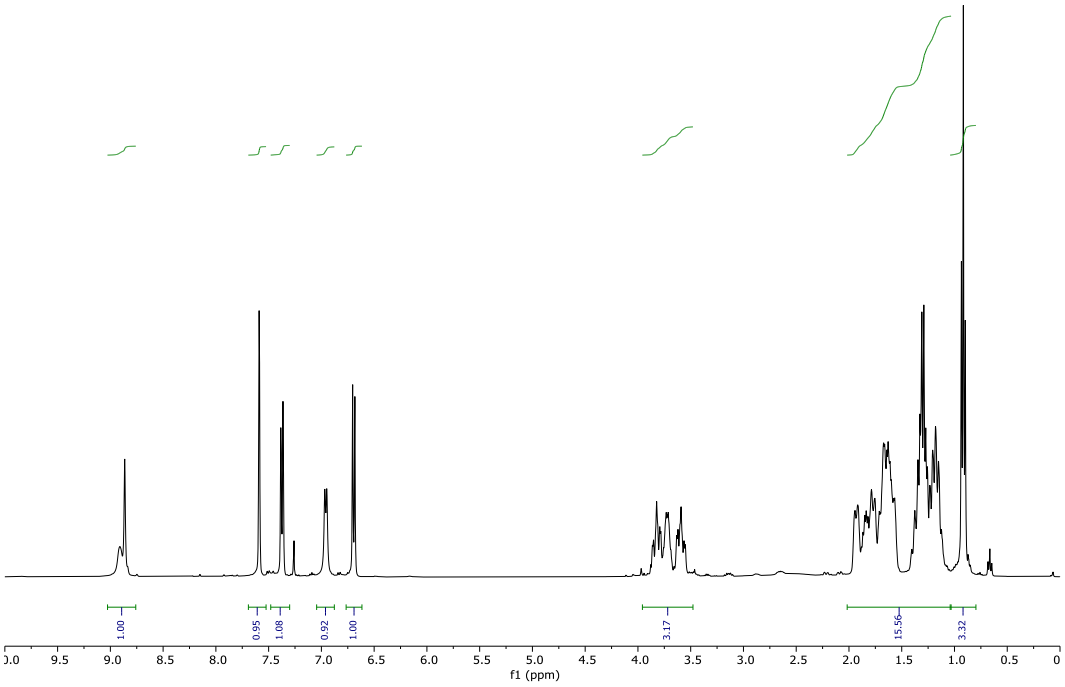


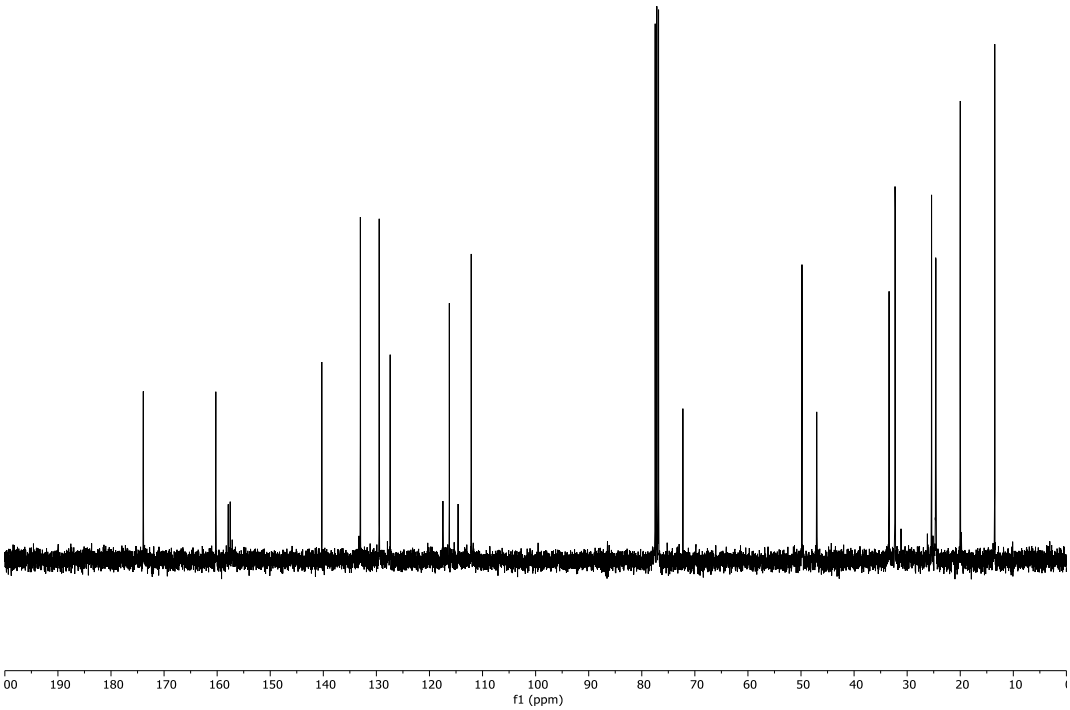

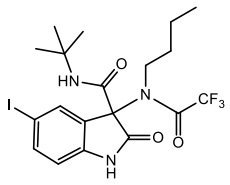
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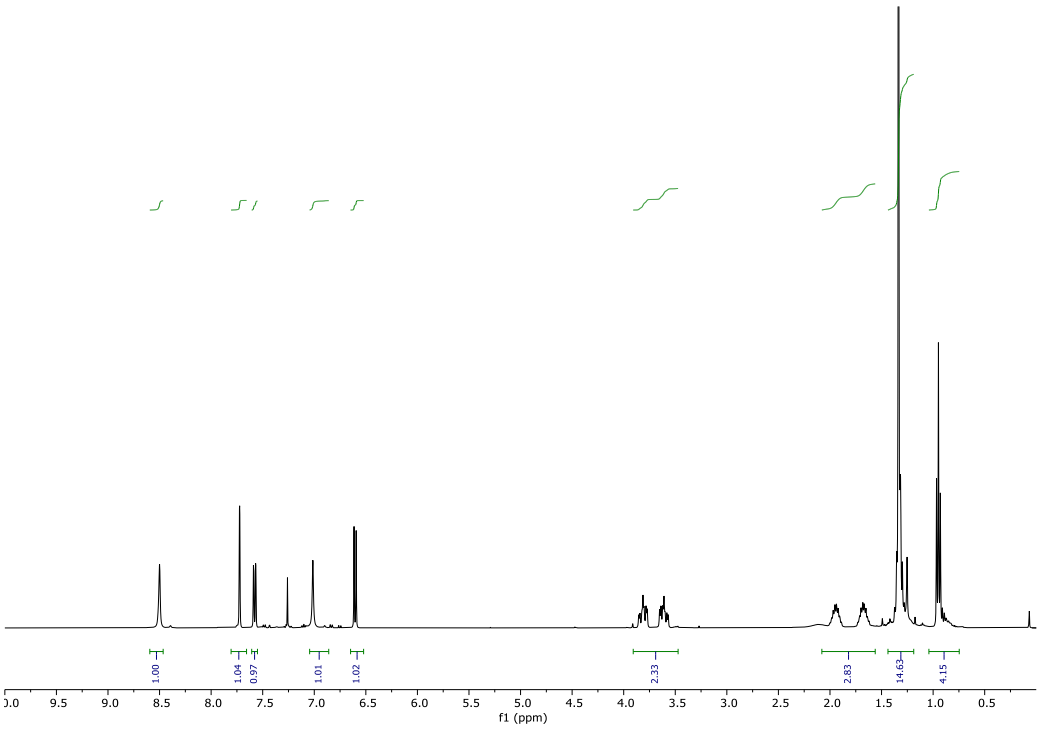
N
CF3
I
O
O
N
H

17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1.01
1.02
2.33
2.83
14.63
4.15
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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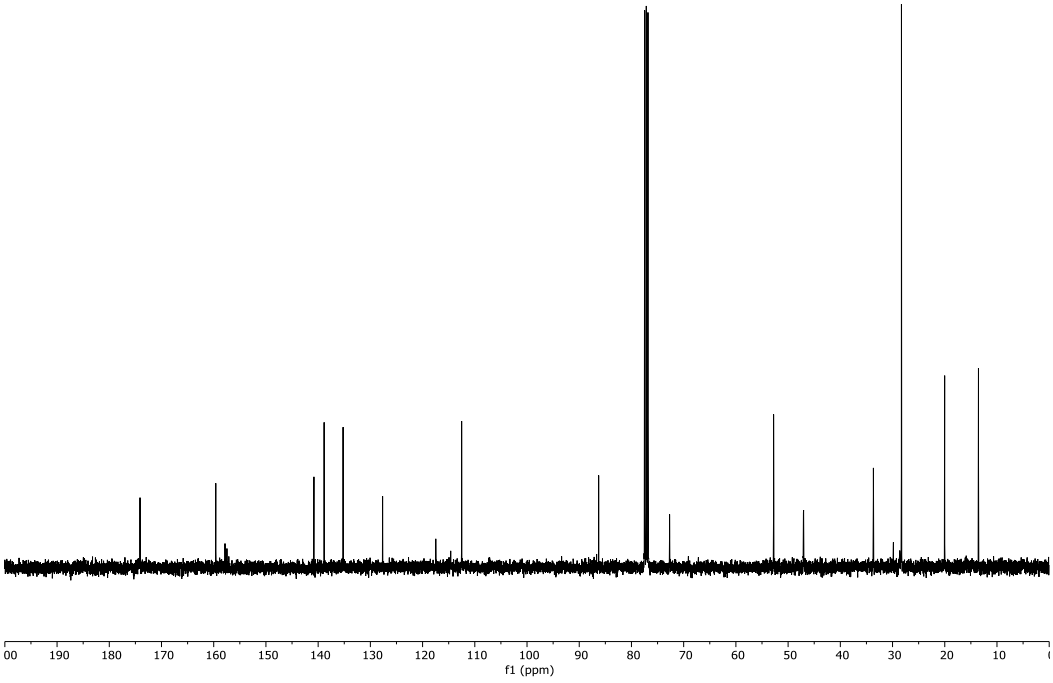
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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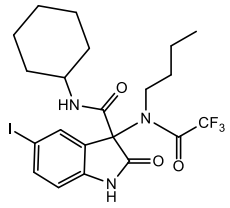
70
60
50
40
30
20
10
f1 (ppm)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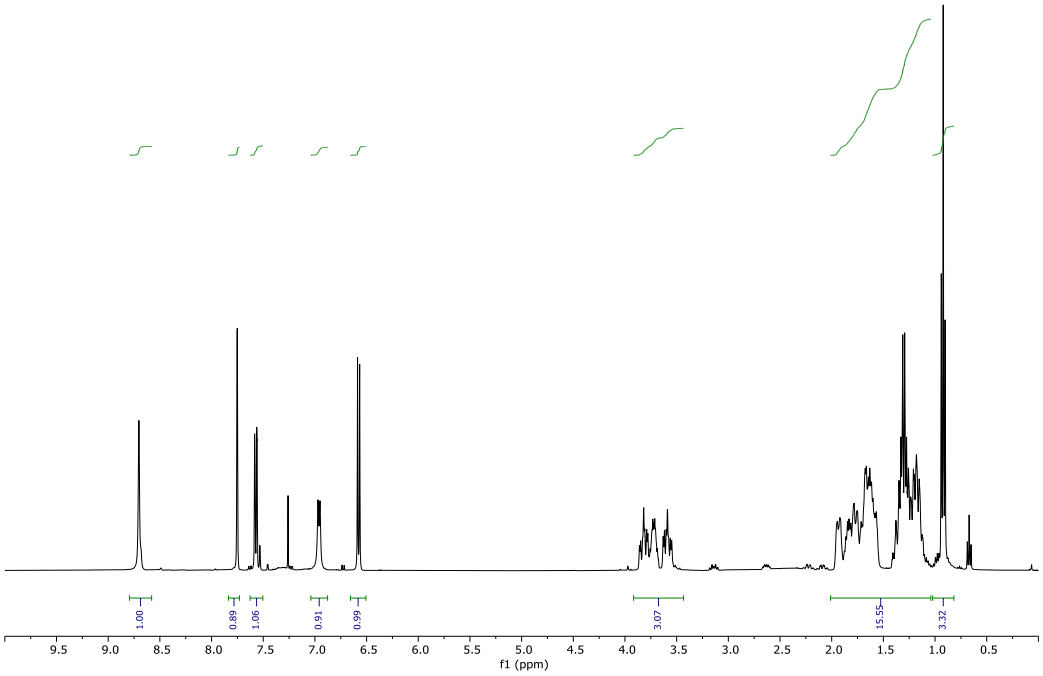
O
N
CF3
O
I
O
N
H

18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1.06
0.91
0.99
3.07
15.55
3.32
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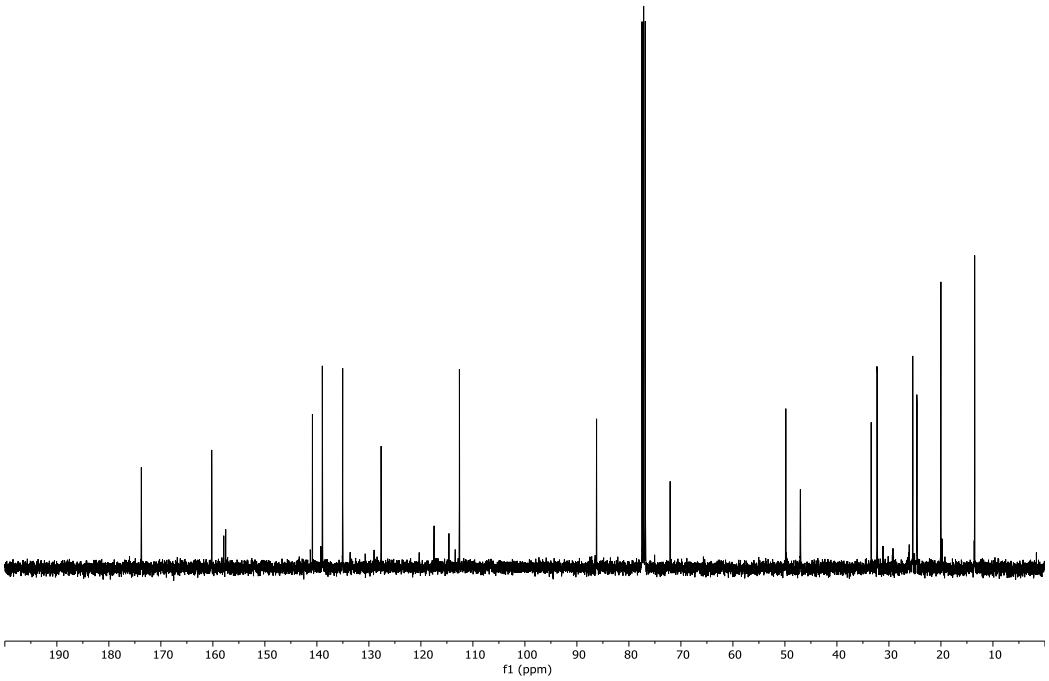
f1 (ppm)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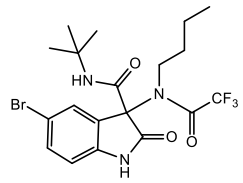
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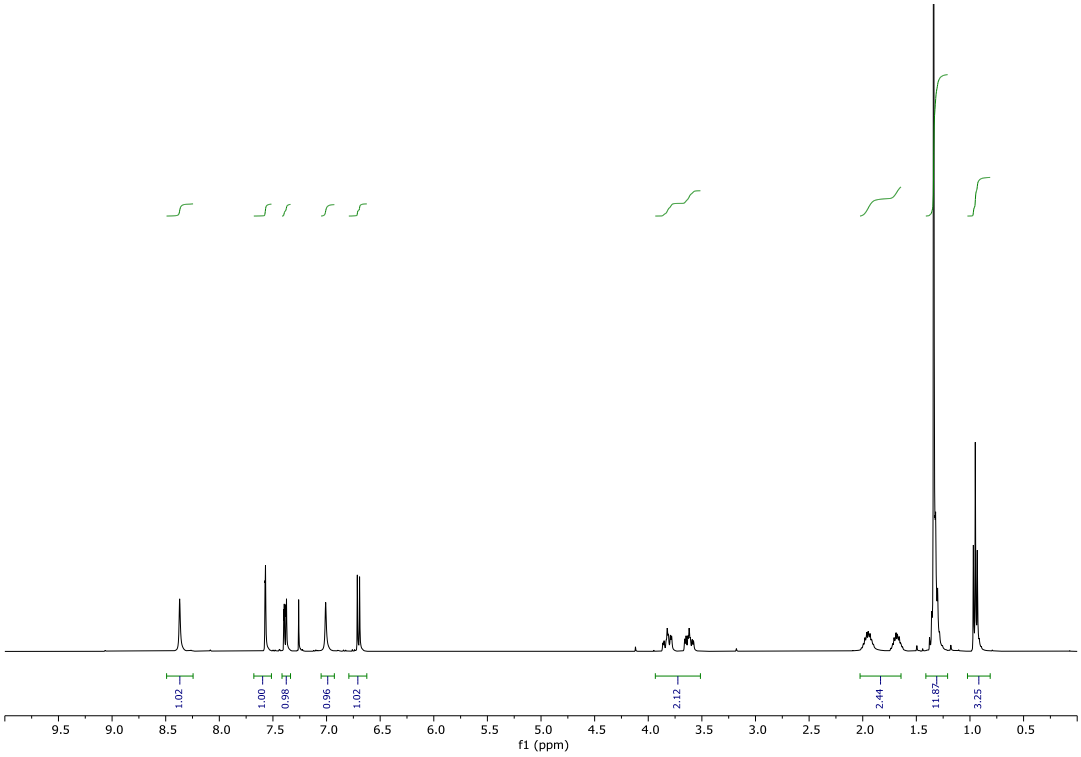
O
N
CF3
Br
O
O
N
H

19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11.87
3.25
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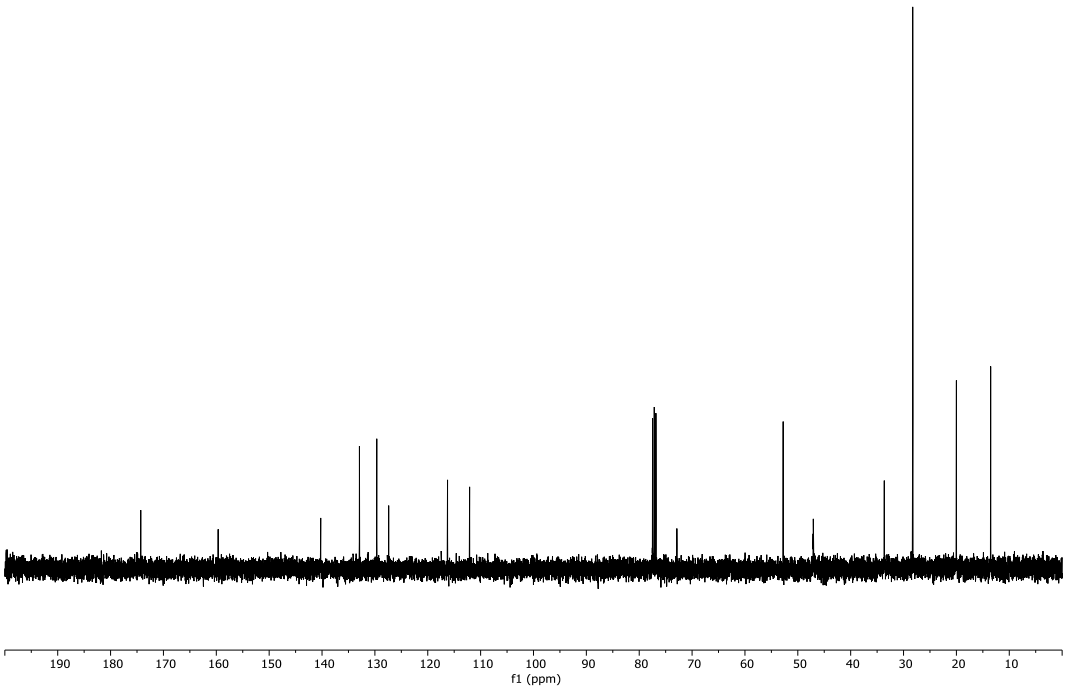
f1 (ppm)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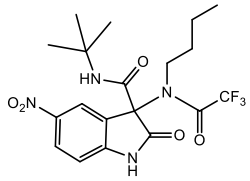
f1 (ppm)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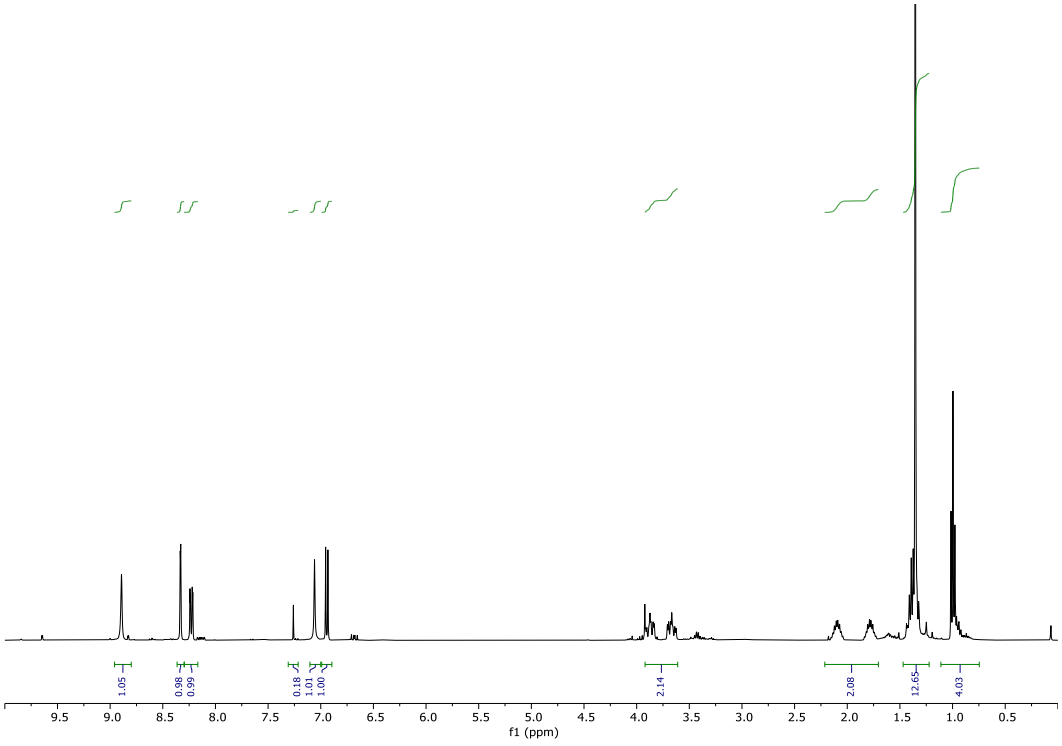
N
CF3
O2N
O
O
N
H

20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4.03
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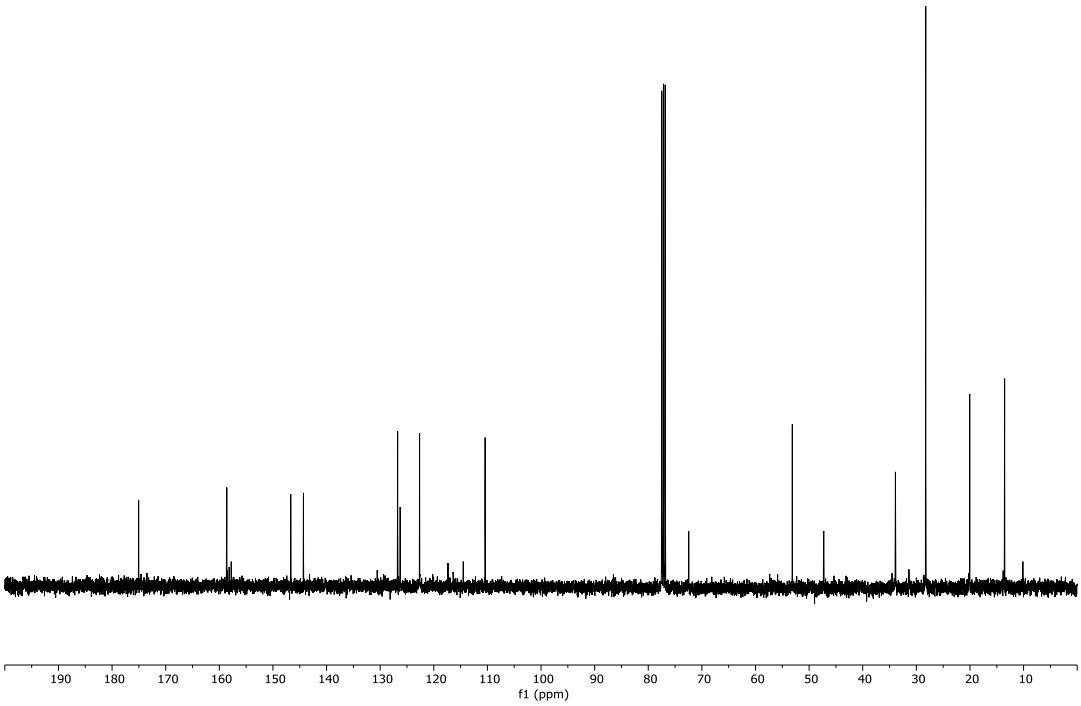
f1 (ppm)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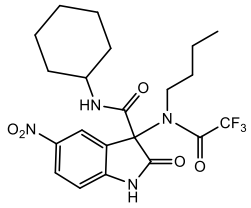
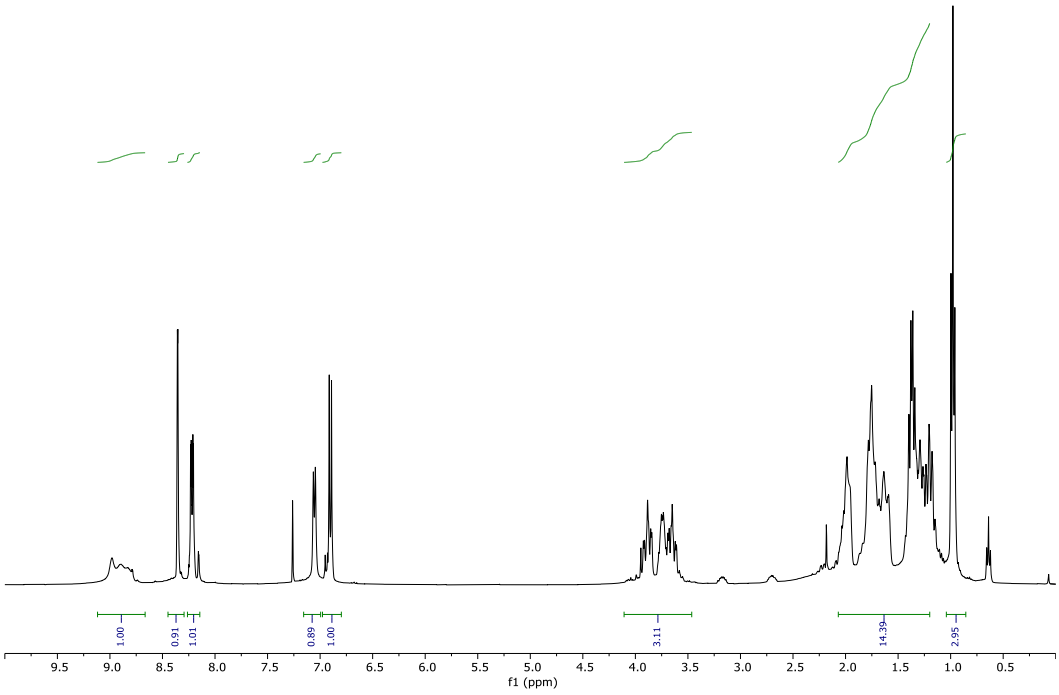
f1 (ppm)


