

DESIGN AND SYNTHESIS OF CXCR4-SPECIFIC TRACERS FOR POSITRON EMISSION TOMOGRAPHY

—SUPPLEMENTARY INFORMATION—

A thesis submitted by

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in partial fulfilment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Comprehensive Cancer Imaging Centre

Department of Surgery and Oncology

Hammersmith Hospital Campus

Du Cane Road

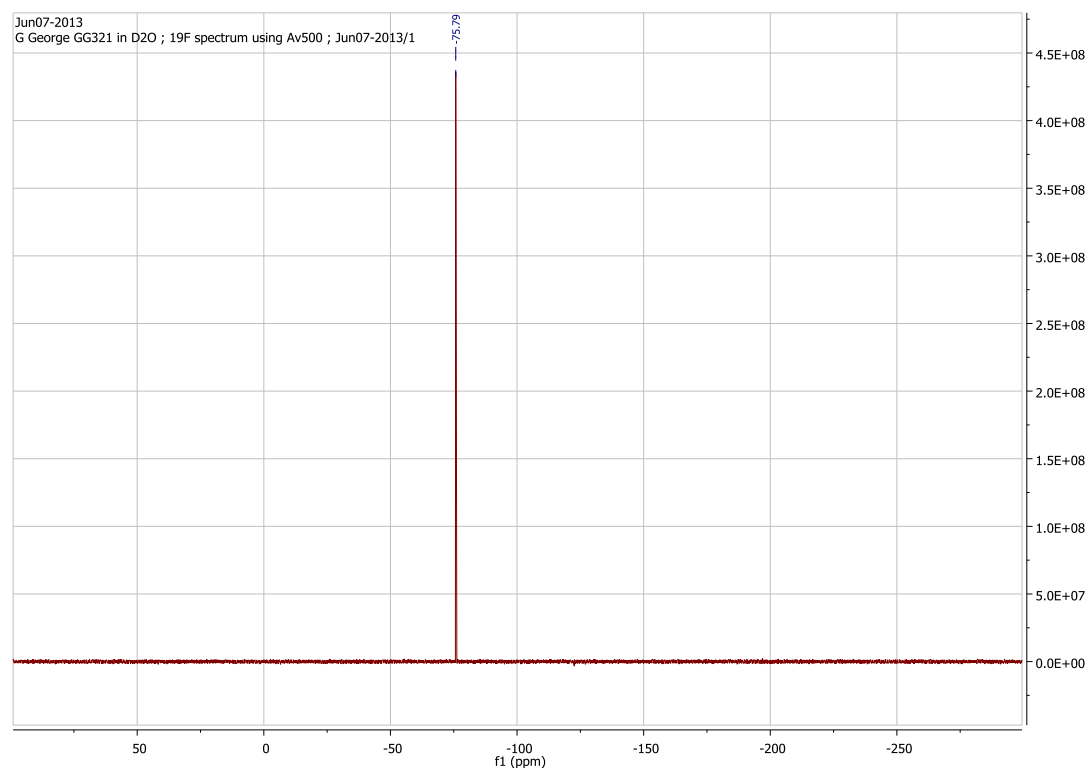
London W12 0NN

United Kingdom

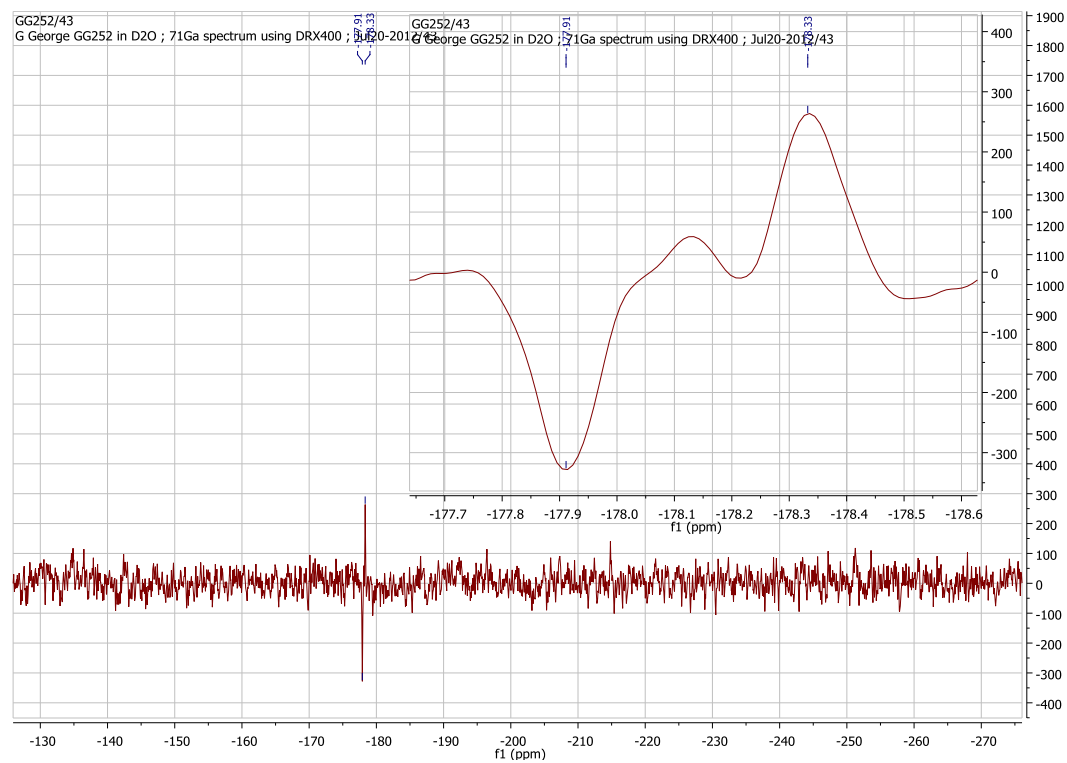
September 2013

22. Ga^{III}-NOT2A – Arg – Arg – Nal – Cys – Tyr – Cit – Lys – D-Lys – Pro – Tyr – Arg – Cit – Cys – Arg – NH₂ 4.4-4.12-disulfide

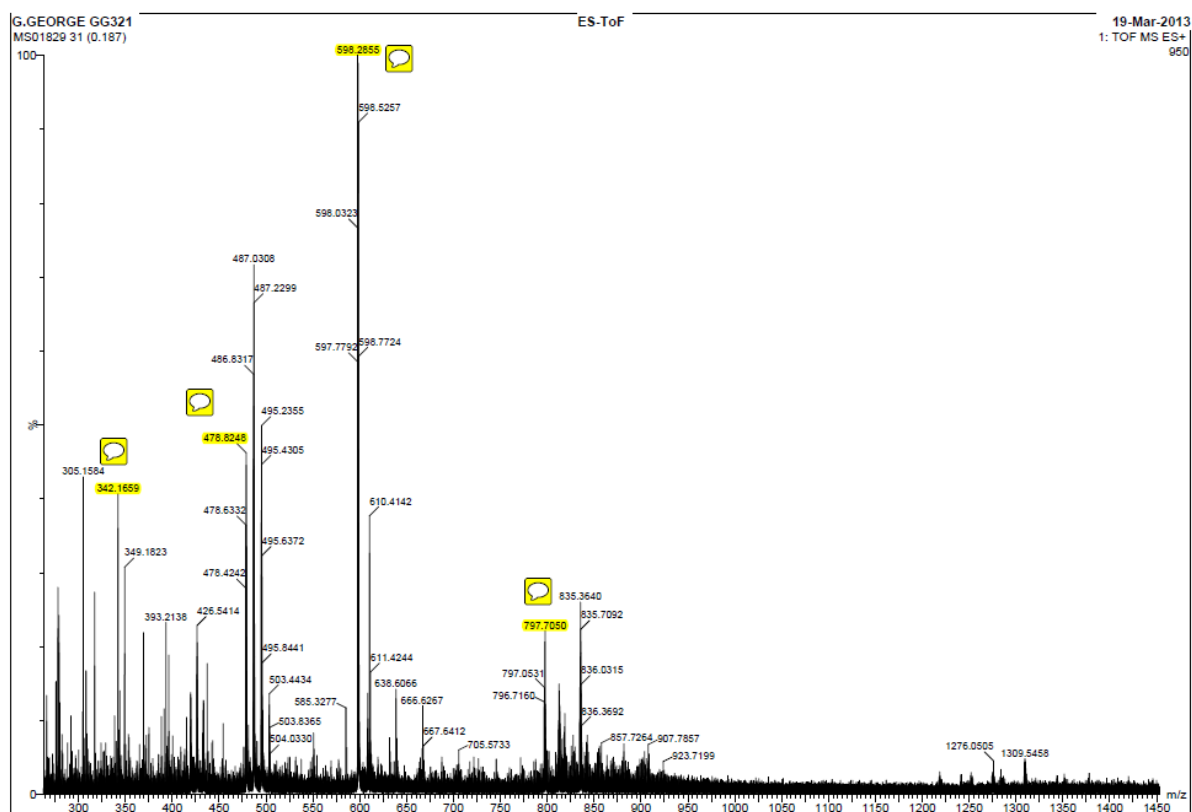
¹⁹F NMR spectrum



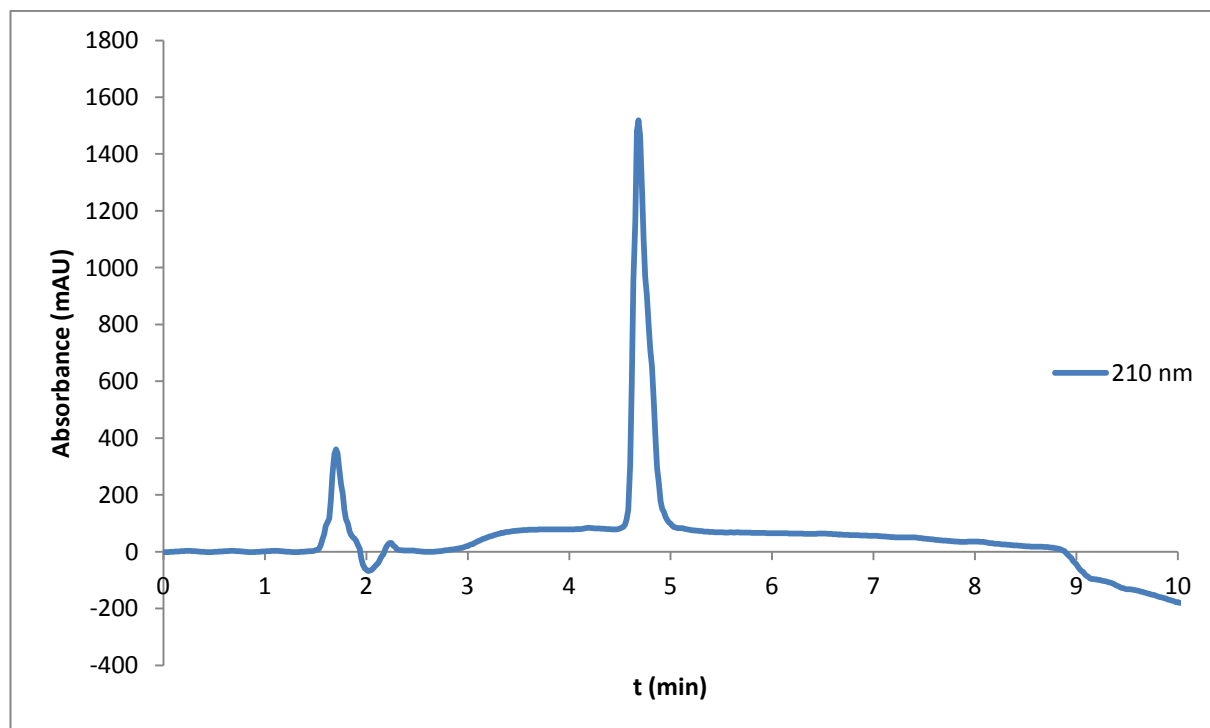
⁷¹Ga NMR spectrum



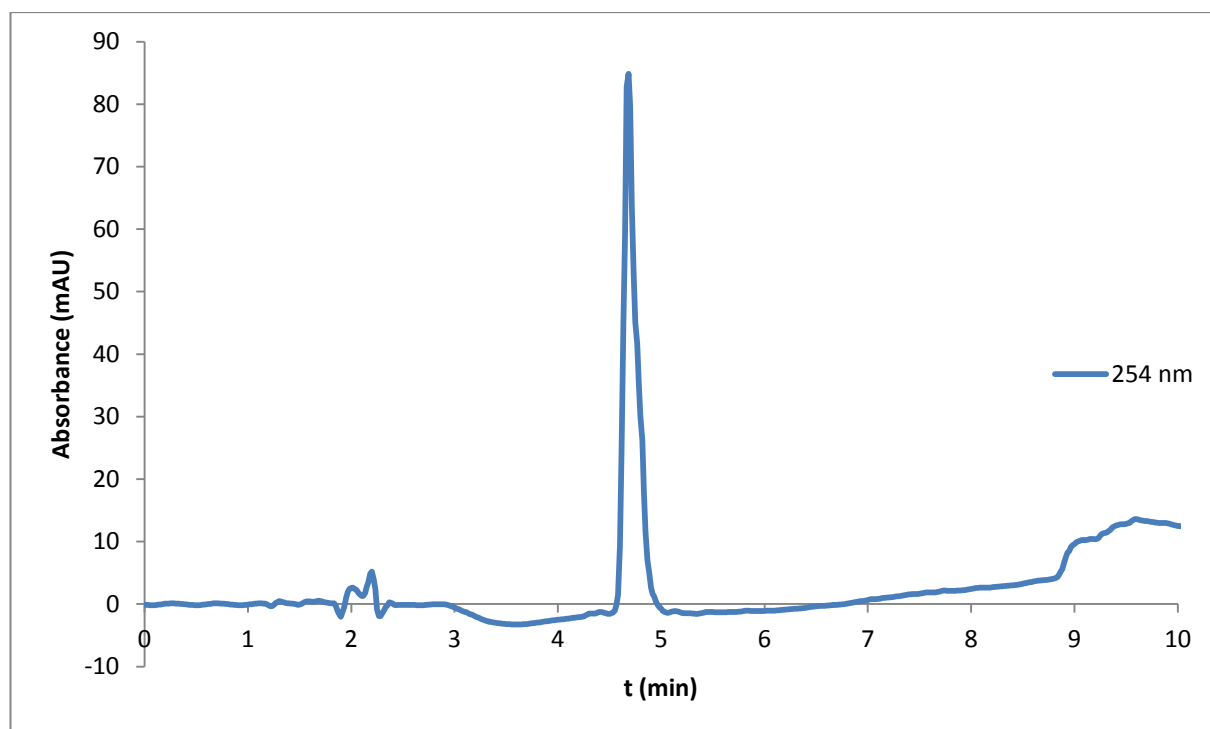
HRMS



HPLC trace at 210 nm

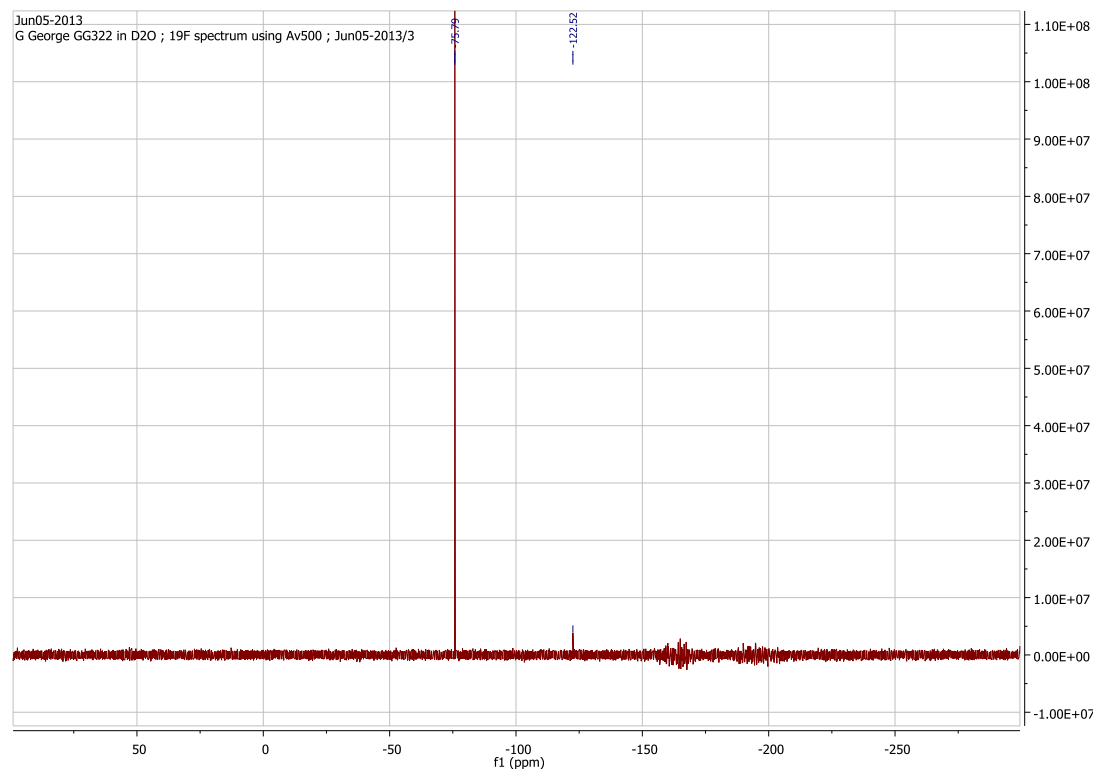


HPLC trace at 254 nm

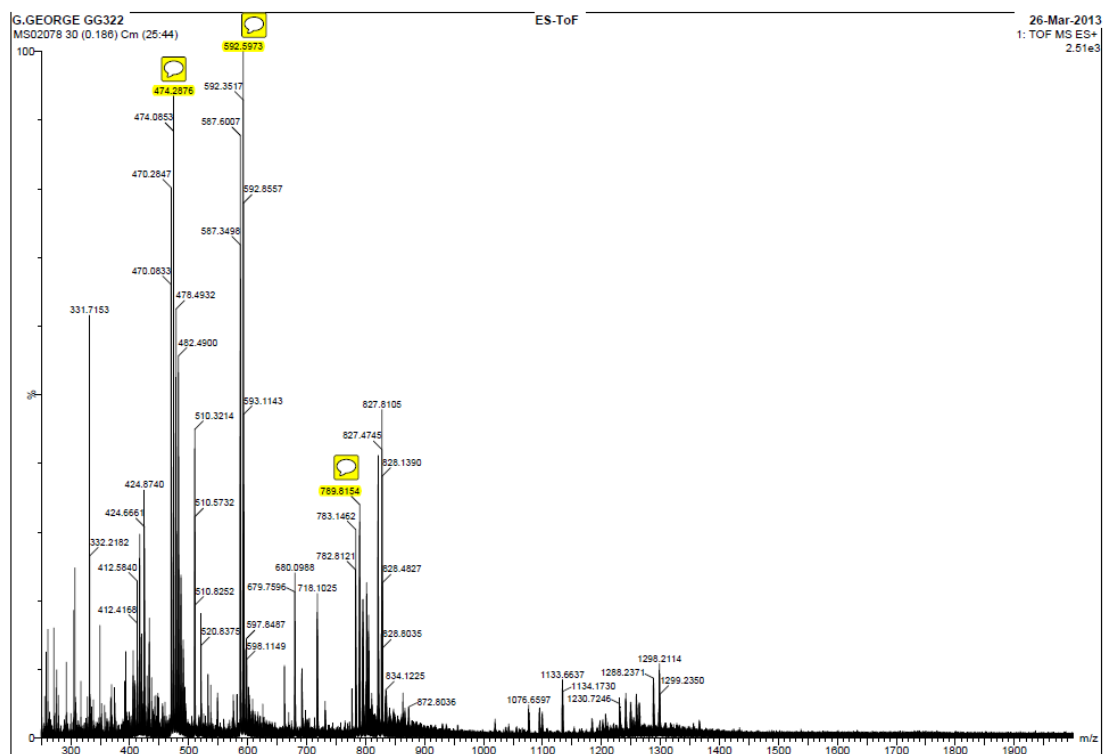


23. Al^{III}F-NOT2A – Arg – Arg – Nal – Cys – Tyr – Cit – Lys – D-Lys – Pro – Tyr – Arg – Cit – Cys – Arg – NH₂ 4.4-4.12-disulfide

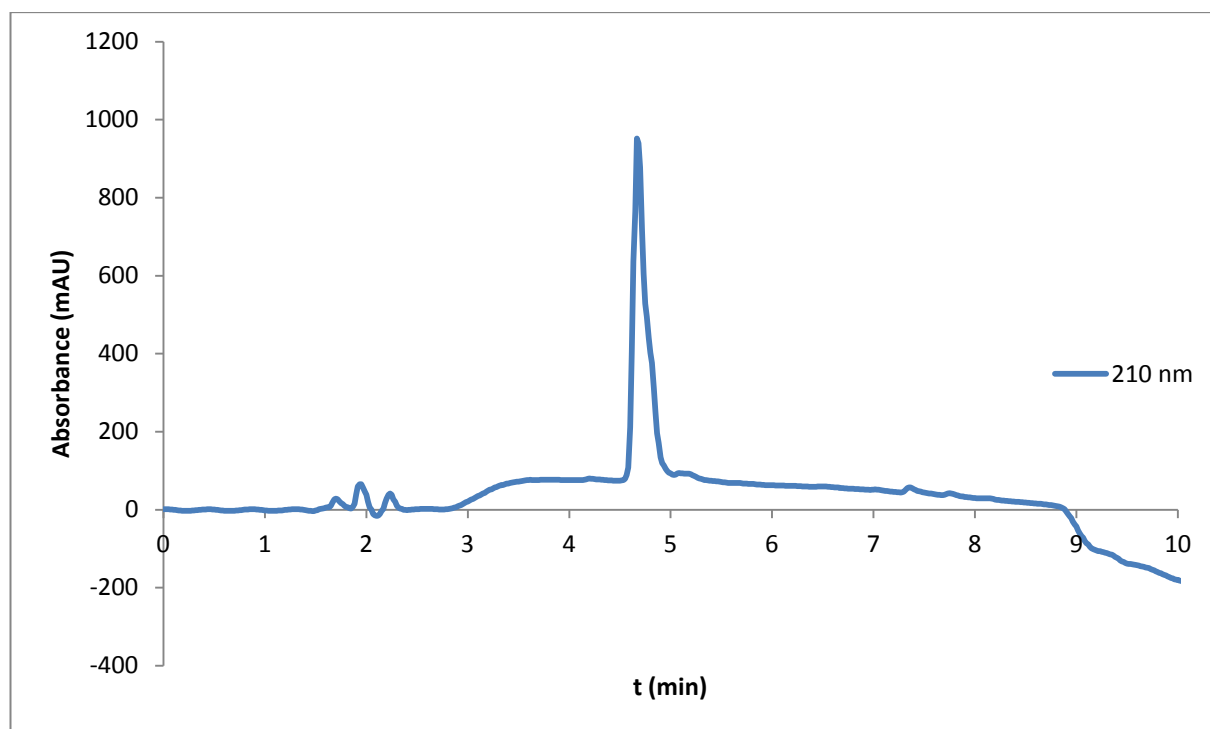
¹⁹F NMR spectrum



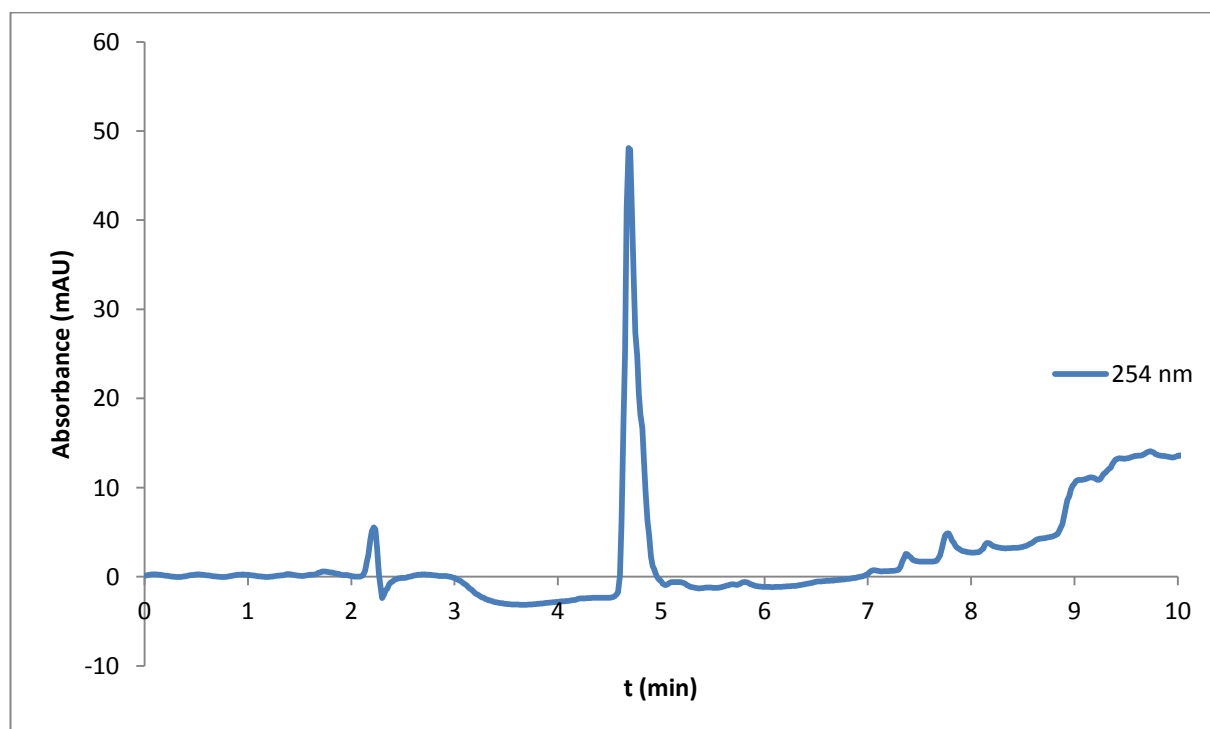
HRMS



HPLC trace at 210 nm

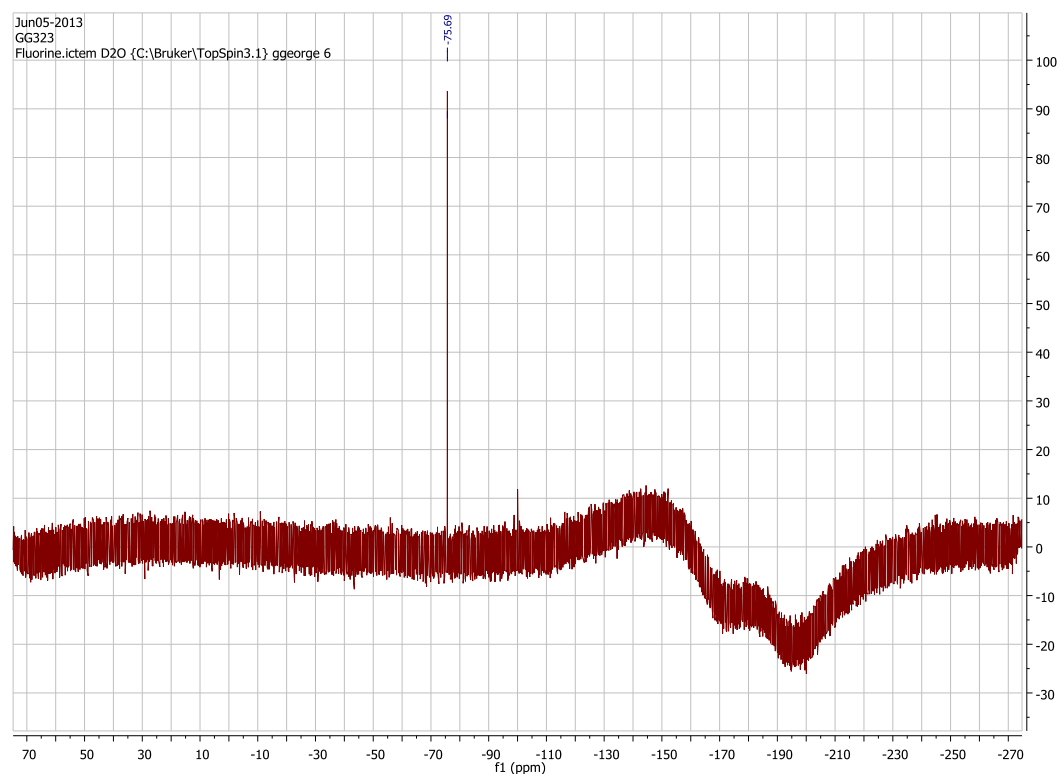


HPLC trace at 254 nm

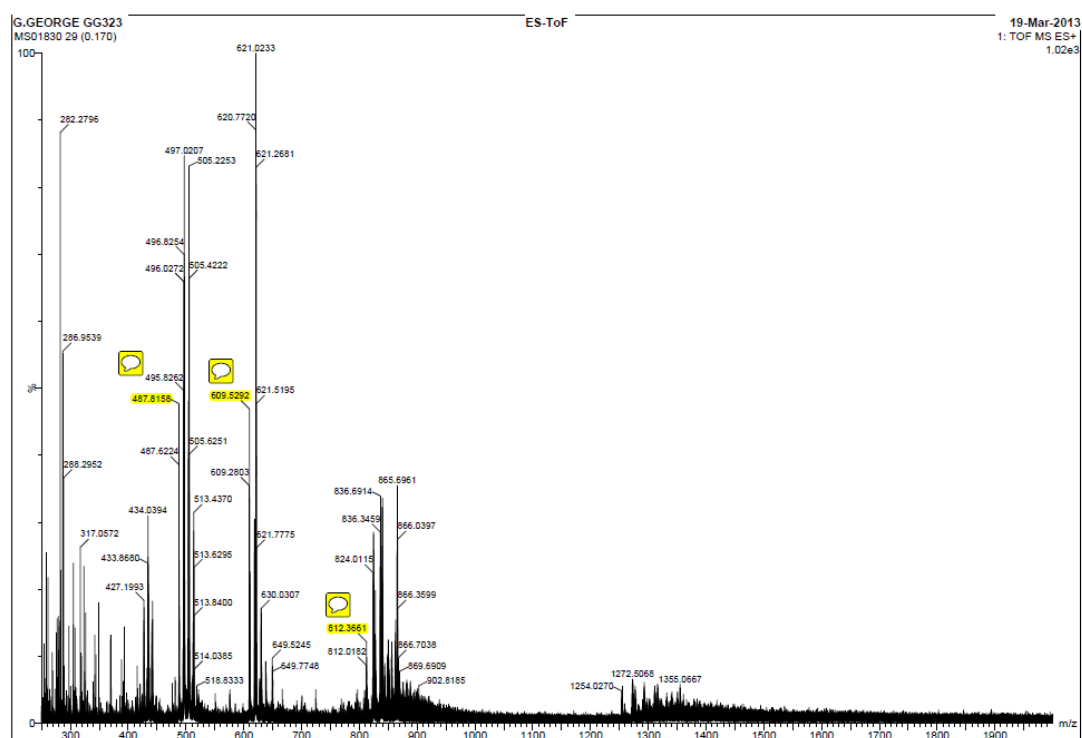


24. In^{III}-NOT2A – Arg – Arg – Nal – Cys – Tyr – Cit – Lys – D-Lys – Pro – Tyr – Arg – Cit – Cys – Arg – NH₂ 4.4-4.12-disulfide