HN
O
N
CF3
O2N
O
O
N
H

21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0.89
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3.11
14.39
2.95
9.5
9.0
8.5
8.0
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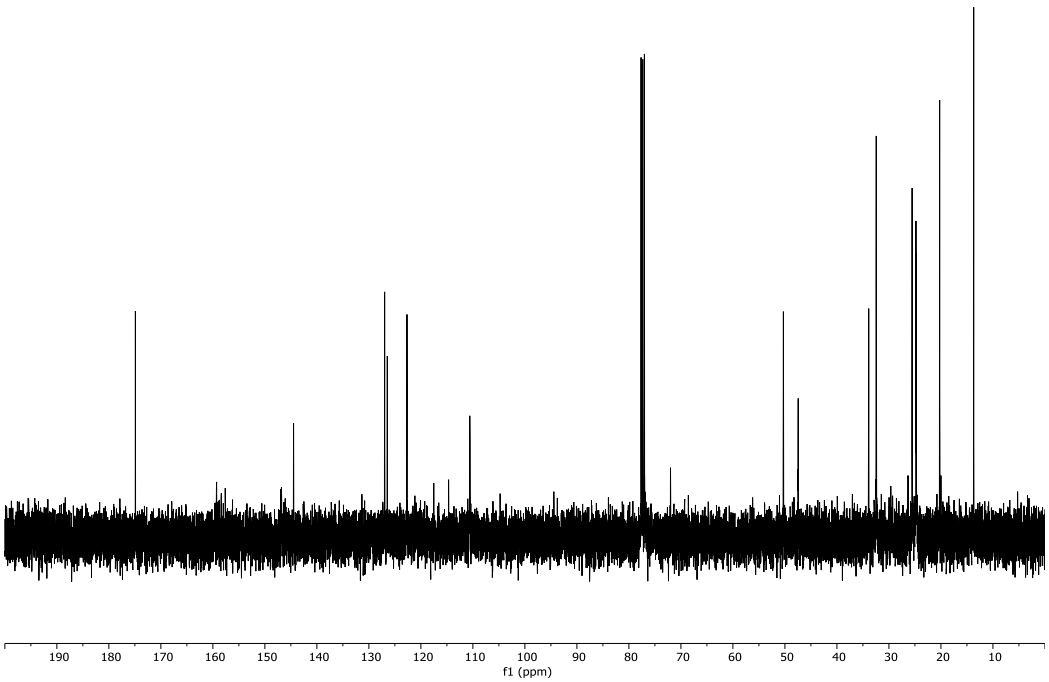
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


190
180
170
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150
140
130
120
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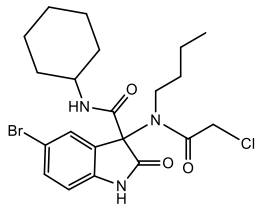
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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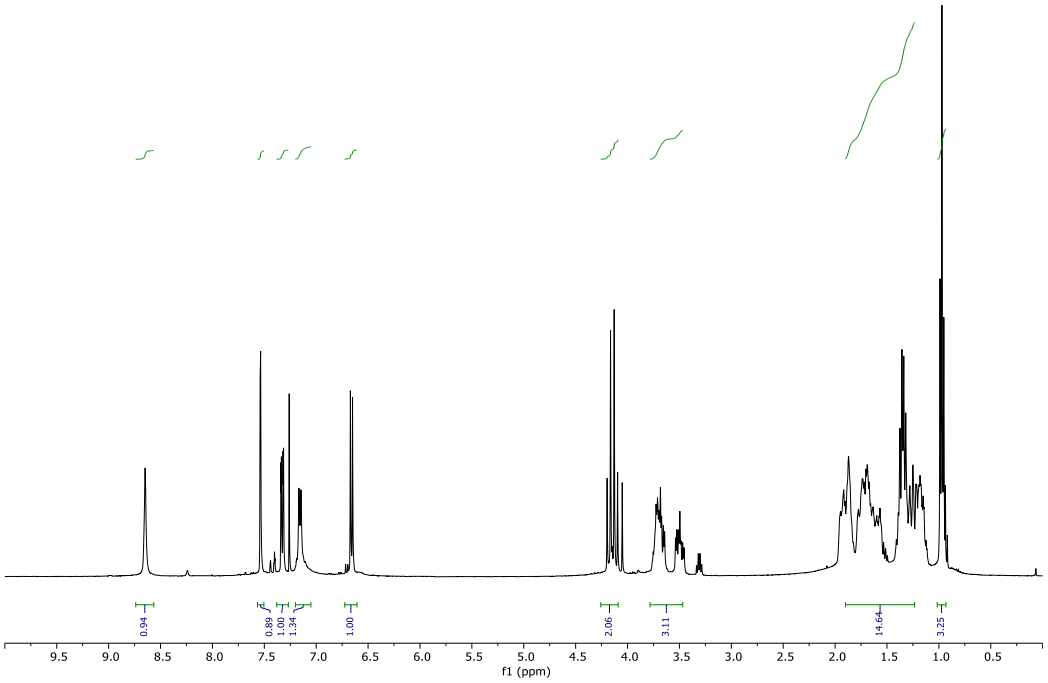
O
N
Br
Cl
O
O
O
N
H

22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1.00
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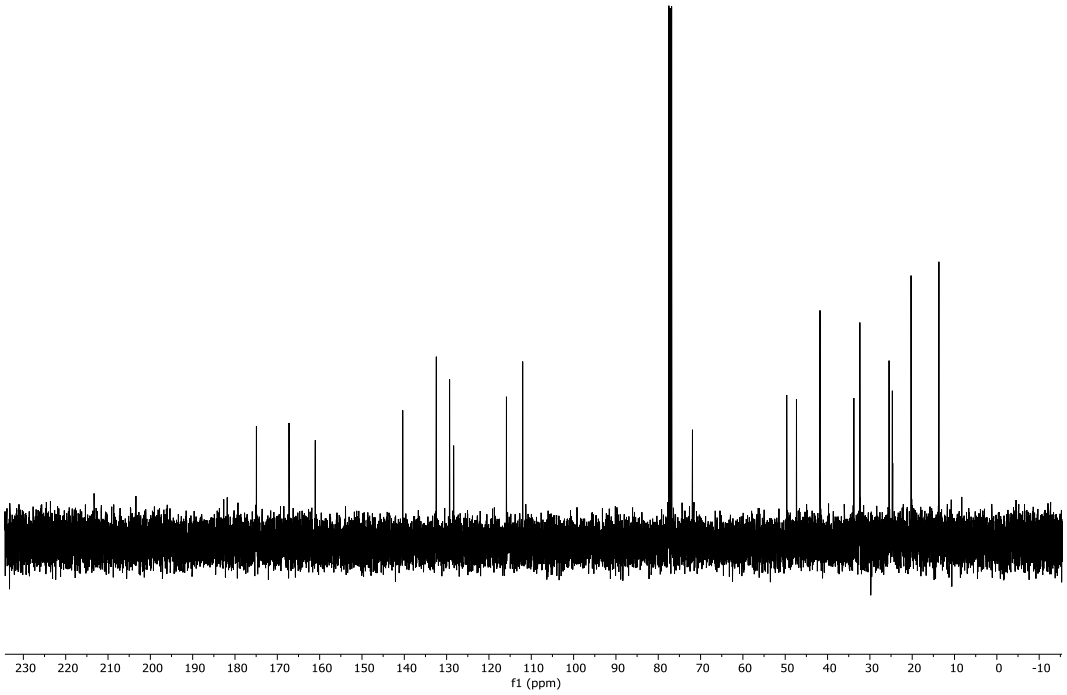
3.11
14.64
3.25
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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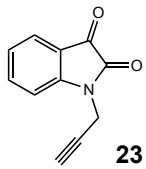

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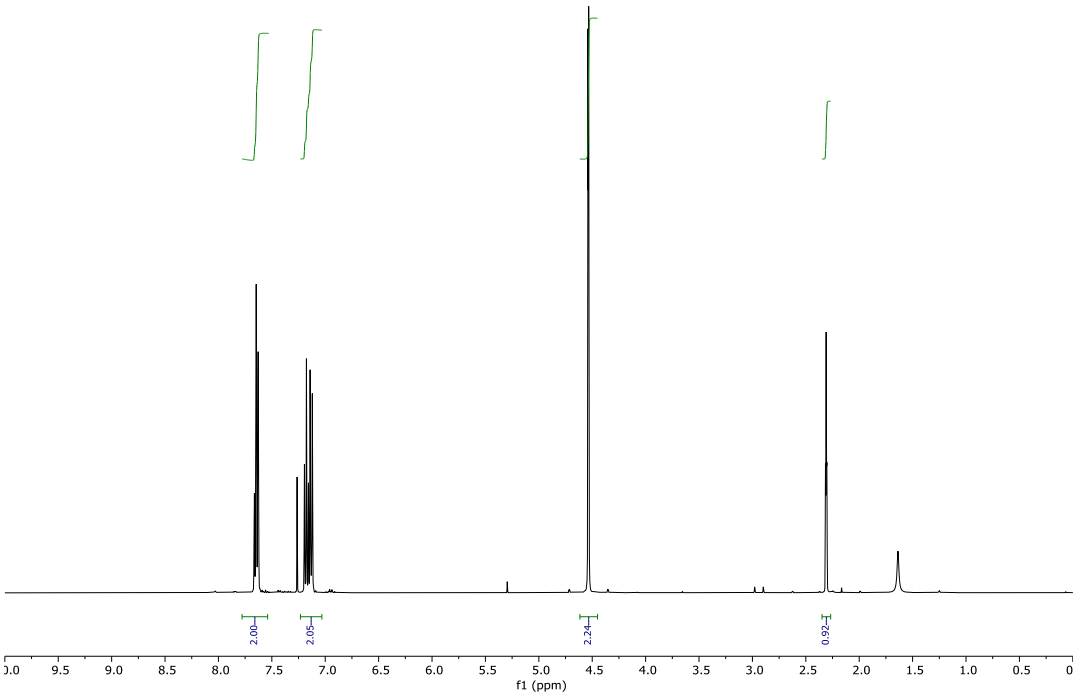
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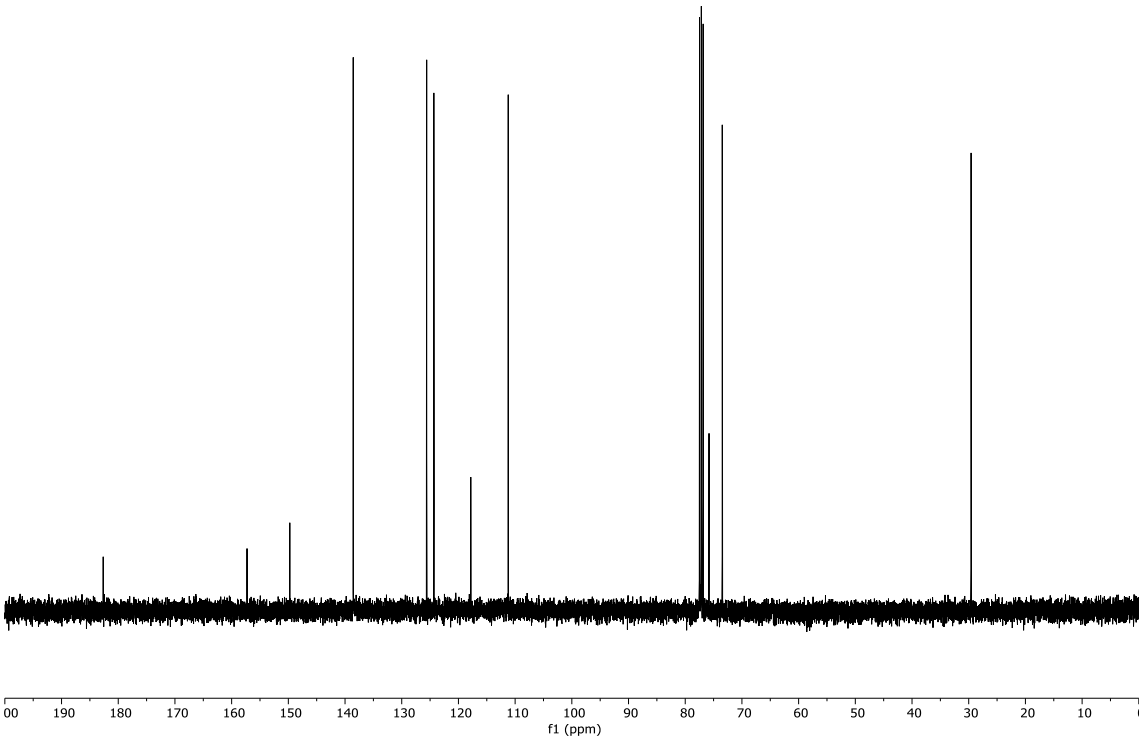
0.92
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
f1 (ppm)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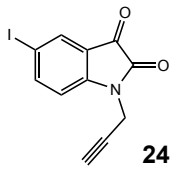
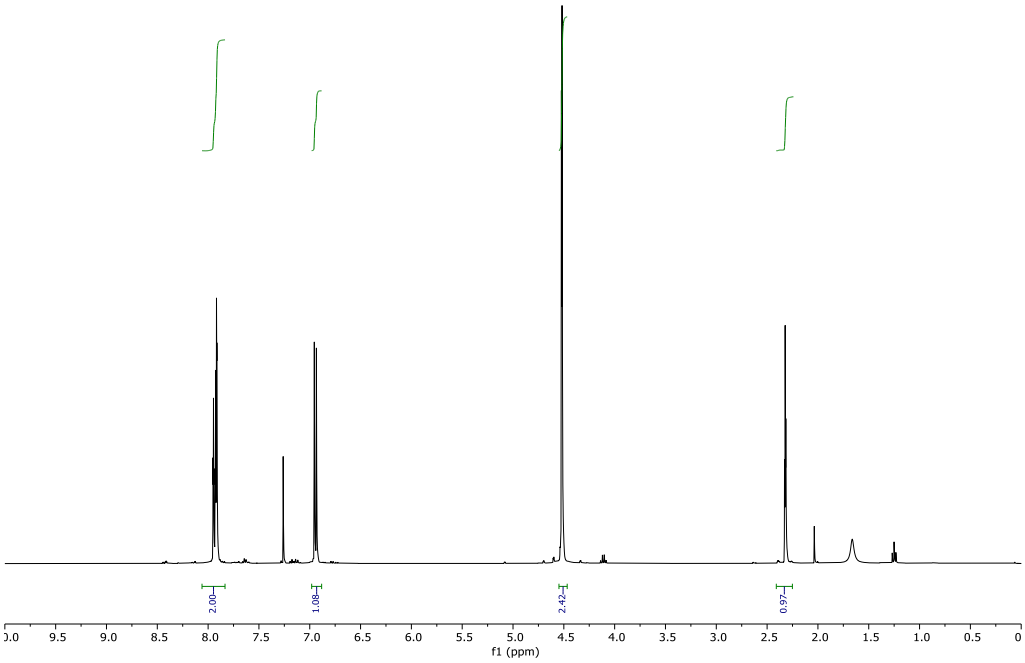
f1 (ppm)


O
I
O
N

**24**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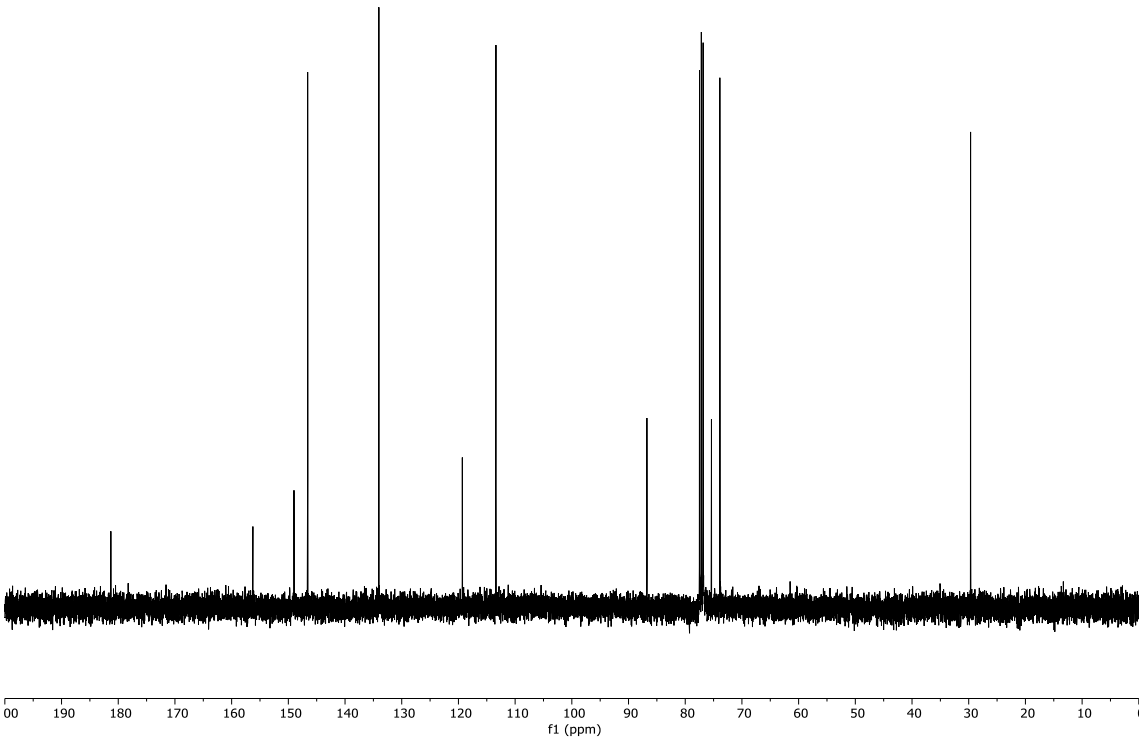
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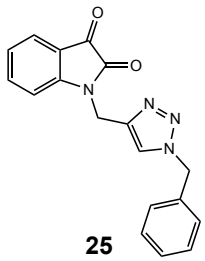

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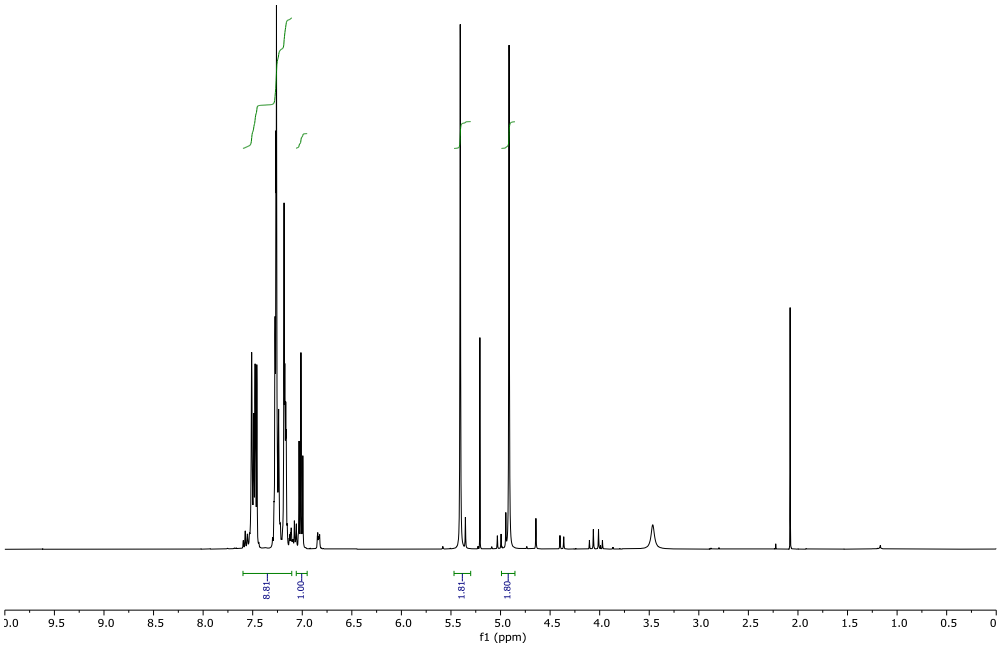


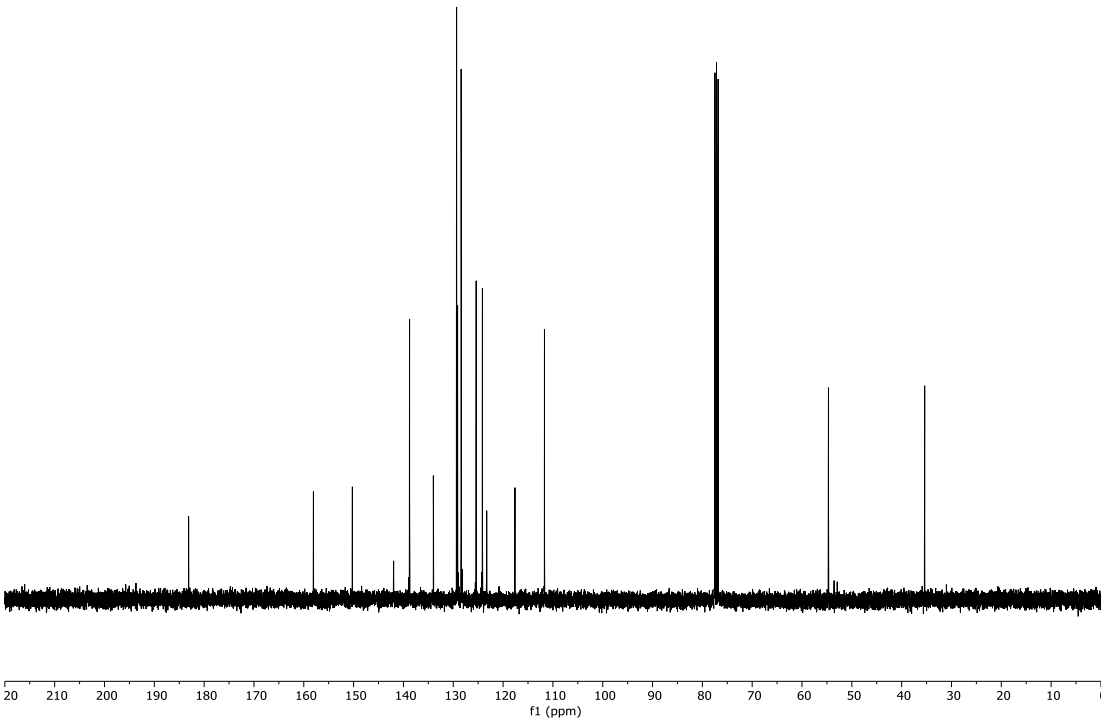

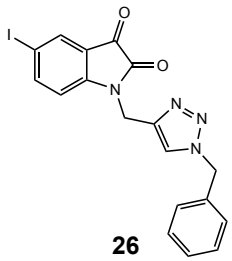

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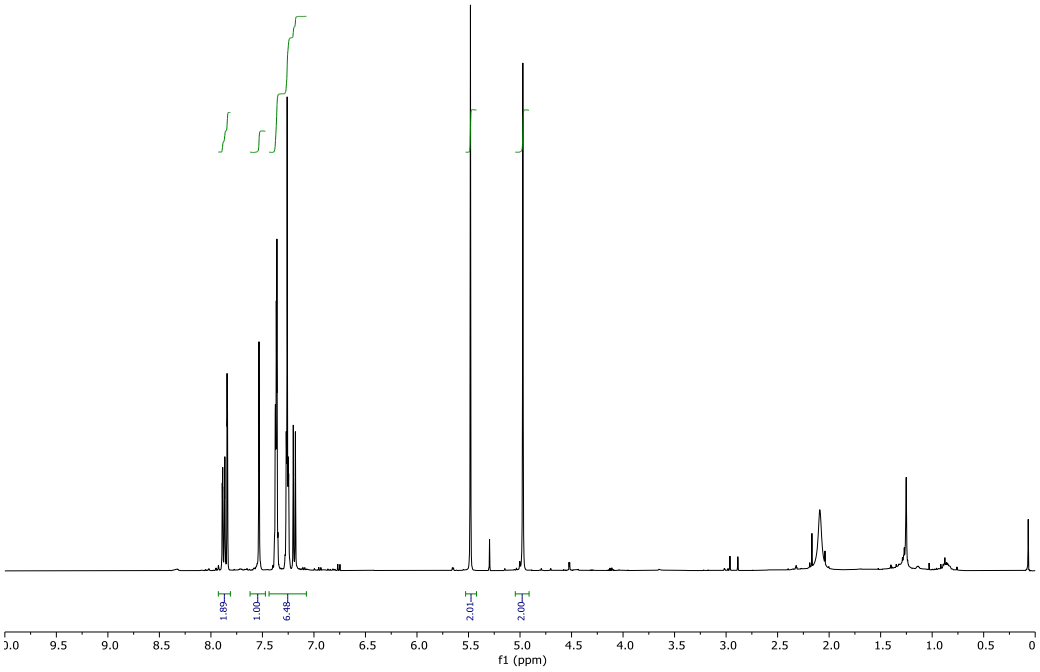