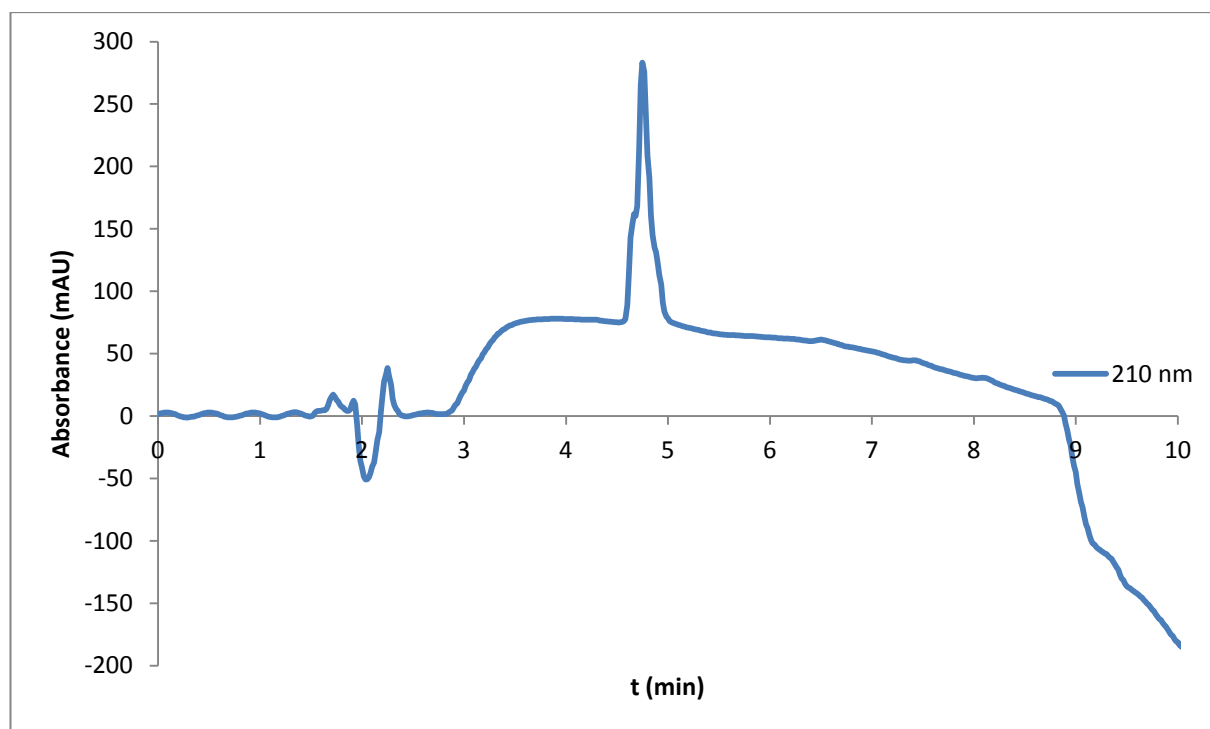
¹⁹F NMR spectrum



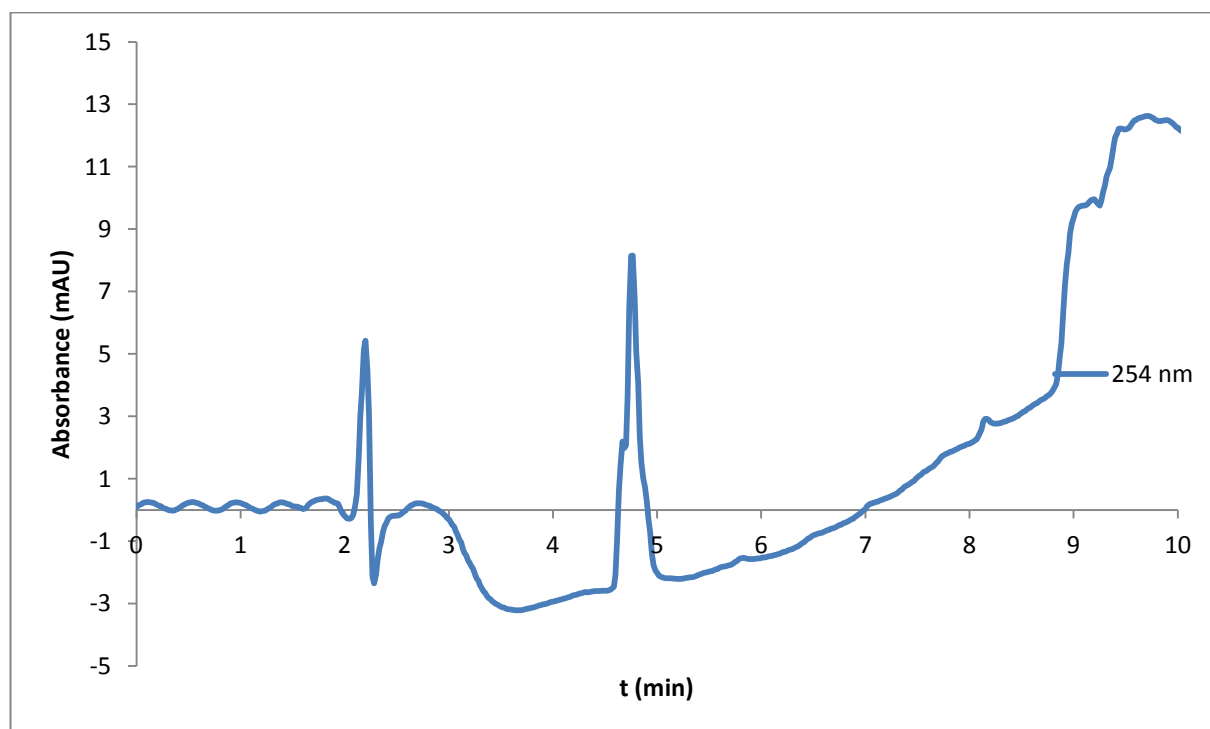
HRMS



HPLC trace at 210 nm

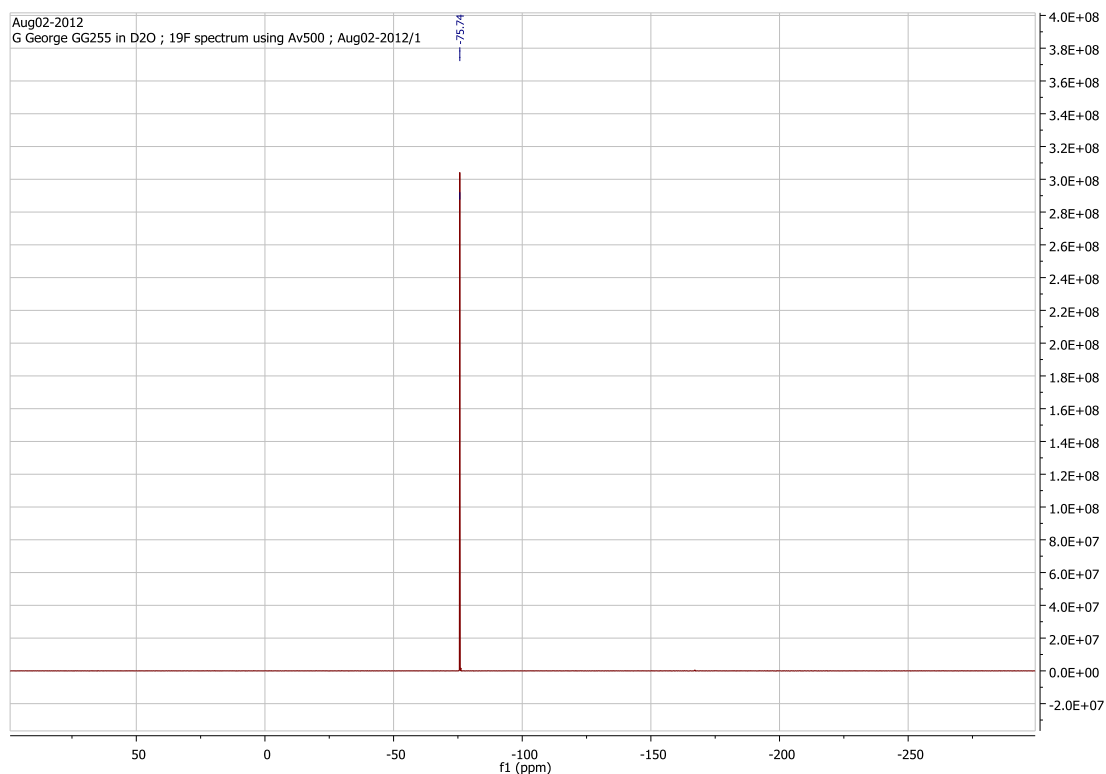


HPLC trace at 254 nm

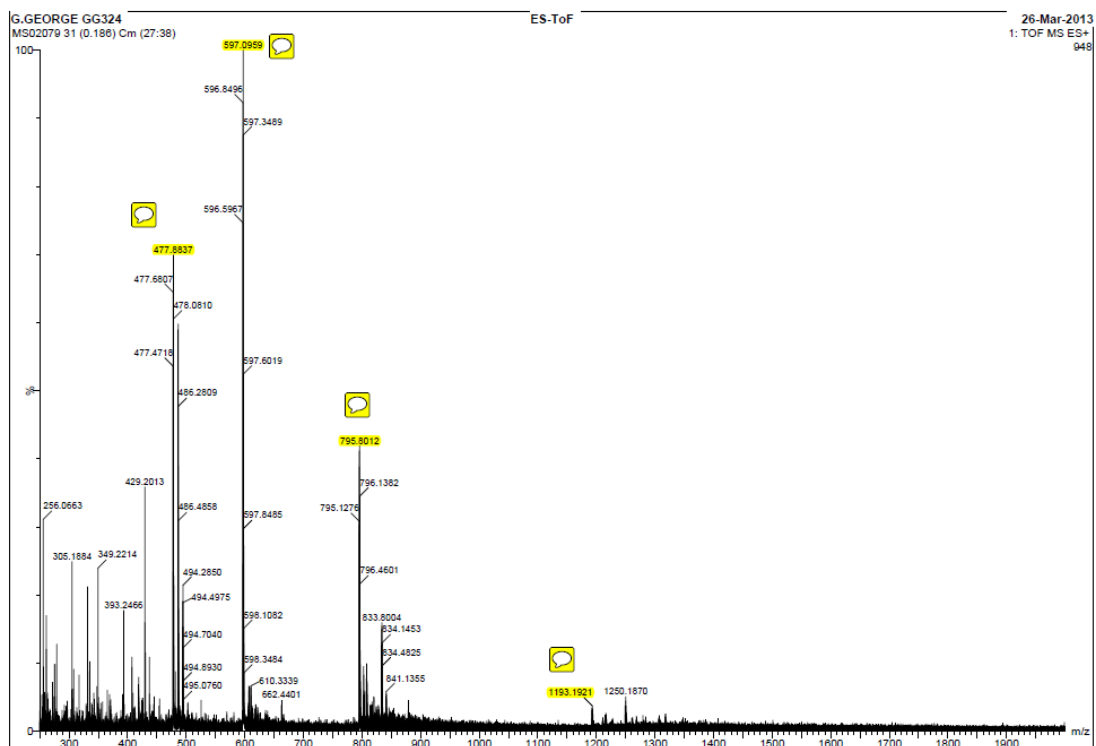


25. Cu^{II}-NOT2A – Arg – Arg – Nal – Cys – Tyr – Cit – Lys – D-Lys – Pro – Tyr – Arg – Cit – Cys – Arg – NH₂ 4.4-4.12-disulfide

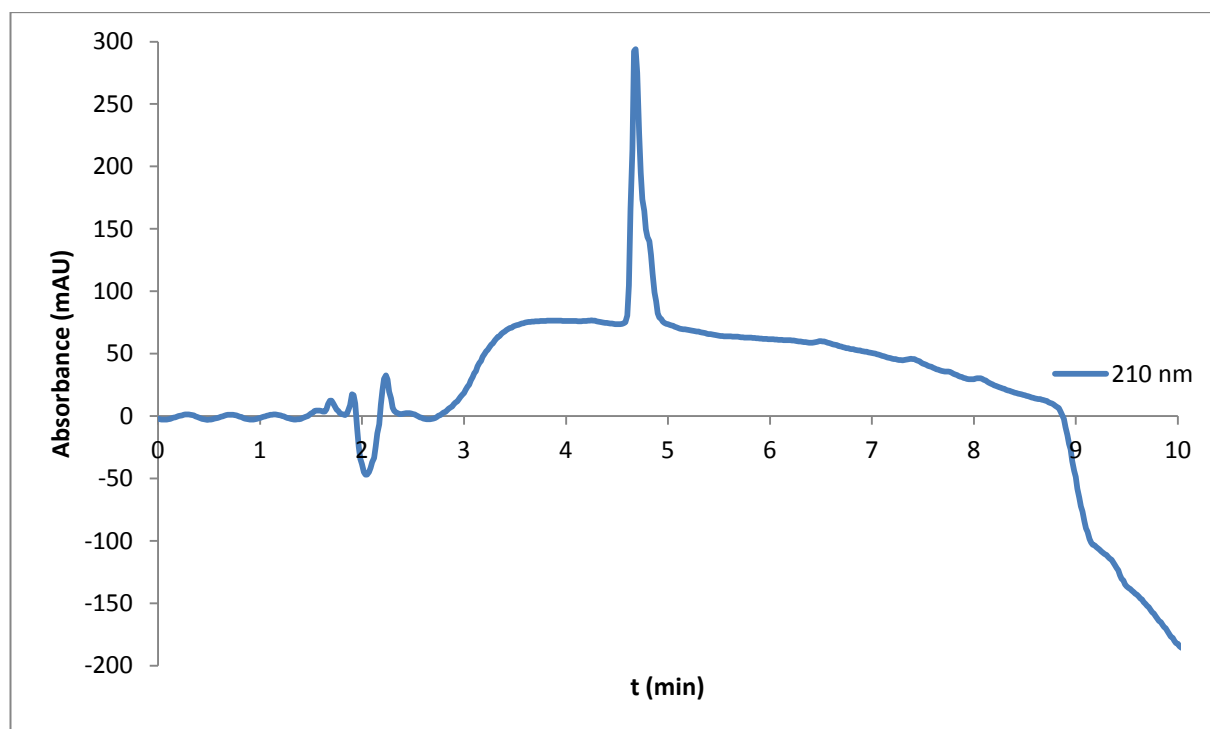
¹⁹F NMR spectrum



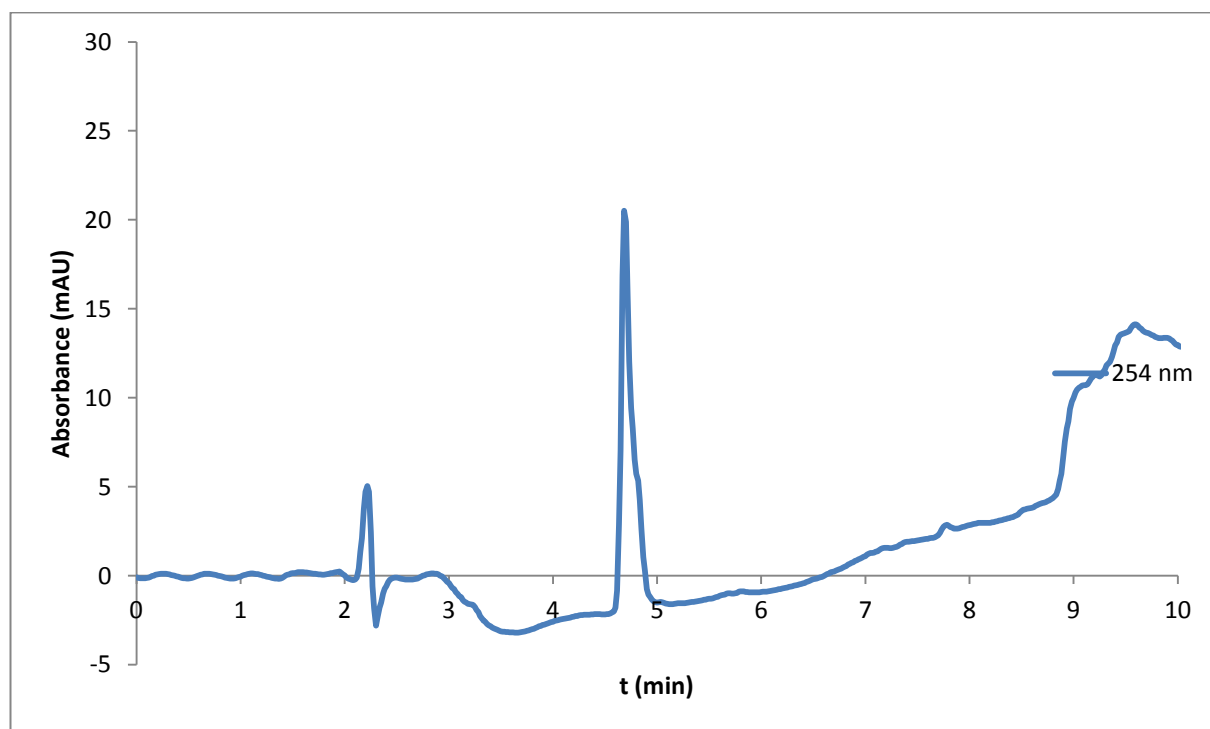
HRMS



HPLC trace at 210 nm



HPLC trace at 254 nm



26. cyclo(- Gly - d-Tyr - Prg - Arg - Nal -).TFA

HRMS

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Odd and Even Electron Ions

317 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 35-35 H: 5-300 N: 0-14 O: 0-10 Na: 0-1

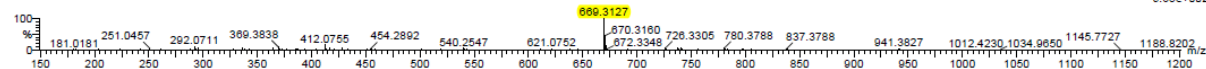
G.GEORGE GG242

LCT28946 5 (0.172) Cm (5-29:30)

12:48:04 11-Sep-2012

1: TOF MS ES+

9.63e+002



Minimum:

Maximum:

5.0

10.0

-1.5

50.0

Mass

Calc. Mass

mDa

PPM

DBE

1-FIT

1-FIT (Norm)