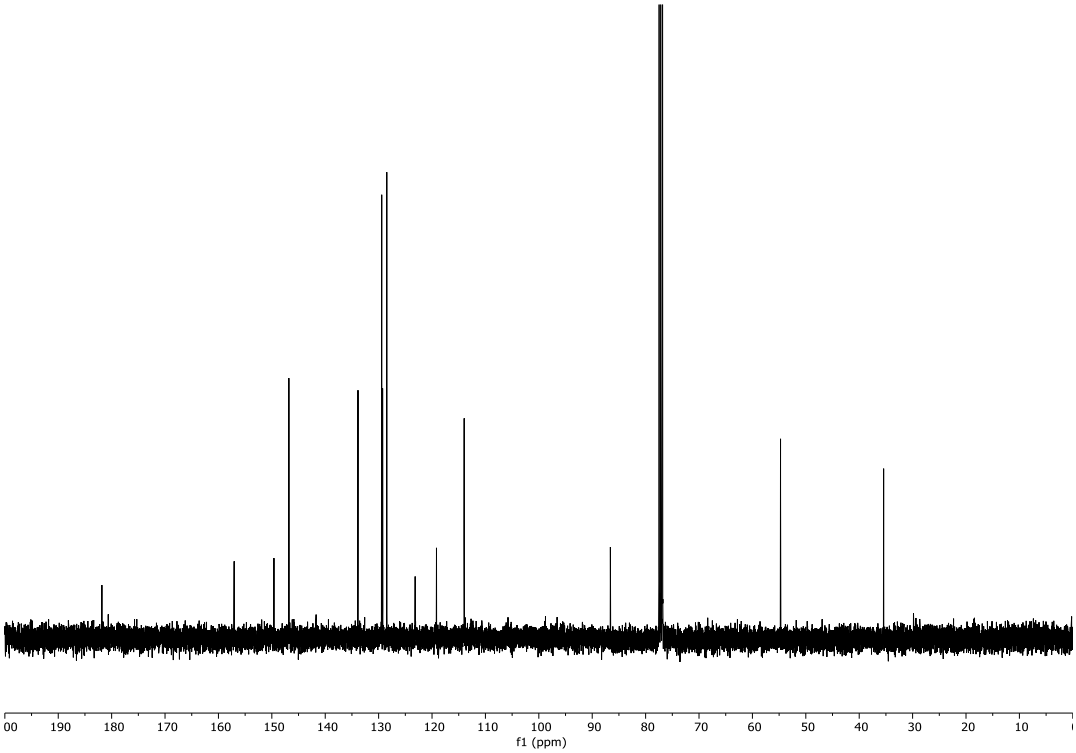

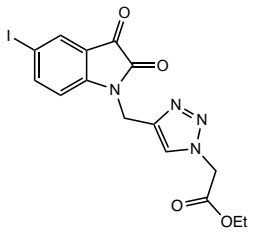


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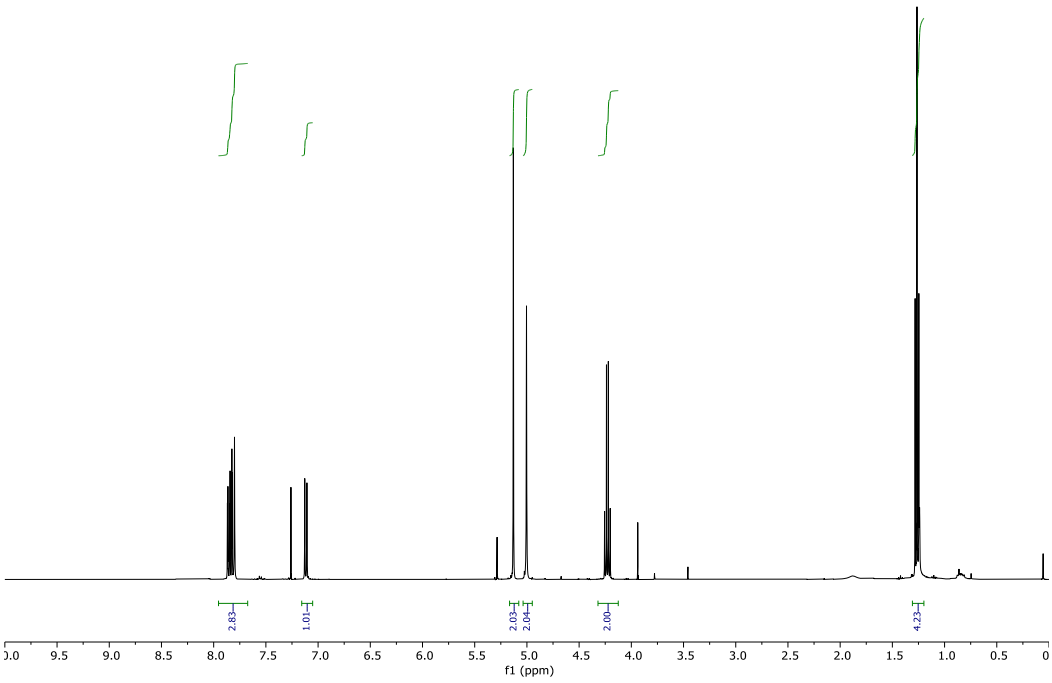


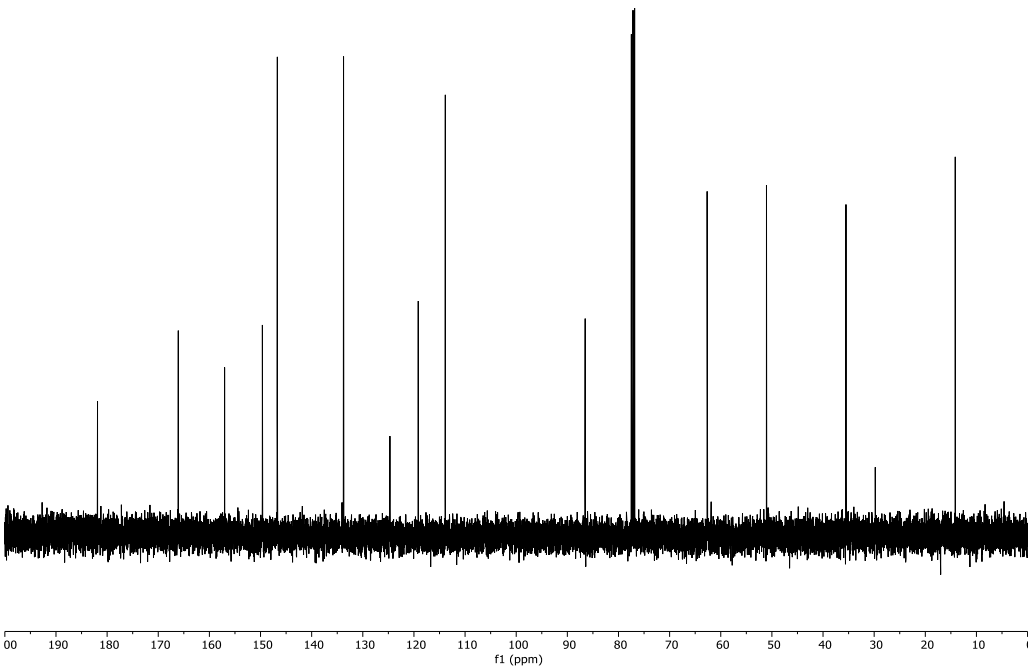

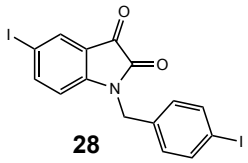


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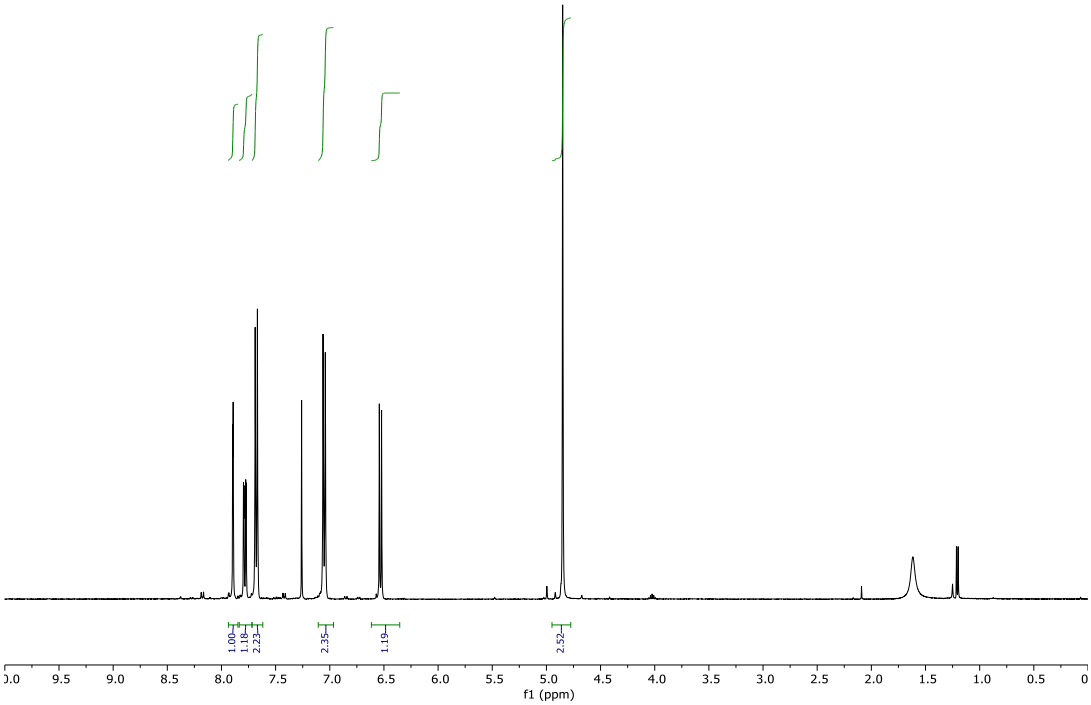
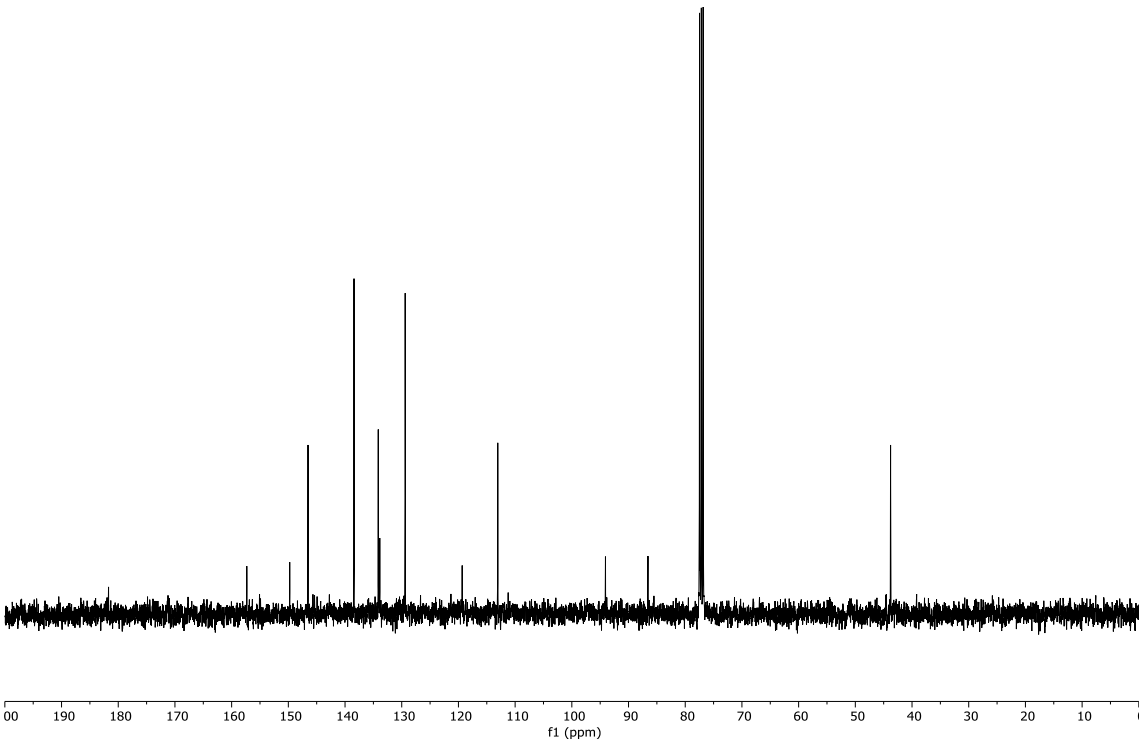
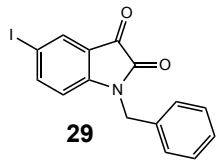
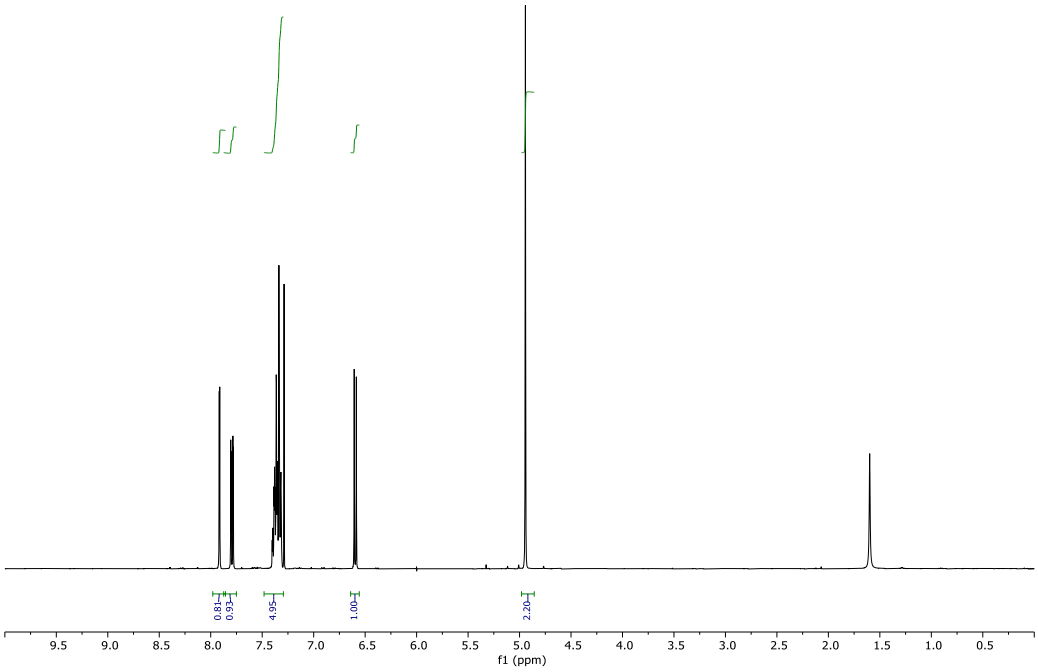



I
O
O
N

**29**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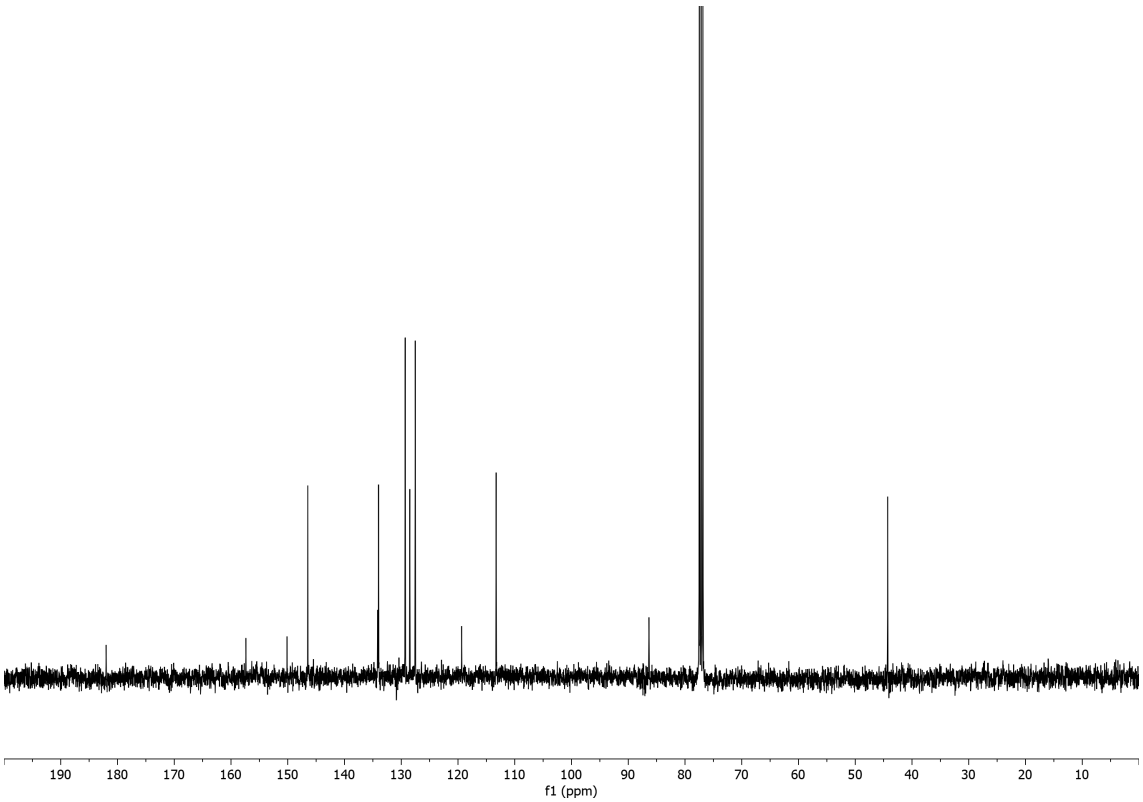
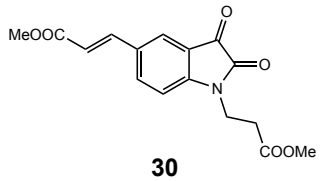
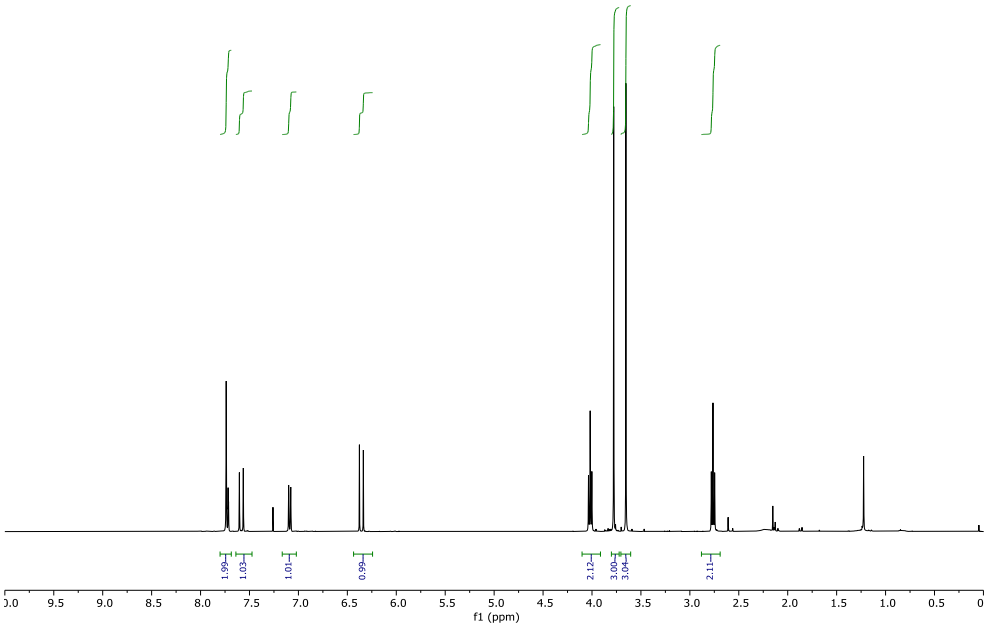
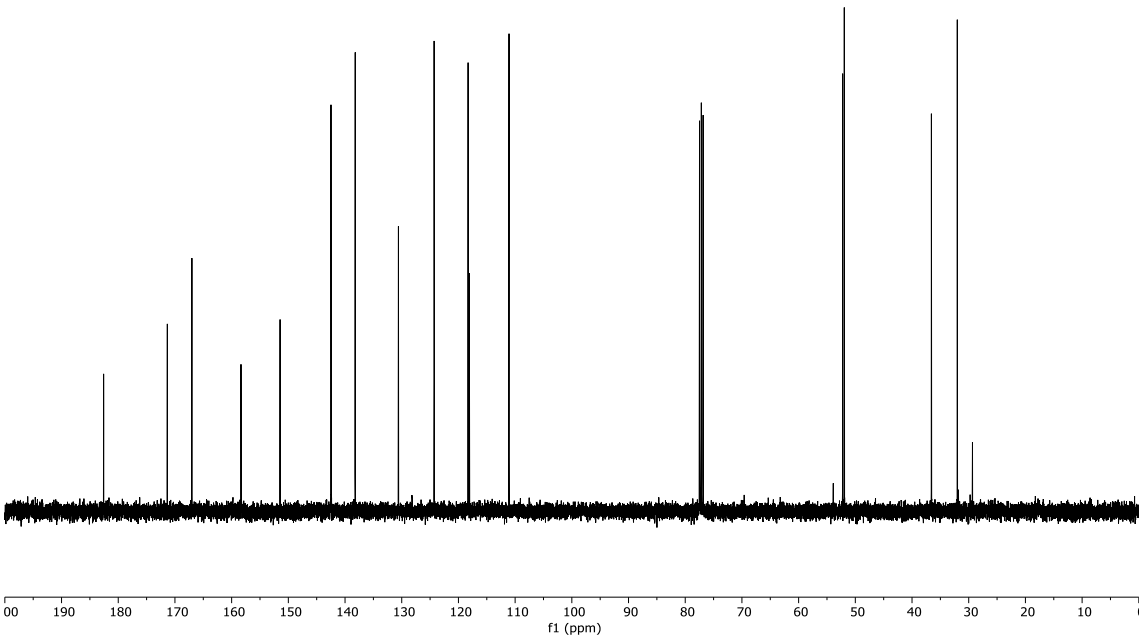
0.93
4.95
1.00
2.20
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