Formula

669.3127

669.3138

-1.1

-1.6

16.0

68.0

0.7

C35 H44 N5 O7 Na

669.3149

-2.2

-3.3

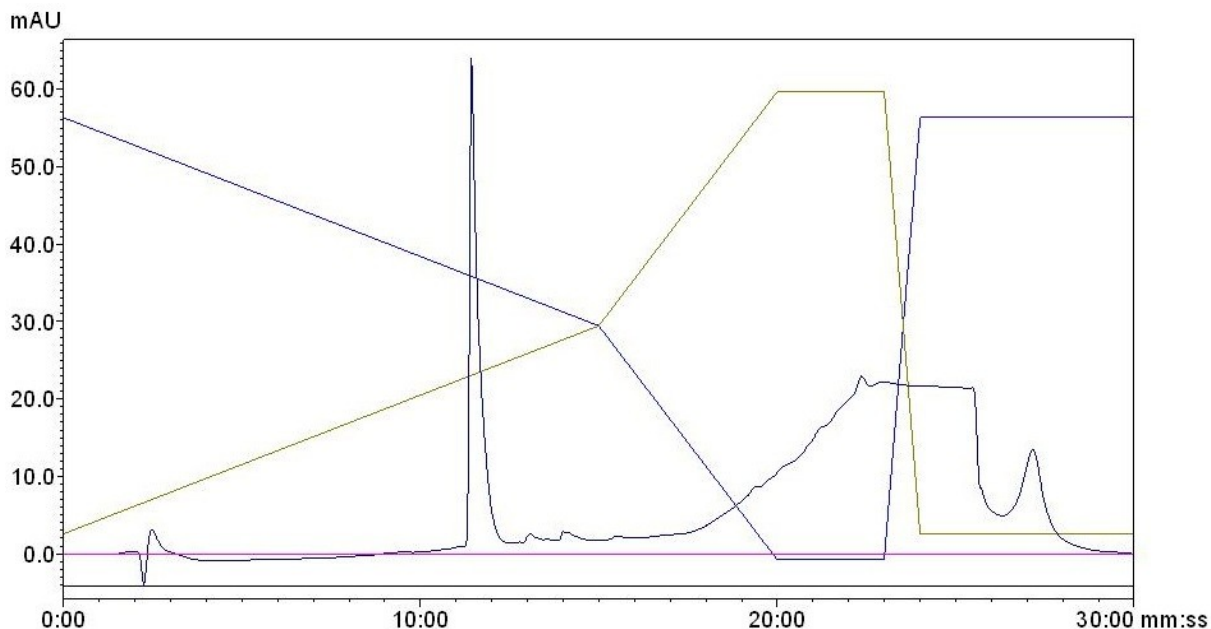
19.5

68.0

0.7

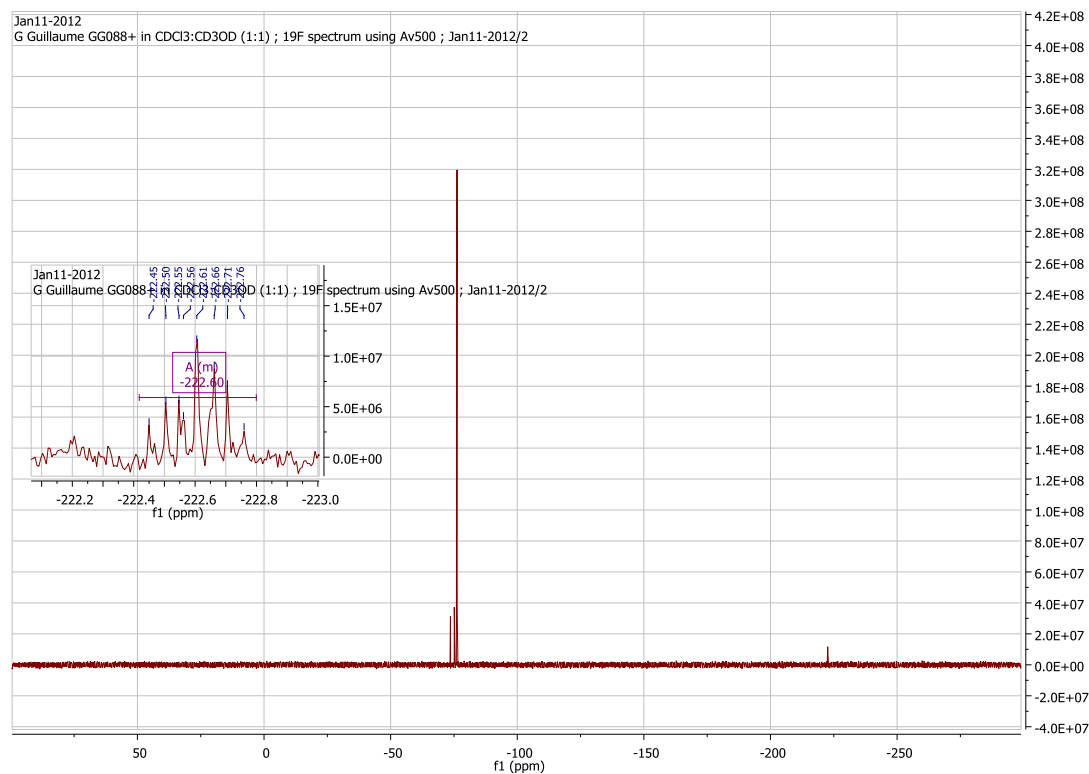
C35 H41 N8 O6

HPLC trace

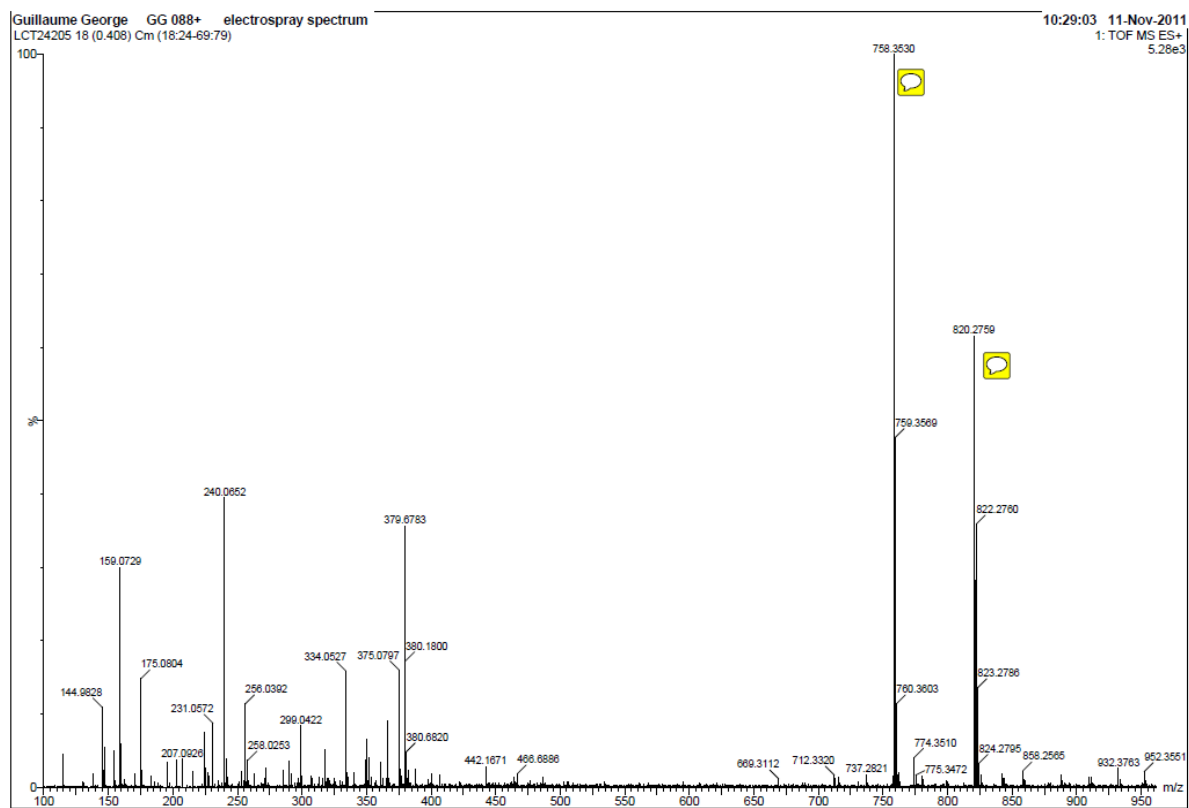


28. cyclo(- Gly - d-Tyr - Fta - Arg - Nal -).TFA

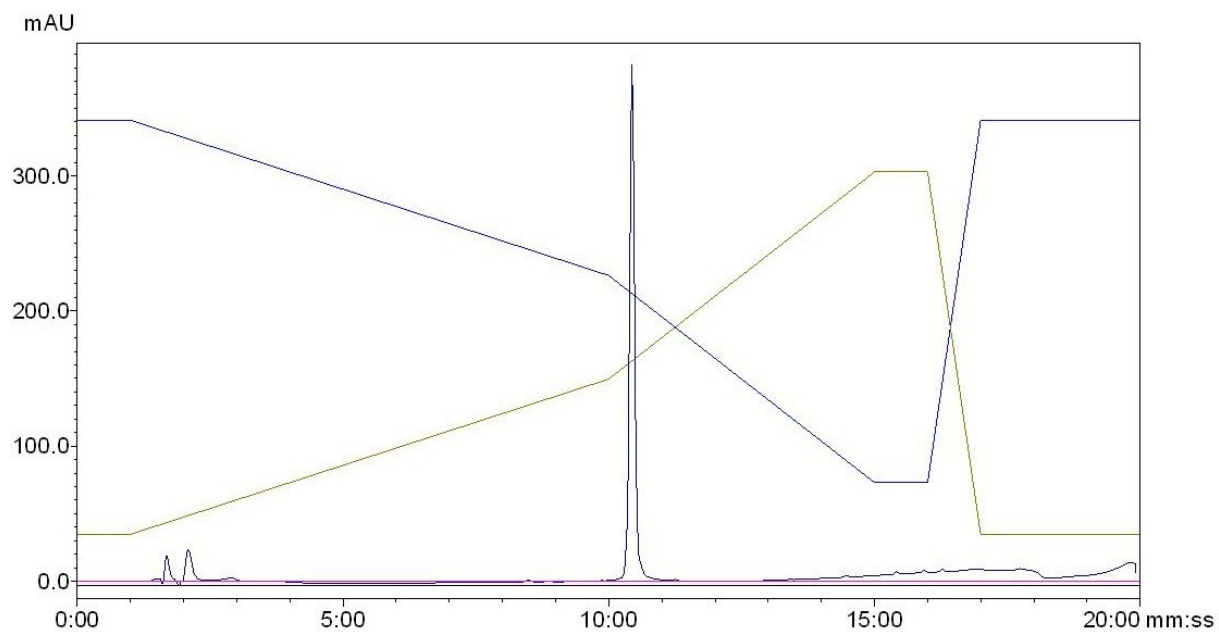
¹⁹F NMR spectrum



HRMS

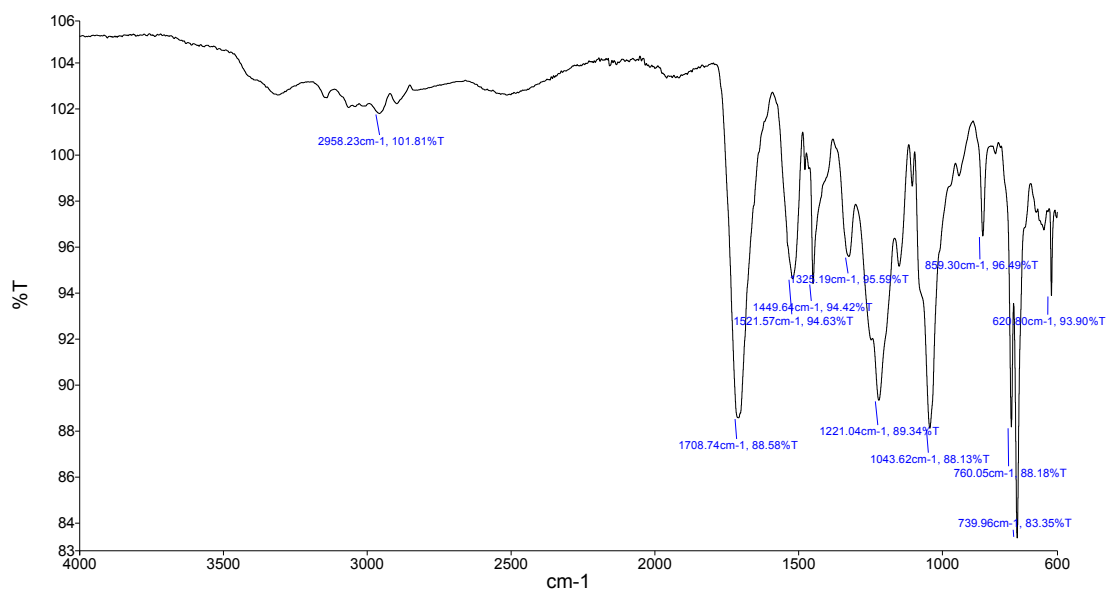


HPLC trace

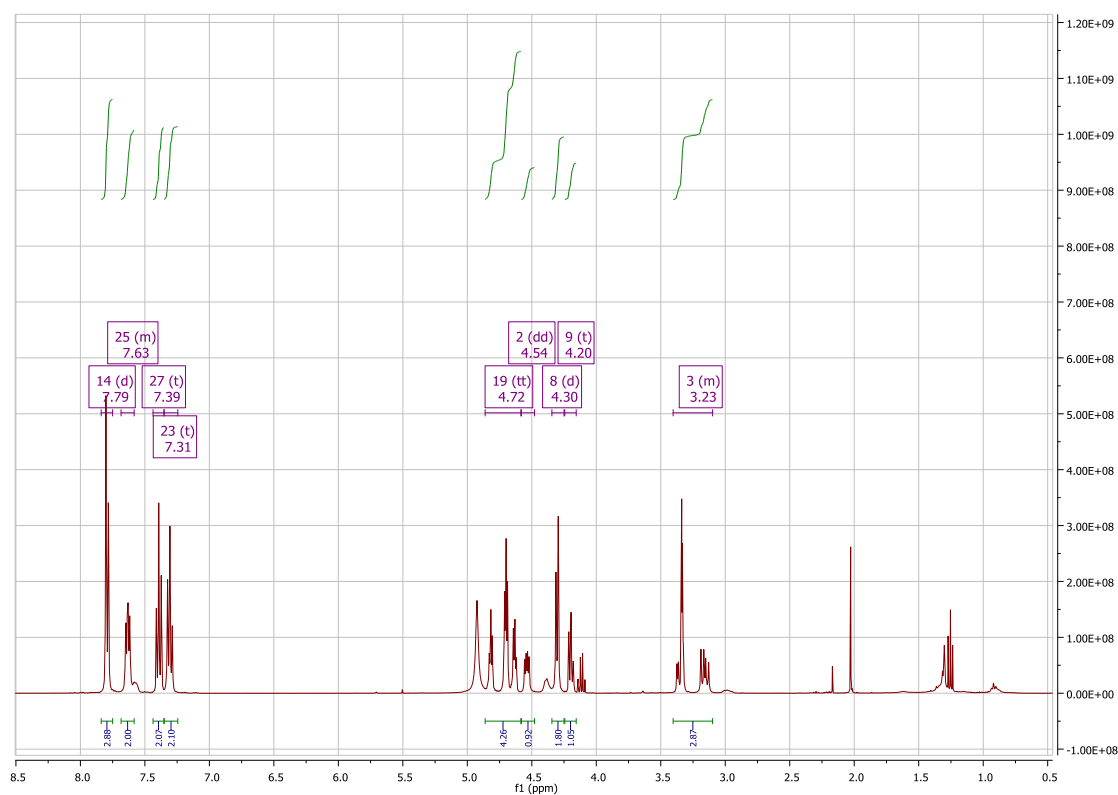


32. (2S)-2-{[(9H-Fluoren-9-ylmethoxy)carbonyl]amino}-3-[1-(2-fluoroethyl)-1H-1,2,3-triazol-4-yl]propanoic acid.

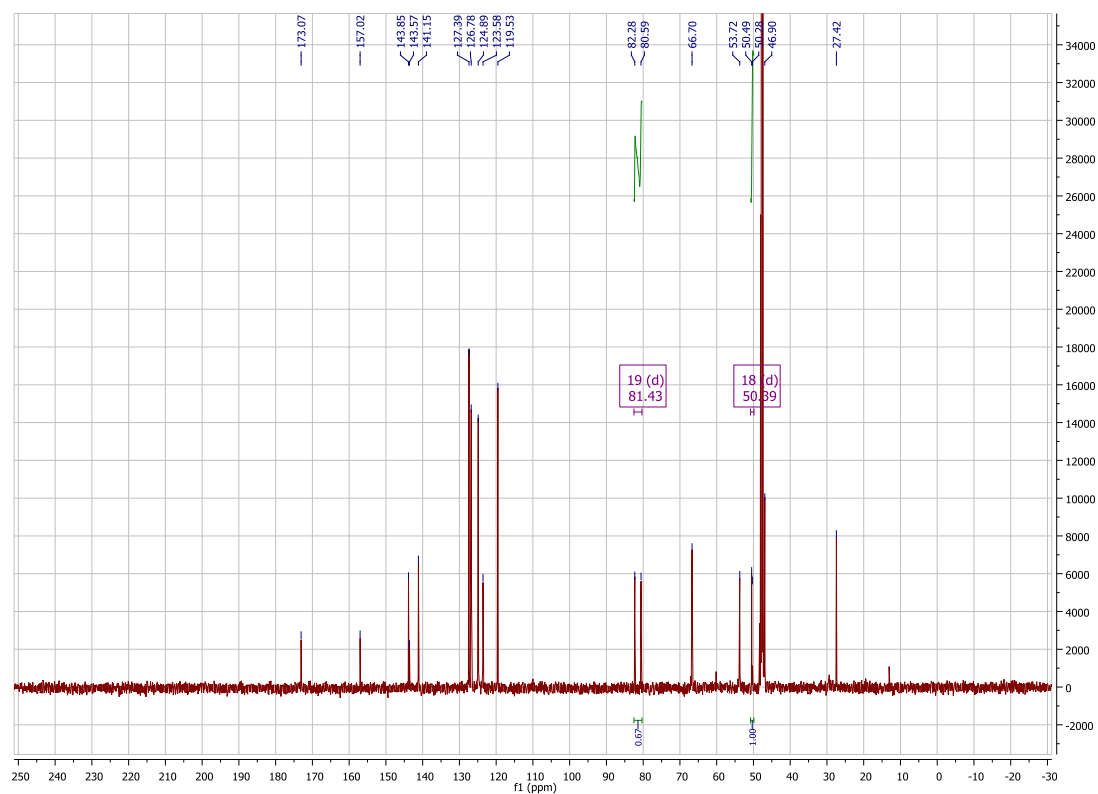
Infrared spectrum



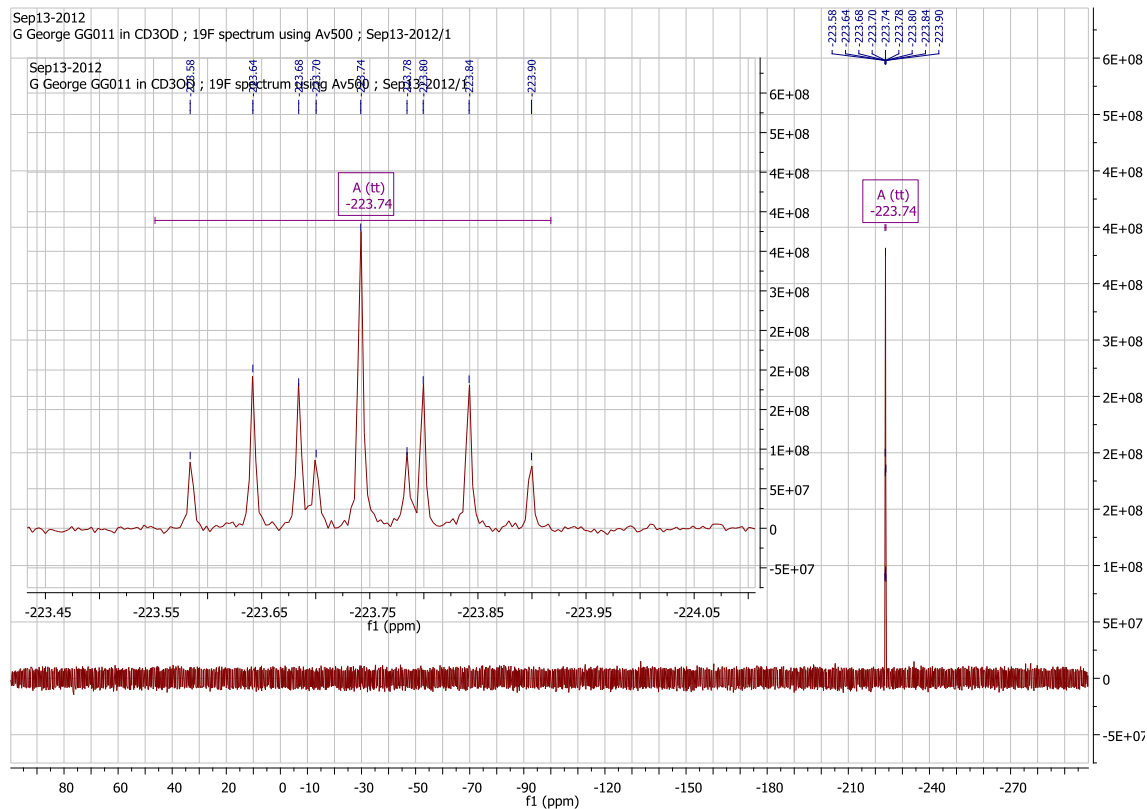
¹H NMR spectrum



^{13}C NMR spectrum

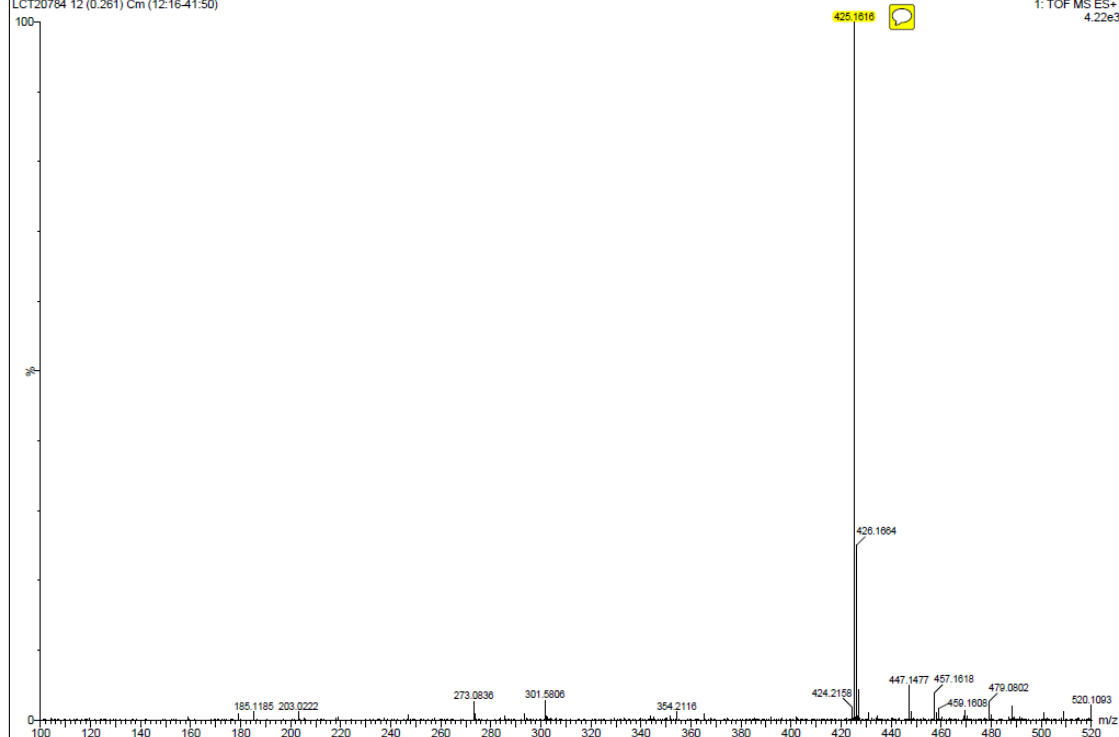


^{19}F NMR spectrum



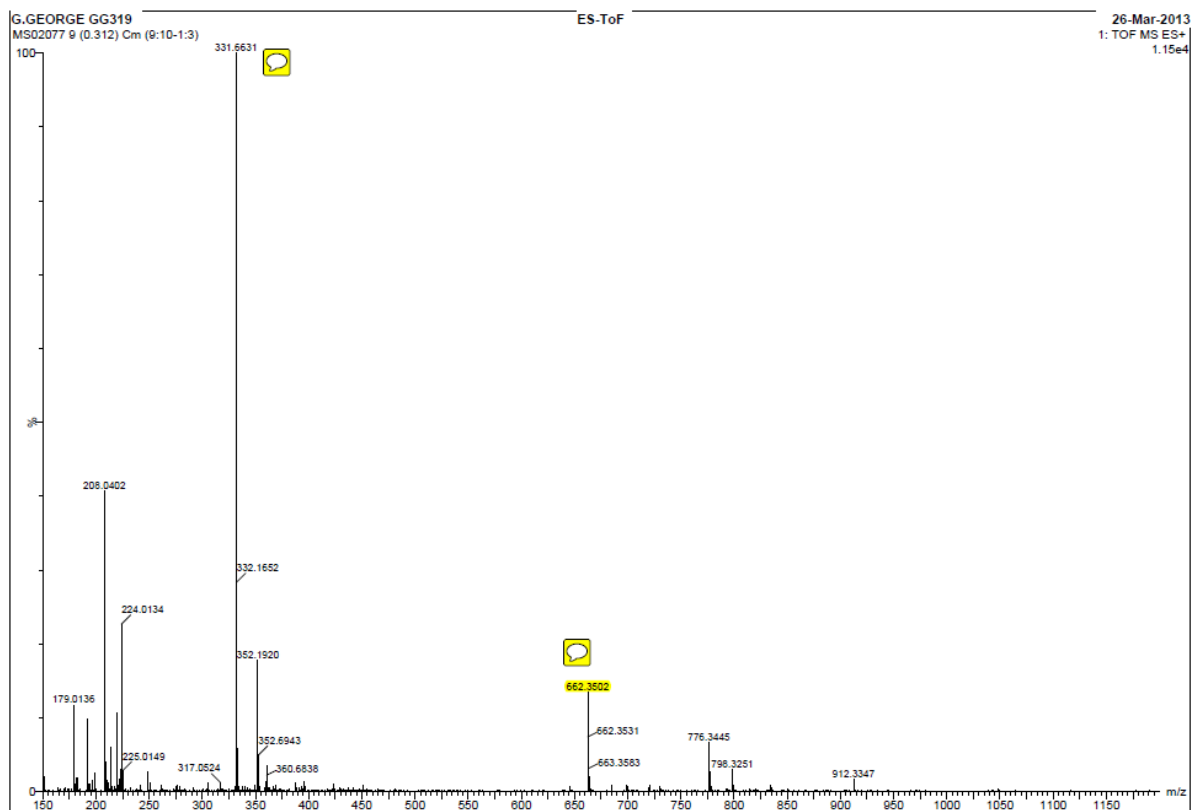
Guillaume George GG011 electrospray spectrum
LCT20784 12 (0.261) Cm (12:16:41:50)

14:21:20 18-Mar-2011
1: TOF MS ES+
4.22e3

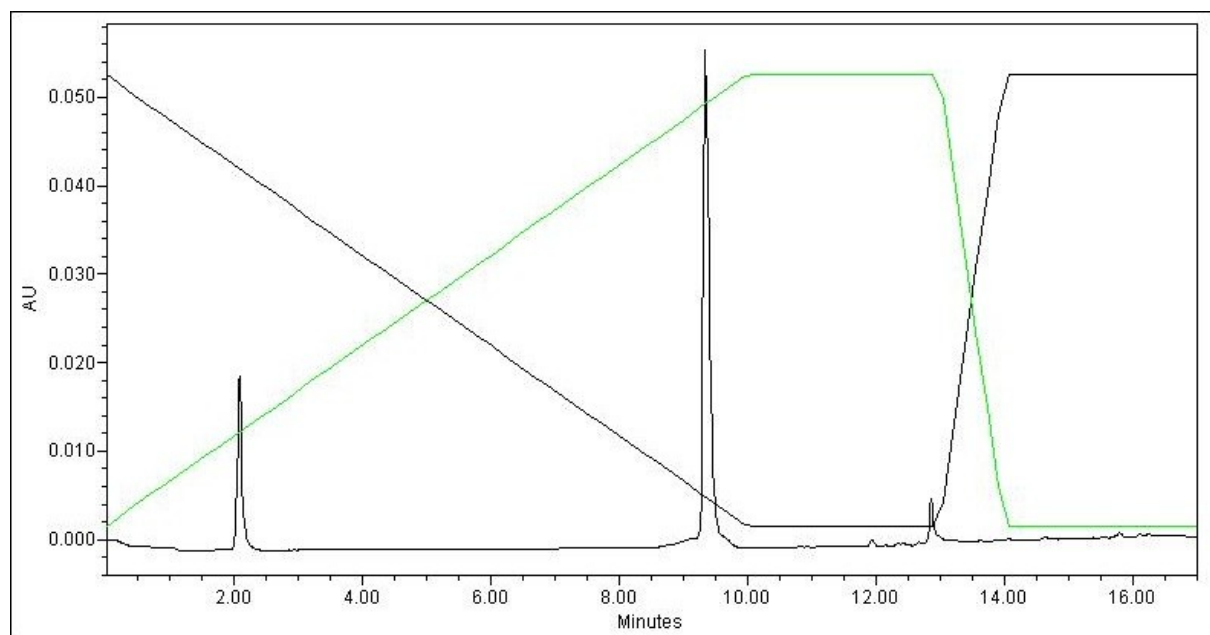


34. *cyclo*(– Gly – Prg – Arg – Arg – Nal –).2TFA

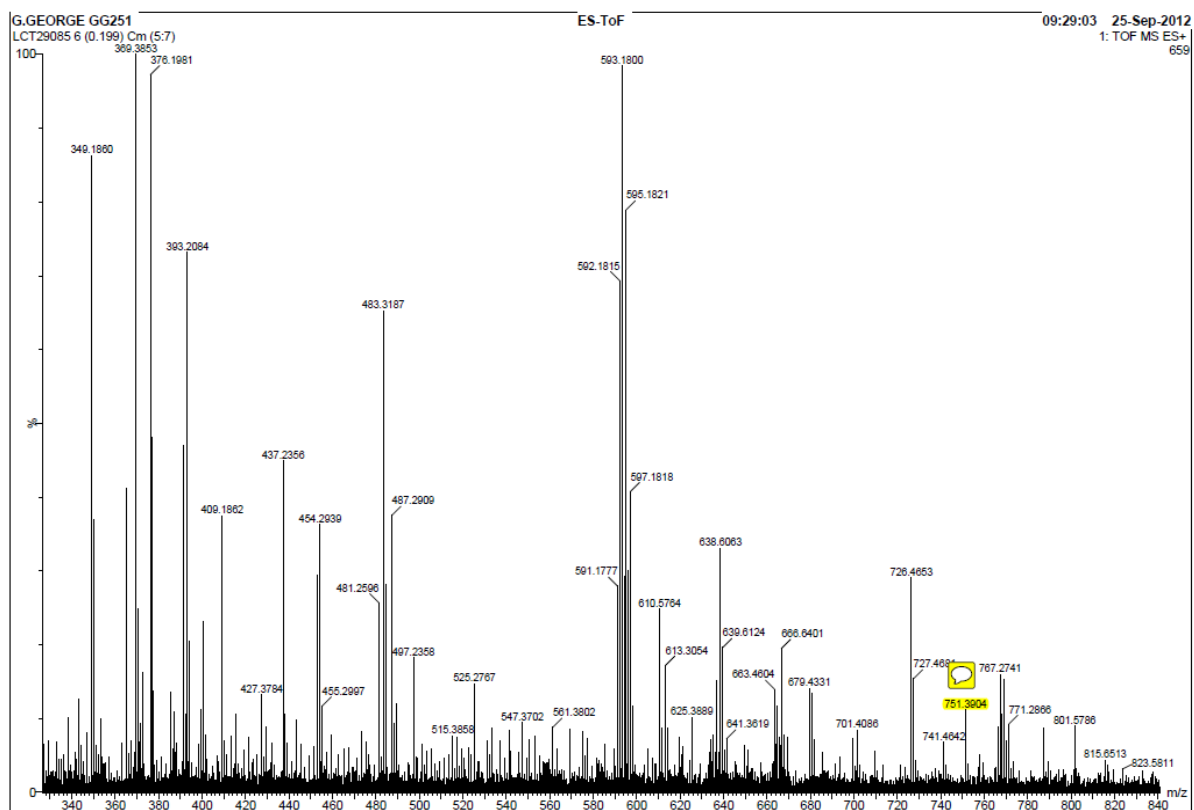
HRMS



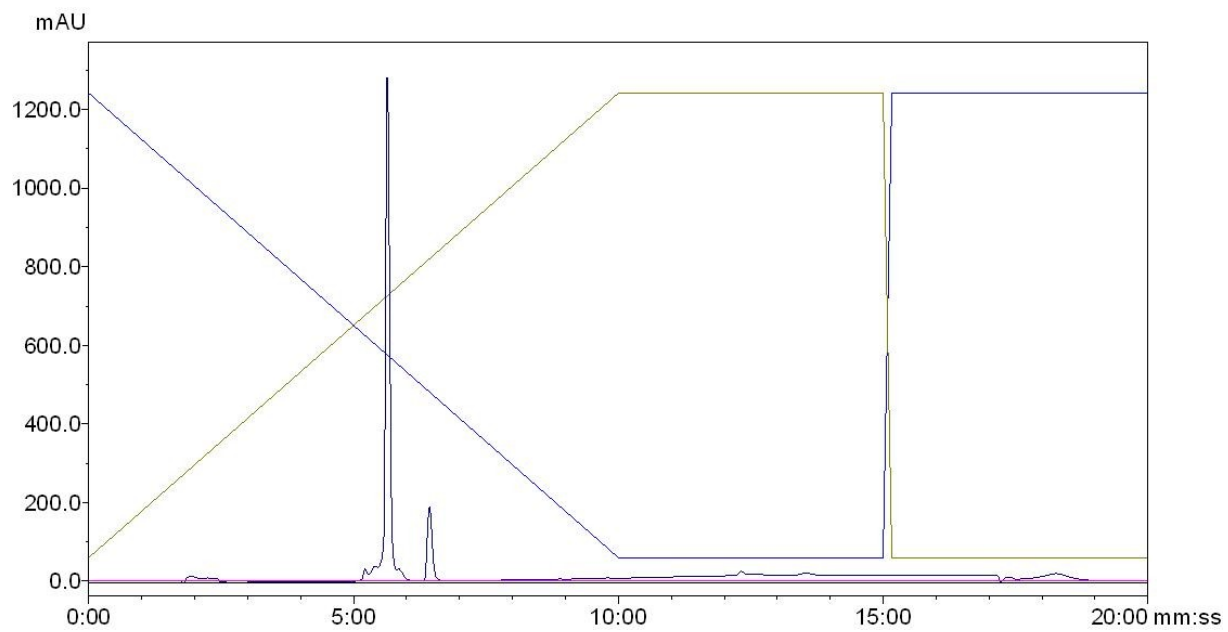
HPLC trace



¹⁹F NMR spectrum



HPLC trace



36. *cyclo(- Gly - D-Tyr - Arg - Prg - Nal -).TFA*

HRMS

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

317 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 35-35 H: 5-300 N: 0-14 O: 0-10 Na: 0-1

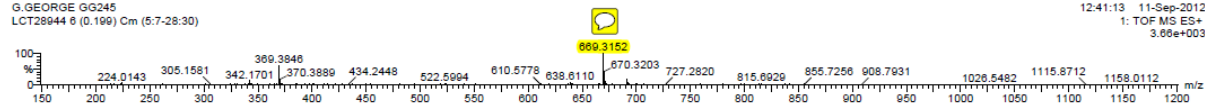
G.GEORGE GG245

LCT28944 6 (0.199) Cm (5:7-28:30)

12:41:13 11-Sep-2012

1: TOF MS ES+

3.86e+003



Minimum:

Maximum:

5.0

10.0

-1.5

50.0

Mass

Calc. Mass

mDa

PPM

DBE

1-FIT

1-FIT (Norm)