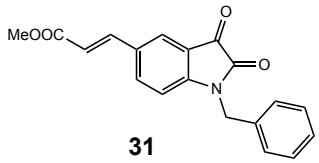

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



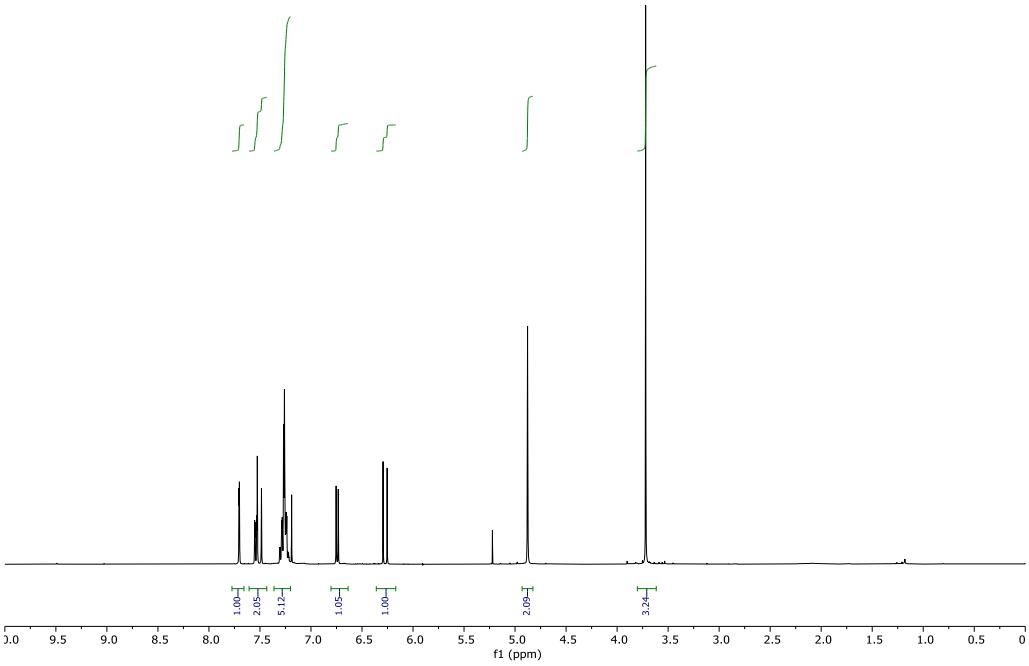

**30**

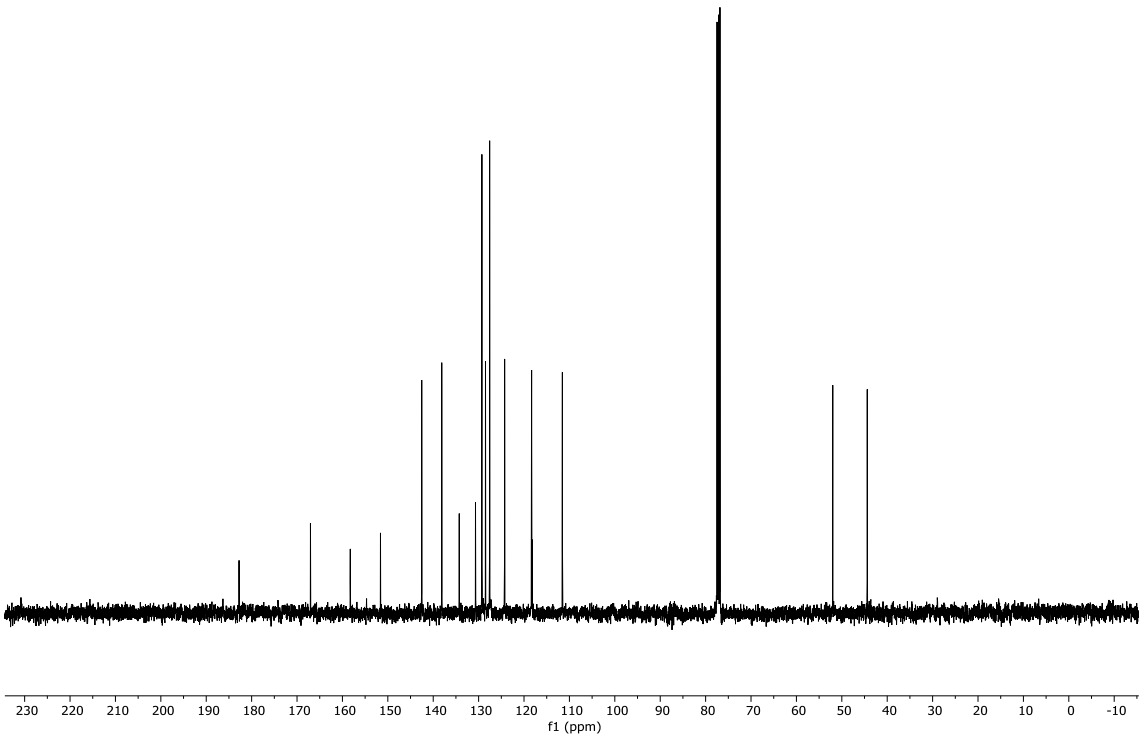


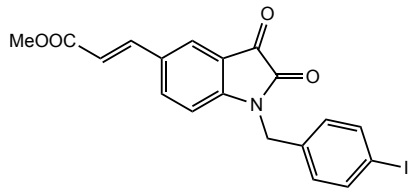





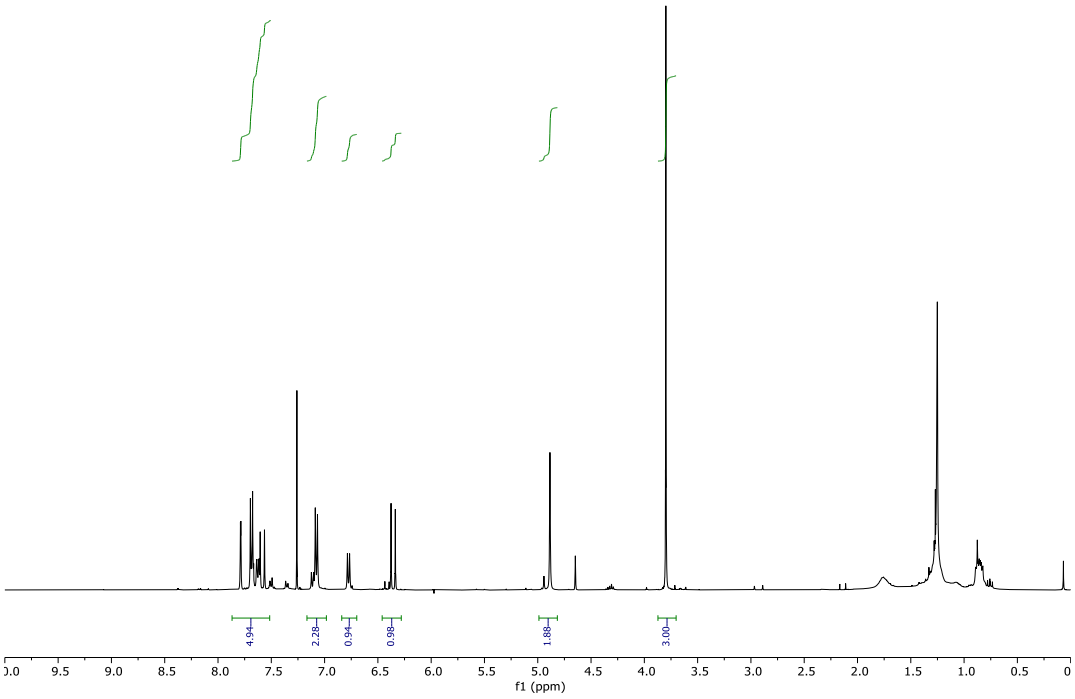

**31**

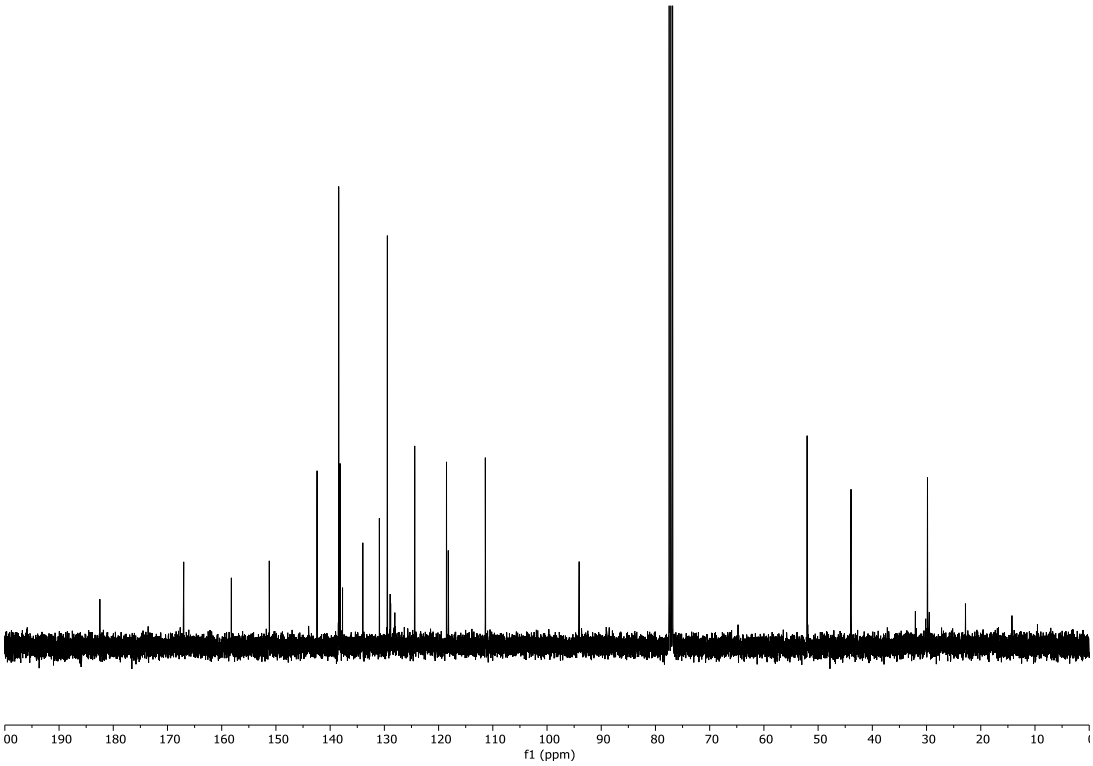


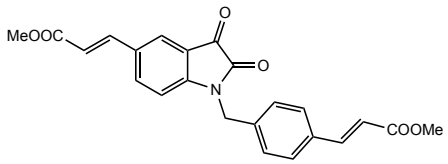





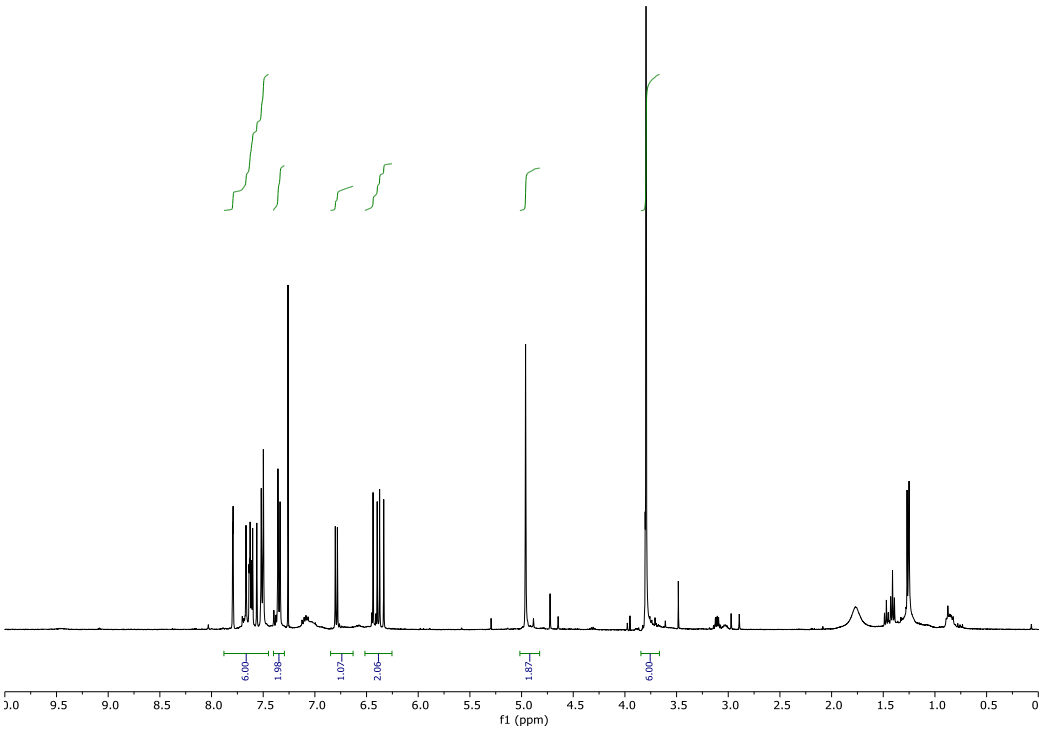

32

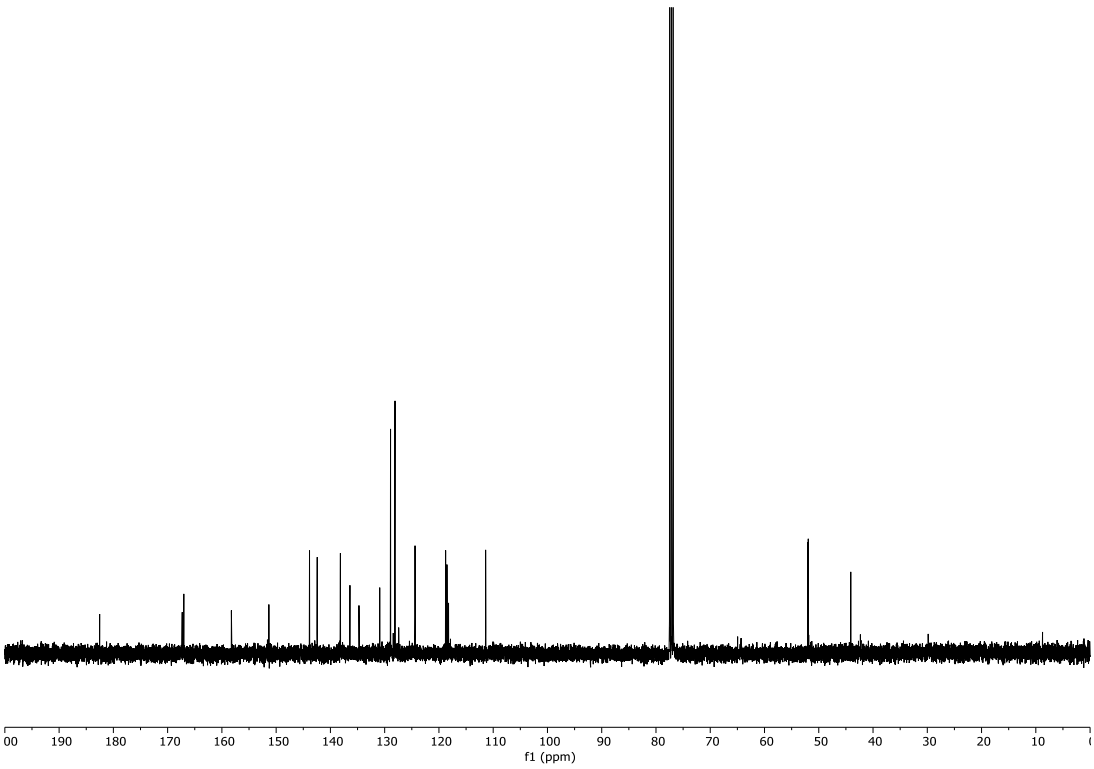





33



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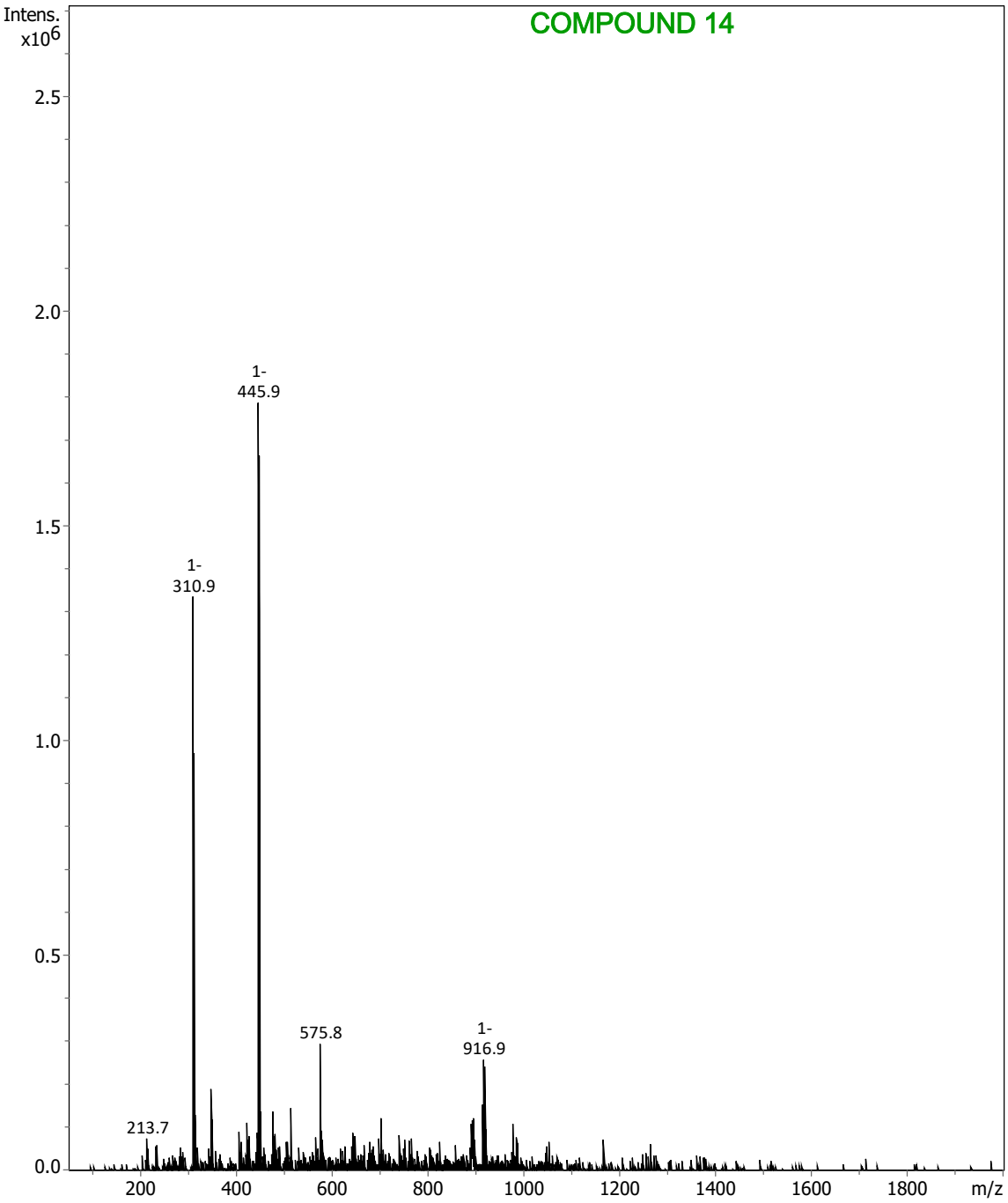
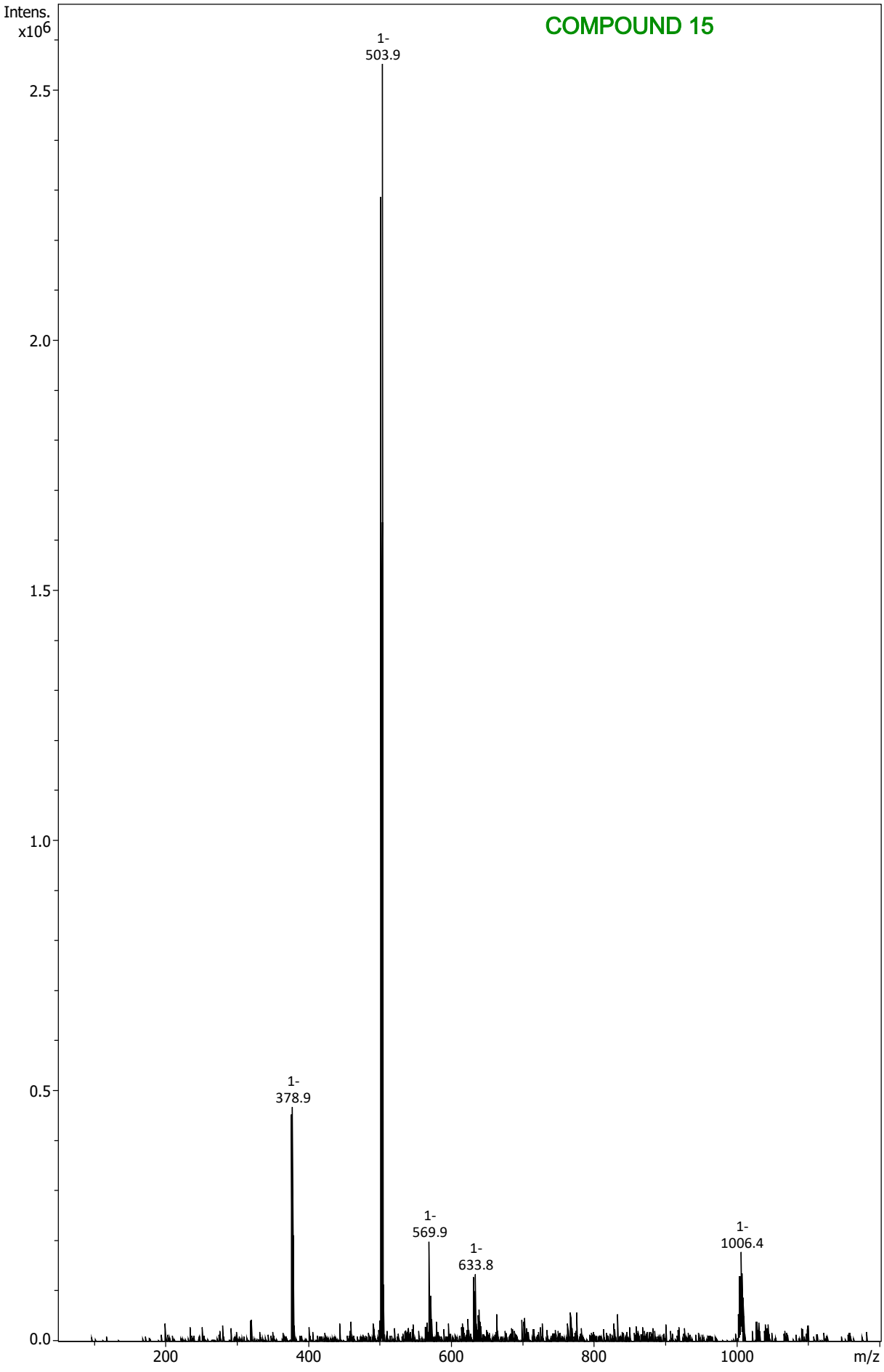
# ESPECTRO ESI-FIA-TOF

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 4 ESI MF3-CM-96 447_42_01_803.d | Acquisition Date | 04/06/2022 11:18:52 |
| Method | 803.m **COMPOUND 14** | Instrument | amaZon ETD |




COMPOUND 15
Intens.
x10^6
2.5
2.0
1.5
1.0
0.5
0.0
1-
503.9
1-
378.9
1-
569.9
1-
633.8
1-
1006.4
200
400
600
800
1000
m/z

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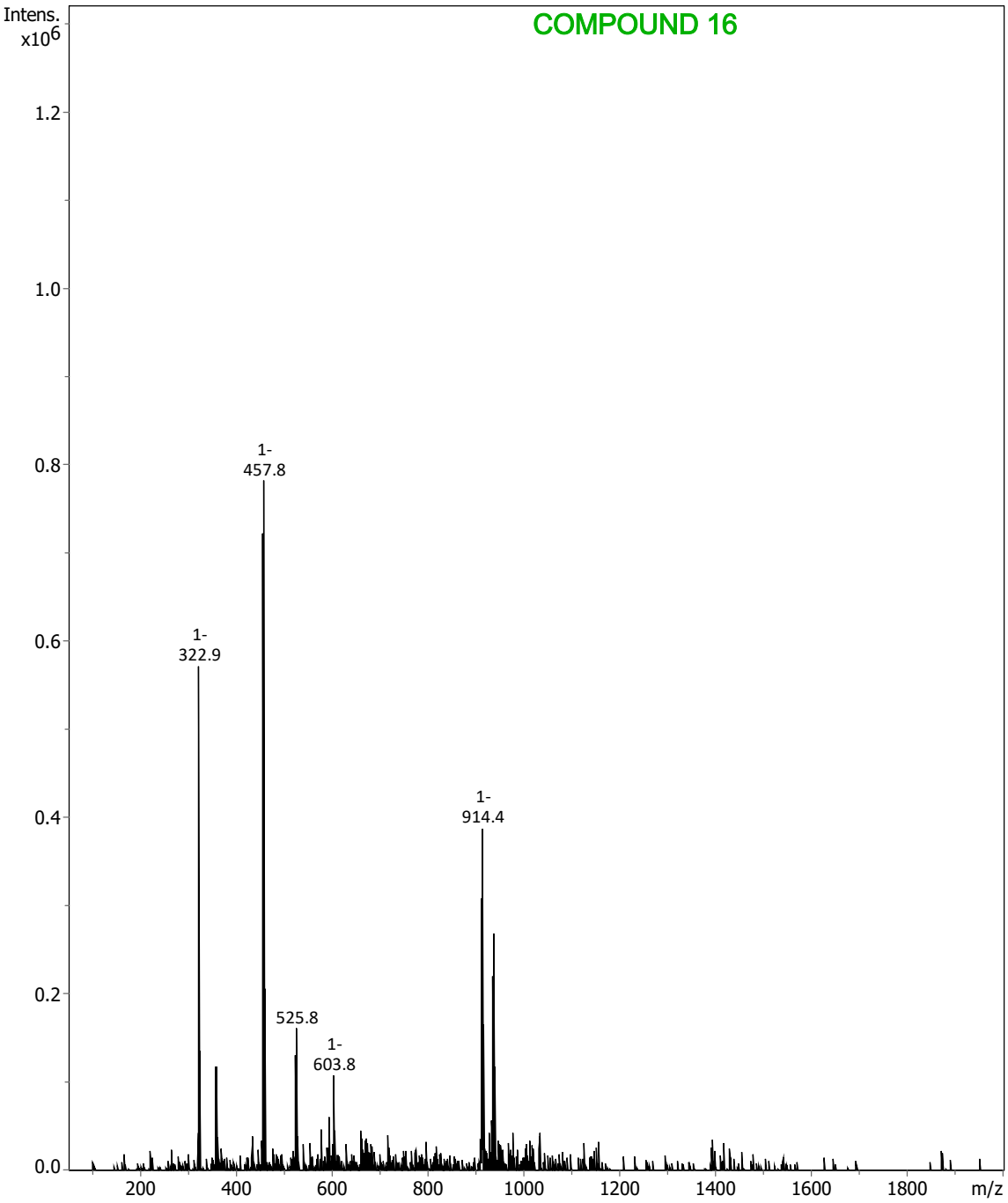
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 16 ESI MF-CM-117 457_51_01_824.d | Acquisition Date | 04/18/2022 16:36:47 |
| Method | 824.m **COMPOUND 16** | Instrument | amaZon ETD |