Formula

669.3152

669.3149

0.3

0.4

19.5

121.6

0.0

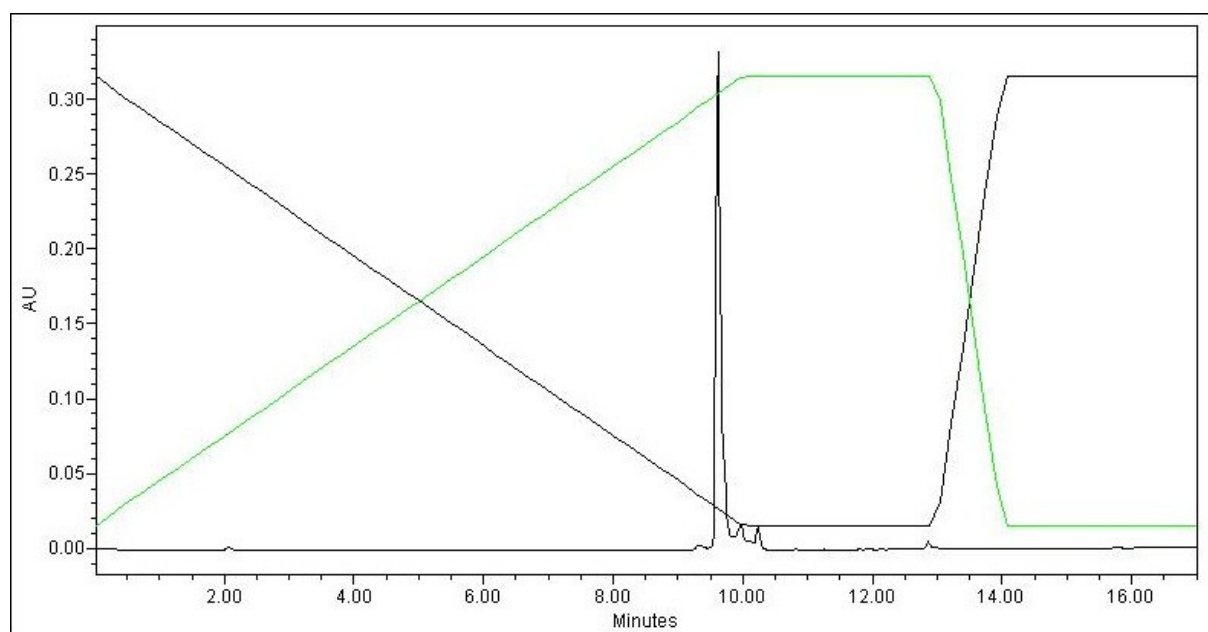
C35

H41

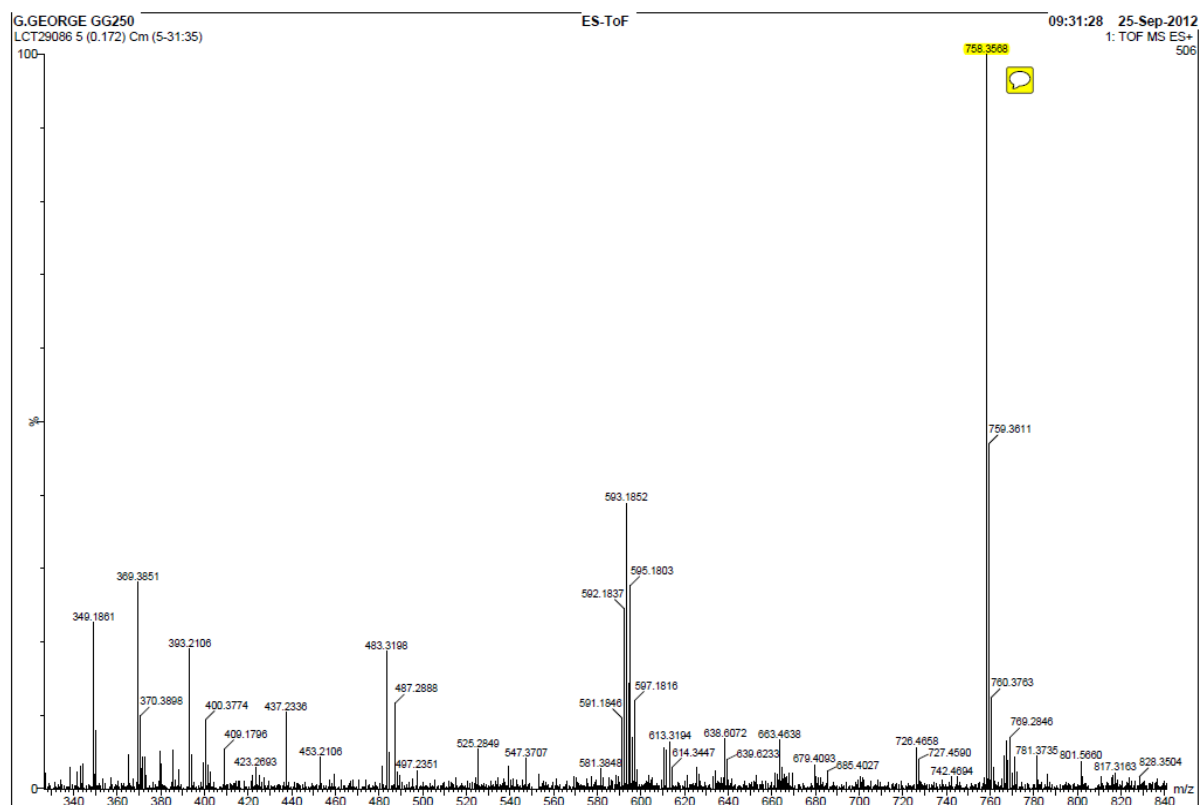
N8

O6

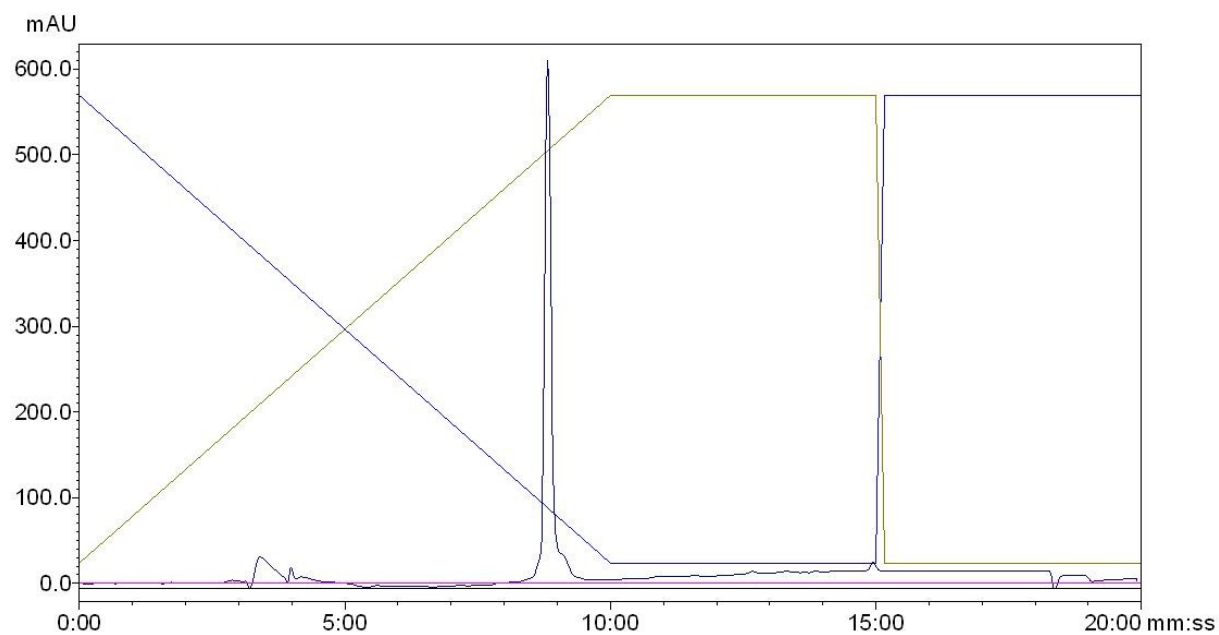
HPLC trace



¹⁹F NMR spectrum



HPLC trace



38. *cyclo*(- Gly - D-Tyr - Arg - Arg - Prg -).2TFA

HRMS

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

322 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 28-28 H: 5-300 N: 0-14 O: 0-10 Na: 0-1

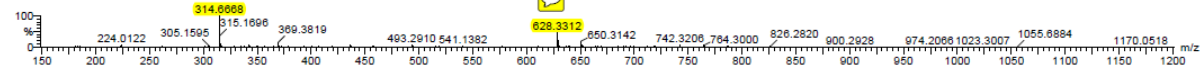
G.GEORGE GG244

LCT28945 7 (0.228) Cm (7.8-1.3)

12:43:38 11-Sep-2012

1: TOF MS ES+

1.94e+003



Minimum:

Maximum:

5.0

10.0

-1.5

50.0

Mass

Calc. Mass

mDa

PPM

DBE

1-FIT

1-FIT (Norm)

Formula

628.3312

628.3320

-0.8

-1.3

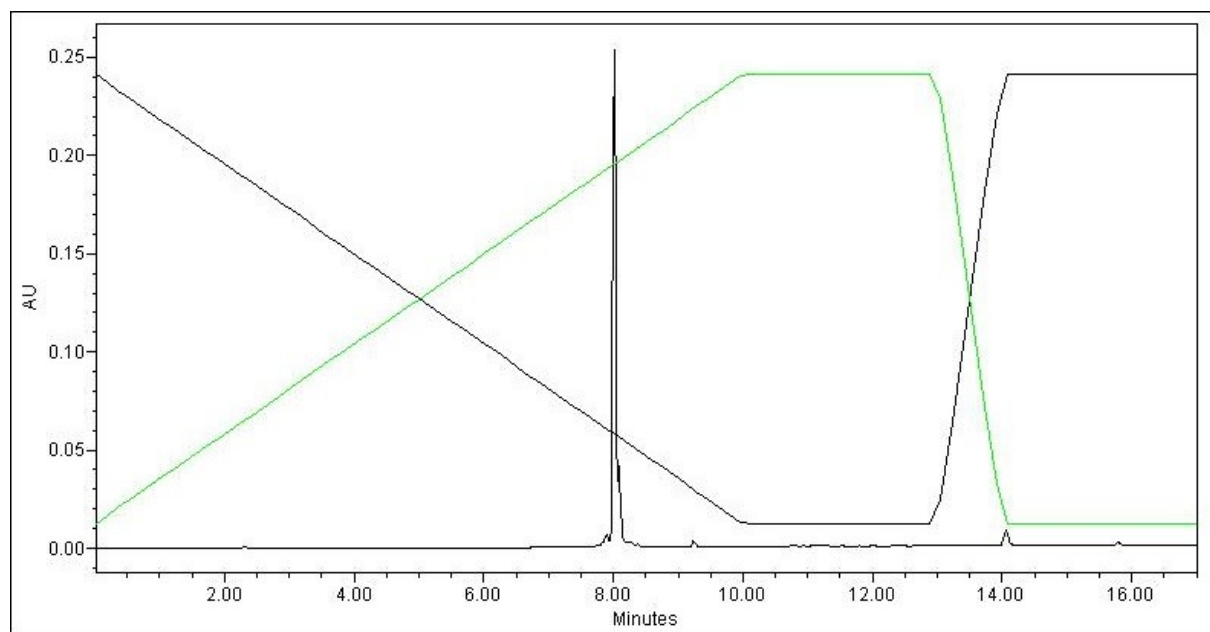
13.5

70.3

0.0

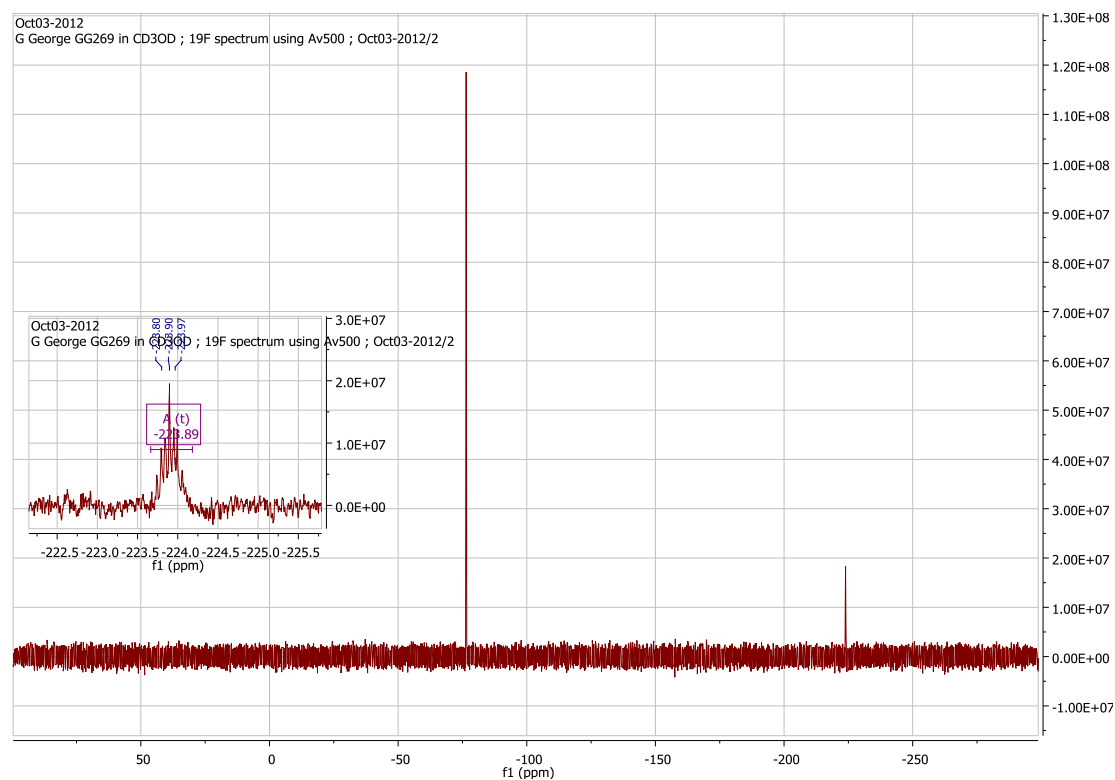
C28 H42 N11 O6

HPLC trace

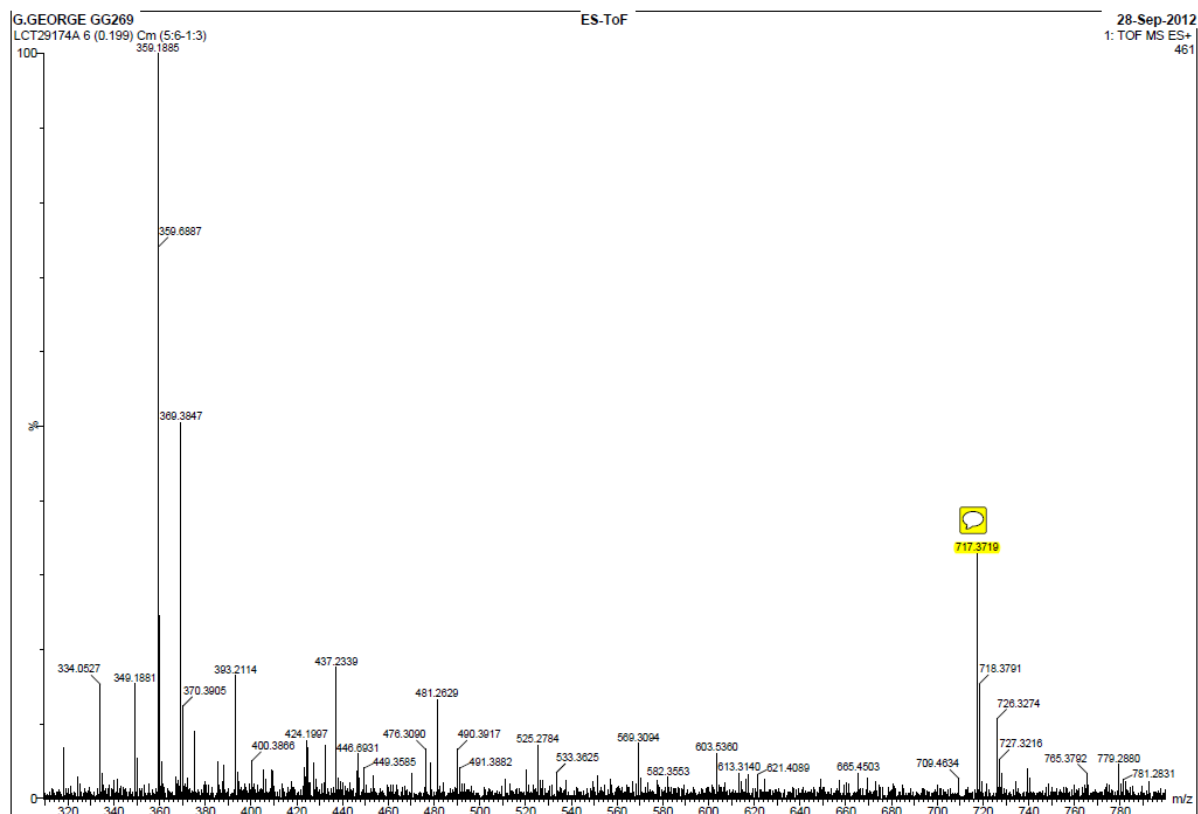


39. *cyclo*(- Gly - D-Tyr - Arg - Arg - Fta -).2TFA

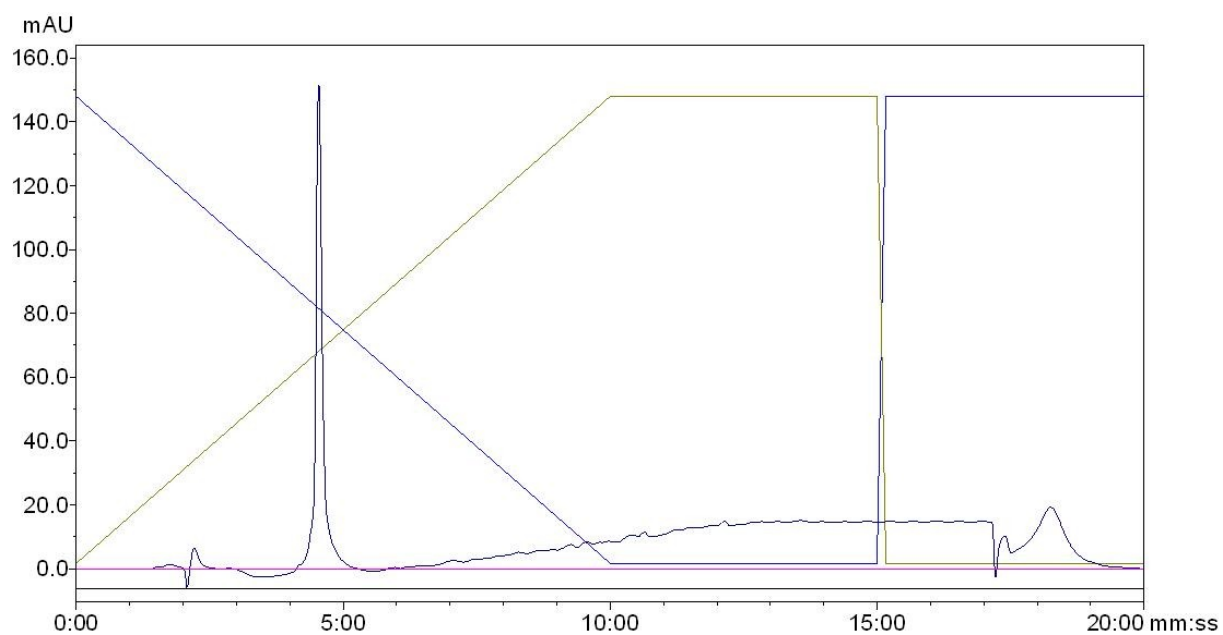
^{19}F NMR spectrum



HRMS

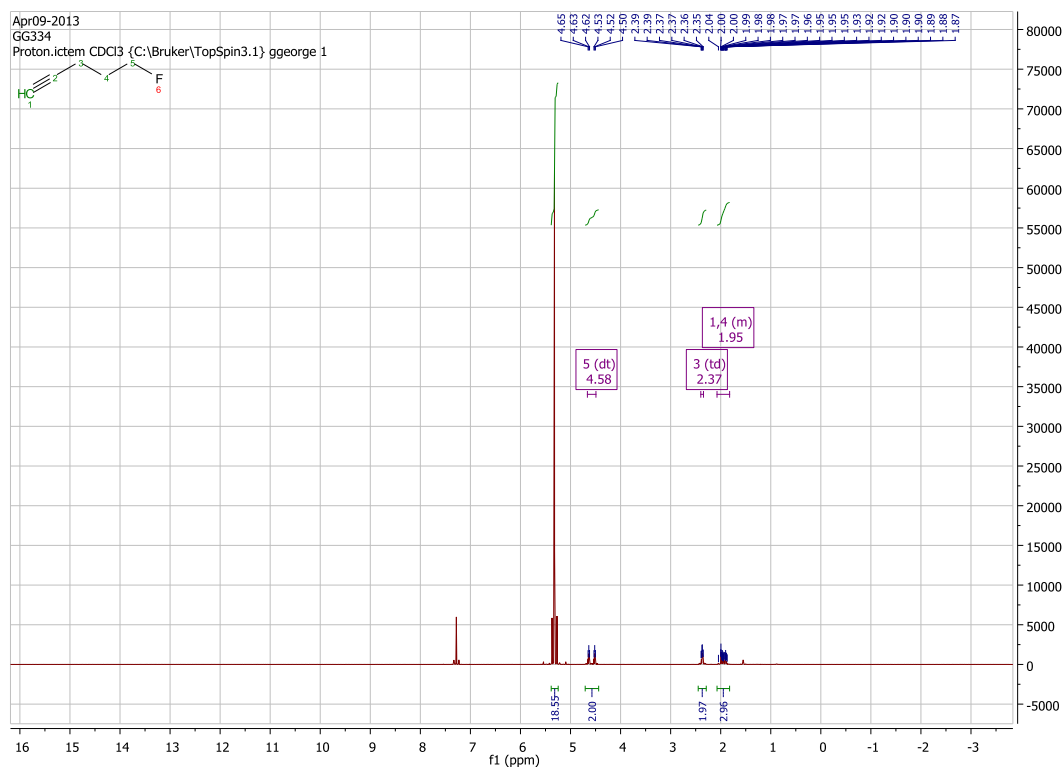


HPLC trace

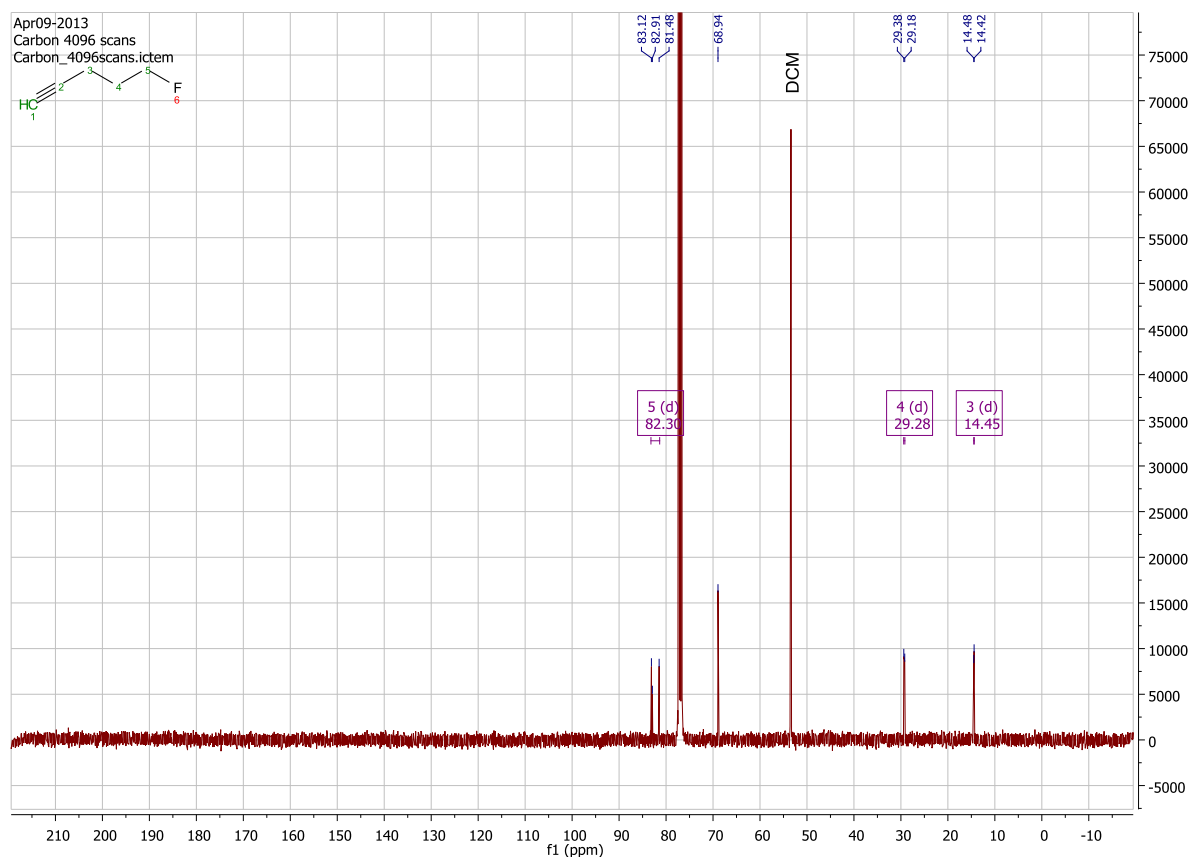


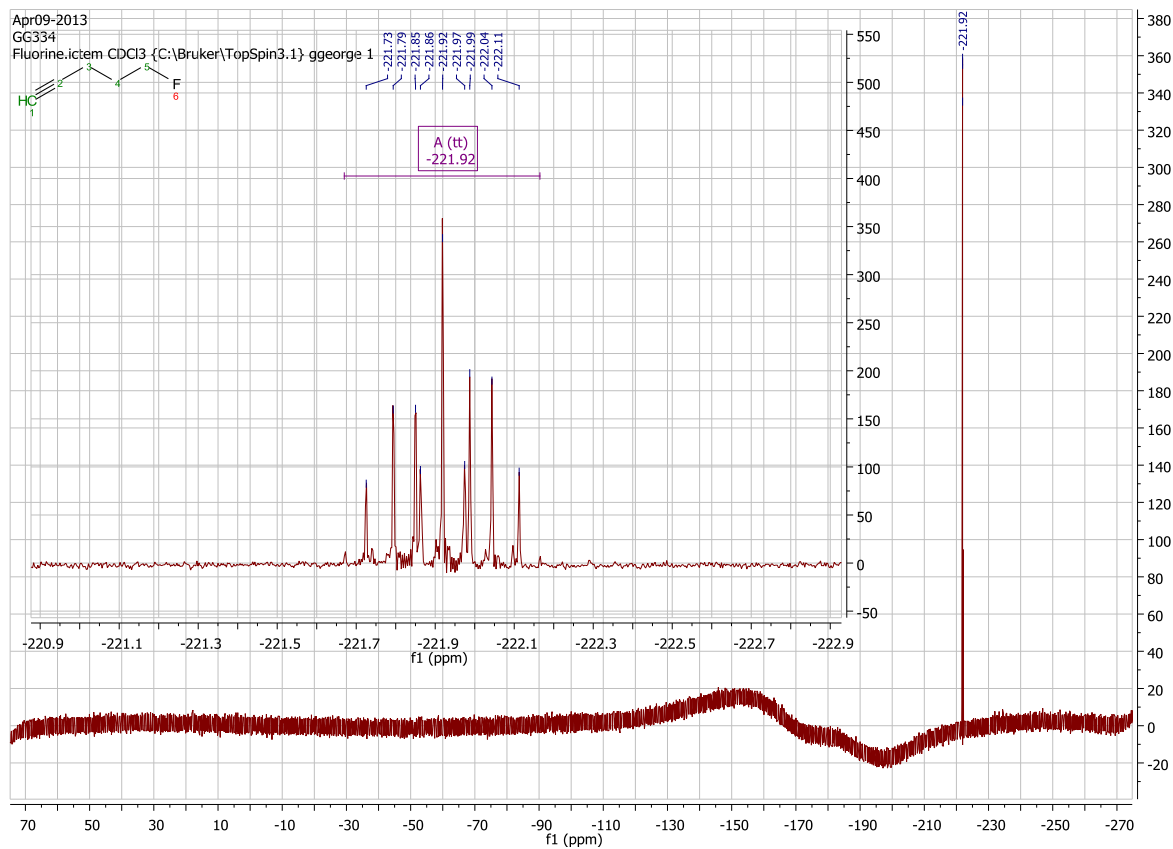
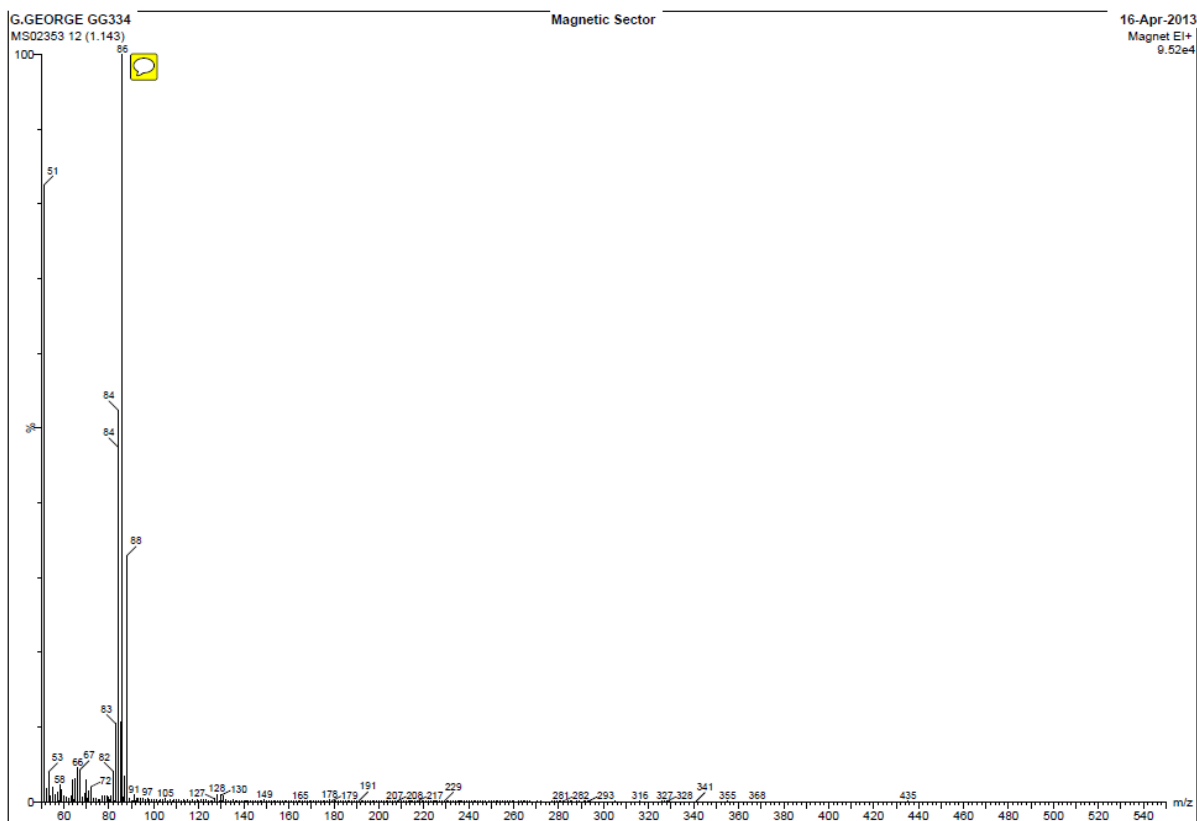
40. 5-Fluoropent-1-yne