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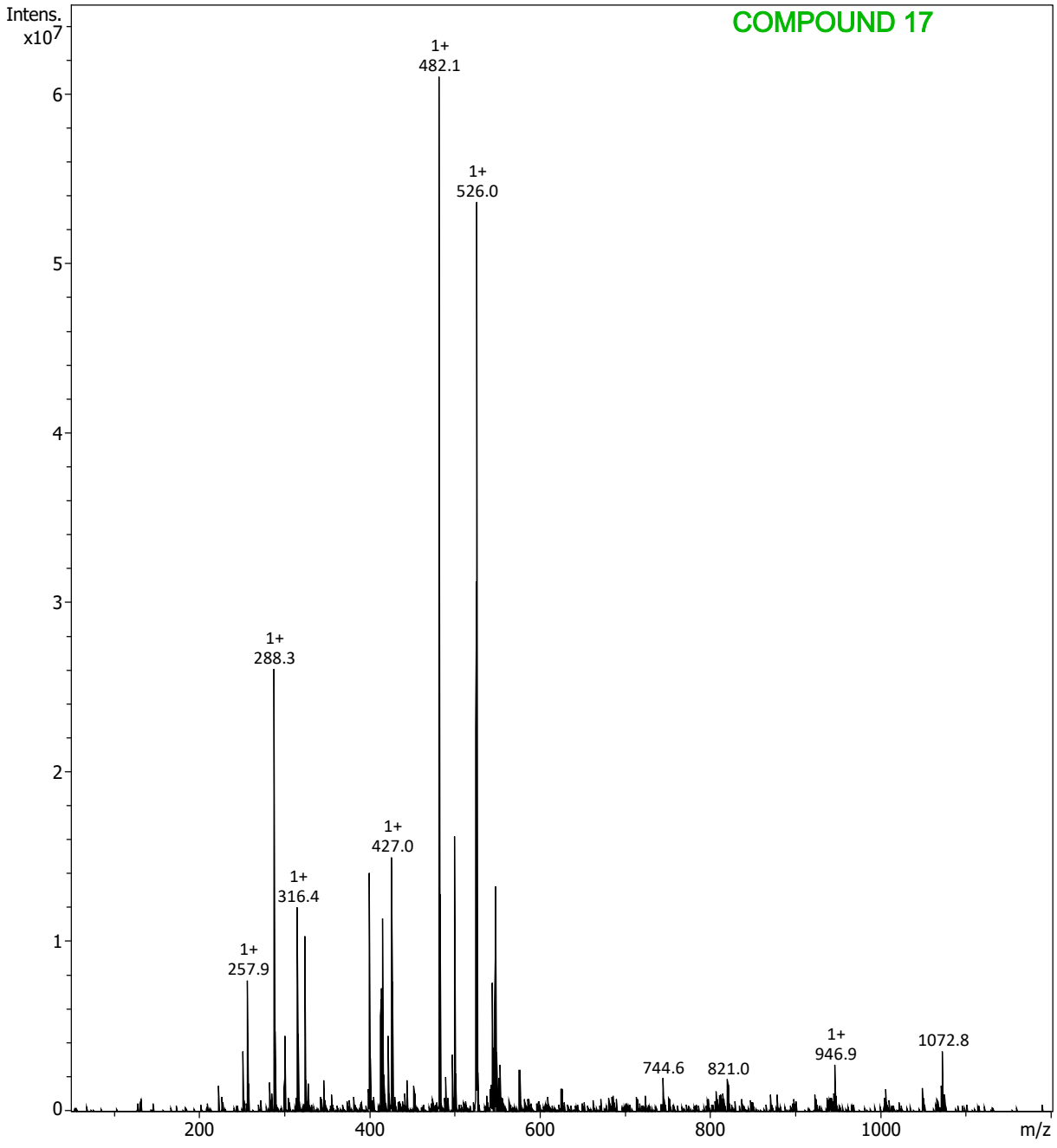
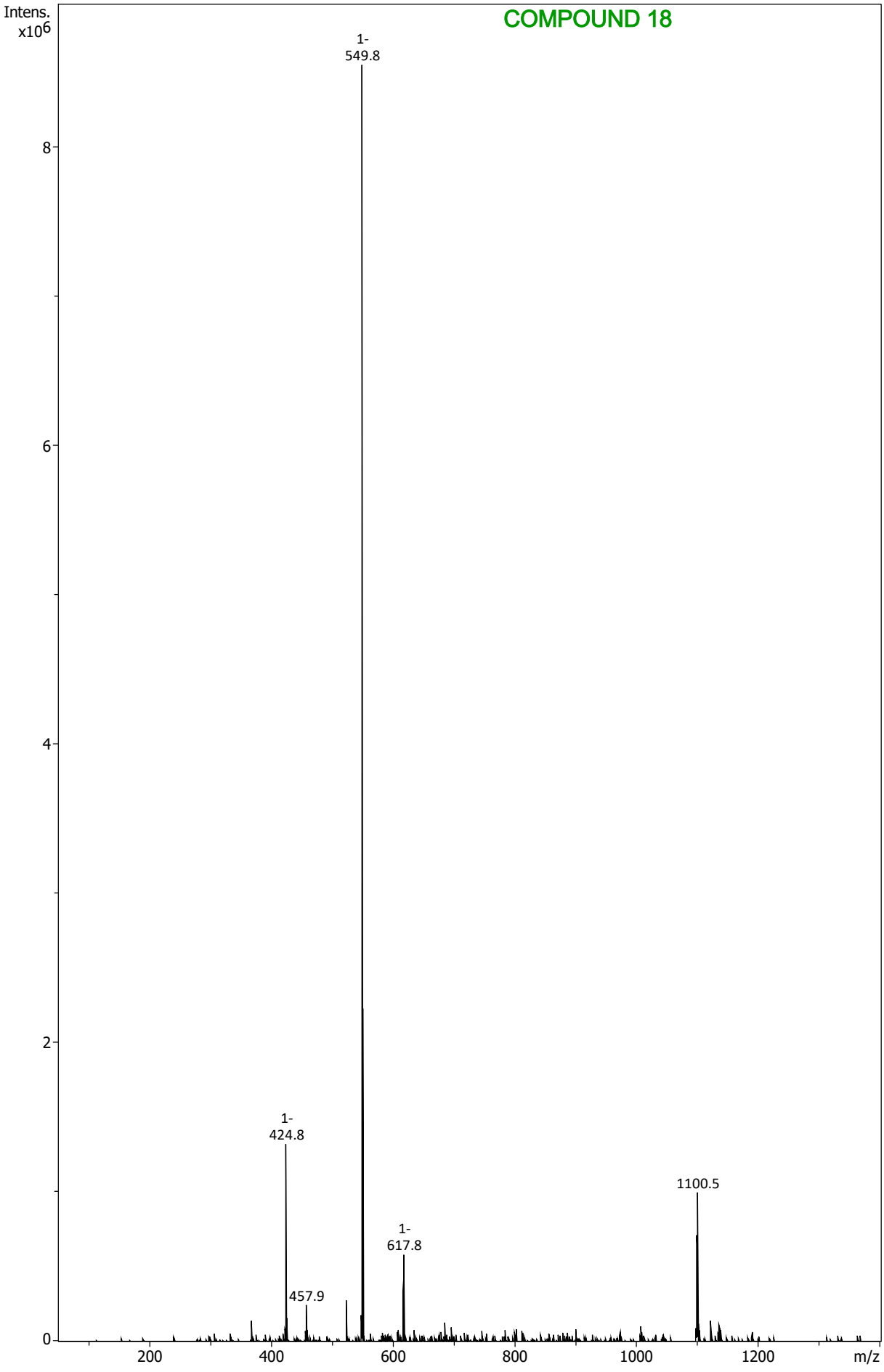
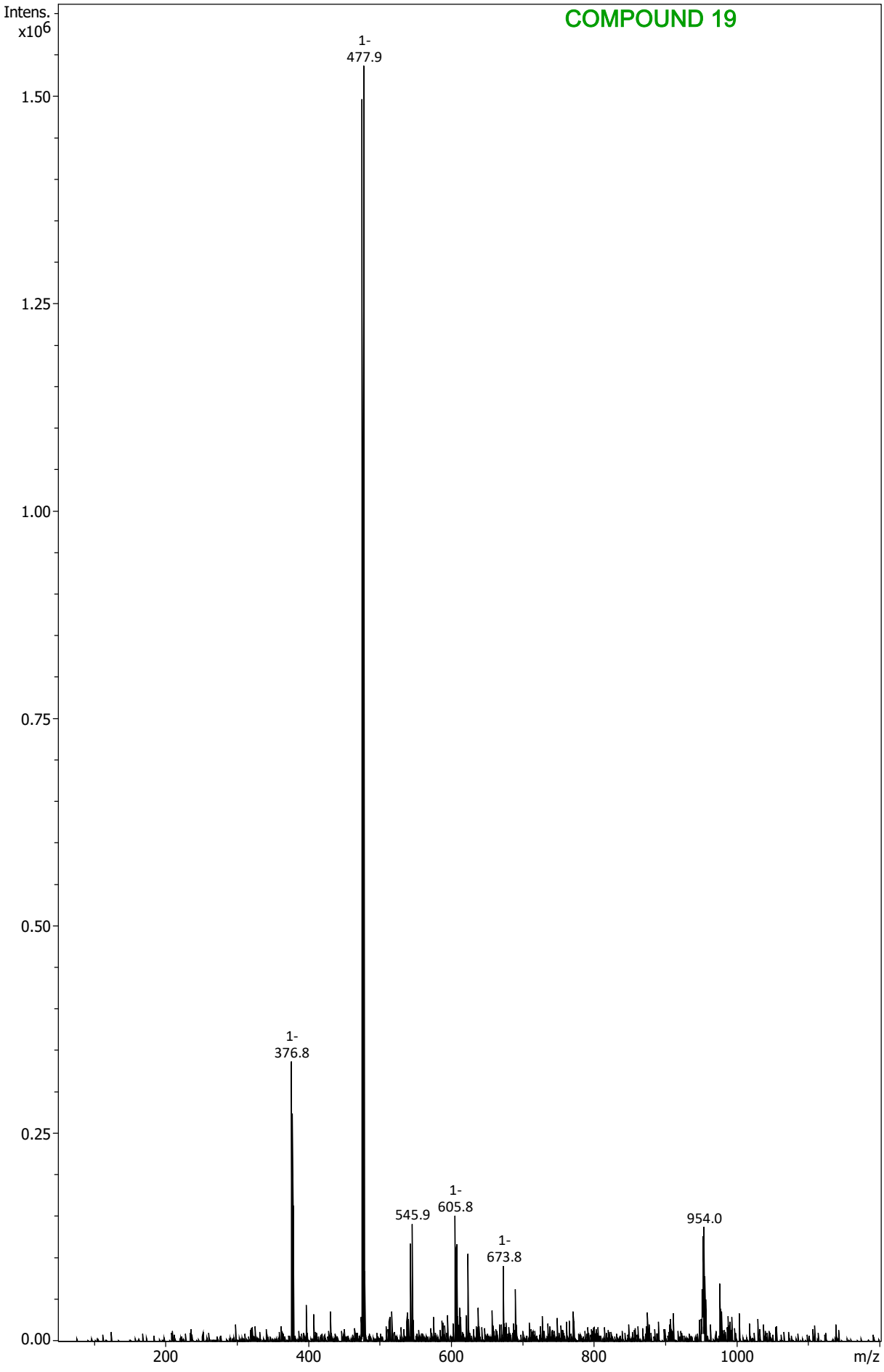
# ESPECTRO ESI-FIA-Ion Trap

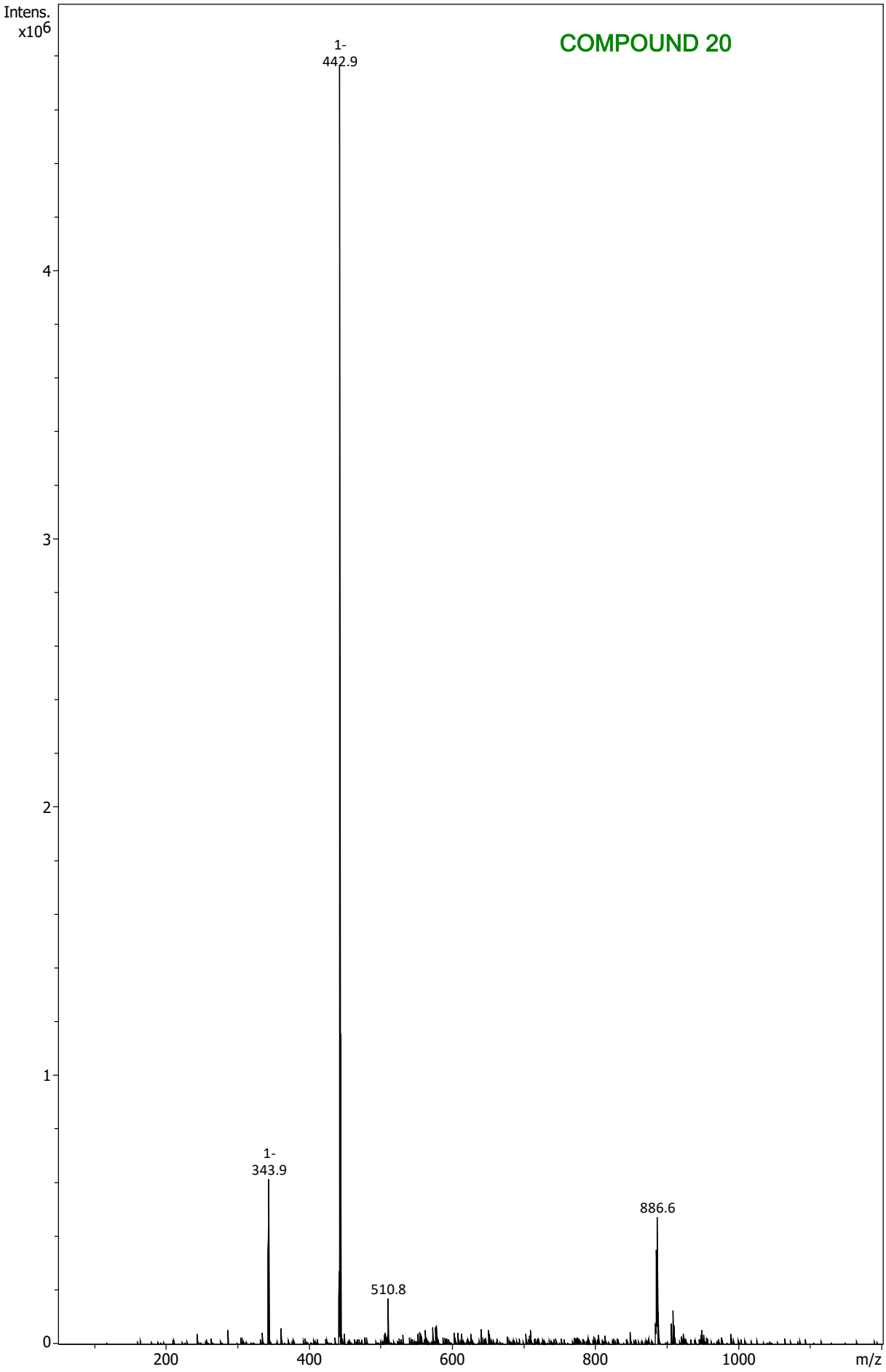
**Analysis Info**

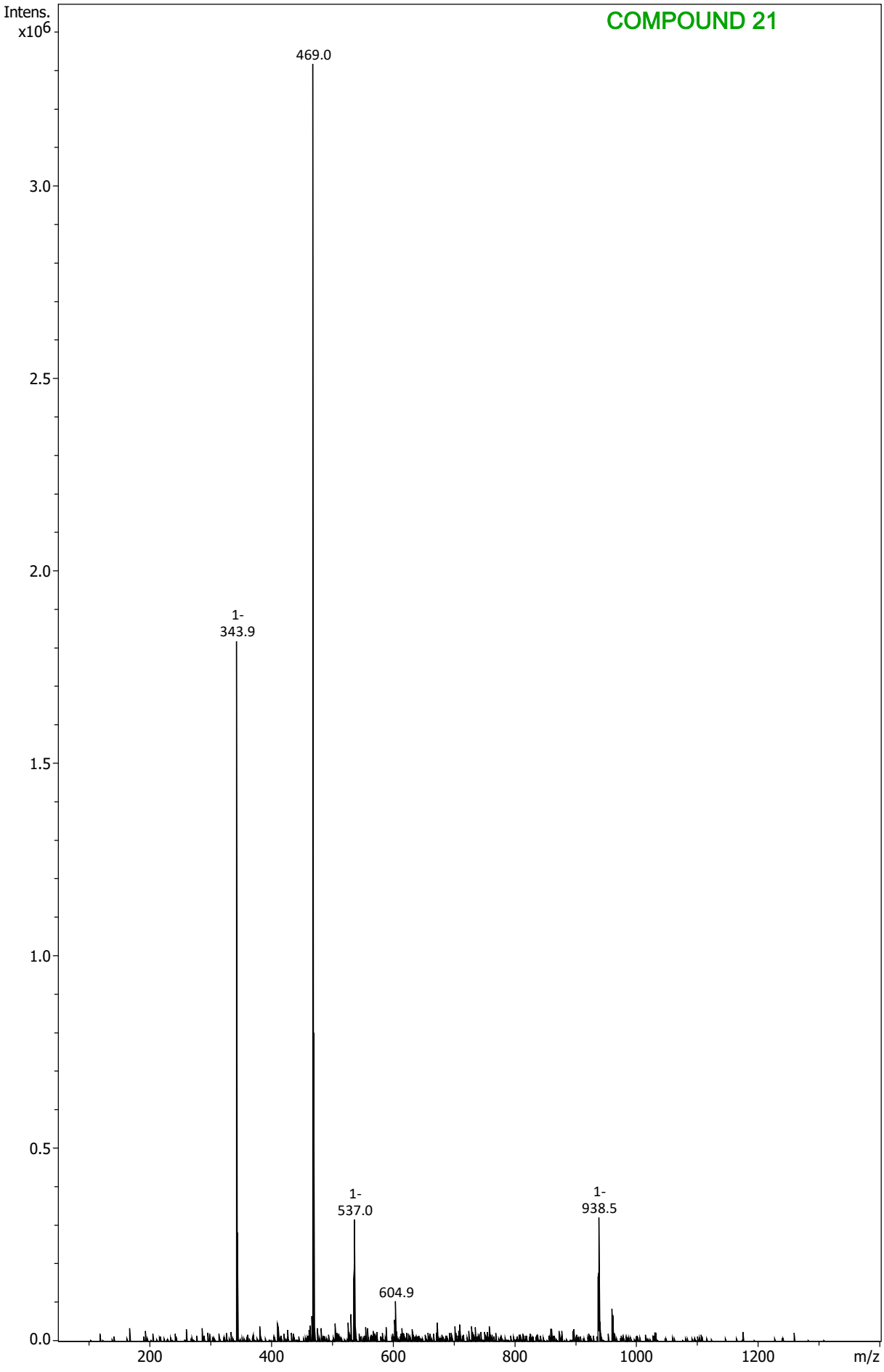
| | | | |
|---|---|---|---|
| Sample Name | 5 ESI MF3-CM-146 525_45_01_836.d | Acquisition Date | 04/22/2022 13:55:33 |
| Method | 836.m **COMPOUND 17** | Instrument | amaZon ETD |

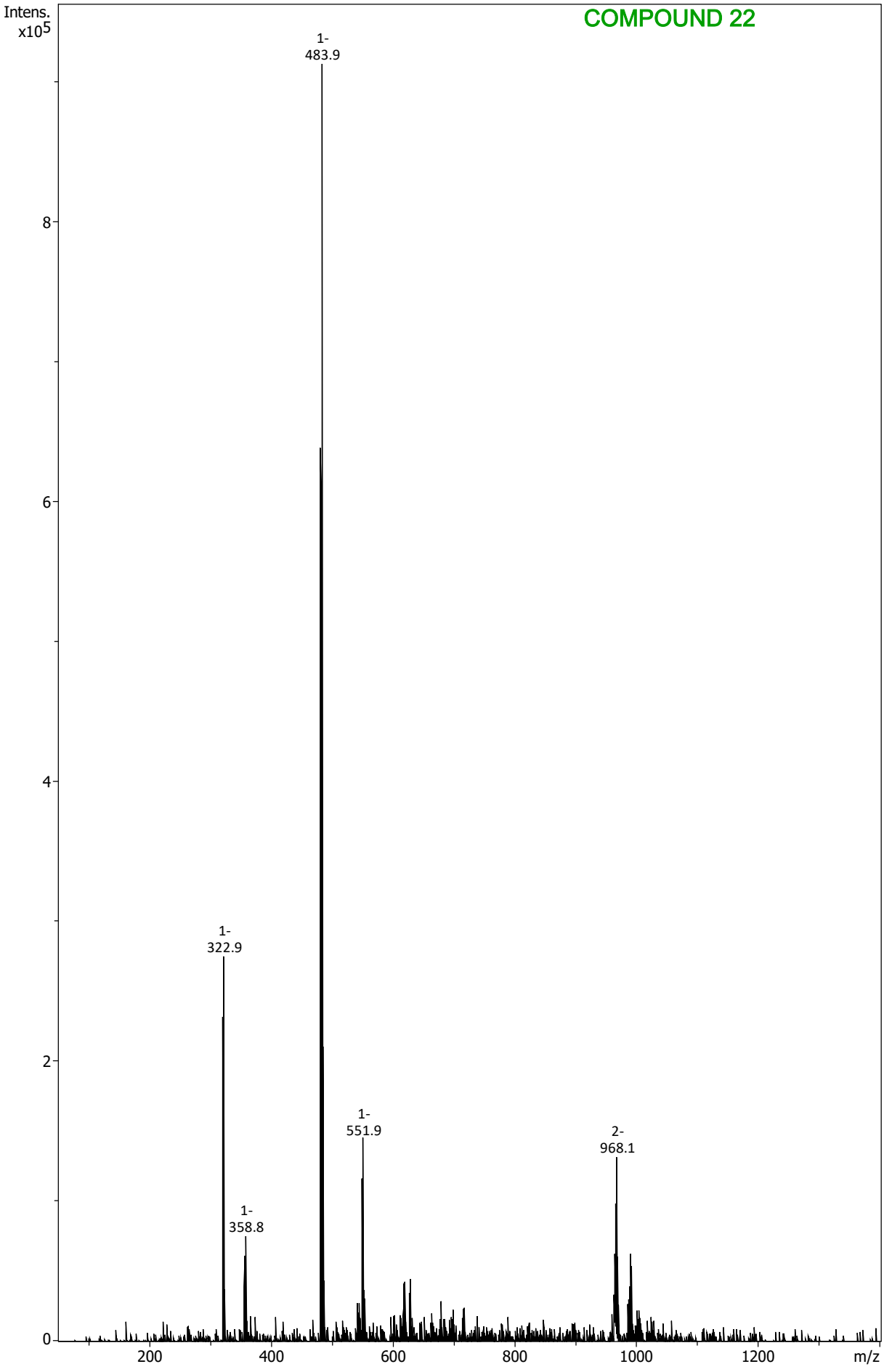



COMPOUND 18
Intens.
x10^6
1-
549.8
1-
424.8
457.9
1-
617.8
1100.5
0
2
4
6
8
200
400
600
800
1000
1200
m/z


COMPOUND 19
Intens.
x10^6
1.50
1.25
1.00
0.75
0.50
0.25
0.00
1-
477.9
1-
376.8
545.9
1-
605.8
1-
673.8
954.0
200
400
600
800
1000
m/z


Intens.
x10^6
COMPOUND 20
1-
442.9
1-
343.9
510.8
886.6
0
1
2
3
4
200
400
600
800
1000
m/z


COMPOUND 21
Intens.
x10^6
469.0
3.0
2.5
2.0
1-
343.9
1.5
1.0
1-
537.0
604.9
0.5
1-
938.5
0.0
200
400
600
800
1000
1200
m/z


COMPOUND 22
Intens.
x10^5
1-
483.9
1-
322.9
1-
358.8
1-
551.9
2-
968.1
0
2
4
6
8
200
400
600
800
1000
1200
m/z

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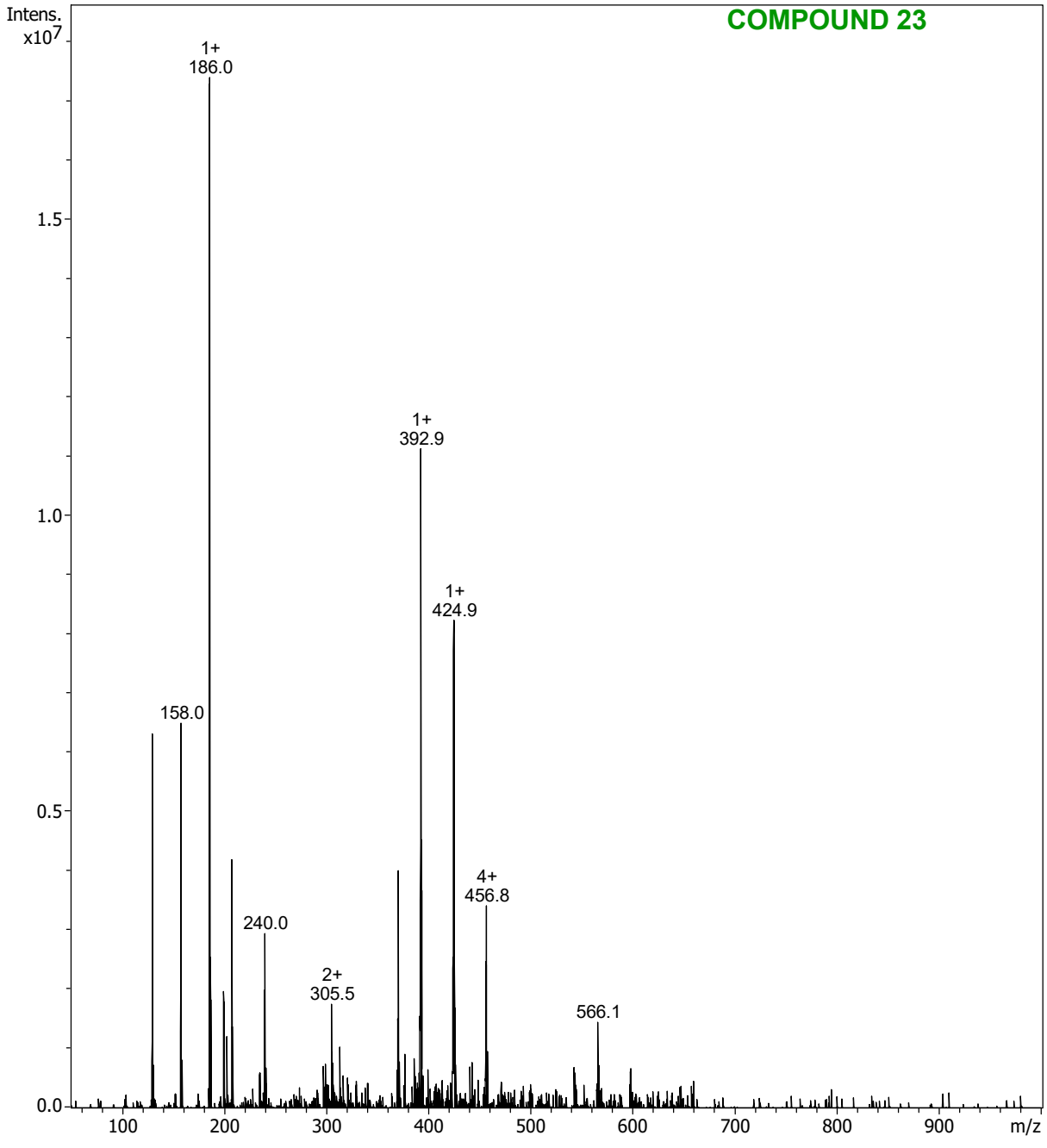
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 17 ESI MF3-CM-179 317_59_01_1156.d | Acquisition Date | 12/1/2022 4:00:32 PM |
| Method | 1156.m | Instrument | amaZon ETD |