^1H NMR spectrum



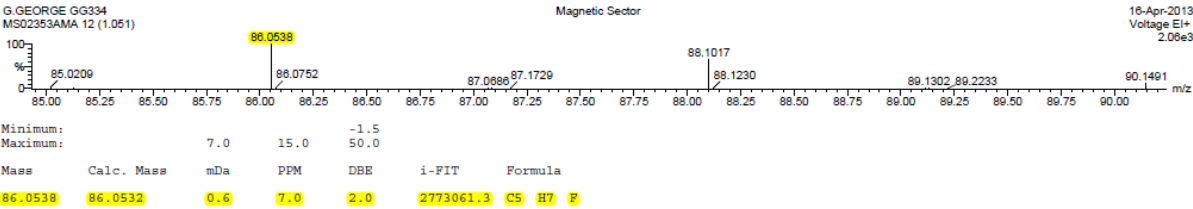
^{13}C NMR spectrum



^{19}F NMR spectrum*HRMS*

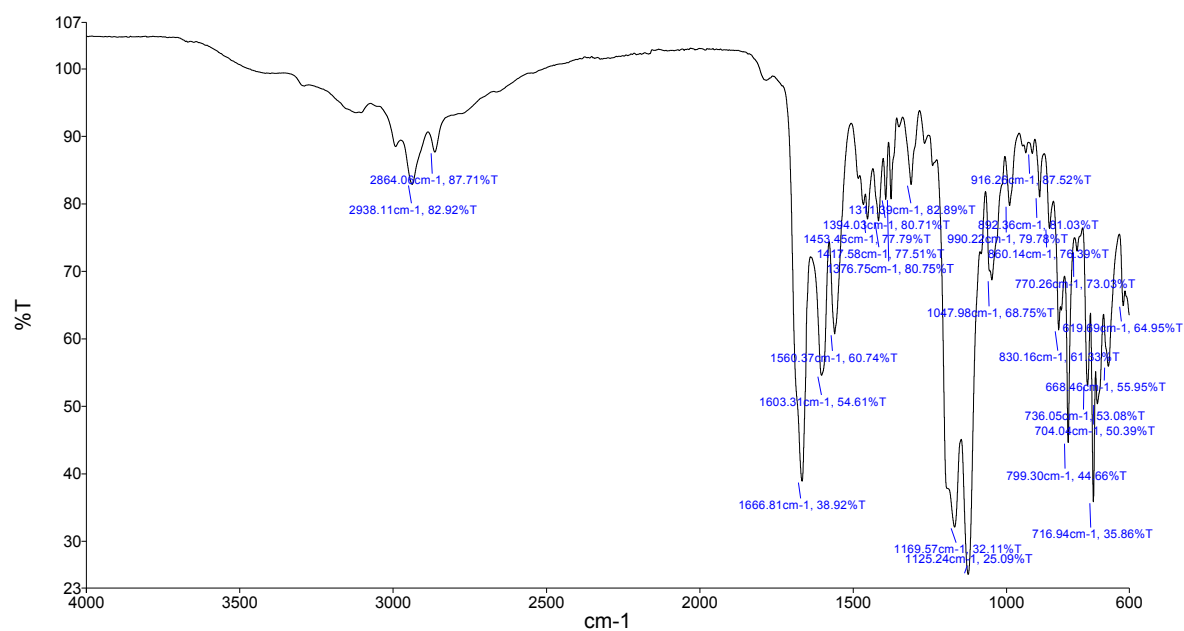
Single Mass Analysis (displaying only valid results)
Tolerance = 15.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
25 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 5-5 H: 0-500 11B: 0-1 N: 0-10 O: 0-10 80Se: 0-1 I: 0-2 F: 1-1

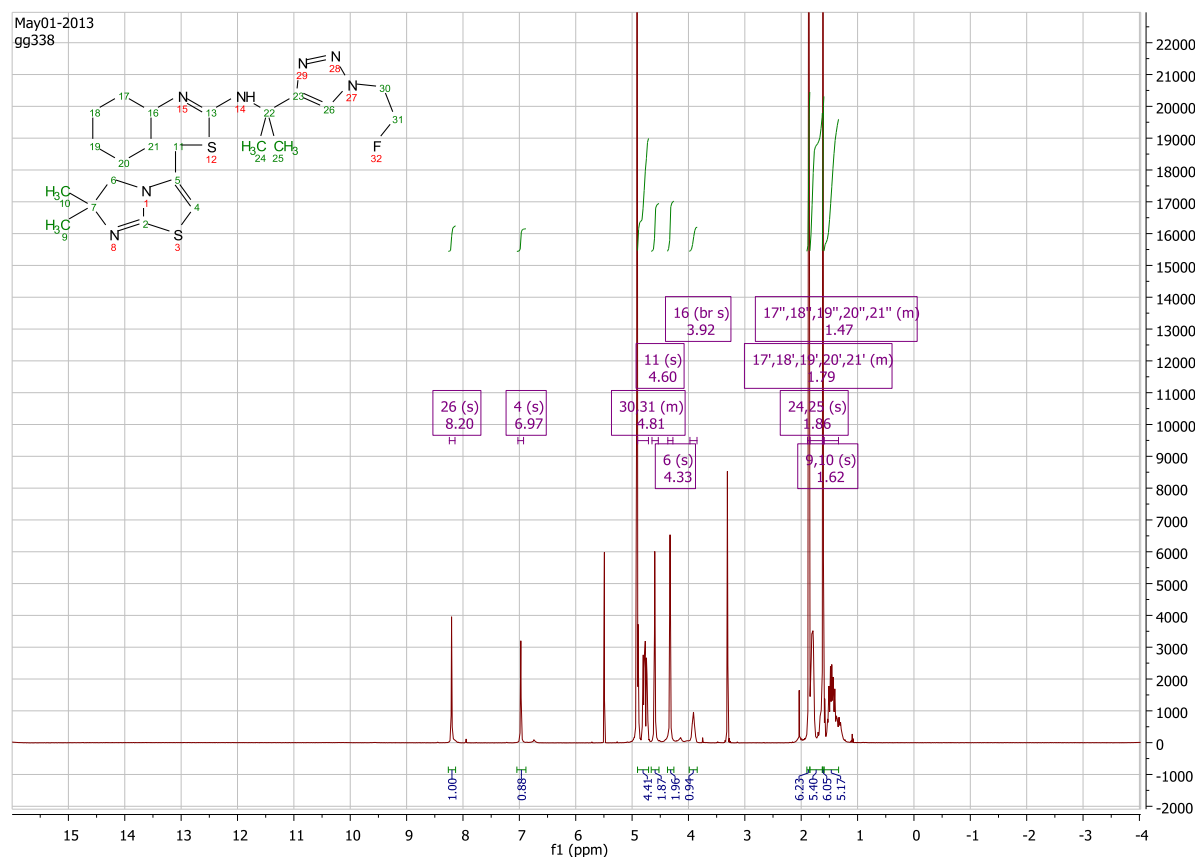


45. *N*'-Cyclohexyl-*N*-{2-[1-(2-fluoroethyl)-1*H*-1,2,3-triazol-4-yl]propan-2-yl}[(6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidamide .3TFA

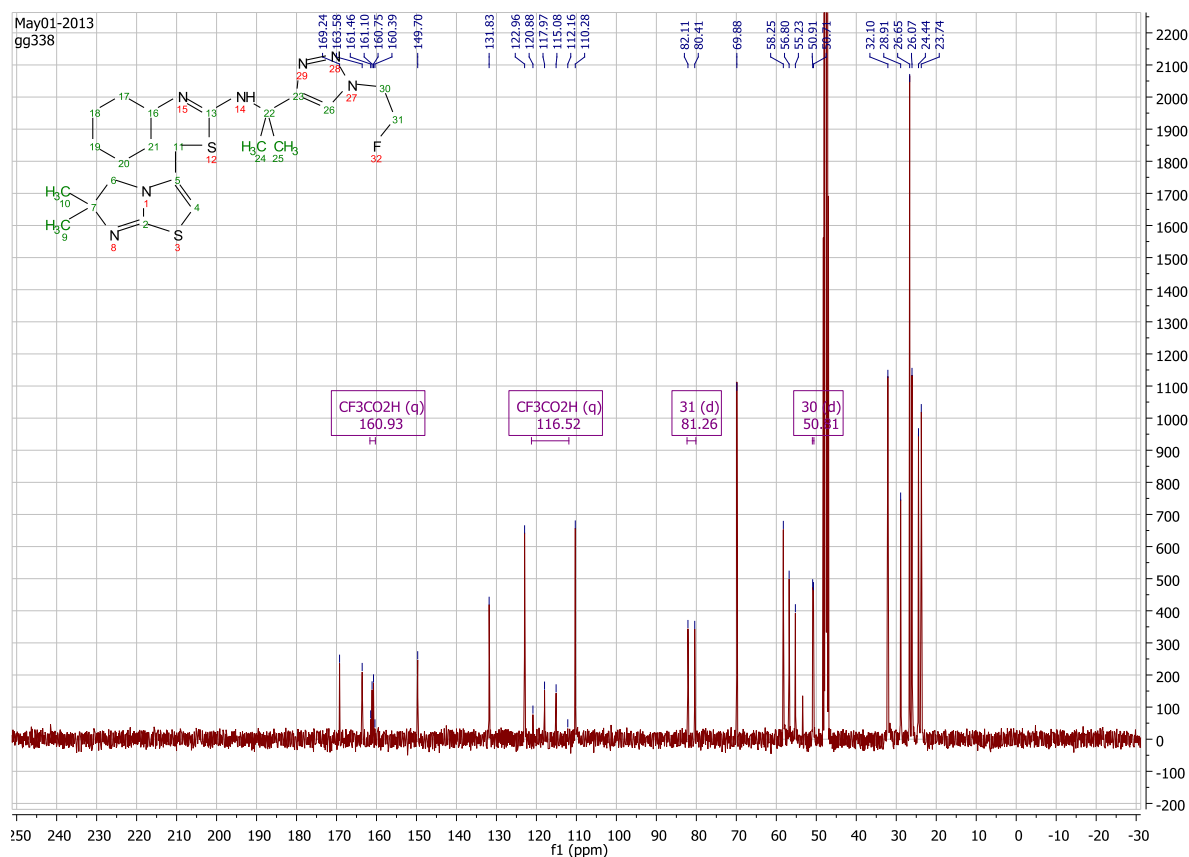
Infrared spectrum



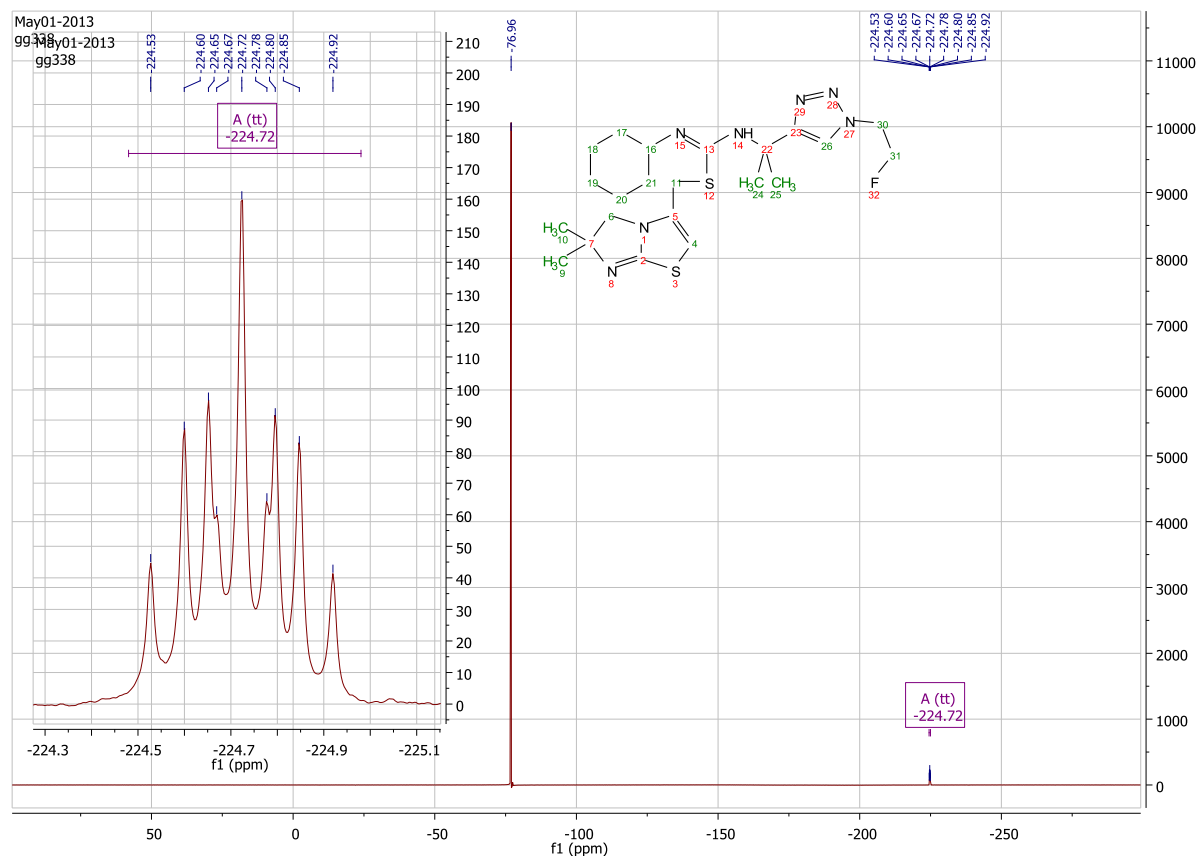
¹H NMR spectrum



^{13}C NMR spectrum



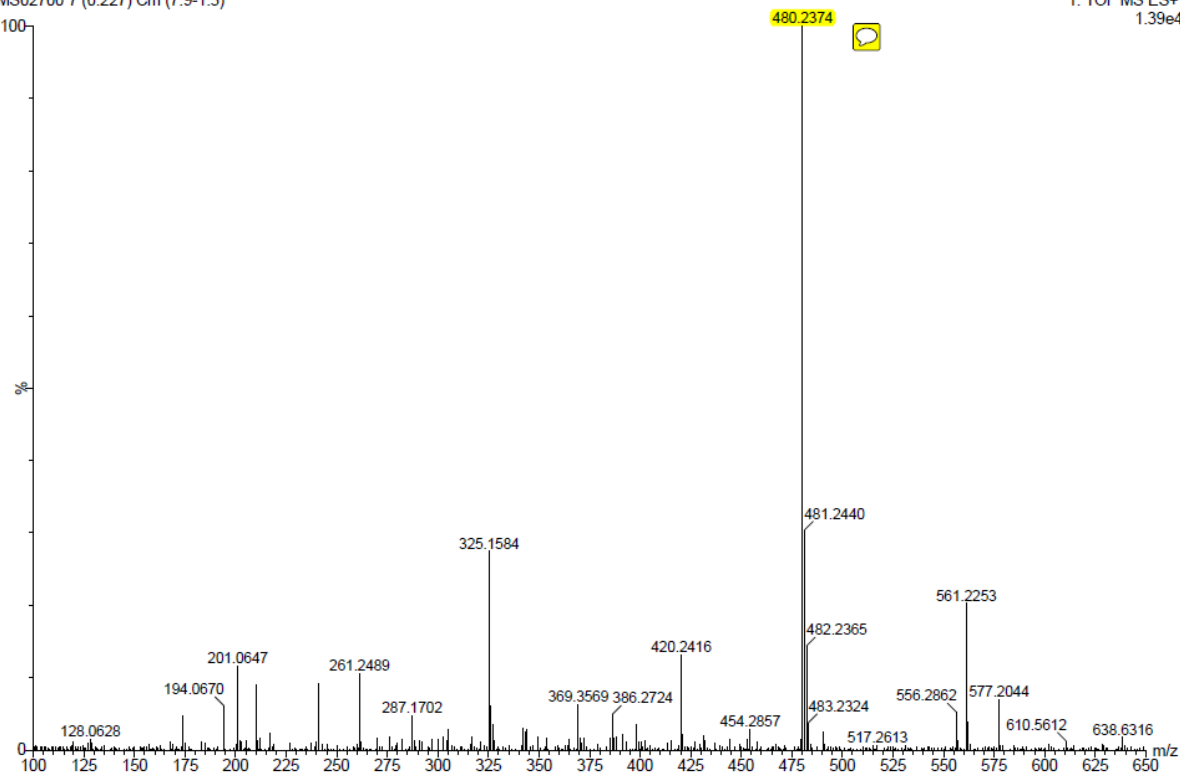
^{19}F NMR spectrum



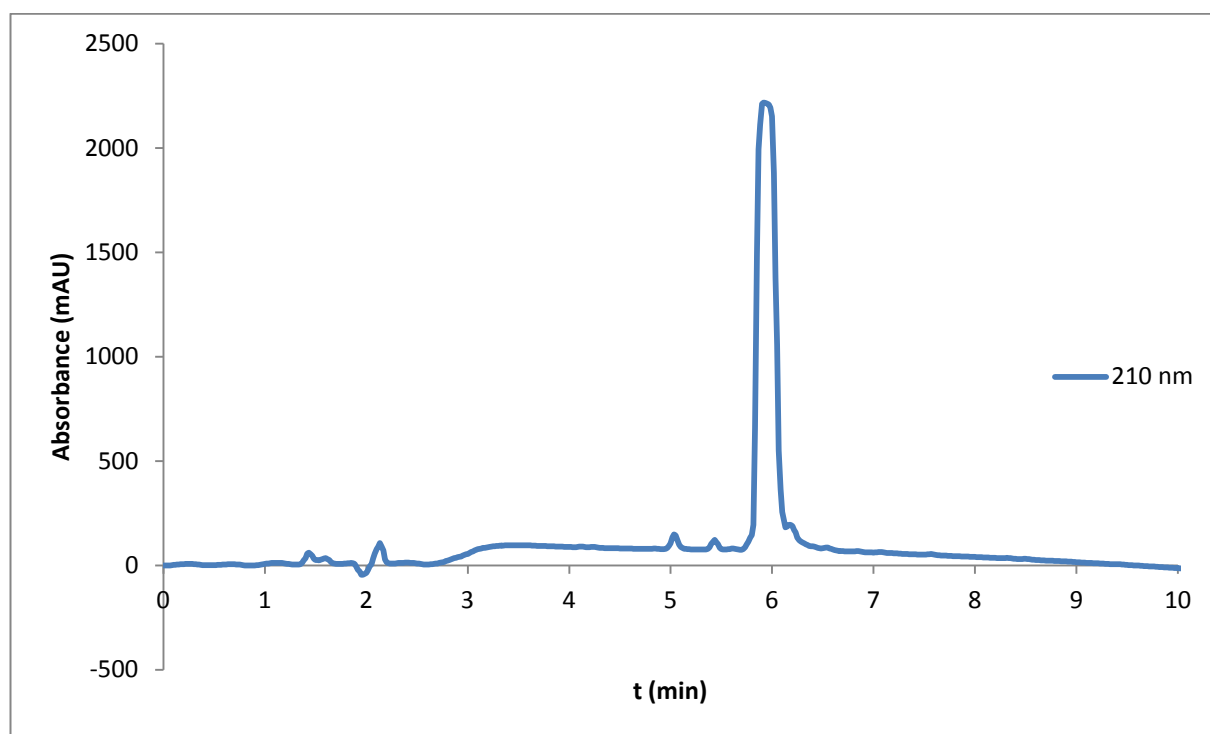
HRMS

G.GEORGE GG338-6MIN
MS02700 7 (0.227) Cm (7:9-1:3)

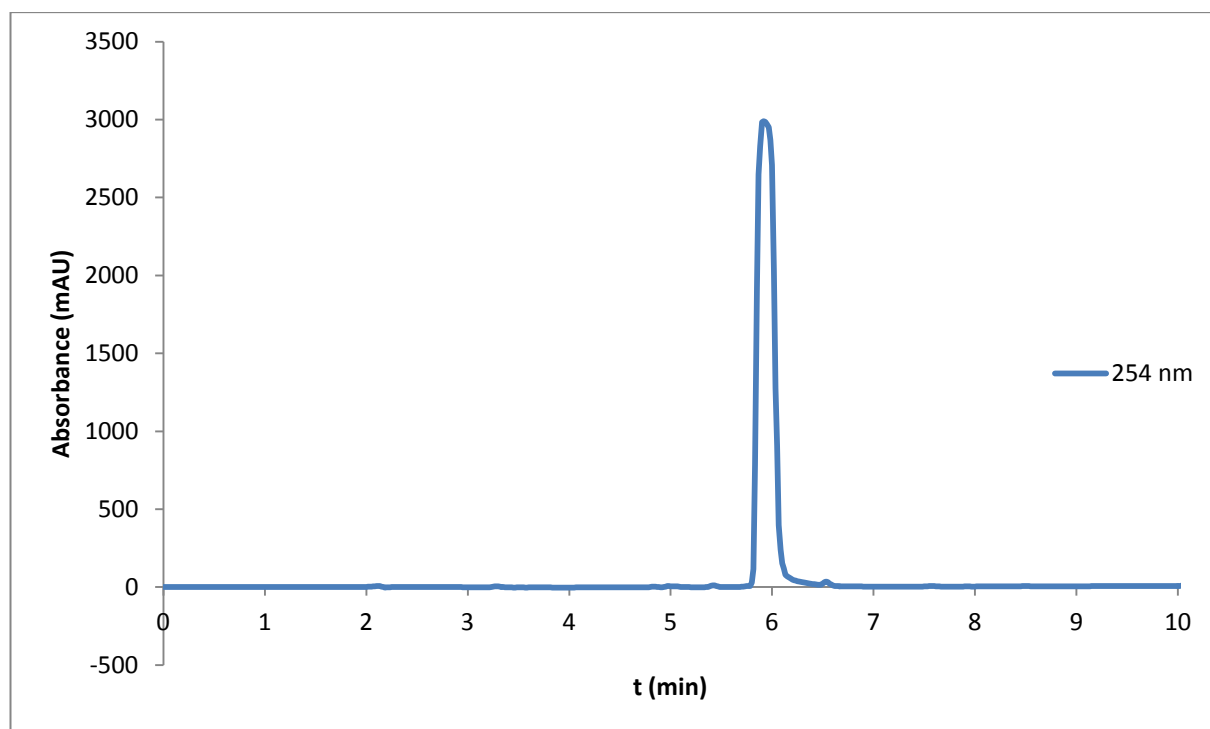
25-Apr-2013
1: TOF MS ES+
1.39e4



HPLC trace at 210 nm

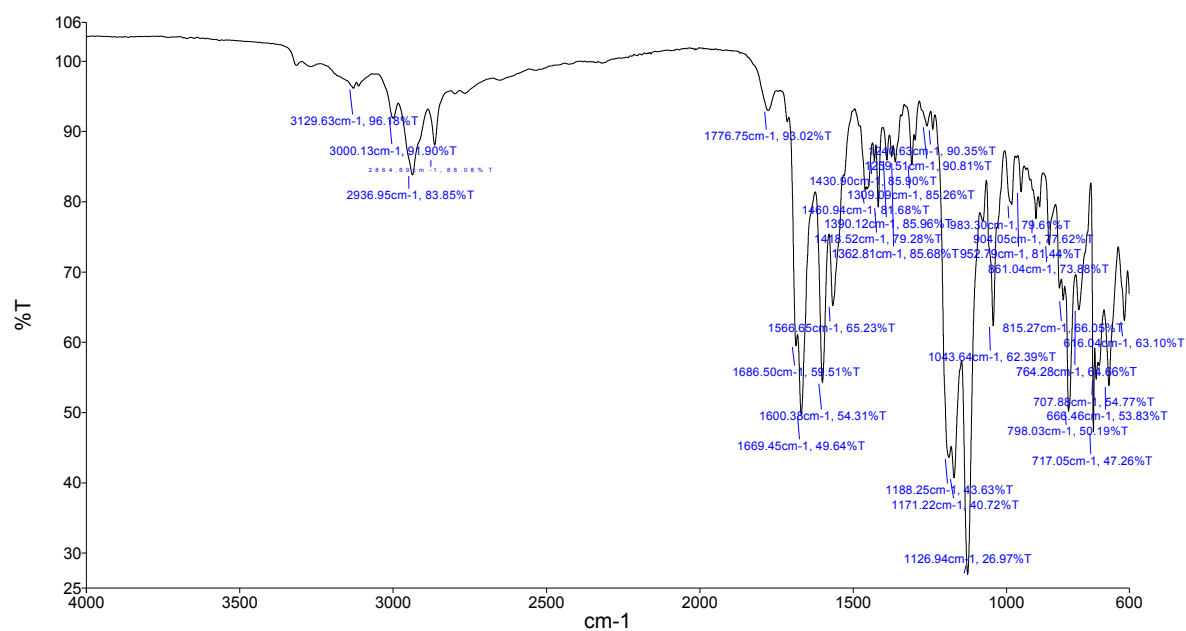


HPLC trace at 254 nm

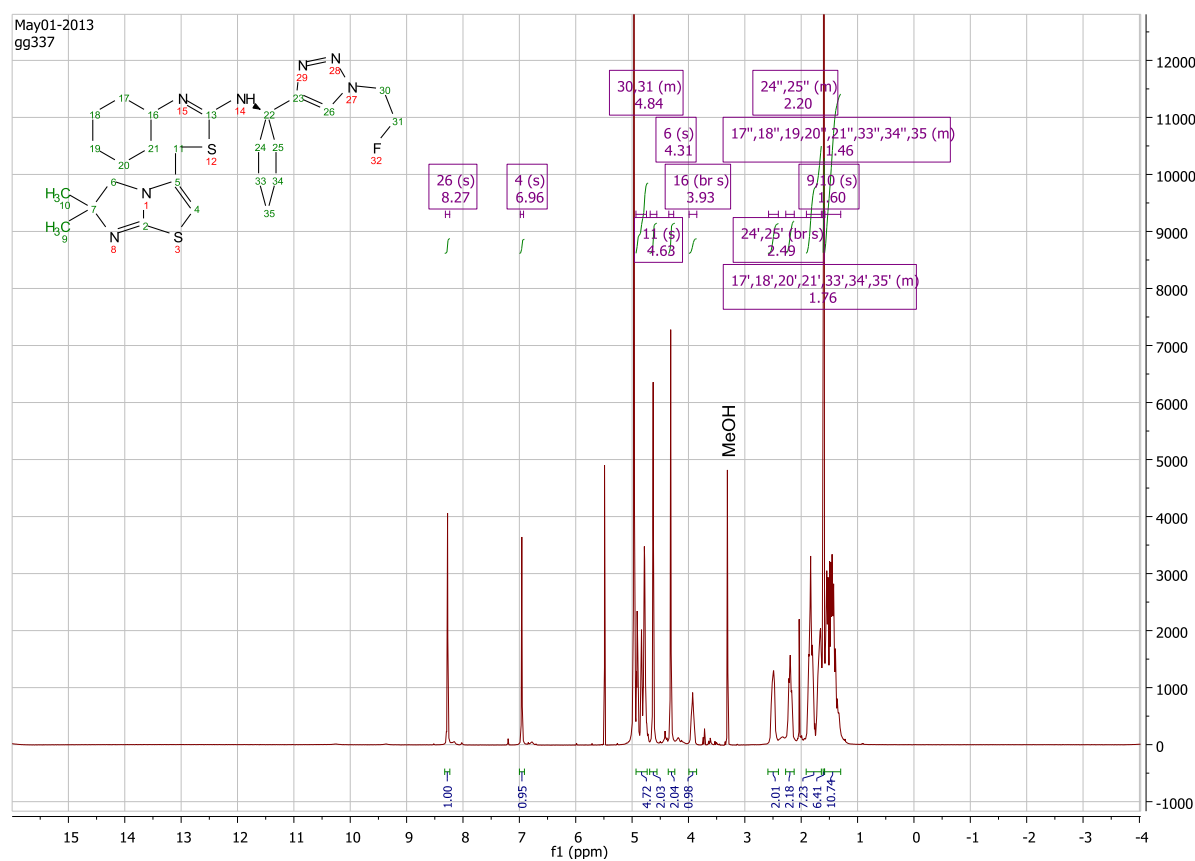


46. *N*'-Cyclohexyl-*N*-{1-[1-(2-fluoroethyl)-1*H*-1,2,3-triazol-4-yl]cyclohexyl}[(6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidamide .3TFA

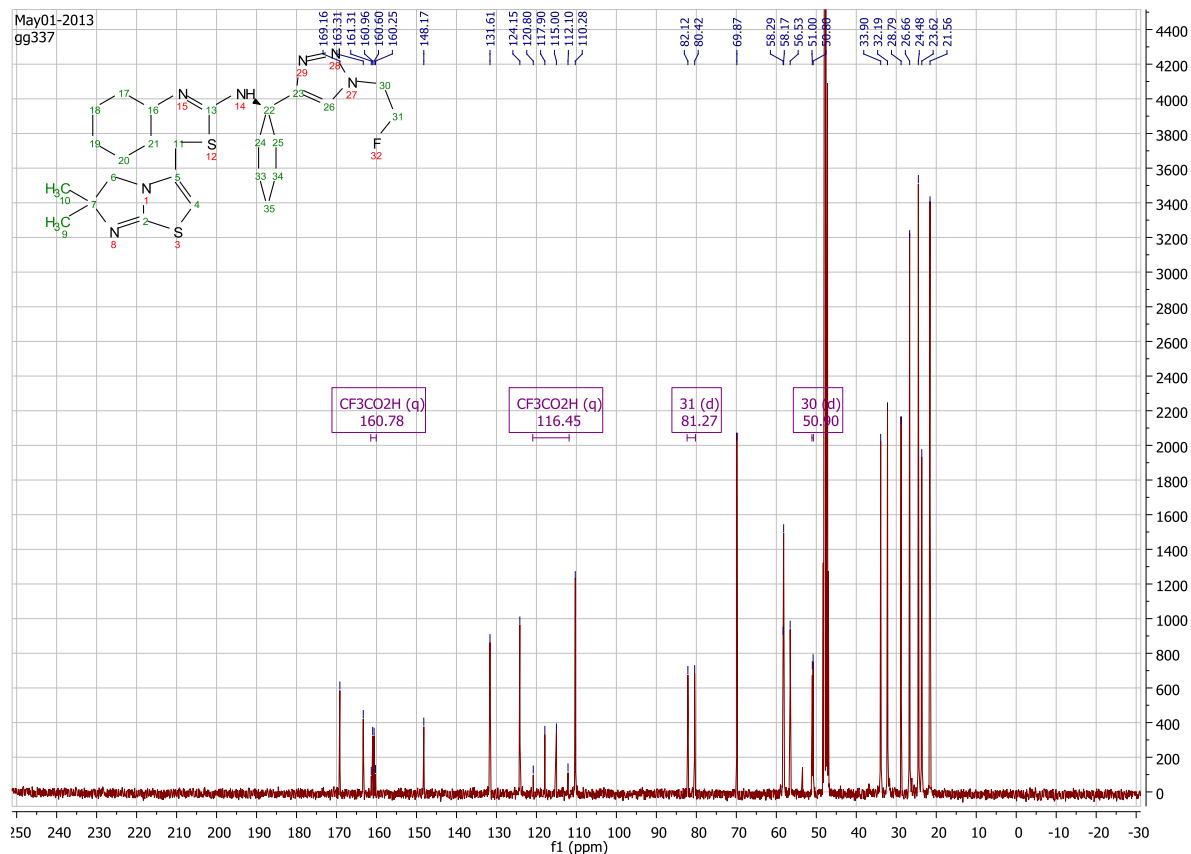
Infrared spectrum



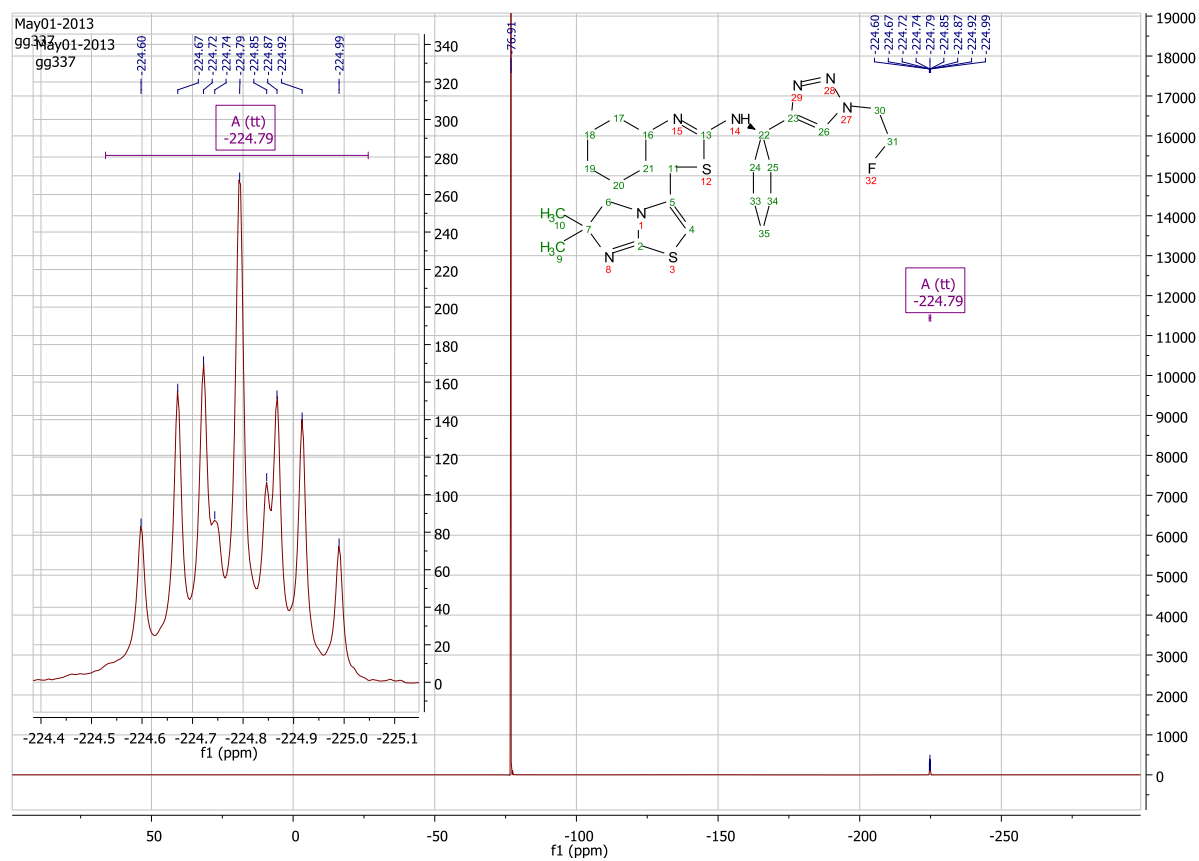
¹H NMR spectrum



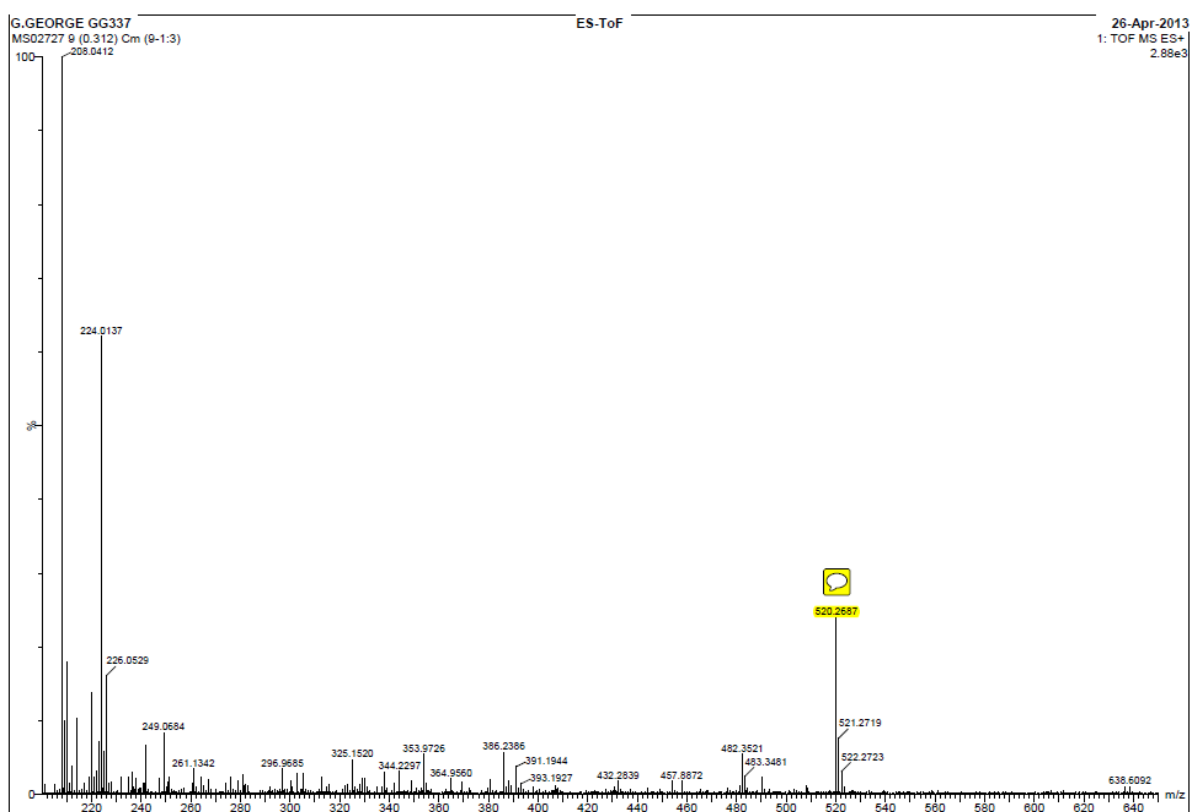
^{13}C NMR spectrum



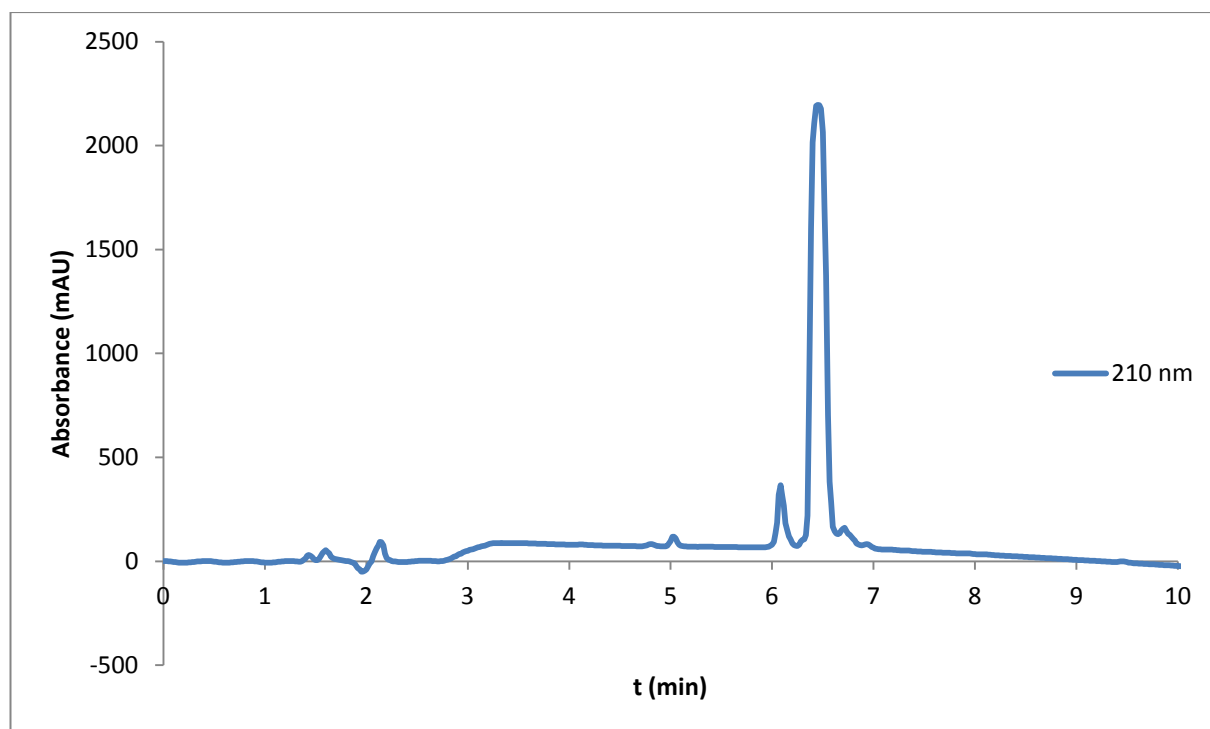
^{19}F NMR spectrum



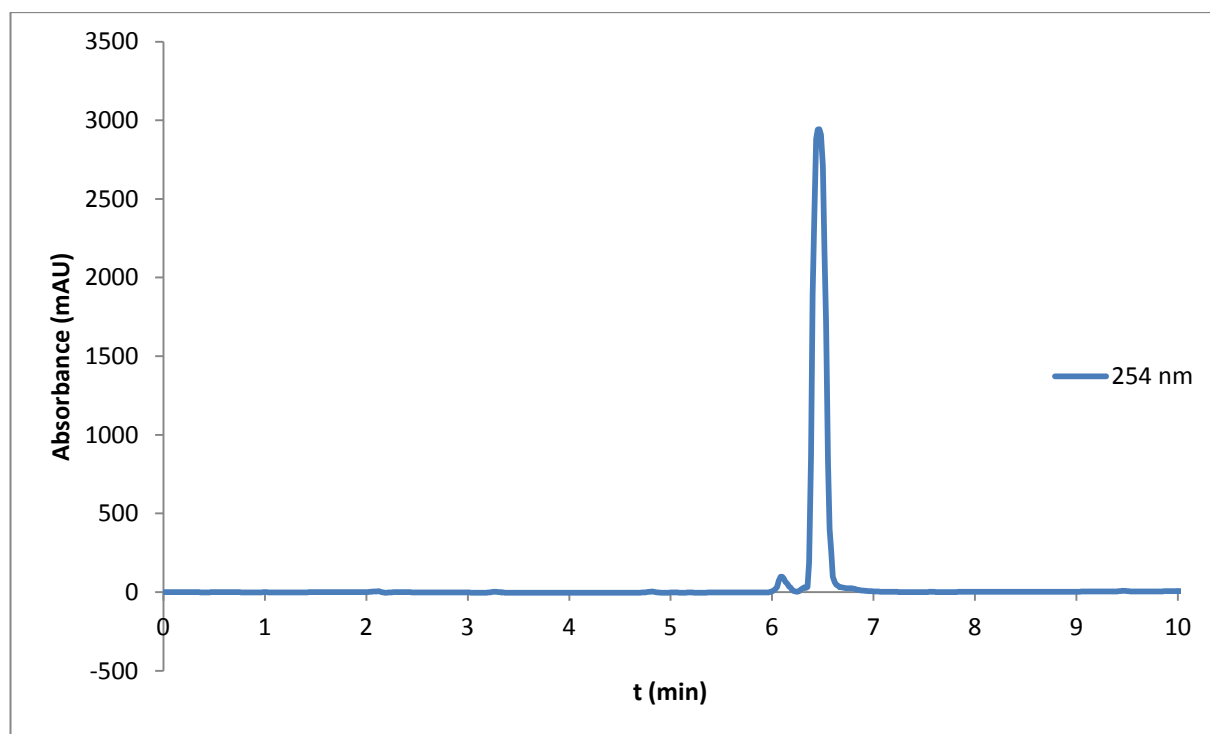
HRMS



HPLC at 210 nm

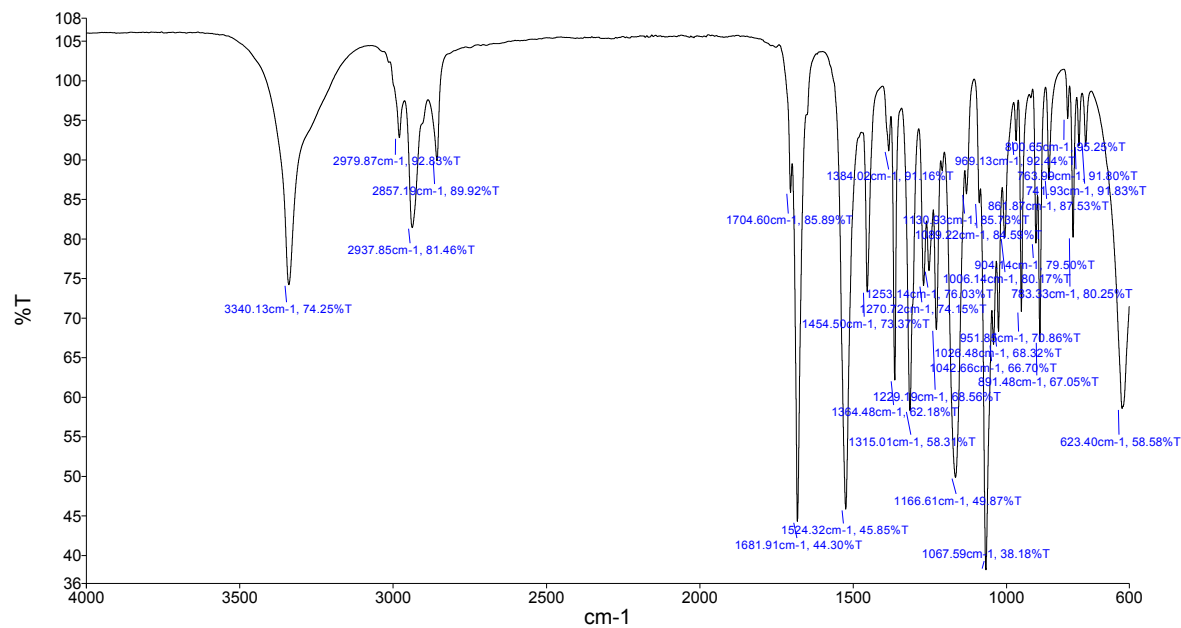


HPLC at 254 nm



47. *tert*-Butyl *N*-[(1*r*,4*r*)-4-hydroxycyclohexyl]carbamate

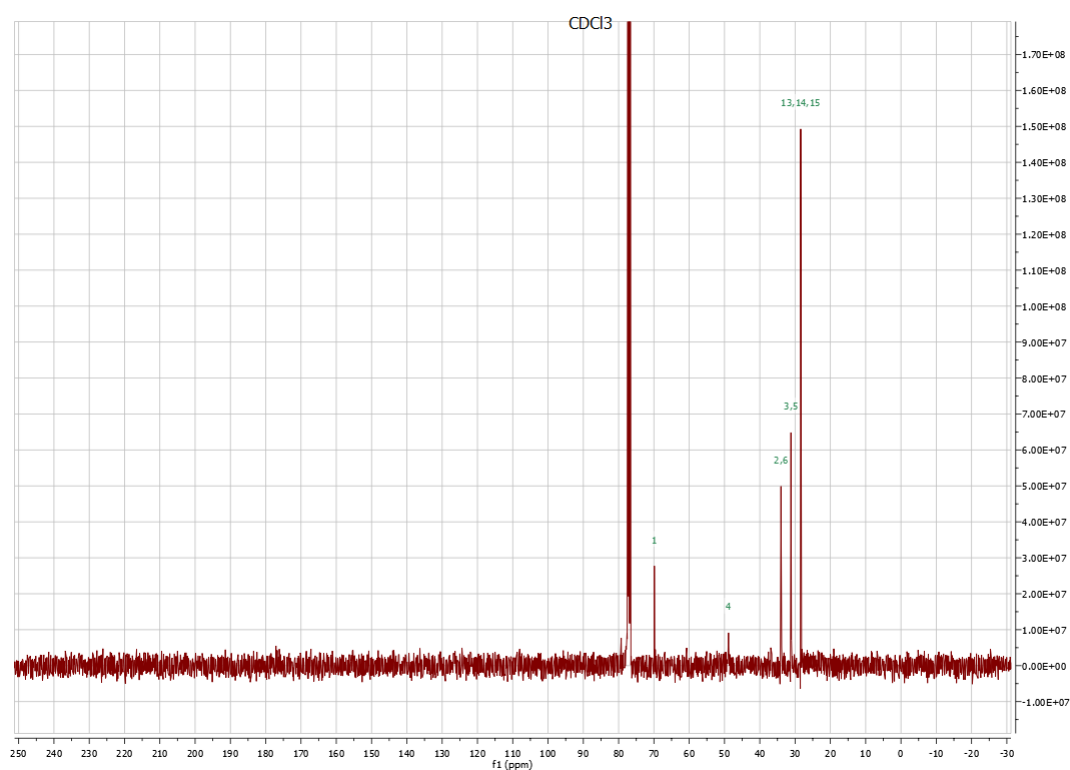
Infrared spectrum



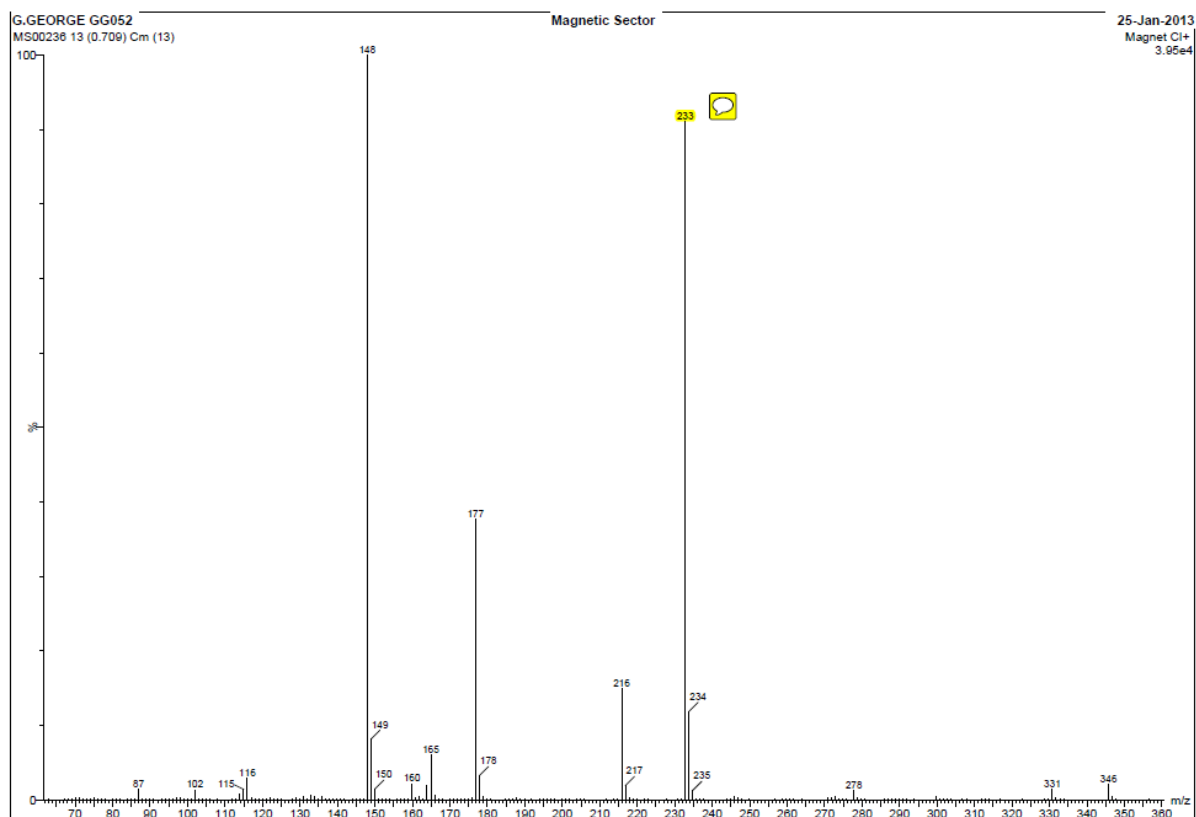
^1H NMR spectrum



^{13}C NMR spectrum

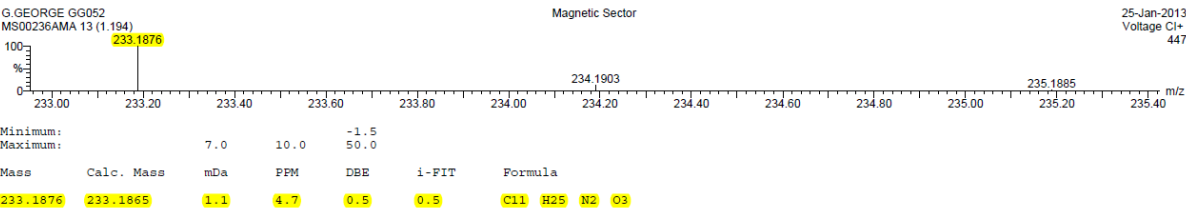


HRMS



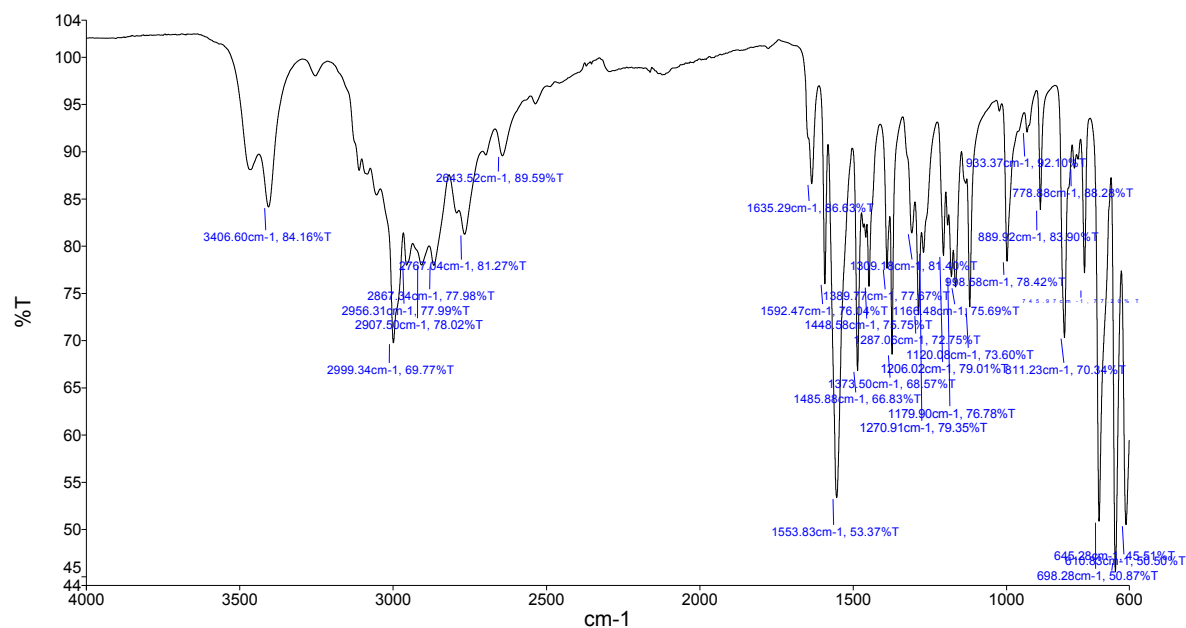
Single Mass Analysis (displaying only valid results)
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
137 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 11-11 H: 0-500 N: 0-10 O: 0-10 I: 0-2