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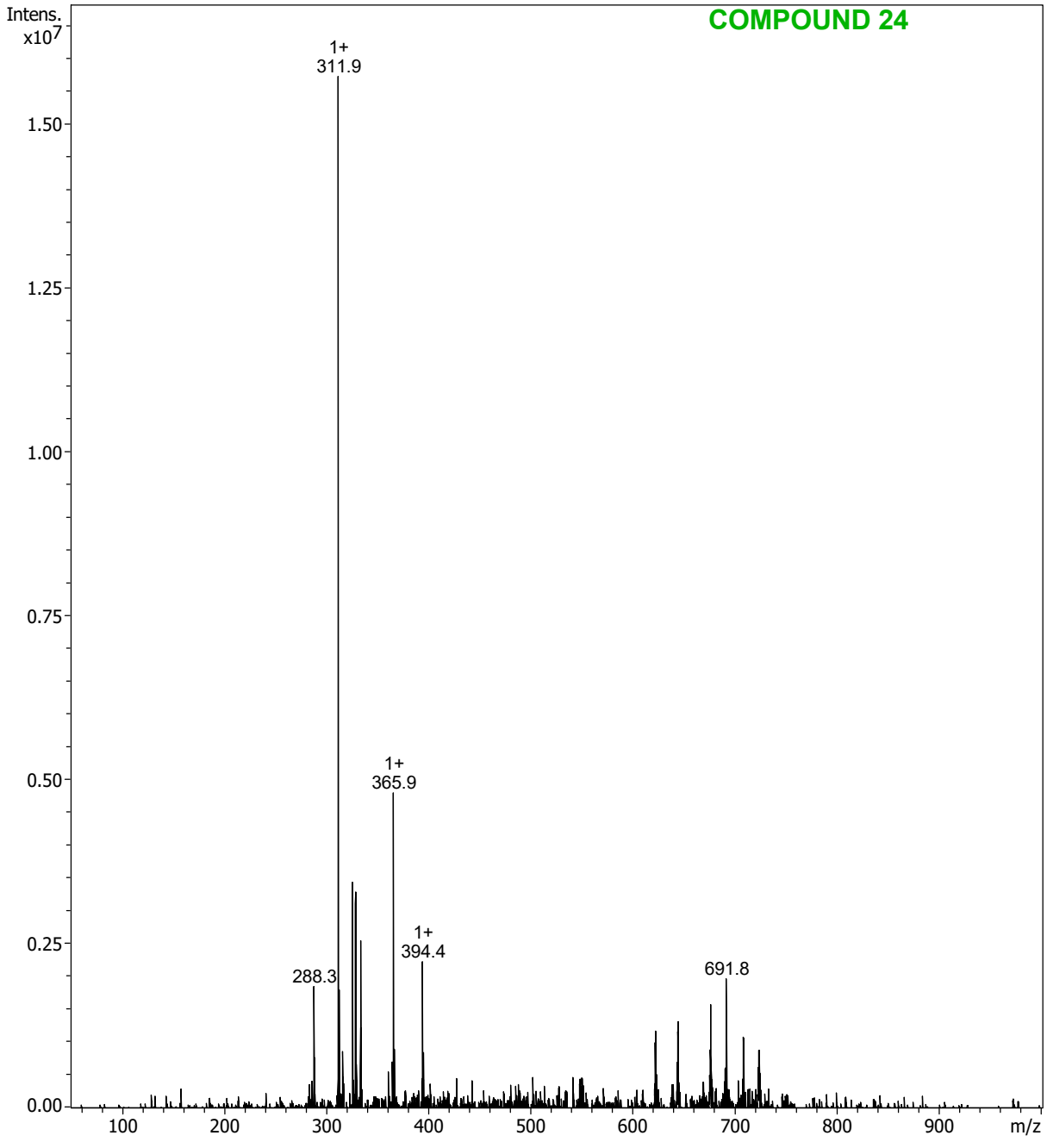
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 9 ESI MF3-CM-181 310_52_01_1148.d | Acquisition Date | 12/1/2022 2:31:54 PM |
| Method | 1148.m | Instrument | amaZon ETD |



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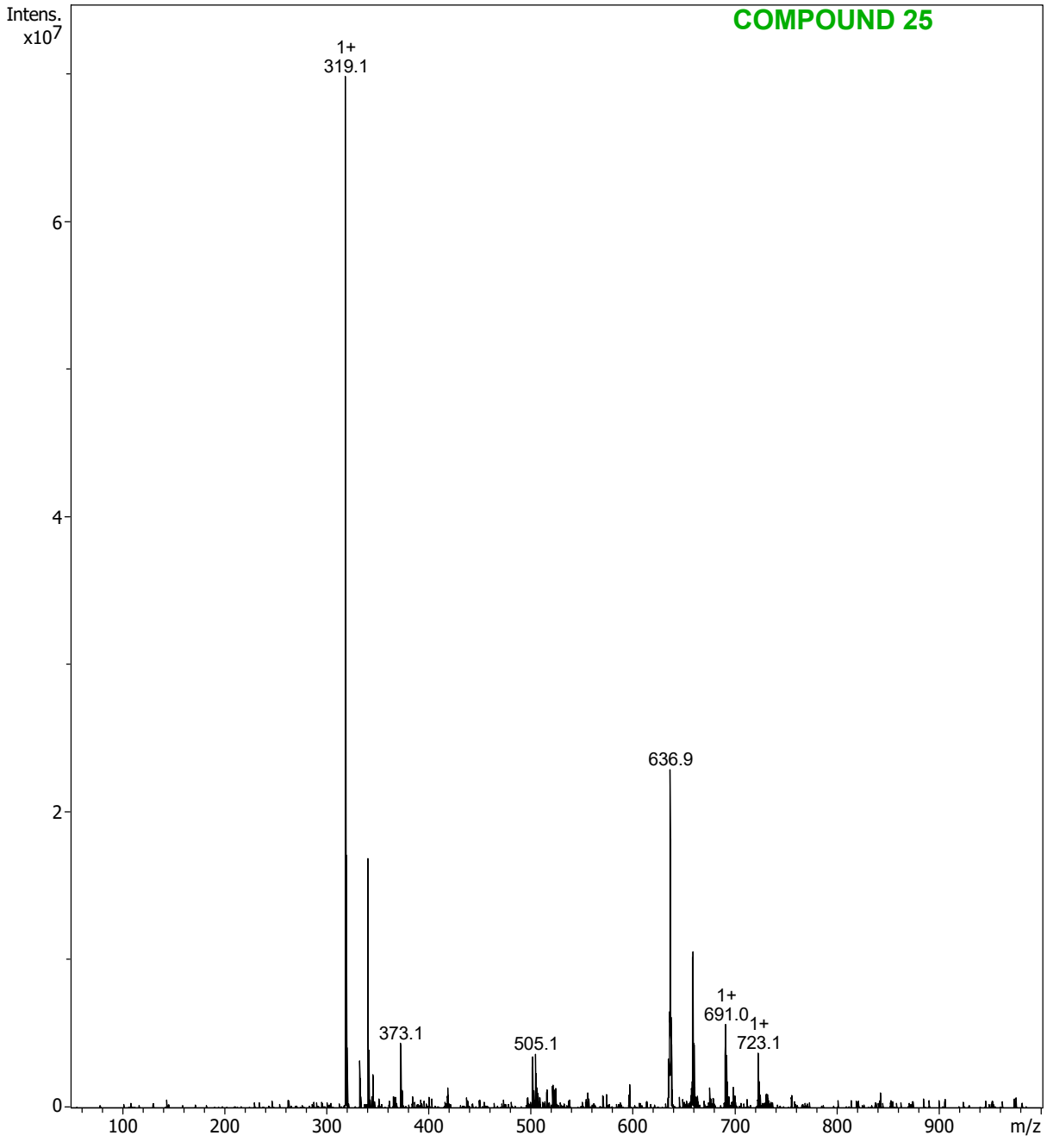
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 15 ESI MF3-CM-193 318_57_01_1154.d | Acquisition Date | 12/1/2022 3:38:22 PM |
| Method | 1154.m | Instrument | amaZon ETD |



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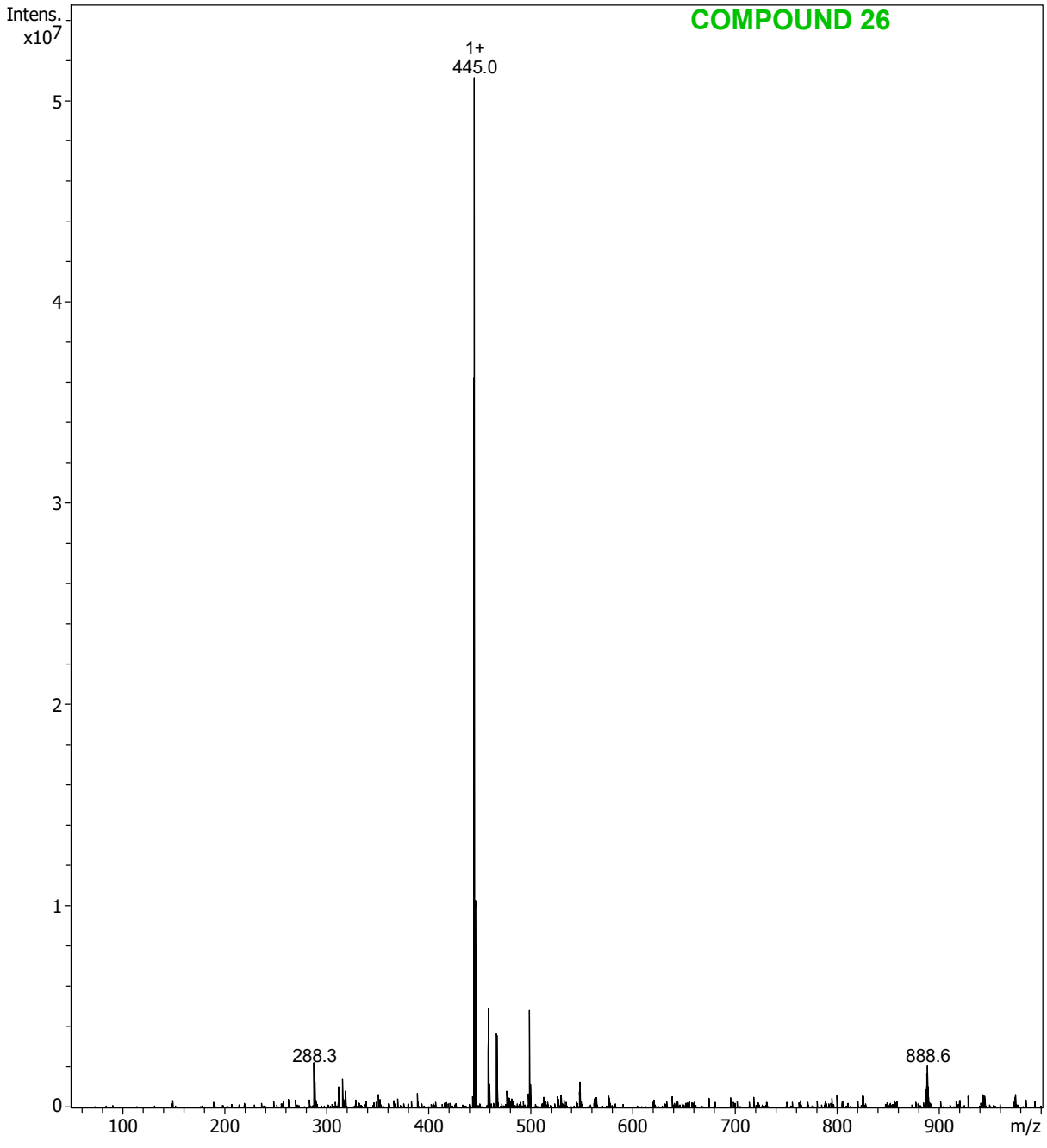
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 10 ESI MF3-CM-182B 444_53_01_1149.d | Acquisition Date | 12/1/2022 2:42:58 PM |
| Method | 1149.m | Instrument | amaZon ETD |



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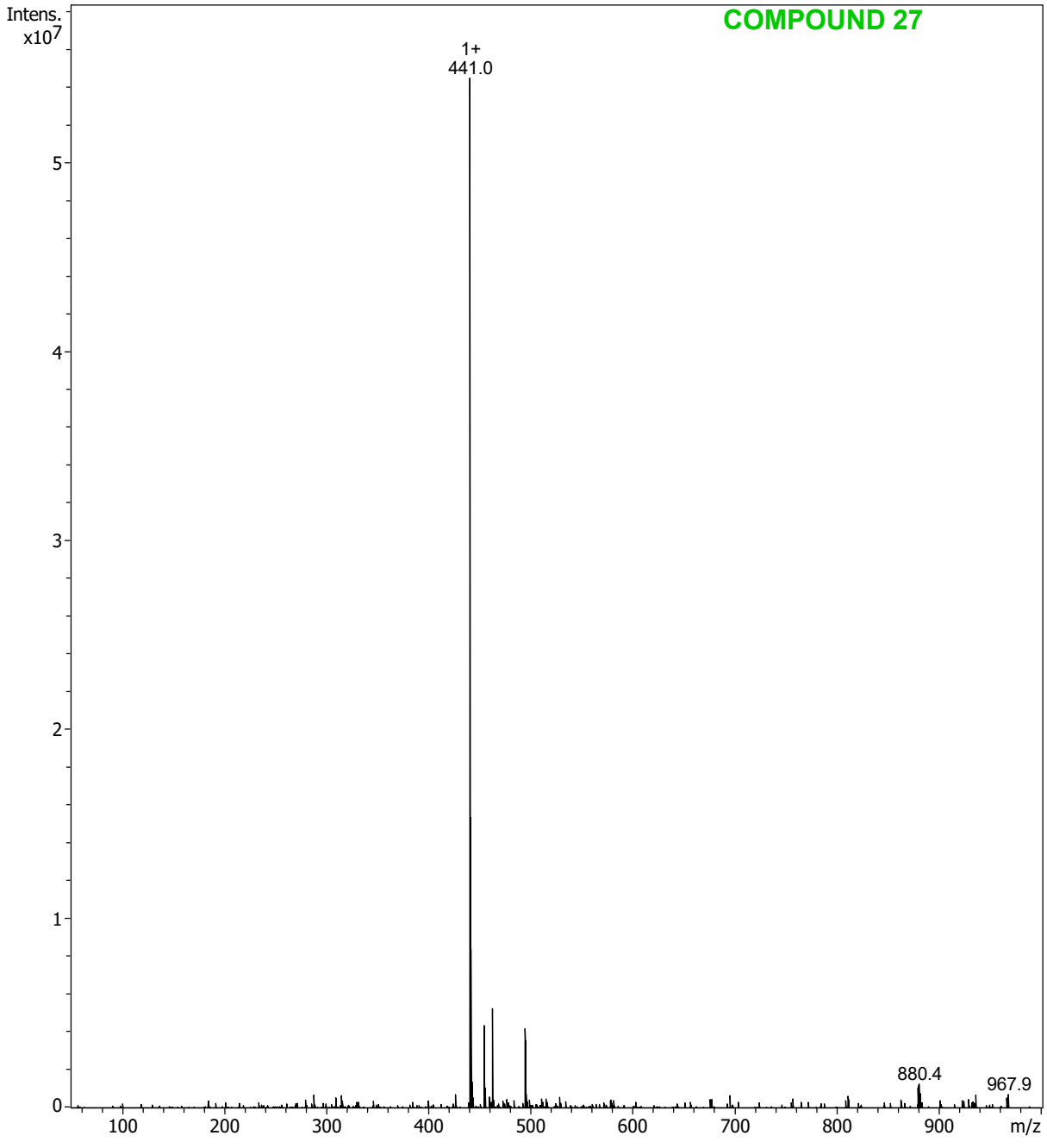
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 11 ESI MF3-CM-190 439_54_01_1150.d | Acquisition Date | 12/1/2022 2:54:01 PM |
| Method | 1150.m | Instrument | amaZon ETD |



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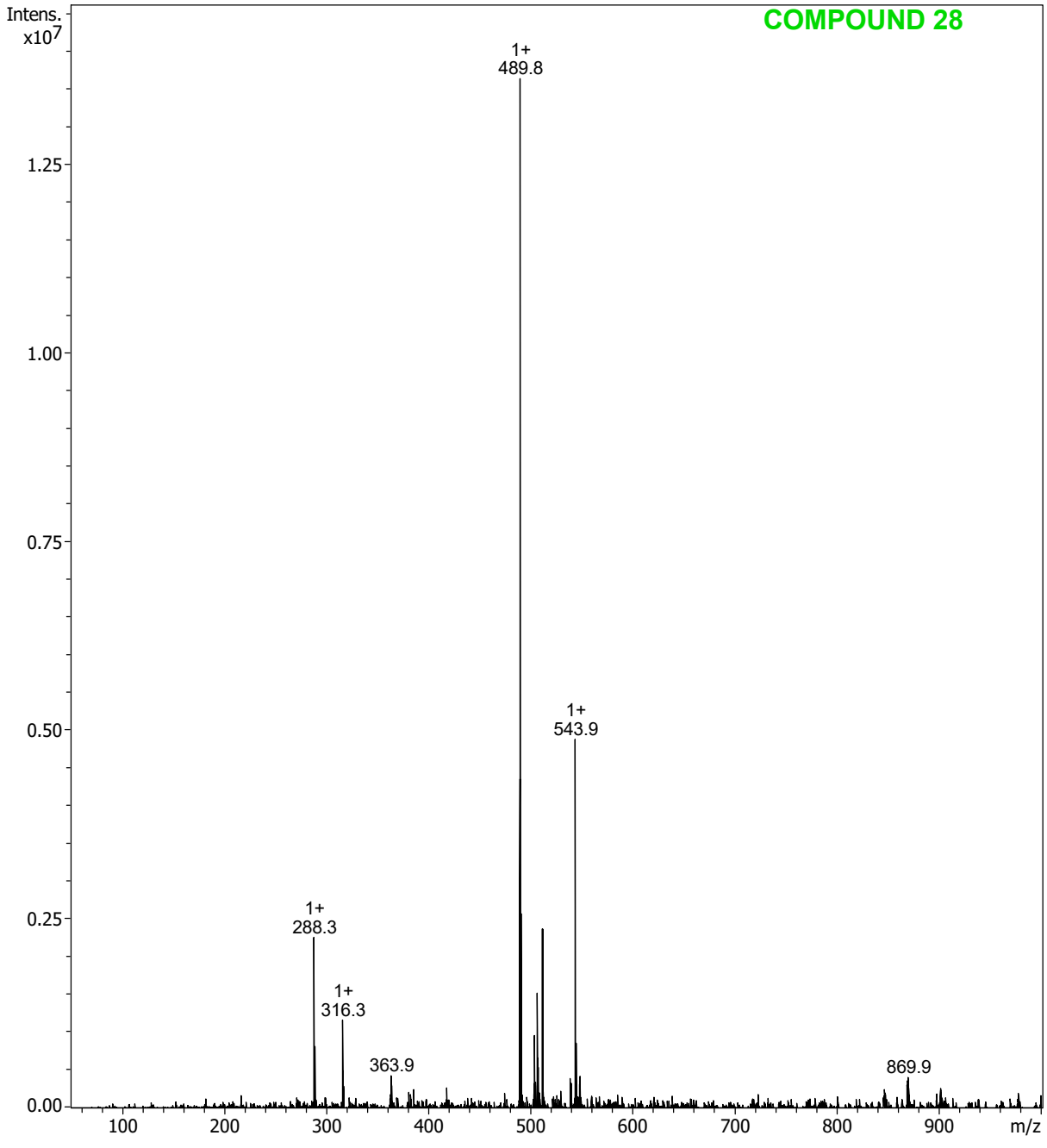
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 12 ESI MF3-CM-186 488_55_01_1151.d | Acquisition Date | 12/1/2022 3:05:06 PM |
| Method | 1151.m | Instrument | amaZon ETD |



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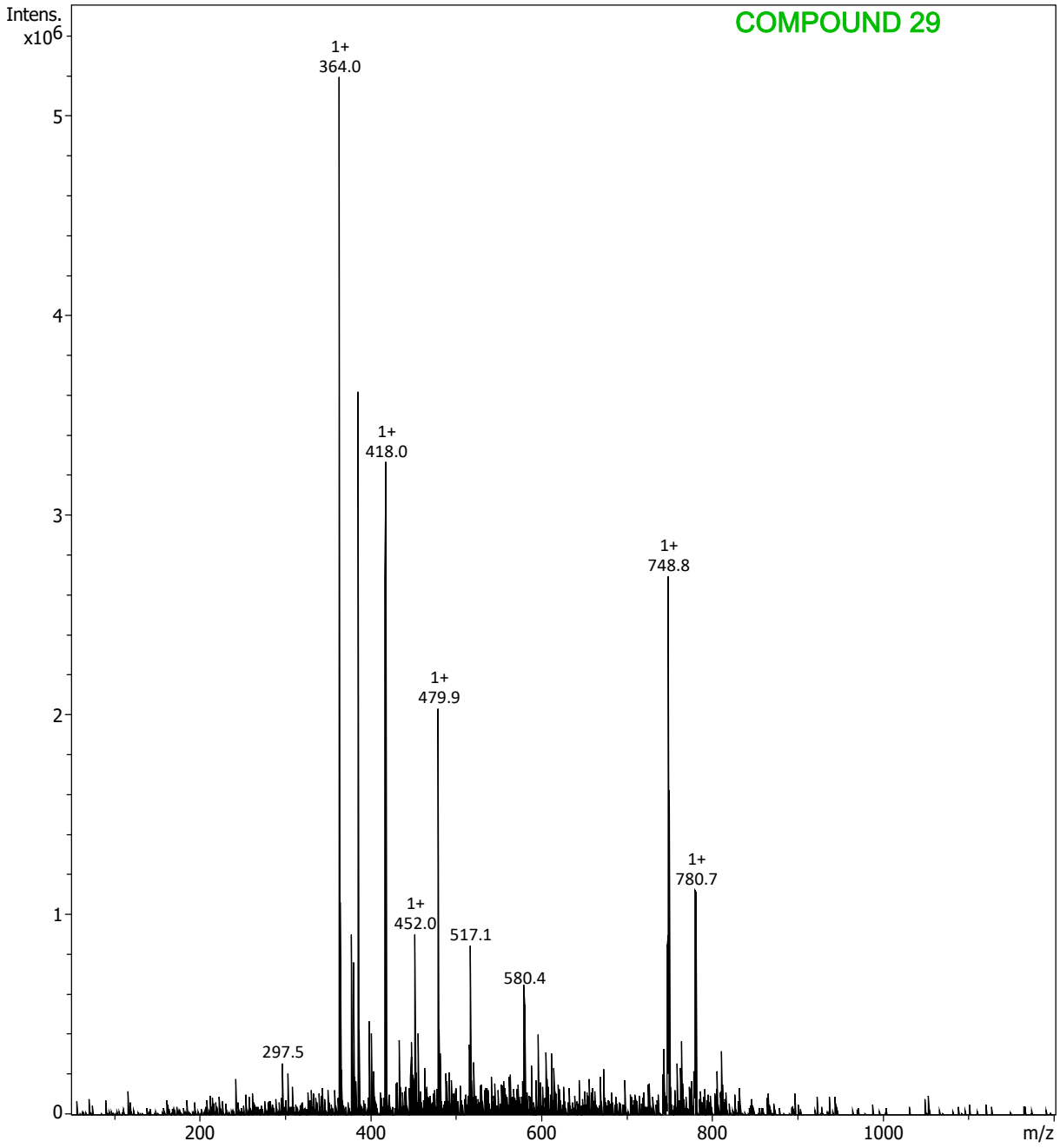
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 4 ESI MF3-CM-105 362_42_01_833.d | Acquisition Date | 04/22/2022 13:22:14 |
| Method | 833.m | Instrument | amaZon ETD |



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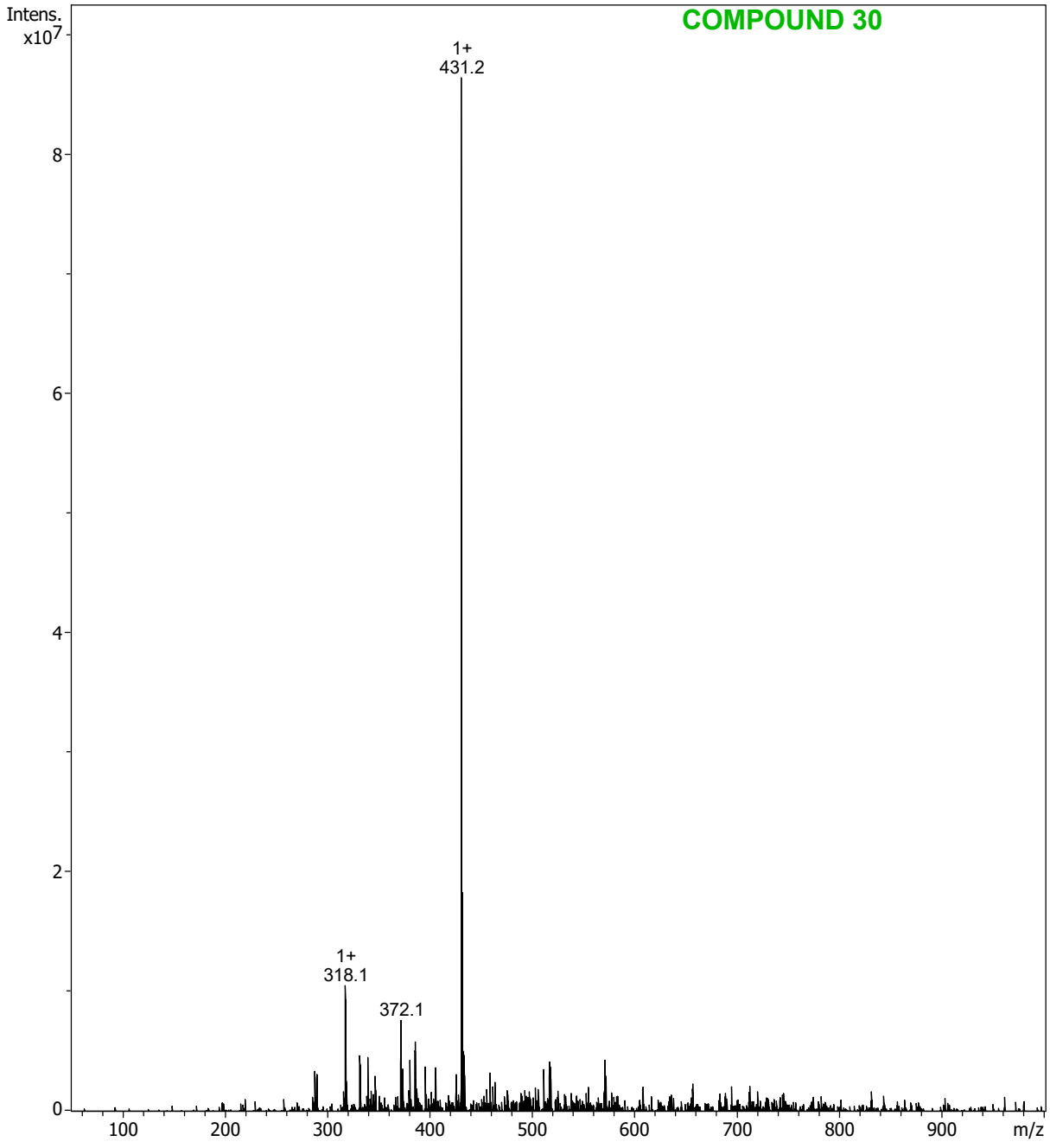
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 3 ESI MF3-CM-171 317_43_01_1187.d | Acquisition Date | 12/21/2022 13:30:09 |
| Method | 1187.m | Instrument | amaZon ETD |