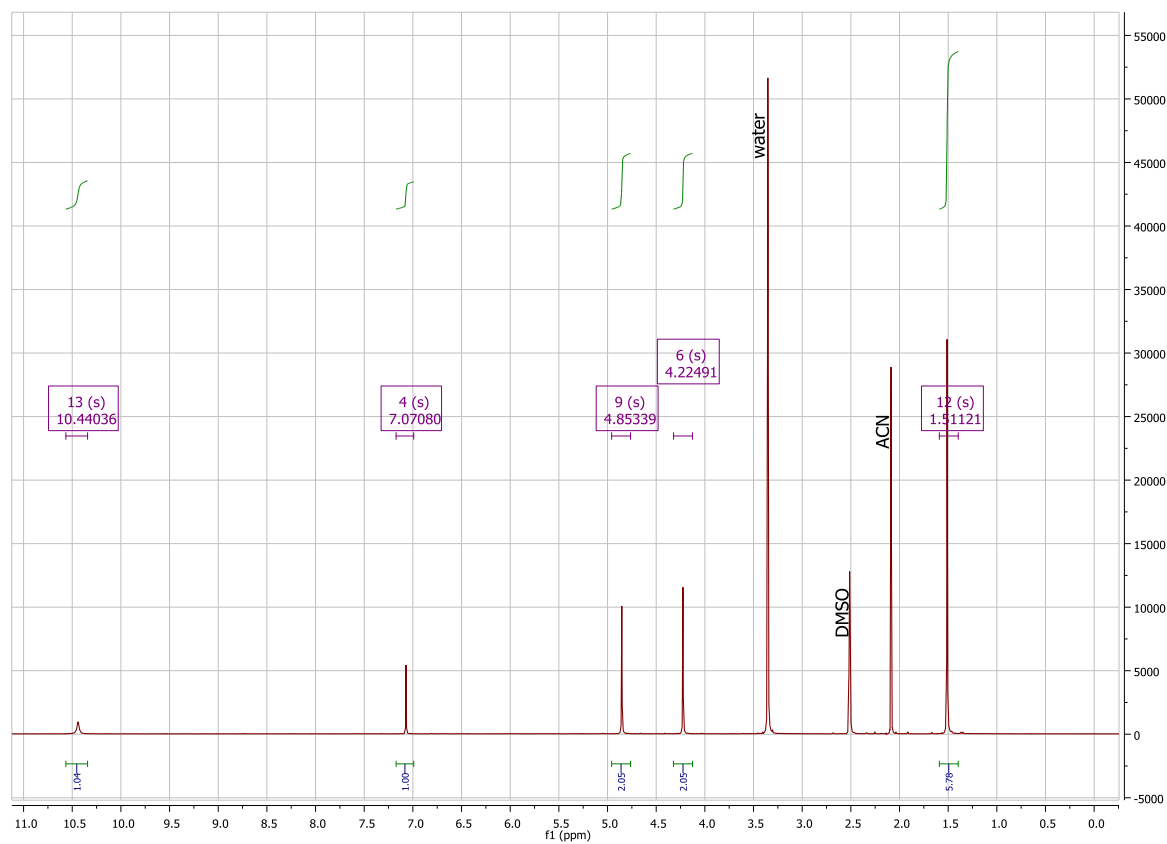


52. 3-(Chloromethyl)-6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazole hydrochloride

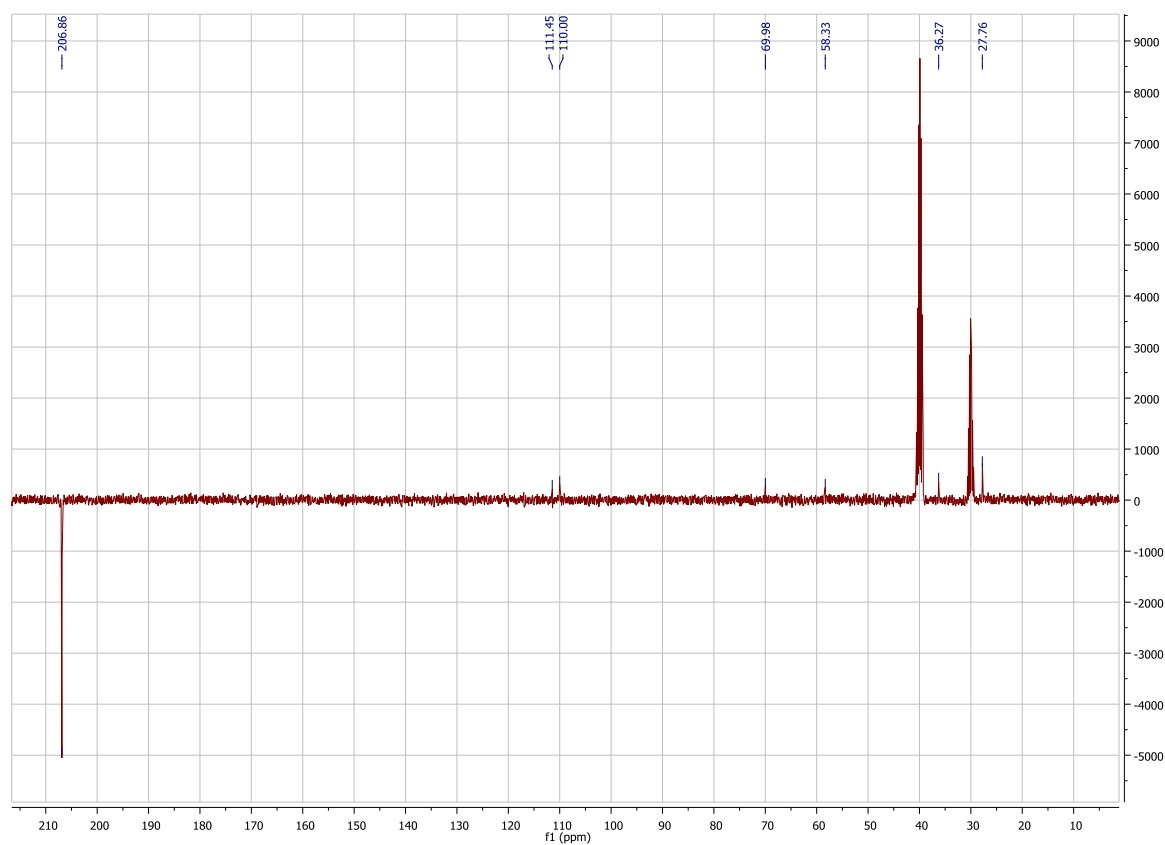
Infrared spectrum



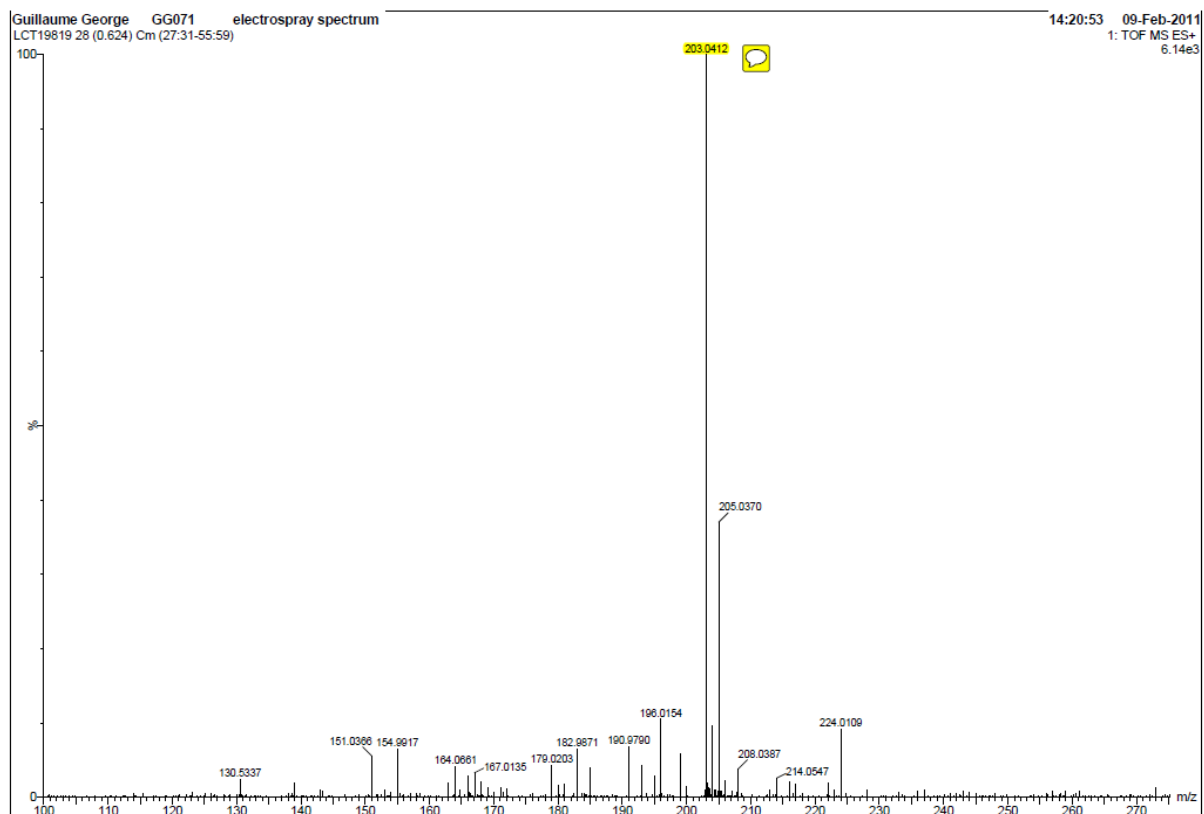
¹H NMR spectrum



^{13}C NMR spectrum

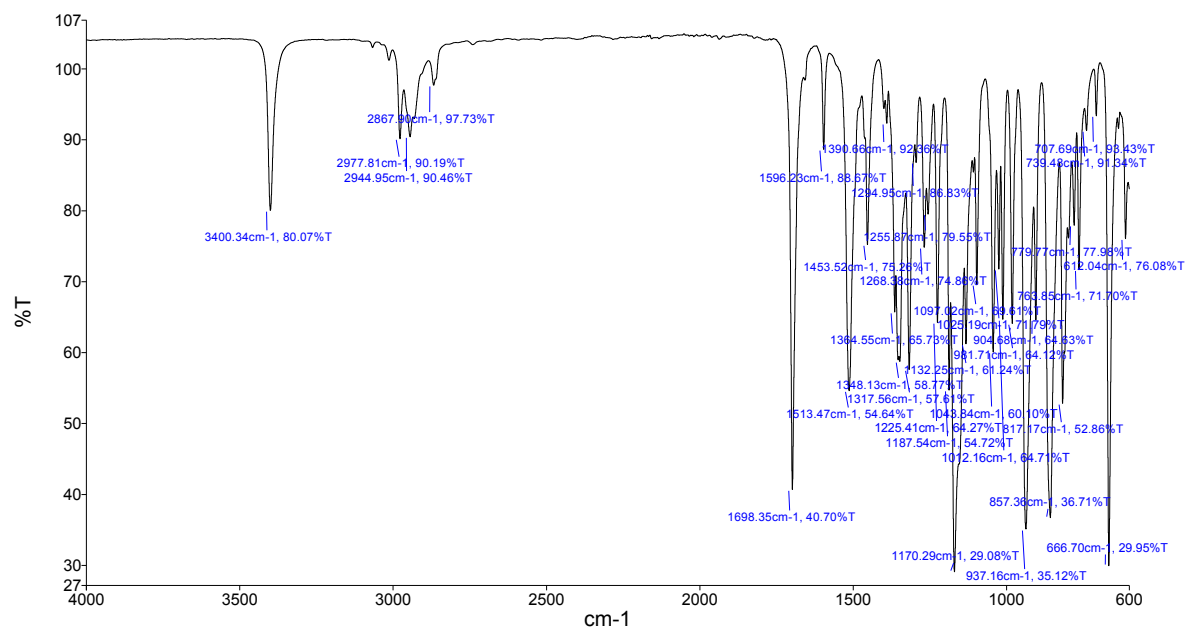


HRMS

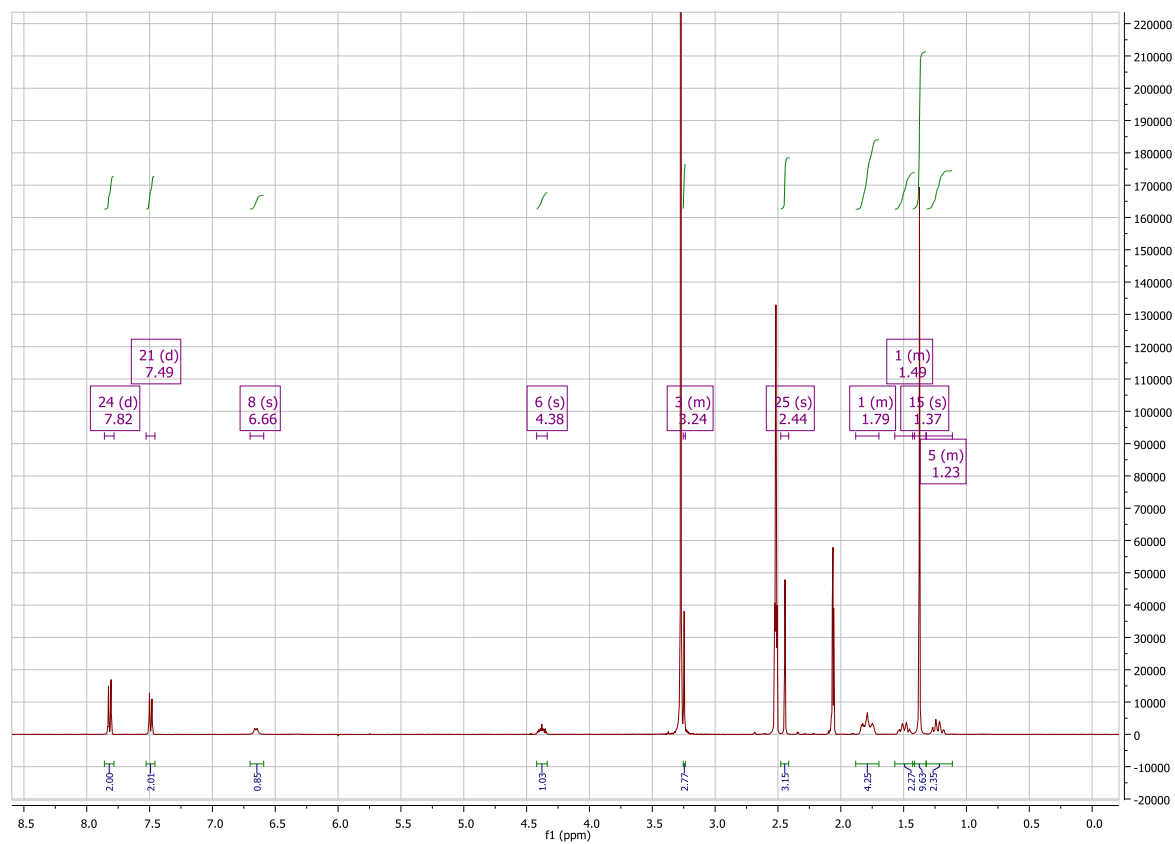


57. tert-Butyl N-[(1r,4r)-4-[(4-methylbenzenesulfonyl)oxy]cyclohexyl]carbamate

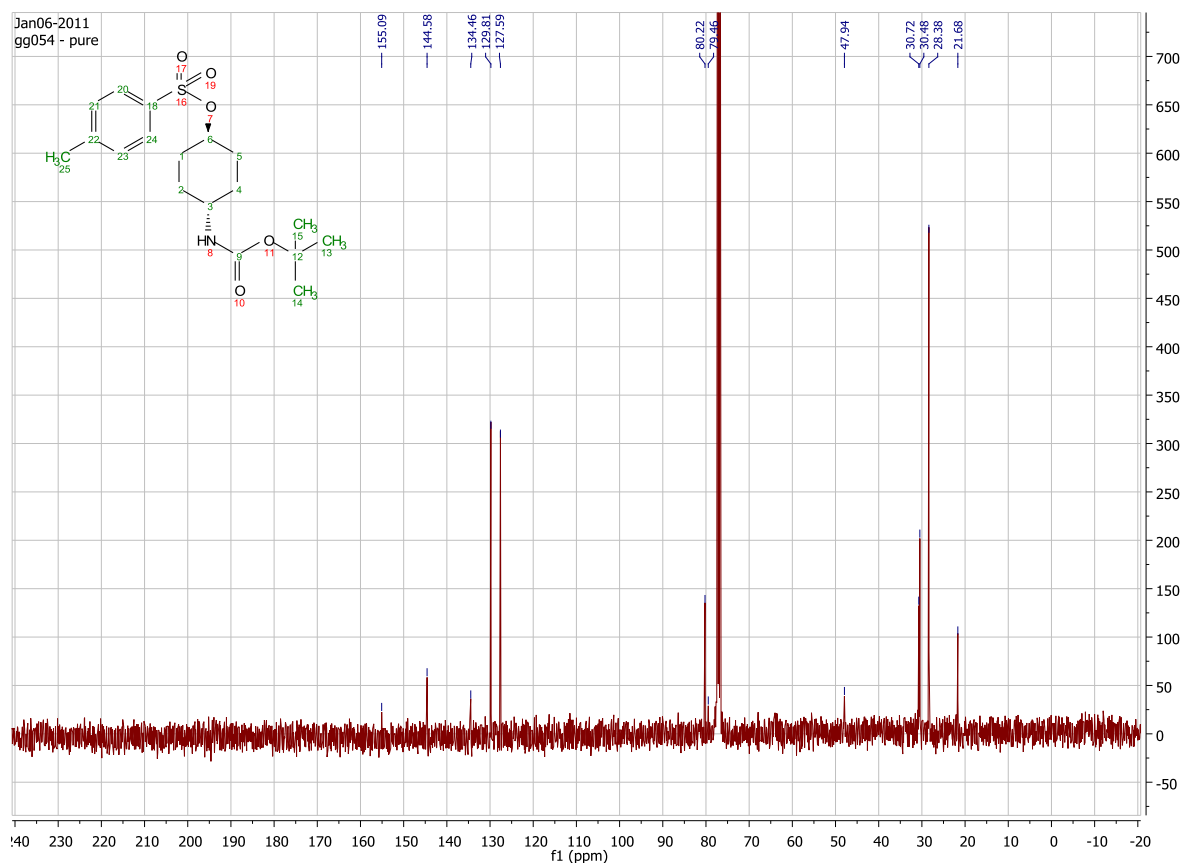
Infrared spectrum



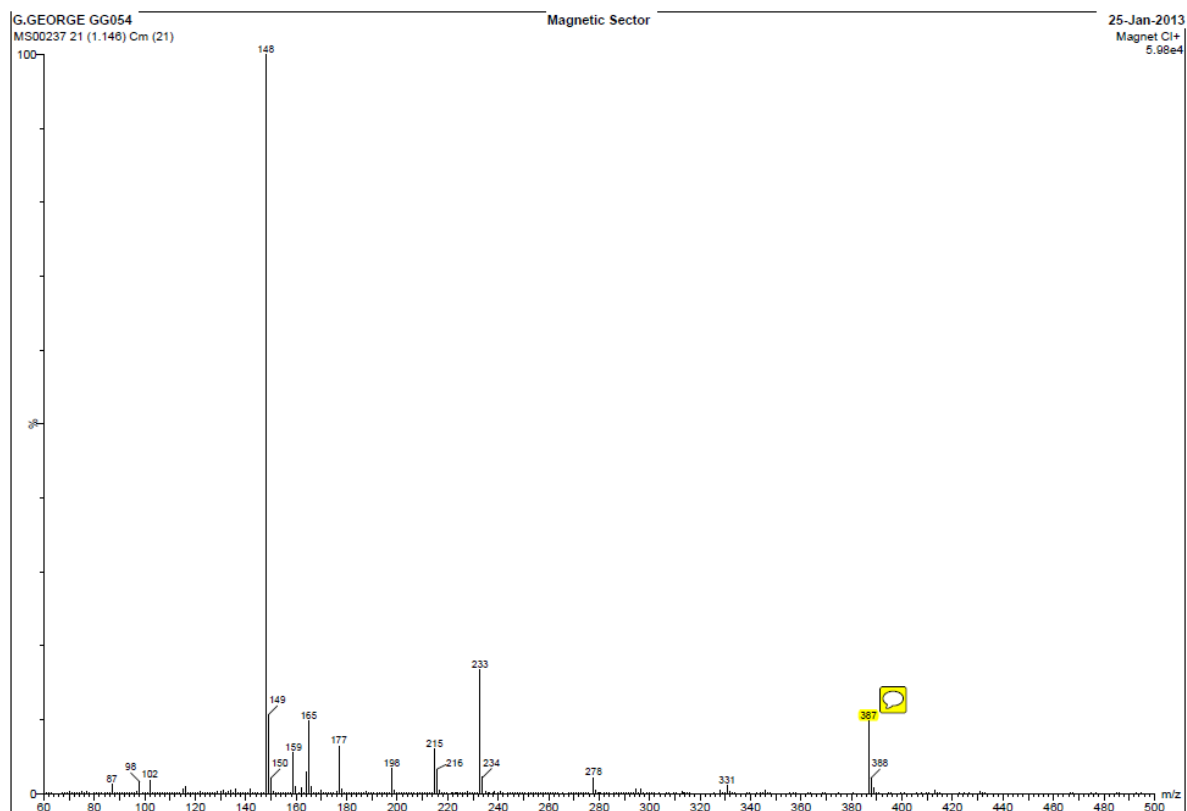
¹H NMR spectrum



¹³C NMR spectrum

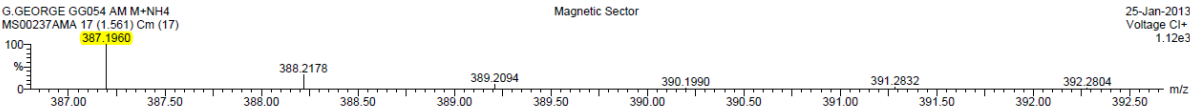


HRMS



Single Mass Analysis (displaying only valid results)
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

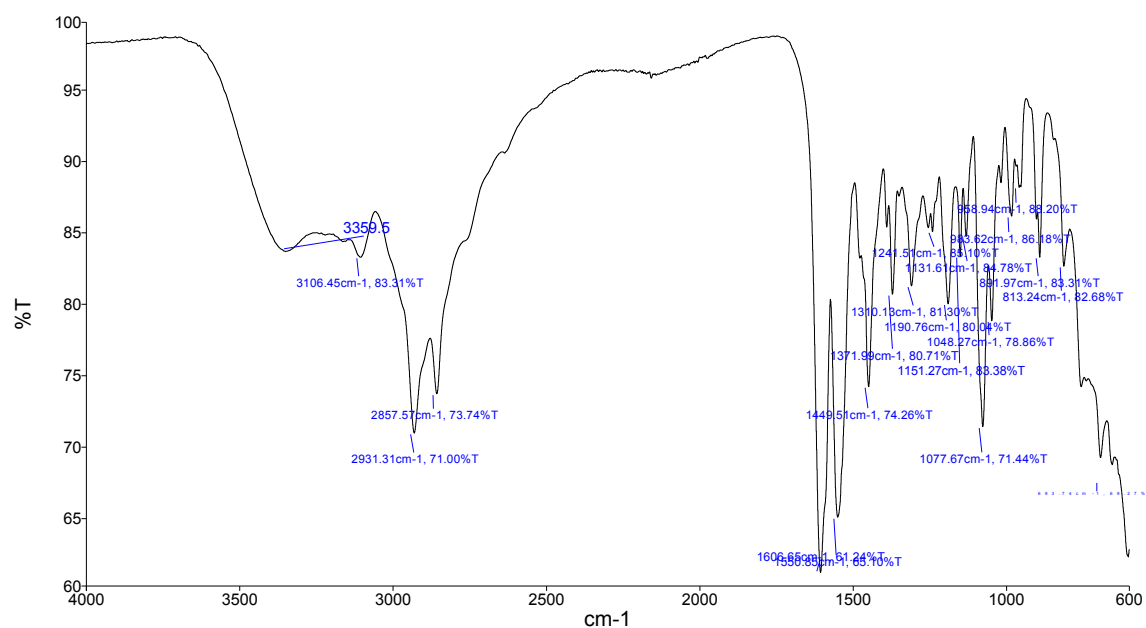
Monoisotopic Mass, Odd and Even Electron Ions
250 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)
Elements Used:
C: 18-18 H: 0-500 N: 0-10 O: 0-10 S: 1-1 I: 0-2



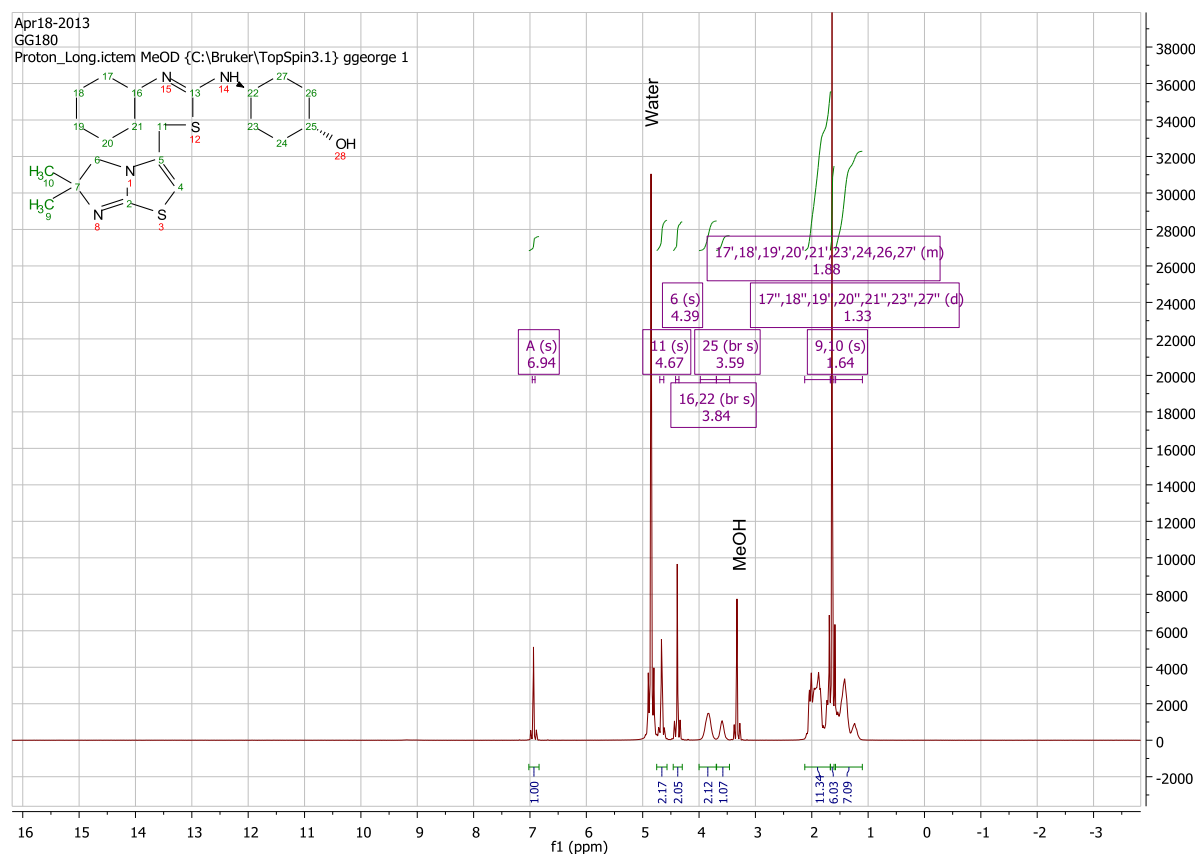
Minimum:				-1.5		
Maximum:		7.0	10.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
387.1960	387.1954	0.6	1.5	4.5	20.5	C18 H31 N2 O5 S

58. *N'*-Cyclohexyl-*N*-[(1*r*,4*r*)-4-hydroxycyclohexyl][({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidamide dihydrochloride