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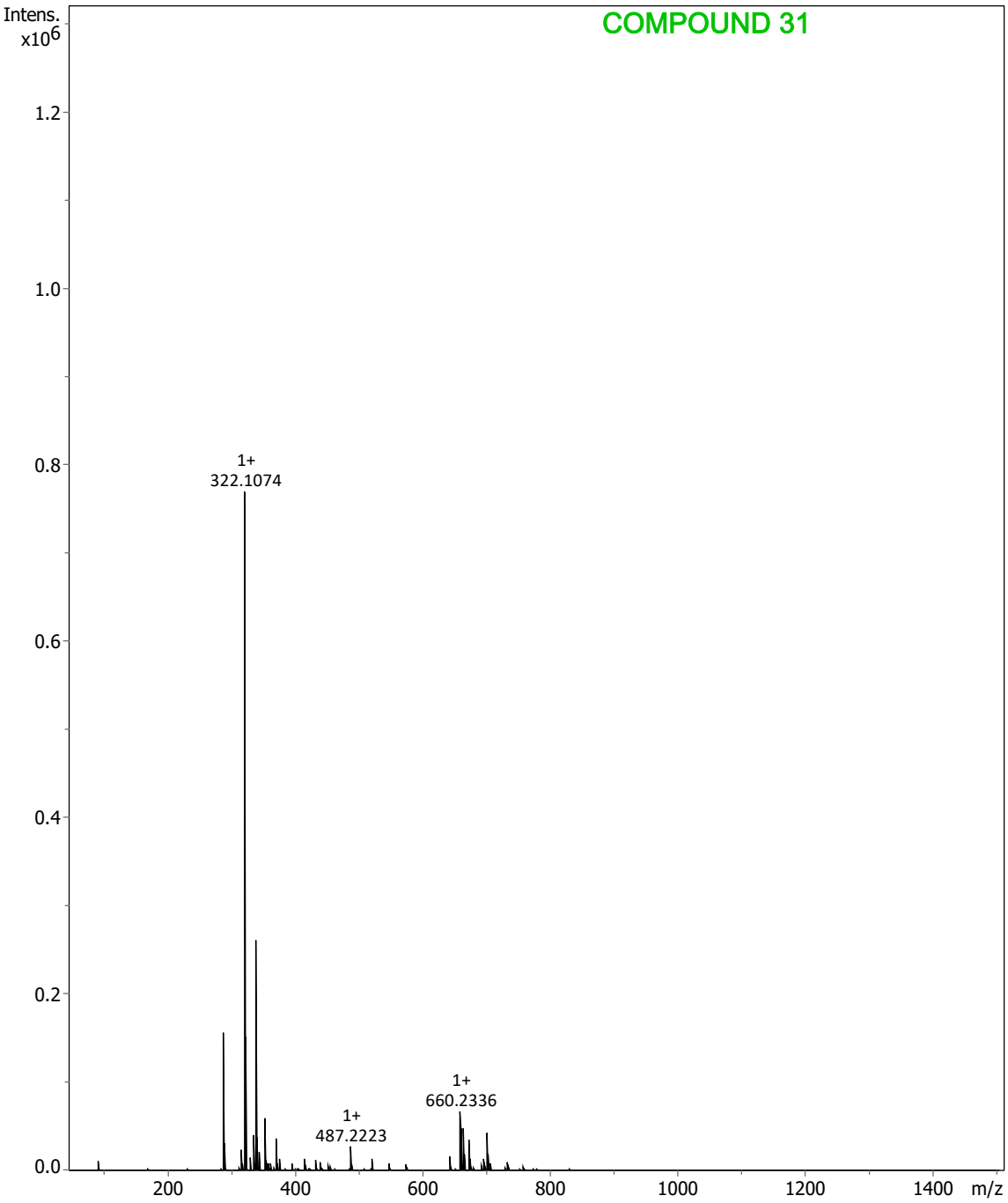
# ESPECTRO ESI-FIA-TOF

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 16 ESI MF3-CM-164 321_362_1_3271.d | Acquisition Date | 05/13/2022 13:08:38 |
| Method | FIA ESI POSITIVO 50-1500.m | Instrument | impact II |



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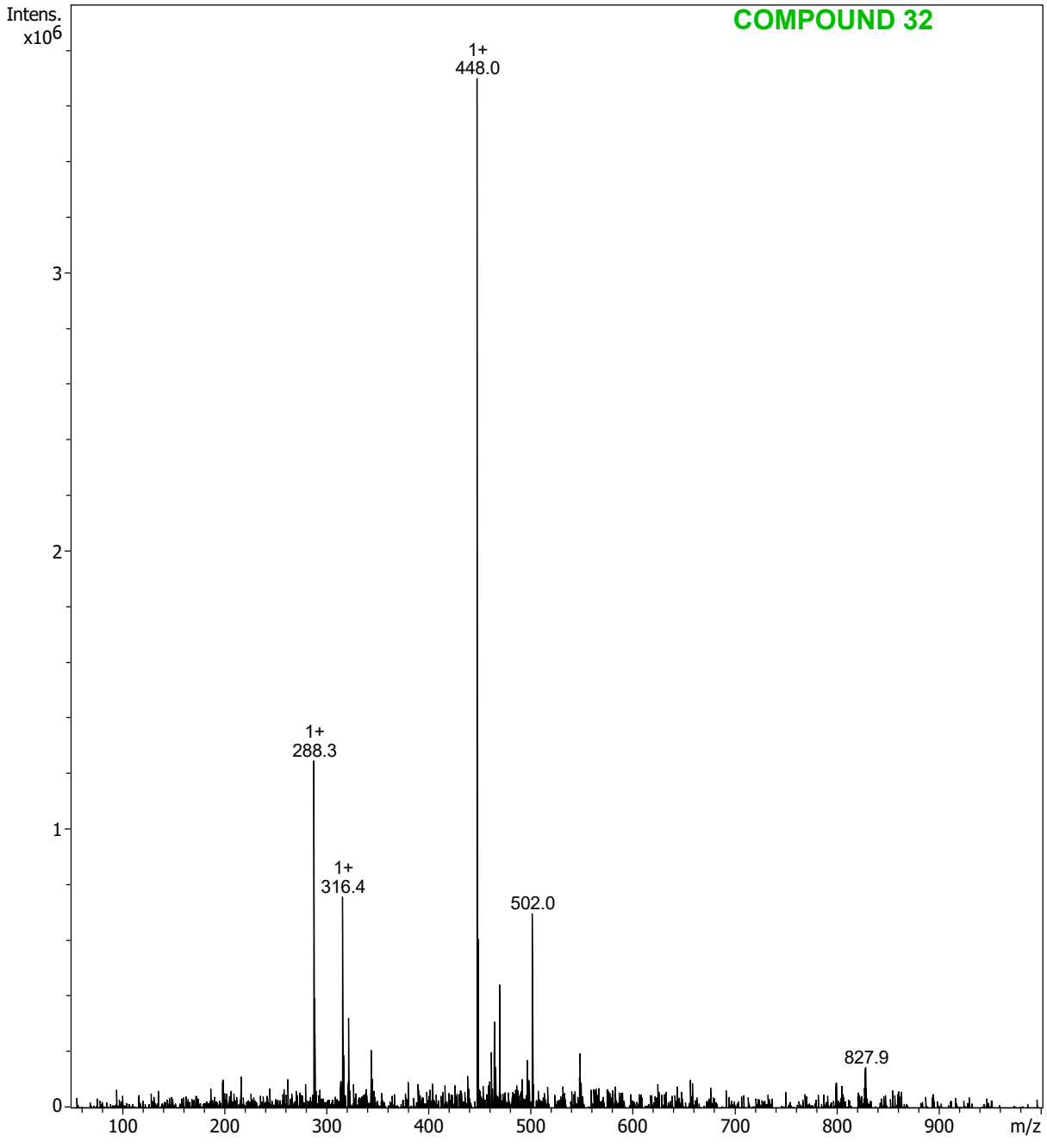
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 14 ESI MF3-CM-188A 447_56_01_1153.d | Acquisition Date | 12/1/2022 3:27:17 PM |
| Method | 1153.m | Instrument | amaZon ETD |



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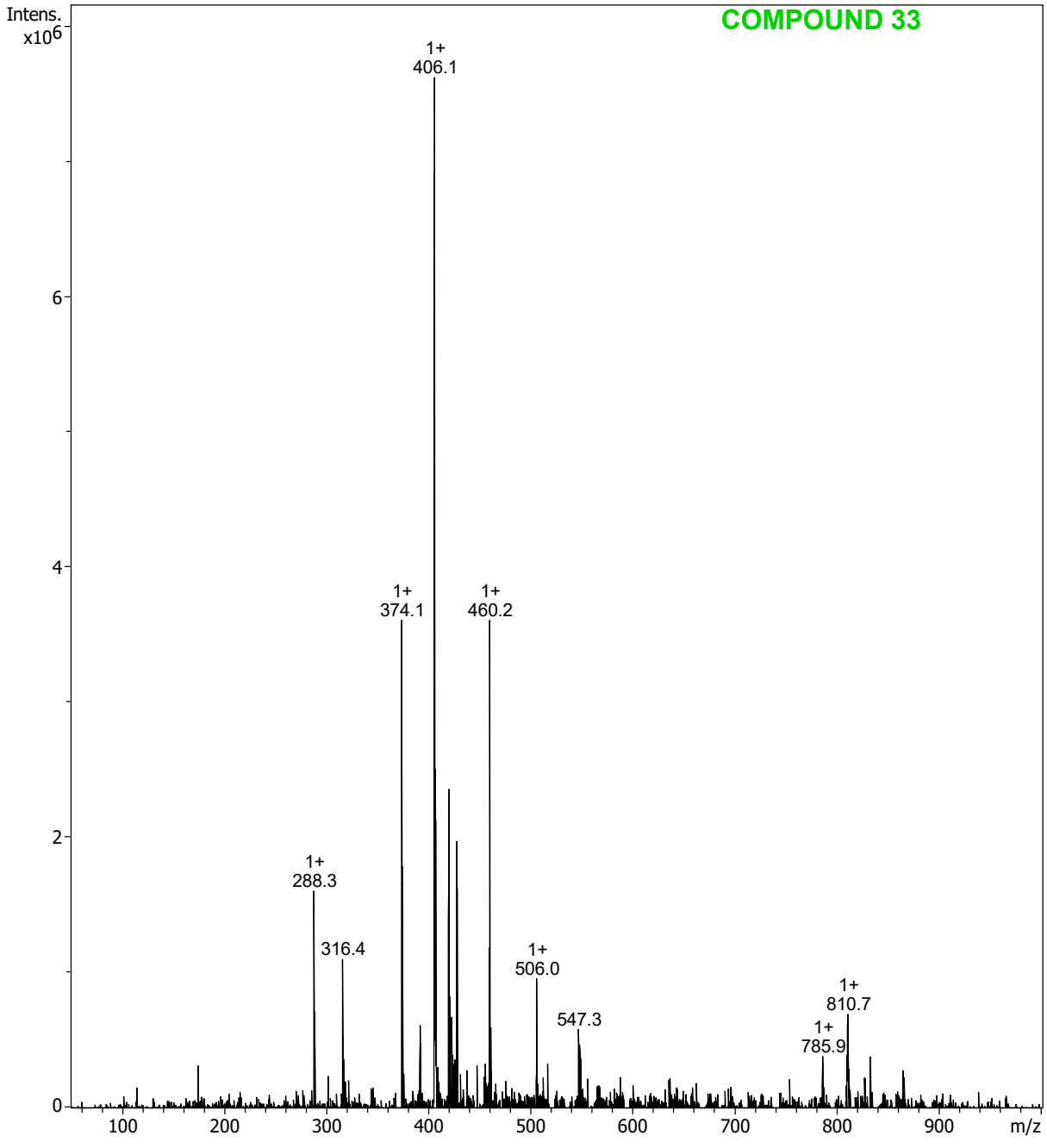
# ESPECTRO ESI-FIA-Ion Trap

**Analysis Info**

| | | | |
|---|---|---|---|
| Sample Name | 16 ESI MF3-CM-188b 446_58_01_1155.d | Acquisition Date | 12/1/2022 3:49:27 PM |
| Method | 1155.m | Instrument | amaZon ETD |



# 5. X-ray crystalographic data of compound 17

## checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 22FMB001

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

## Datablock: 22FMB001

---

```
Bond precision:   C-C = 0.0012 A          Wavelength=0.71073

Cell:           a=6.6703(4)       b=10.0801(6)      c=16.8273(10)
                alpha=83.649(2)   beta=86.849(2)    gamma=74.054(2)
Temperature:    100 K

                Calculated                Reported
Volume          1080.85(11)               1080.85(11)
Space group     P -1                      P -1
Hall group      -P 1                      -P 1
Moiety formula  C19 H23 F3 I N3 O3        C19 H23 F3 I N3 O3
Sum formula     C19 H23 F3 I N3 O3        C19 H23 F3 I N3 O3
Mr              525.30                    525.30
Dx,g cm-3       1.614                     1.614
Z               2                         2
Mu (mm-1)       1.531                     1.531
F000            524.0                     524.0
F000'           523.32
h,k,lmax        11,17,30                  11,17,30
Nref            12881                     12861
Tmin,Tmax       0.745,0.908               0.850,0.910
Tmin'           0.716

Correction method= # Reported T Limits: Tmin=0.850 Tmax=0.910
AbsCorr = MULTI-SCAN

Data completeness= 0.998          Theta(max)= 39.390

R(reflections)= 0.0200( 11865)                wR2(reflections)=
                                              0.0476( 12861)
S = 1.053                 Npar= 331
```

```
The following ALERTS were generated. Each ALERT has the format
      test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.
```

**Alert level C**

```
PLAT220_ALERT_2_C NonSolvent   Resd 1  C   Ueq(max)/Ueq(min) Range        3.1 Ratio
```

**Alert level G**

```
PLAT154_ALERT_1_G The s.u.'s on the Cell Angles are Equal ..(Note)      0.002 Degree
PLAT164_ALERT_4_G Nr. of Refined C-H H-Atoms in Heavy-Atom Struct.         21 Note
PLAT793_ALERT_4_G Model has Chirality at C8          (Centro SPGR)          R Verify
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          2 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         17 Note
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.         11 Info
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   1 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   6 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   2 ALERT type 2 Indicator that the structure model may be wrong or deficient
   1 ALERT type 3 Indicator that the structure quality may be low
   3 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

### Validation response form

Please find below a validation response form (VRF) that can be filled in and pasted into your CIF.

```
# start Validation Reply Form
_vrf_PLAT220_22FMB001
;
PROBLEM: NonSolvent   Resd 1  C   Ueq(max)/Ueq(min) Range        3.1 Ratio
RESPONSE: ...
;
# end Validation Reply Form
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

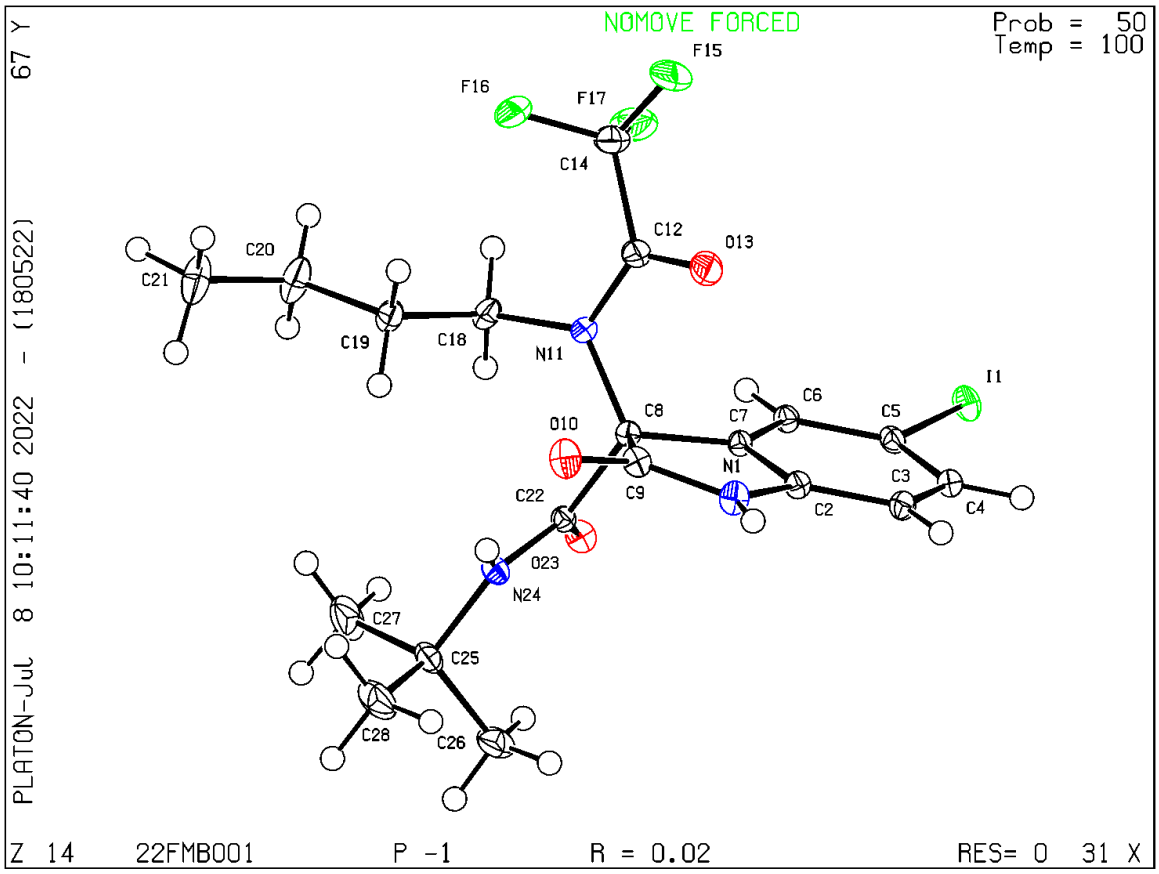
**Publication of your CIF in other journals**

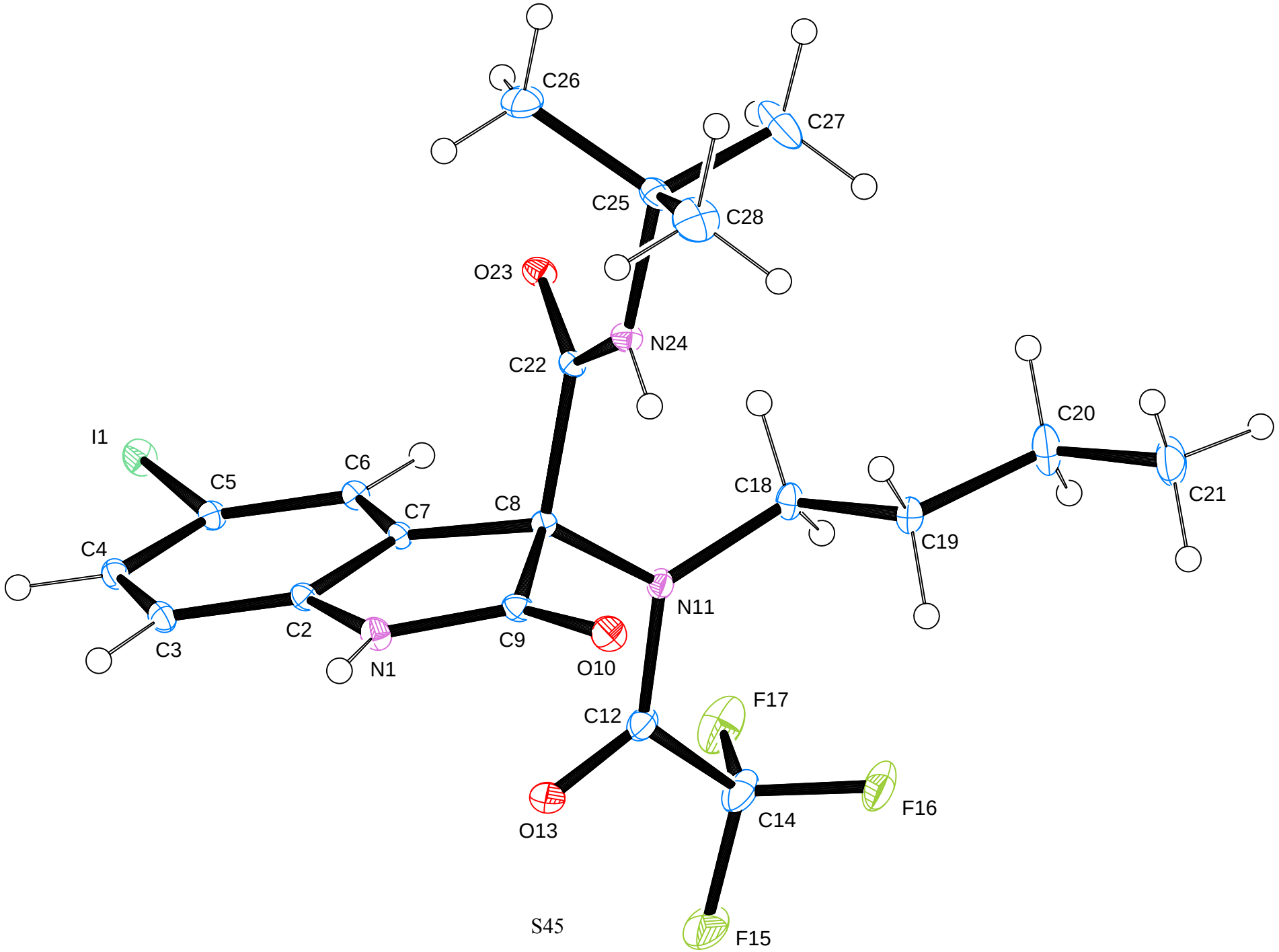
Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

---

**PLATON version of 18/05/2022; check.def file version of 17/05/2022**

**Datablock 22FMB001** - ellipsoid plot




C26
C27
C25
C28
O23
N24
C22
I1
C5
C6
C7
C8
C18
C20
C21
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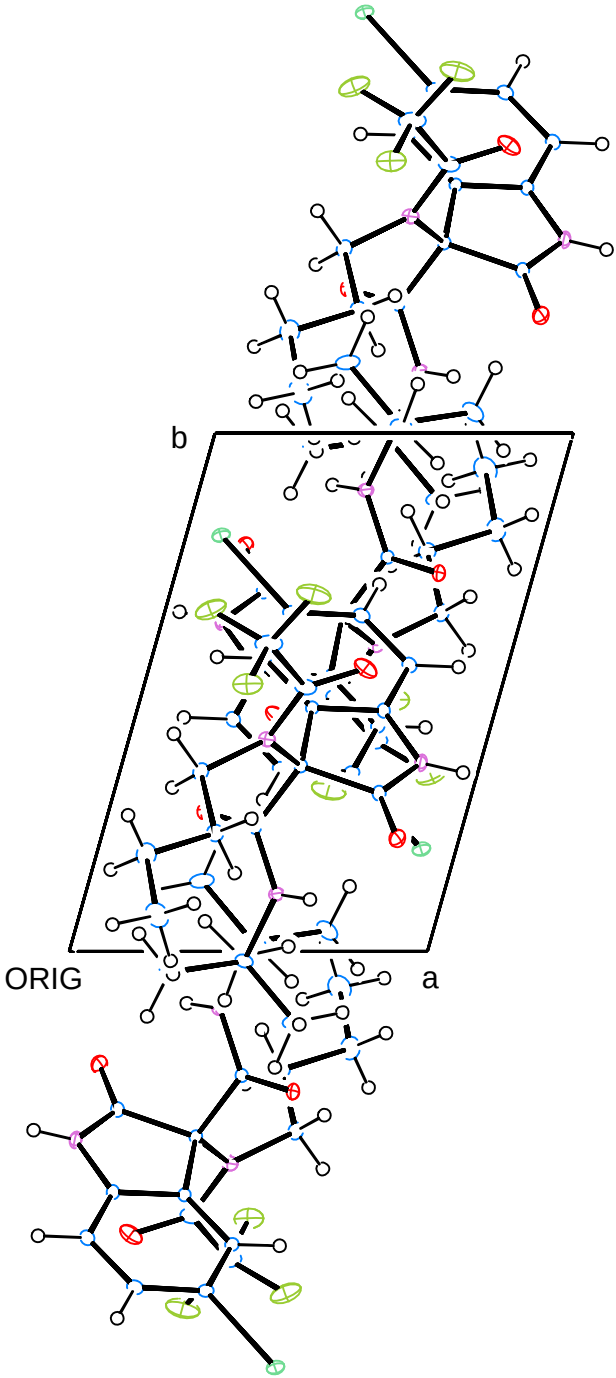
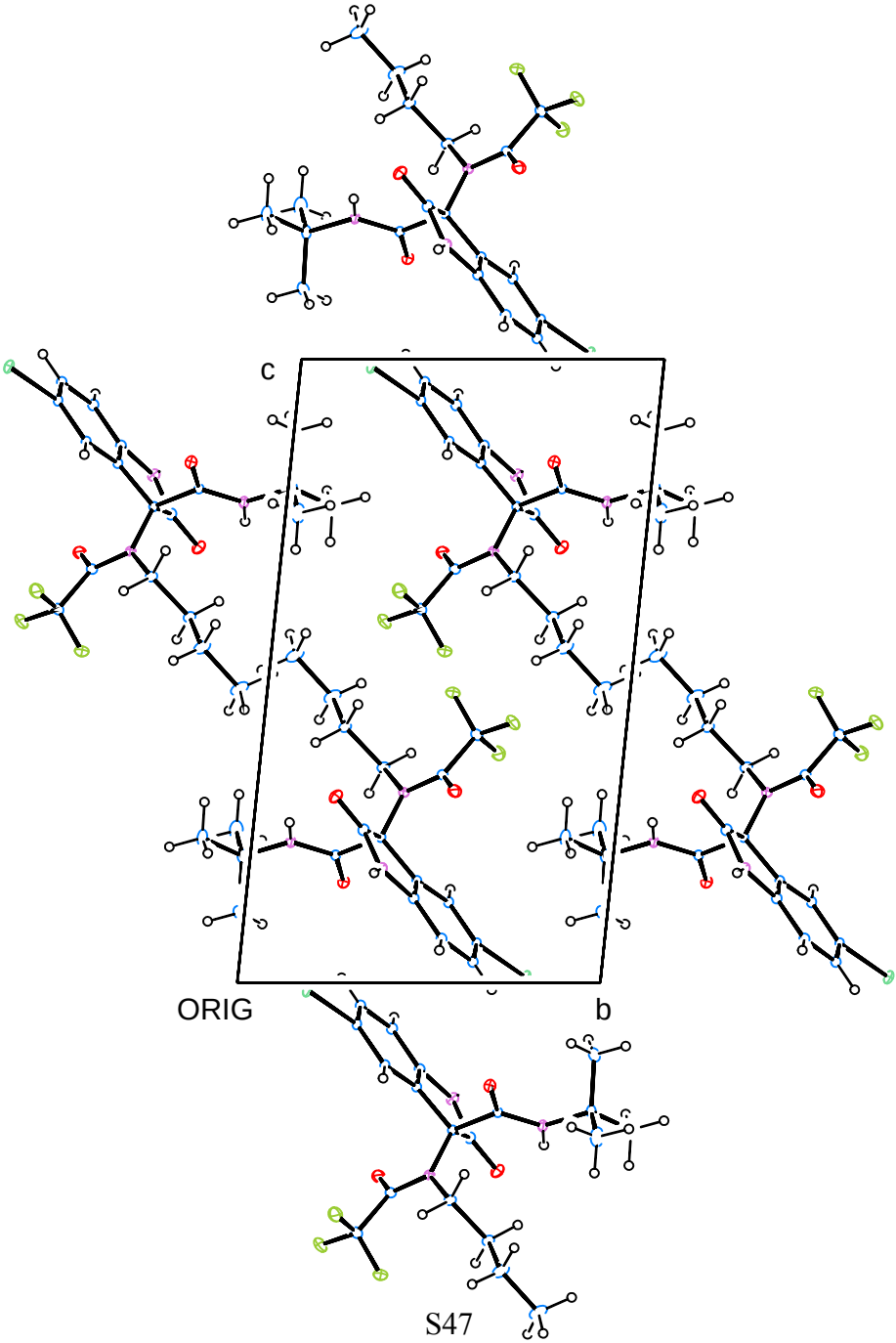
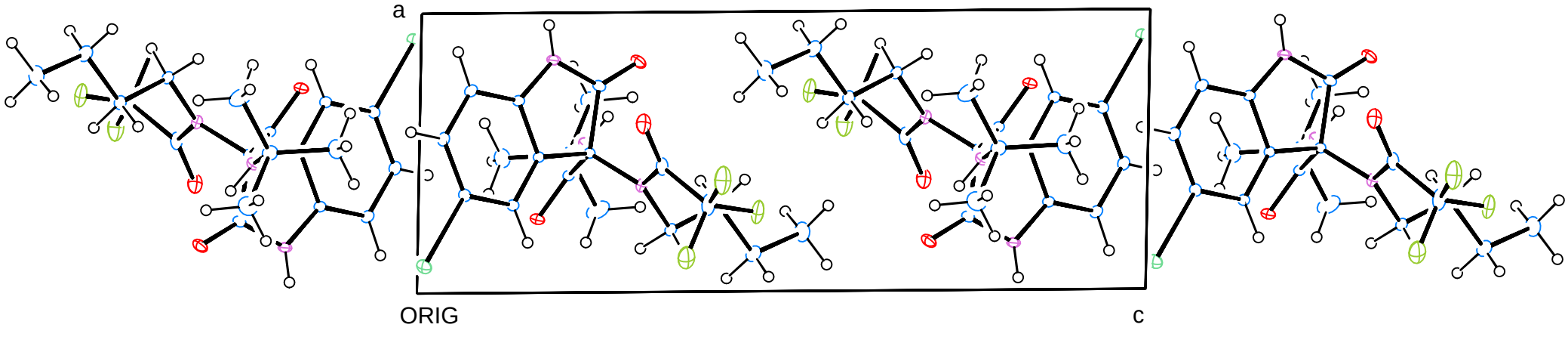
C4
N11
C2
C3
C9
N1
O10
C12
F17
O13
C14
F16
F15


b
ORIG
a


c
ORIG
b


a
ORIG
c

# Title

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## Abstract

## Related literature

## Computing details

Data collection: Bruker *APEX4* software; cell refinement: *SAINT* V8.40B (Bruker AXS LLC, 2019); data reduction: *SAINT* V8.40B (Bruker AXS LLC, 2019); program(s) used to solve structure: *SHELXT* 2018/2 (Sheldrick, 2015); program(s) used to refine structure: *SHELXL2019*/1 (Sheldrick, 2019); molecular graphics: *ORTEP* 2014.1 (Farrugia, 2012); software used to prepare material for publication: IUCr Journals printCIF.

## Acknowledgements

## Funding information

## References

## Figure 1

## (22FMB001)

*Crystal data*

$C_{19}H_{23}F_3IN_3O_3$
$M_r$ = 525.30
Triclinic, $P\overline{1}$
$a$ = 6.6703 (4) Å
$b$ = 10.0801 (6) Å
$c$ = 16.8273 (10) Å
$\alpha$ = 83.649 (2)°
$\beta$ = 86.849 (2)°
$\gamma$ = 74.054 (2)°
$V$ = 1080.85 (11) Å$^3$
$Z$ = 2
$F(000)$ = 524
$D_x$ = 1.614 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9733 reflections
$\theta$ = 2.3–39.3°
$\mu$ = 1.53 mm$^{-1}$
$T$ = 100 K
Block, clear colourless
0.21 × 0.17 × 0.06 mm

*Data collection*

Bruker D8 VENTURE PHOTON-III C14 diffractometer
Radiation source: microfocus sealed tube, Incoatec I$\mu$S 3.0
Multilayer mirror monochromator
Detector resolution: 7.3910 pixels mm$^{-1}$
$\varphi$ or $\omega$ oscillation scans
Absorption correction: multi-scan Krause, L., Herbst-Irmer, R., Sheldrick, G. M., Stalke, D. (2015). "Comparison of silver and molybdenum microfocus X-ray sources for single-crystal structure determination" J. Appl. Cryst. 48, 3-10. doi:10.1107/S1600576714022985
$T_{min}$ = 0.85, $T_{max}$ = 0.91
112325 measured reflections
12861 independent reflections
11865 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036
$\theta_{max}$ = 39.4°, $\theta_{min}$ = 2.3°

$h = -11 \rightarrow 11$
$k = -17 \rightarrow 17$
$l = -30 \rightarrow 30$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.048$
$S = 1.05$
12861 reflections
331 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0198P)^2 + 0.3032P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.66$ e Å$^{-3}$
$\Delta\rho_{min} = -0.70$ e Å$^{-3}$

*Special details*

*Geometry*. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| I1 | 0.09709 (2) | 0.80277 (2) | 0.01168 (2) | 0.01746 (10) |
| N1 | 0.83216 (10) | 0.36529 (7) | 0.18333 (4) | 0.01473 (10) |
| H1 | 0.957 (2) | 0.3465 (14) | 0.1762 (8) | 0.018000* |
| C2 | 0.68732 (11) | 0.46564 (7) | 0.13636 (4) | 0.01198 (10) |
| C3 | 0.72412 (12) | 0.55300 (8) | 0.07117 (5) | 0.01402 (11) |
| H3 | 0.863 (2) | 0.5490 (14) | 0.0526 (8) | 0.017000* |
| C4 | 0.55197 (12) | 0.64821 (7) | 0.03486 (4) | 0.01401 (11) |
| H4 | 0.578 (2) | 0.7077 (14) | −0.0099 (8) | 0.017000* |
| C5 | 0.35109 (11) | 0.65516 (7) | 0.06533 (4) | 0.01265 (10) |
| C6 | 0.31411 (11) | 0.56671 (7) | 0.13132 (4) | 0.01197 (10) |
| H6 | 0.180 (2) | 0.5693 (13) | 0.1525 (8) | 0.014000* |
| C7 | 0.48538 (10) | 0.47110 (7) | 0.16559 (4) | 0.01058 (9) |
| C8 | 0.49842 (10) | 0.35981 (7) | 0.23472 (4) | 0.01074 (10) |
| C9 | 0.73856 (11) | 0.30716 (8) | 0.24616 (5) | 0.01344 (11) |
| O10 | 0.82396 (10) | 0.22072 (7) | 0.29922 (4) | 0.01859 (10) |
| N11 | 0.38239 (10) | 0.41181 (7) | 0.30704 (4) | 0.01345 (10) |
| C12 | 0.44897 (15) | 0.51293 (8) | 0.33515 (5) | 0.01823 (13) |
| O13 | 0.60186 (12) | 0.54880 (7) | 0.30875 (4) | 0.02351 (13) |
| C14 | 0.31135 (19) | 0.59693 (10) | 0.39980 (6) | 0.02614 (18) |
| F15 | 0.39814 (15) | 0.69233 (7) | 0.41920 (4) | 0.03885 (18) |
| F16 | 0.28382 (12) | 0.51971 (7) | 0.46738 (4) | 0.03041 (14) |
| F17 | 0.12288 (13) | 0.66281 (7) | 0.37105 (4) | 0.03554 (16) |
| C18 | 0.22267 (12) | 0.34978 (8) | 0.34804 (5) | 0.01546 (12) |
| H18A | 0.156 (2) | 0.3187 (14) | 0.3066 (8) | 0.019000* |
| H18B | 0.118 (2) | 0.4229 (14) | 0.3712 (8) | 0.019000* |
| C19 | 0.31038 (13) | 0.23197 (8) | 0.41248 (5) | 0.01657 (12) |
| H19A | 0.396 (2) | 0.1545 (15) | 0.3872 (8) | 0.020000* |
| H19B | 0.400 (2) | 0.2600 (15) | 0.4481 (8) | 0.020000* |
| C20 | 0.13447 (16) | 0.19076 (12) | 0.46165 (6) | 0.02675 (18) |
| H20A | 0.052 (3) | 0.2669 (17) | 0.4832 (10) | 0.032000* |
| H20B | 0.047 (2) | 0.1615 (17) | 0.4250 (10) | 0.032000* |

| | | | | |
|---|---|---|---|---|
| C21 | 0.2113 (2) | 0.07467 (13) | 0.52753 (7) | 0.0312 (2) |
| H21A | 0.285 (3) | −0.014 (2) | 0.5037 (11) | 0.047000* |
| H21B | 0.094 (3) | 0.0567 (19) | 0.5619 (11) | 0.047000* |
| H21C | 0.305 (3) | 0.097 (2) | 0.5626 (11) | 0.047000* |
| C22 | 0.41867 (10) | 0.24117 (7) | 0.20629 (4) | 0.01098 (10) |
| O23 | 0.26339 (8) | 0.27194 (6) | 0.16440 (4) | 0.01470 (9) |
| N24 | 0.53178 (10) | 0.11250 (6) | 0.22777 (4) | 0.01359 (10) |
| H24 | 0.629 (2) | 0.1024 (14) | 0.2583 (8) | 0.016000* |
| C25 | 0.49394 (13) | −0.01688 (8) | 0.20490 (5) | 0.01723 (12) |
| C26 | 0.48183 (17) | −0.00927 (10) | 0.11394 (6) | 0.02395 (16) |
| H26A | 0.607 (3) | 0.0101 (18) | 0.0892 (10) | 0.036000* |
| H26B | 0.355 (3) | 0.0588 (18) | 0.0962 (10) | 0.036000* |
| H26C | 0.472 (3) | −0.0933 (18) | 0.1002 (10) | 0.036000* |
| C27 | 0.29397 (19) | −0.03820 (11) | 0.24637 (9) | 0.0326 (2) |
| H27A | 0.179 (3) | 0.036 (2) | 0.2309 (11) | 0.049000* |
| H27B | 0.304 (3) | −0.041 (2) | 0.3043 (12) | 0.049000* |
| H27C | 0.266 (3) | −0.123 (2) | 0.2337 (11) | 0.049000* |
| C28 | 0.68293 (18) | −0.13407 (9) | 0.23339 (7) | 0.02786 (19) |
| H28A | 0.663 (3) | −0.2220 (19) | 0.2190 (10) | 0.042000* |
| H28B | 0.697 (3) | −0.1384 (19) | 0.2908 (11) | 0.042000* |
| H28C | 0.808 (3) | −0.1160 (19) | 0.2081 (11) | 0.042000* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I1 | 0.01787 (2) | 0.01155 (2) | 0.02040 (2) | −0.00108 (10) | −0.00367 (2) | 0.00356 (10) |
| N1 | 0.0085 (2) | 0.0175 (3) | 0.0174 (3) | −0.00300 (18) | −0.00130 (19) | 0.0009 (2) |
| C2 | 0.0100 (2) | 0.0128 (2) | 0.0133 (3) | −0.00359 (19) | −0.00009 (19) | −0.0011 (2) |
| C3 | 0.0136 (3) | 0.0139 (3) | 0.0152 (3) | −0.0053 (2) | 0.0024 (2) | −0.0013 (2) |
| C4 | 0.0171 (3) | 0.0121 (2) | 0.0133 (3) | −0.0052 (2) | 0.0015 (2) | −0.0010 (2) |
| C5 | 0.0142 (3) | 0.0094 (2) | 0.0137 (3) | −0.00236 (19) | −0.0016 (2) | −0.00014 (19) |
| C6 | 0.0111 (2) | 0.0109 (2) | 0.0134 (3) | −0.00243 (19) | −0.0003 (2) | −0.0004 (2) |
| C7 | 0.0098 (2) | 0.0106 (2) | 0.0113 (2) | −0.00281 (18) | −0.00002 (18) | −0.00058 (19) |
| C8 | 0.0099 (2) | 0.0108 (2) | 0.0112 (2) | −0.00230 (18) | −0.00049 (18) | −0.00059 (19) |
| C9 | 0.0110 (2) | 0.0139 (3) | 0.0158 (3) | −0.0038 (2) | −0.0030 (2) | −0.0004 (2) |
| O10 | 0.0155 (2) | 0.0193 (2) | 0.0200 (3) | −0.00409 (19) | −0.00721 (19) | 0.0045 (2) |
| N11 | 0.0165 (2) | 0.0126 (2) | 0.0110 (2) | −0.00398 (19) | 0.00177 (19) | −0.00155 (18) |
| C12 | 0.0285 (4) | 0.0144 (3) | 0.0123 (3) | −0.0063 (3) | −0.0008 (3) | −0.0018 (2) |
| O13 | 0.0349 (4) | 0.0228 (3) | 0.0187 (3) | −0.0169 (3) | −0.0005 (2) | −0.0037 (2) |
| C14 | 0.0437 (5) | 0.0170 (3) | 0.0157 (3) | −0.0041 (3) | 0.0017 (3) | −0.0045 (3) |
| F15 | 0.0735 (6) | 0.0236 (3) | 0.0241 (3) | −0.0180 (3) | 0.0022 (3) | −0.0115 (2) |
| F16 | 0.0496 (4) | 0.0247 (3) | 0.0138 (2) | −0.0056 (3) | 0.0057 (2) | −0.0031 (2) |
| F17 | 0.0456 (4) | 0.0247 (3) | 0.0256 (3) | 0.0092 (3) | 0.0019 (3) | −0.0059 (2) |
| C18 | 0.0136 (3) | 0.0182 (3) | 0.0128 (3) | −0.0026 (2) | 0.0022 (2) | 0.0006 (2) |
| C19 | 0.0178 (3) | 0.0176 (3) | 0.0133 (3) | −0.0046 (2) | 0.0009 (2) | 0.0015 (2) |
| C20 | 0.0231 (4) | 0.0326 (5) | 0.0212 (4) | −0.0074 (3) | 0.0044 (3) | 0.0086 (3) |
| C21 | 0.0360 (5) | 0.0366 (5) | 0.0216 (4) | −0.0161 (4) | −0.0009 (4) | 0.0113 (4) |
| C22 | 0.0099 (2) | 0.0108 (2) | 0.0123 (2) | −0.00317 (18) | 0.00061 (19) | −0.00101 (19) |
| O23 | 0.00987 (19) | 0.0156 (2) | 0.0188 (2) | −0.00359 (16) | −0.00267 (17) | −0.00076 (18) |
| N24 | 0.0142 (2) | 0.0103 (2) | 0.0158 (3) | −0.00238 (18) | −0.00209 (19) | −0.00066 (18) |
| C25 | 0.0200 (3) | 0.0106 (3) | 0.0219 (3) | −0.0059 (2) | 0.0013 (3) | −0.0017 (2) |
| C26 | 0.0333 (5) | 0.0180 (3) | 0.0235 (4) | −0.0095 (3) | −0.0026 (3) | −0.0070 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C27 | 0.0308 (5) | 0.0219 (4) | 0.0482 (7) | −0.0153 (4) | 0.0141 (5) | −0.0037 (4) |
| C28 | 0.0328 (5) | 0.0119 (3) | 0.0351 (5) | 0.0003 (3) | −0.0043 (4) | −0.0006 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| I1—C5 | 2.0941 (7) | C18—H18B | 0.967 (14) |
| N1—C9 | 1.3574 (10) | C19—C20 | 1.5228 (12) |
| N1—C2 | 1.3969 (10) | C19—H19A | 0.960 (14) |
| N1—H1 | 0.807 (14) | C19—H19B | 0.983 (14) |
| C2—C3 | 1.3837 (10) | C20—C21 | 1.5189 (14) |
| C2—C7 | 1.3970 (9) | C20—H20A | 0.910 (16) |
| C3—C4 | 1.3981 (11) | C20—H20B | 0.990 (16) |
| C3—H3 | 0.954 (13) | C21—H21A | 1.007 (19) |
| C4—C5 | 1.3943 (10) | C21—H21B | 0.992 (19) |
| C4—H4 | 0.948 (13) | C21—H21C | 0.968 (19) |
| C5—C6 | 1.4002 (10) | C22—O23 | 1.2334 (9) |
| C6—C7 | 1.3824 (10) | C22—N24 | 1.3306 (9) |
| C6—H6 | 0.940 (13) | N24—C25 | 1.4884 (10) |
| C7—C8 | 1.5127 (10) | N24—H24 | 0.824 (13) |
| C8—N11 | 1.4728 (9) | C25—C27 | 1.5279 (13) |
| C8—C9 | 1.5584 (10) | C25—C28 | 1.5288 (13) |
| C8—C22 | 1.5639 (9) | C25—C26 | 1.5295 (13) |
| C9—O10 | 1.2214 (9) | C26—H26A | 0.971 (17) |
| N11—C12 | 1.3559 (10) | C26—H26B | 0.971 (17) |
| N11—C18 | 1.4805 (10) | C26—H26C | 0.924 (17) |
| C12—O13 | 1.2188 (11) | C27—H27A | 0.94 (2) |
| C12—C14 | 1.5523 (12) | C27—H27B | 0.98 (2) |
| C14—F15 | 1.3277 (13) | C27—H27C | 0.975 (19) |
| C14—F17 | 1.3388 (14) | C28—H28A | 0.989 (18) |
| C14—F16 | 1.3389 (11) | C28—H28B | 0.971 (18) |
| C18—C19 | 1.5269 (11) | C28—H28C | 0.969 (18) |
| C18—H18A | 0.970 (14) | | |

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| C9—N1—C2 | 111.75 (6) | C20—C19—C18 | 110.60 (7) |
| C9—N1—H1 | 123.2 (10) | C20—C19—H19A | 110.6 (8) |
| C2—N1—H1 | 125.0 (10) | C18—C19—H19A | 108.8 (8) |
| C3—C2—N1 | 128.49 (7) | C20—C19—H19B | 109.3 (8) |
| C3—C2—C7 | 121.66 (7) | C18—C19—H19B | 110.5 (8) |
| N1—C2—C7 | 109.83 (6) | H19A—C19—H19B | 107.1 (11) |
| C2—C3—C4 | 117.91 (7) | C21—C20—C19 | 113.28 (9) |
| C2—C3—H3 | 120.6 (8) | C21—C20—H20A | 109.8 (10) |
| C4—C3—H3 | 121.5 (8) | C19—C20—H20A | 108.7 (10) |
| C5—C4—C3 | 120.16 (7) | C21—C20—H20B | 109.4 (9) |
| C5—C4—H4 | 122.2 (8) | C19—C20—H20B | 108.3 (9) |
| C3—C4—H4 | 117.7 (8) | H20A—C20—H20B | 107.2 (14) |
| C4—C5—C6 | 121.85 (7) | C20—C21—H21A | 110.2 (11) |
| C4—C5—I1 | 119.27 (5) | C20—C21—H21B | 111.4 (11) |
| C6—C5—I1 | 118.88 (5) | H21A—C21—H21B | 107.8 (15) |
| C7—C6—C5 | 117.37 (6) | C20—C21—H21C | 111.8 (11) |
| C7—C6—H6 | 119.5 (8) | H21A—C21—H21C | 109.1 (15) |
| C5—C6—H6 | 123.1 (8) | H21B—C21—H21C | 106.3 (15) |
| C6—C7—C2 | 121.02 (6) | O23—C22—N24 | 125.00 (7) |

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| C6—C7—C8 | 130.44 (6) | O23—C22—C8 | 119.06 (6) |
| C2—C7—C8 | 108.54 (6) | N24—C22—C8 | 115.90 (6) |
| N11—C8—C7 | 113.38 (6) | C22—N24—C25 | 125.99 (7) |
| N11—C8—C9 | 113.12 (6) | C22—N24—H24 | 117.7 (9) |
| C7—C8—C9 | 101.34 (5) | C25—N24—H24 | 116.2 (9) |
| N11—C8—C22 | 110.95 (6) | N24—C25—C27 | 109.74 (7) |
| C7—C8—C22 | 108.31 (5) | N24—C25—C28 | 105.71 (7) |
| C9—C8—C22 | 109.25 (5) | C27—C25—C28 | 110.65 (8) |
| O10—C9—N1 | 127.07 (7) | N24—C25—C26 | 109.87 (7) |
| O10—C9—C8 | 125.08 (7) | C27—C25—C26 | 111.30 (9) |
| N1—C9—C8 | 107.62 (6) | C28—C25—C26 | 109.42 (8) |
| C12—N11—C8 | 113.23 (6) | C25—C26—H26A | 109.4 (10) |
| C12—N11—C18 | 124.07 (7) | C25—C26—H26B | 109.6 (10) |
| C8—N11—C18 | 122.48 (6) | H26A—C26—H26B | 113.2 (14) |
| O13—C12—N11 | 124.06 (8) | C25—C26—H26C | 109.0 (10) |
| O13—C12—C14 | 117.79 (8) | H26A—C26—H26C | 109.5 (14) |
| N11—C12—C14 | 117.93 (8) | H26B—C26—H26C | 106.0 (14) |
| F15—C14—F17 | 107.65 (8) | C25—C27—H27A | 110.7 (12) |
| F15—C14—F16 | 107.35 (8) | C25—C27—H27B | 109.8 (11) |
| F17—C14—F16 | 107.85 (9) | H27A—C27—H27B | 107.1 (16) |
| F15—C14—C12 | 109.69 (9) | C25—C27—H27C | 111.9 (11) |
| F17—C14—C12 | 110.10 (8) | H27A—C27—H27C | 108.0 (16) |
| F16—C14—C12 | 113.97 (7) | H27B—C27—H27C | 109.2 (16) |
| N11—C18—C19 | 113.67 (6) | C25—C28—H28A | 107.9 (10) |
| N11—C18—H18A | 106.4 (8) | C25—C28—H28B | 110.8 (11) |
| C19—C18—H18A | 111.5 (8) | H28A—C28—H28B | 110.1 (15) |
| N11—C18—H18B | 107.8 (8) | C25—C28—H28C | 109.1 (11) |
| C19—C18—H18B | 110.1 (8) | H28A—C28—H28C | 111.5 (14) |
| H18A—C18—H18B | 107.0 (11) | H28B—C28—H28C | 107.5 (15) |

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| C9—N1—C2—C3 | 175.13 (7) | C9—C8—N11—C12 | 55.44 (8) |
| C9—N1—C2—C7 | −3.42 (9) | C22—C8—N11—C12 | 178.62 (6) |
| N1—C2—C3—C4 | −178.33 (7) | C7—C8—N11—C18 | 125.90 (7) |
| C7—C2—C3—C4 | 0.06 (11) | C9—C8—N11—C18 | −119.43 (7) |
| C2—C3—C4—C5 | 1.28 (11) | C22—C8—N11—C18 | 3.75 (9) |
| C3—C4—C5—C6 | −1.31 (11) | C8—N11—C12—O13 | −6.82 (12) |
| C3—C4—C5—I1 | 178.23 (5) | C18—N11—C12—O13 | 167.96 (8) |
| C4—C5—C6—C7 | −0.06 (10) | C8—N11—C12—C14 | 167.56 (7) |
| I1—C5—C6—C7 | −179.60 (5) | C18—N11—C12—C14 | −17.66 (11) |
| C5—C6—C7—C2 | 1.42 (10) | O13—C12—C14—F15 | −4.22 (12) |
| C5—C6—C7—C8 | −177.99 (7) | N11—C12—C14—F15 | −178.96 (8) |
| C3—C2—C7—C6 | −1.46 (11) | O13—C12—C14—F17 | 114.07 (10) |
| N1—C2—C7—C6 | 177.21 (6) | N11—C12—C14—F17 | −60.67 (11) |
| C3—C2—C7—C8 | 178.07 (6) | O13—C12—C14—F16 | −124.61 (10) |
| N1—C2—C7—C8 | −3.27 (8) | N11—C12—C14—F16 | 60.65 (12) |
| C6—C7—C8—N11 | −51.43 (10) | C12—N11—C18—C19 | −84.56 (9) |
| C2—C7—C8—N11 | 129.10 (6) | C8—N11—C18—C19 | 89.74 (8) |
| C6—C7—C8—C9 | −172.97 (7) | N11—C18—C19—C20 | 170.01 (7) |
| C2—C7—C8—C9 | 7.57 (7) | C18—C19—C20—C21 | −179.43 (9) |
| C6—C7—C8—C22 | 72.17 (9) | N11—C8—C22—O23 | 83.48 (8) |
| C2—C7—C8—C22 | −107.29 (6) | C7—C8—C22—O23 | −41.57 (8) |
| C2—N1—C9—O10 | −176.98 (8) | C9—C8—C22—O23 | −151.13 (7) |

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| C2—N1—C9—C8 | 8.36 (8) | N11—C8—C22—N24 | −98.81 (7) |
| N11—C8—C9—O10 | 53.96 (10) | C7—C8—C22—N24 | 136.14 (6) |
| C7—C8—C9—O10 | 175.67 (7) | C9—C8—C22—N24 | 26.58 (8) |
| C22—C8—C9—O10 | −70.16 (9) | O23—C22—N24—C25 | 0.64 (12) |
| N11—C8—C9—N1 | −131.25 (6) | C8—C22—N24—C25 | −176.91 (7) |
| C7—C8—C9—N1 | −9.54 (7) | C22—N24—C25—C27 | −69.02 (11) |
| C22—C8—C9—N1 | 104.63 (7) | C22—N24—C25—C28 | 171.63 (8) |
| C7—C8—N11—C12 | −59.23 (8) | C22—N24—C25—C26 | 53.66 (10) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O23$^{i}$ | 0.807 (14) | 1.981 (14) | 2.7819 (8) | 171.6 (14) |
| C18—H18*A*···O23 | 0.970 (14) | 2.518 (14) | 3.2505 (10) | 132.3 (10) |
| C18—H18*B*···F17 | 0.967 (14) | 2.428 (14) | 3.1045 (11) | 126.7 (10) |
| N24—H24···O10 | 0.824 (13) | 2.175 (13) | 2.8548 (9) | 139.8 (12) |
| C26—H26*B*···O23 | 0.971 (17) | 2.460 (17) | 3.0011 (11) | 114.9 (12) |

Symmetry code: (i) *x*+1, *y*, *z*.