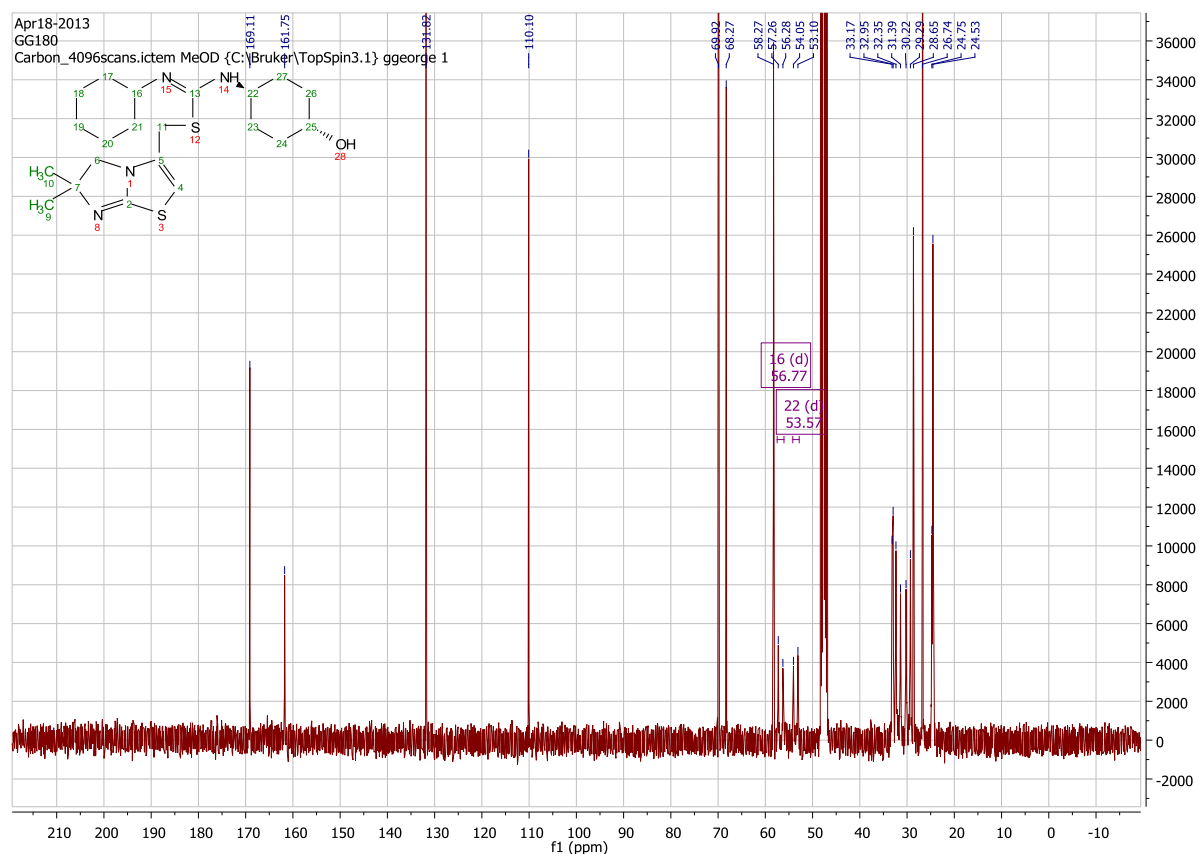
Infrared spectrum



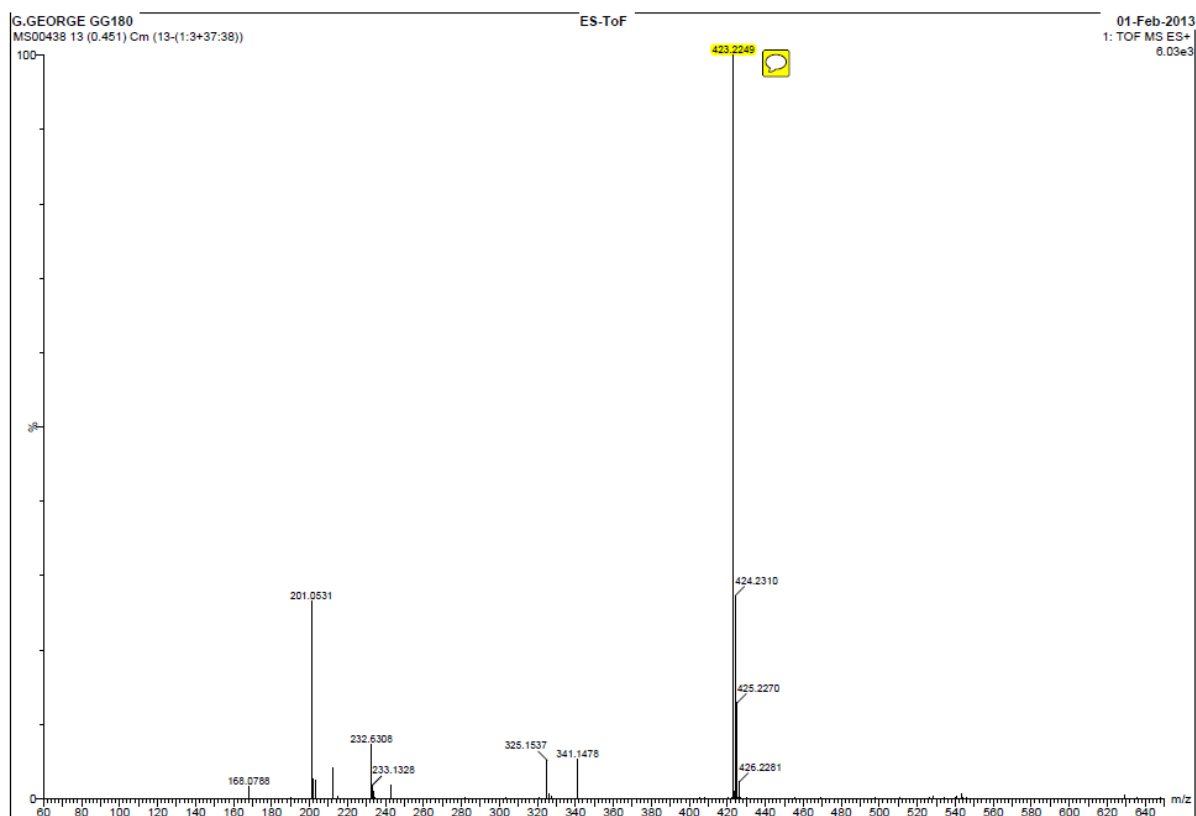
¹H NMR spectrum



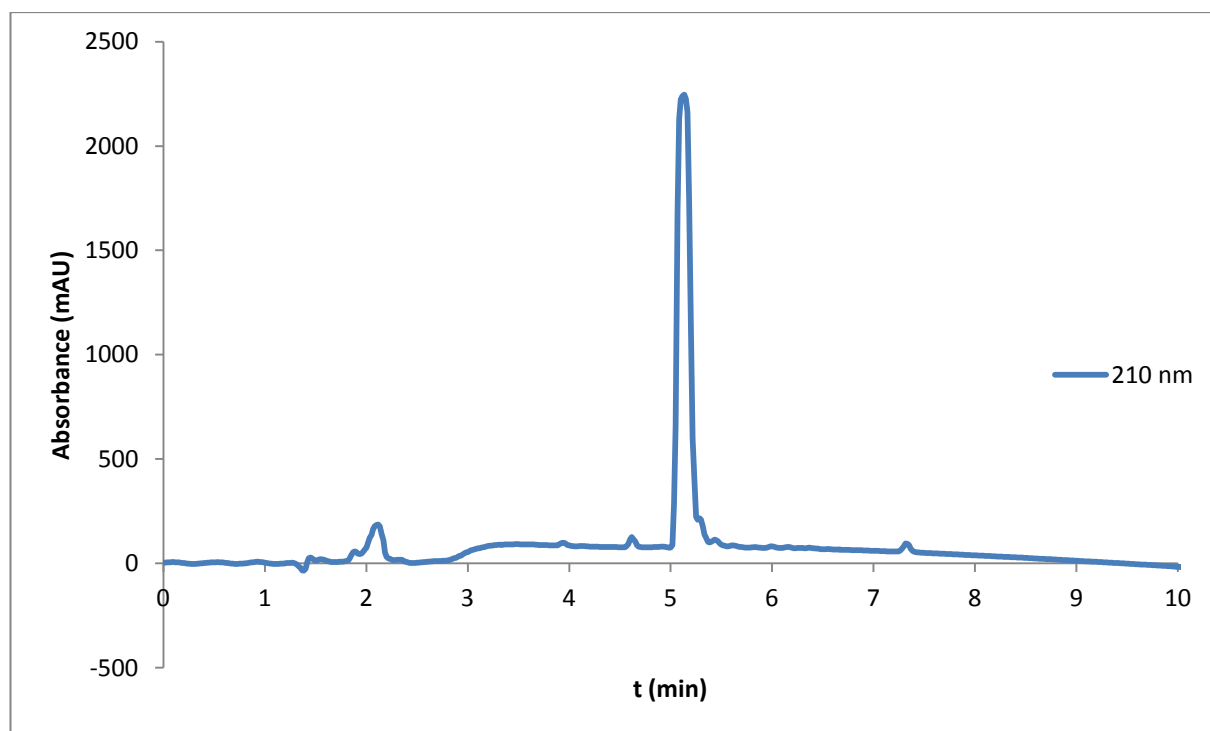
^{13}C NMR spectrum



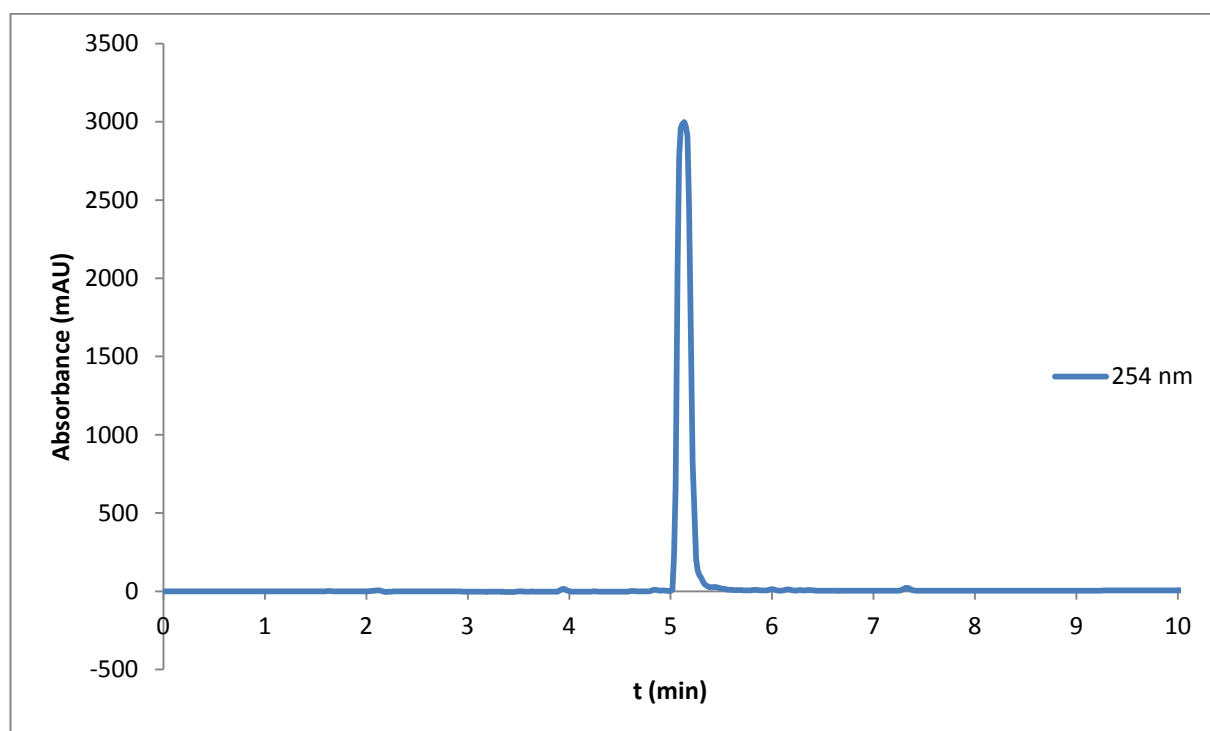
HRMS



HPLC at 210 nm

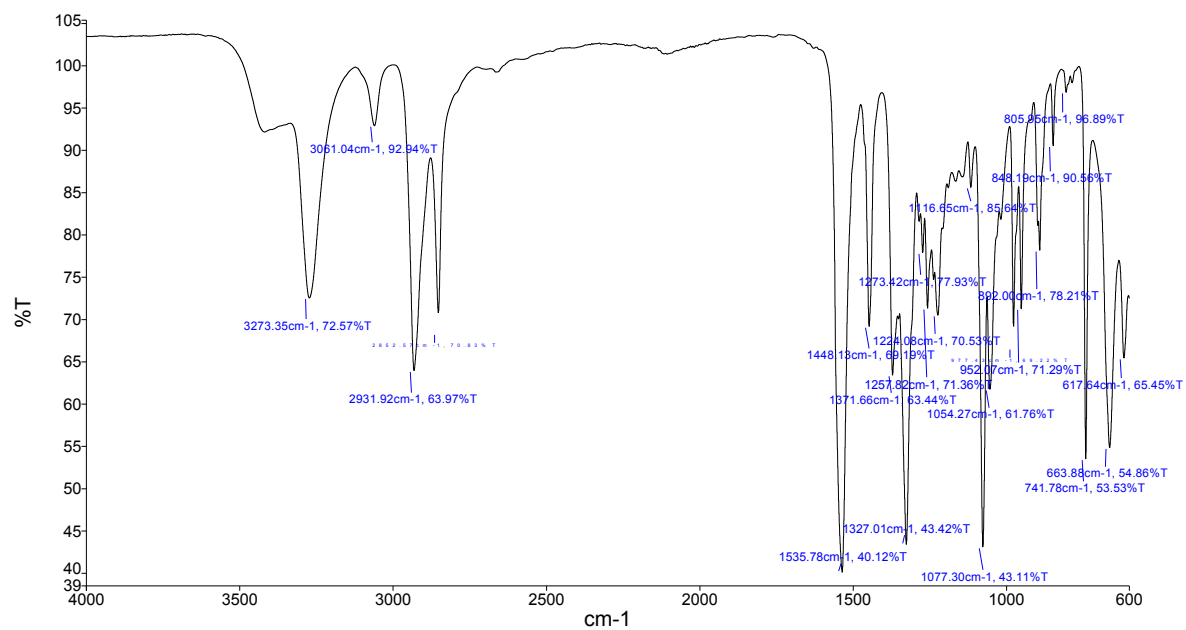


HPLC at 254 nm

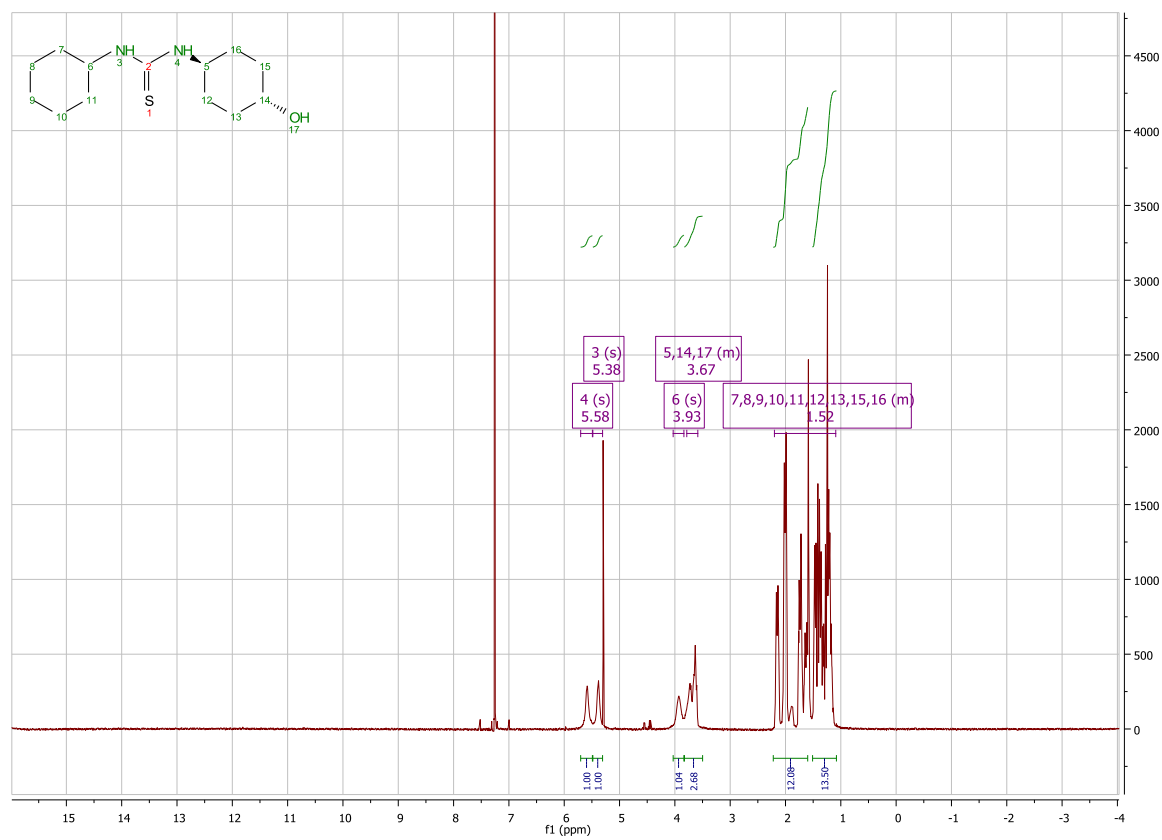


59. 3-Cyclohexyl-1-[(1r,4r)-4-hydroxycyclohexyl]thiourea

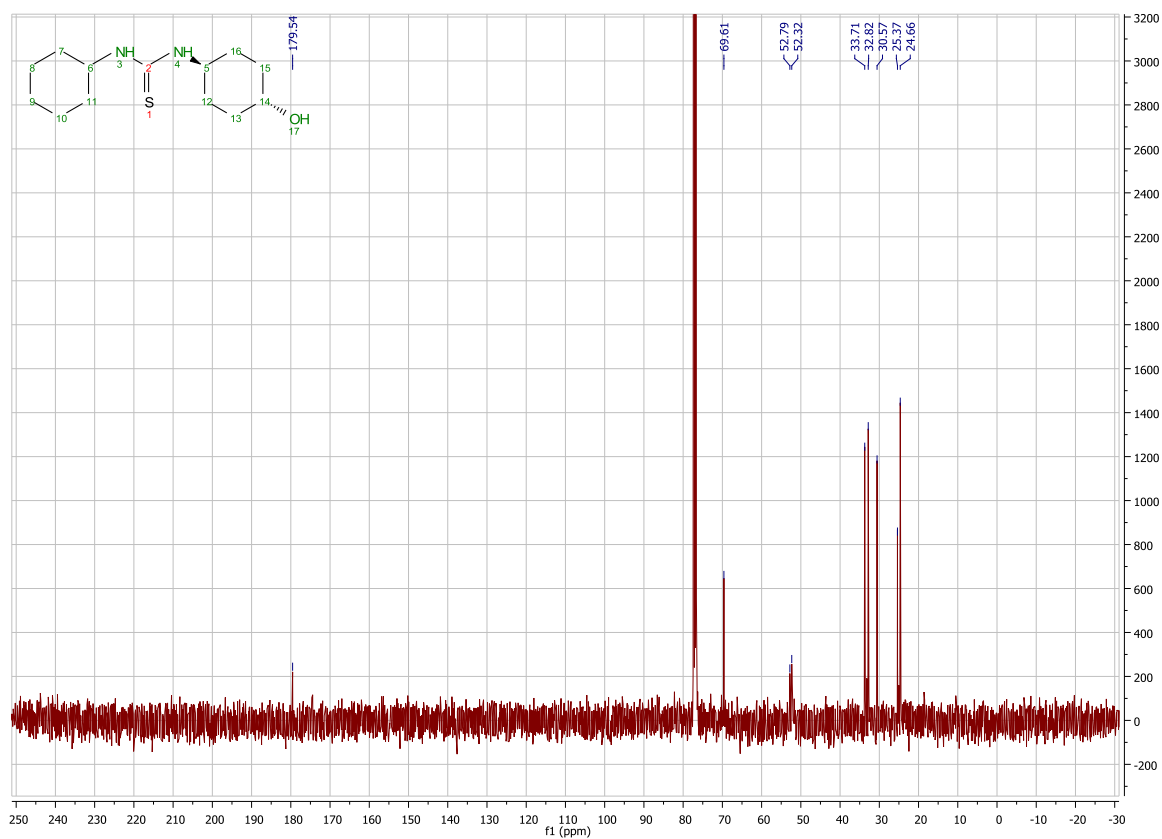
Infrared spectrum



^1H NMR spectrum



^{13}C NMR spectrum

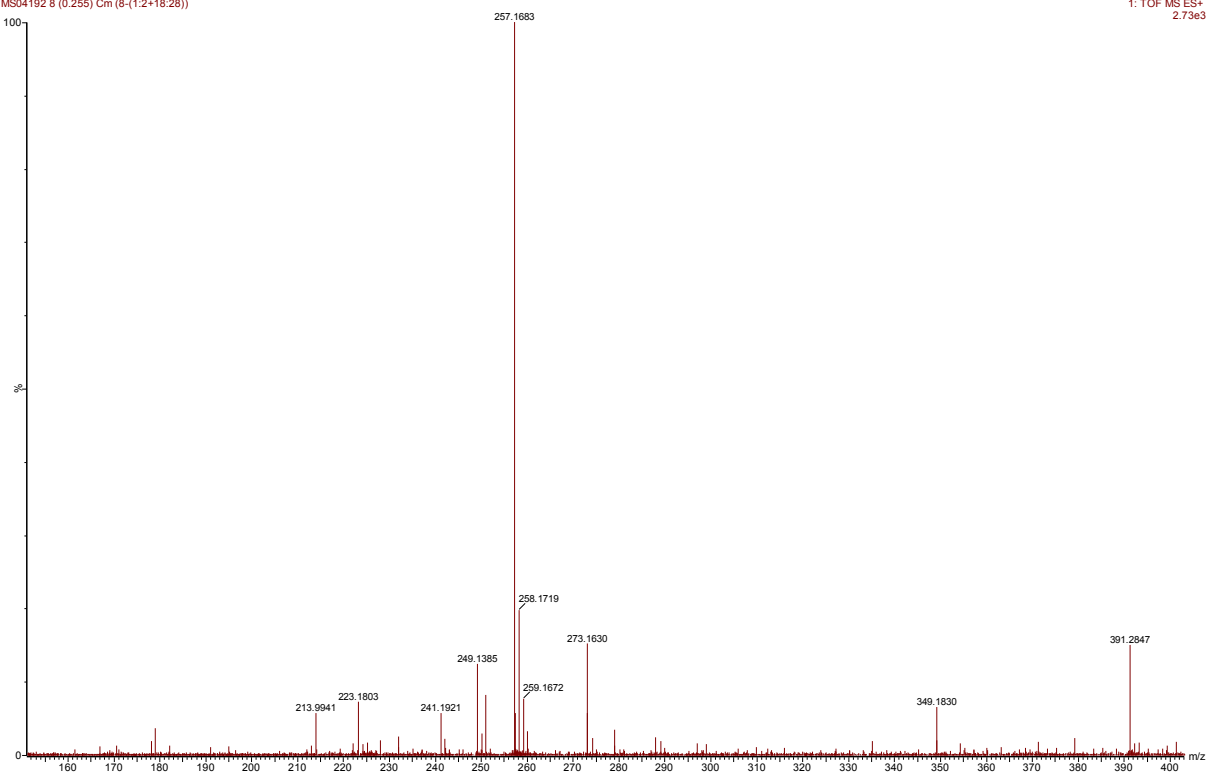


HRMS

G.GEORGE GG102

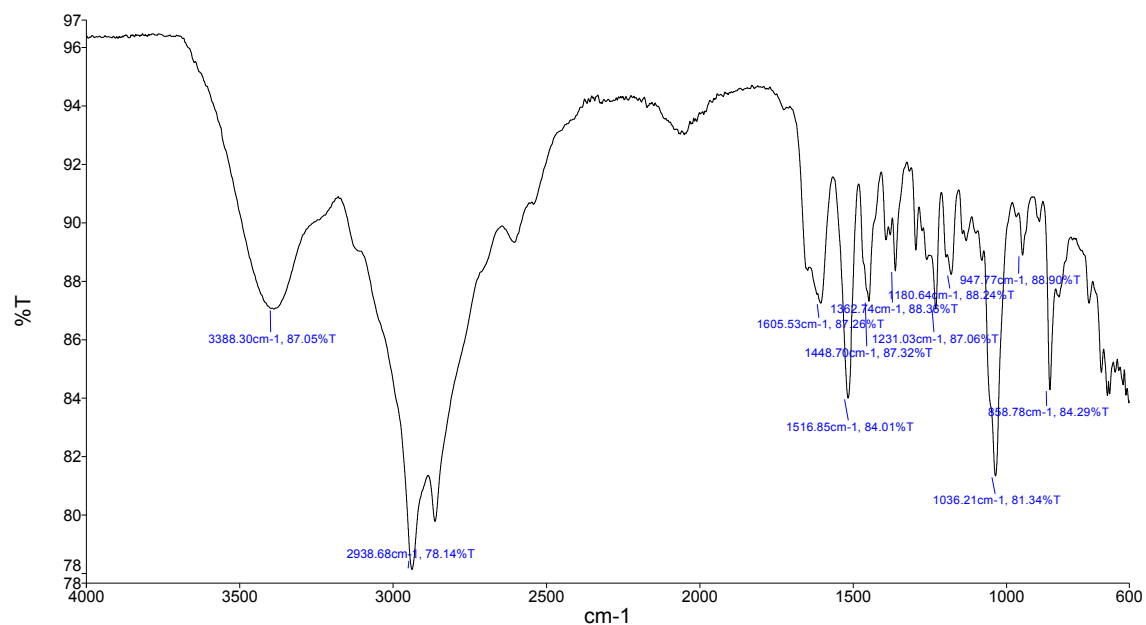
MS04192 8 (0.255) Cm (8-(1,2+18,28))

1: TOF MS ES+
2.73e3

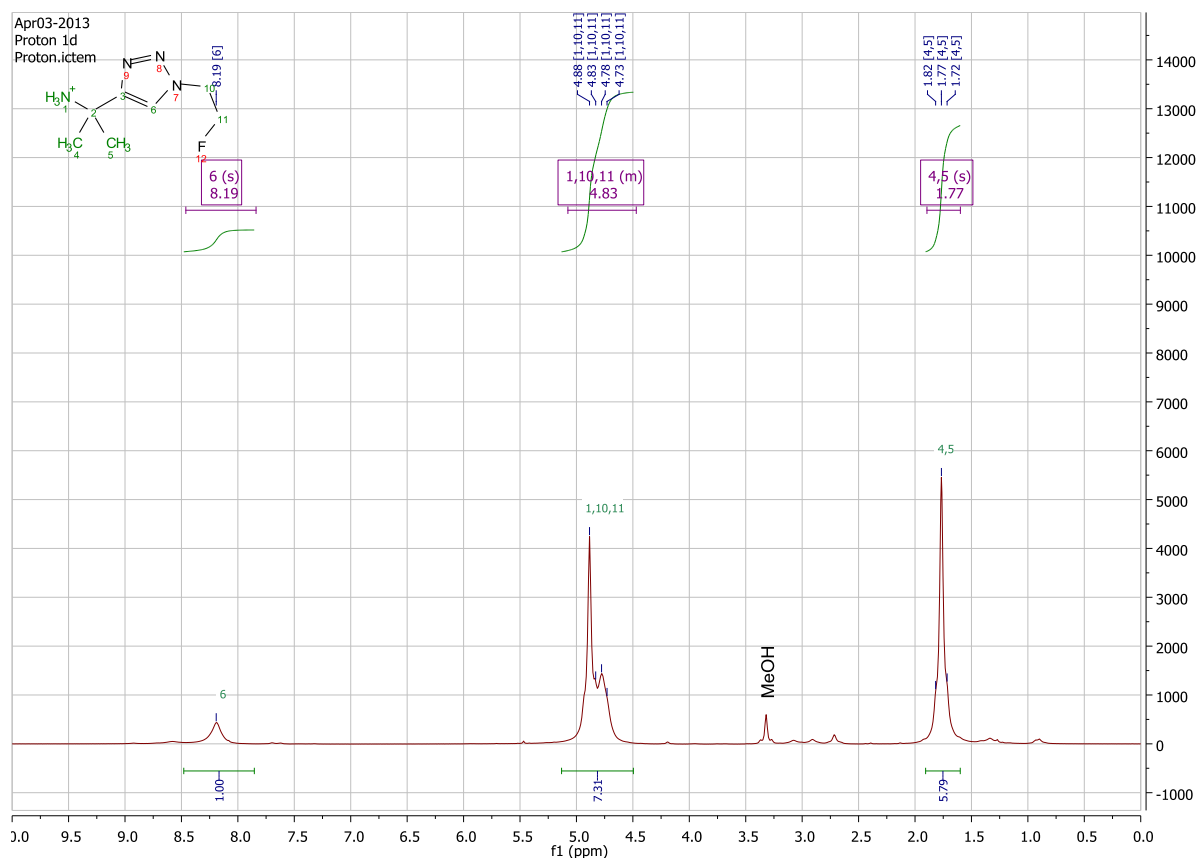


64. 2-[1-(2-fluoroethyl)-1*H*-1,2,3-triazol-4-yl]propan-2-aminium chloride

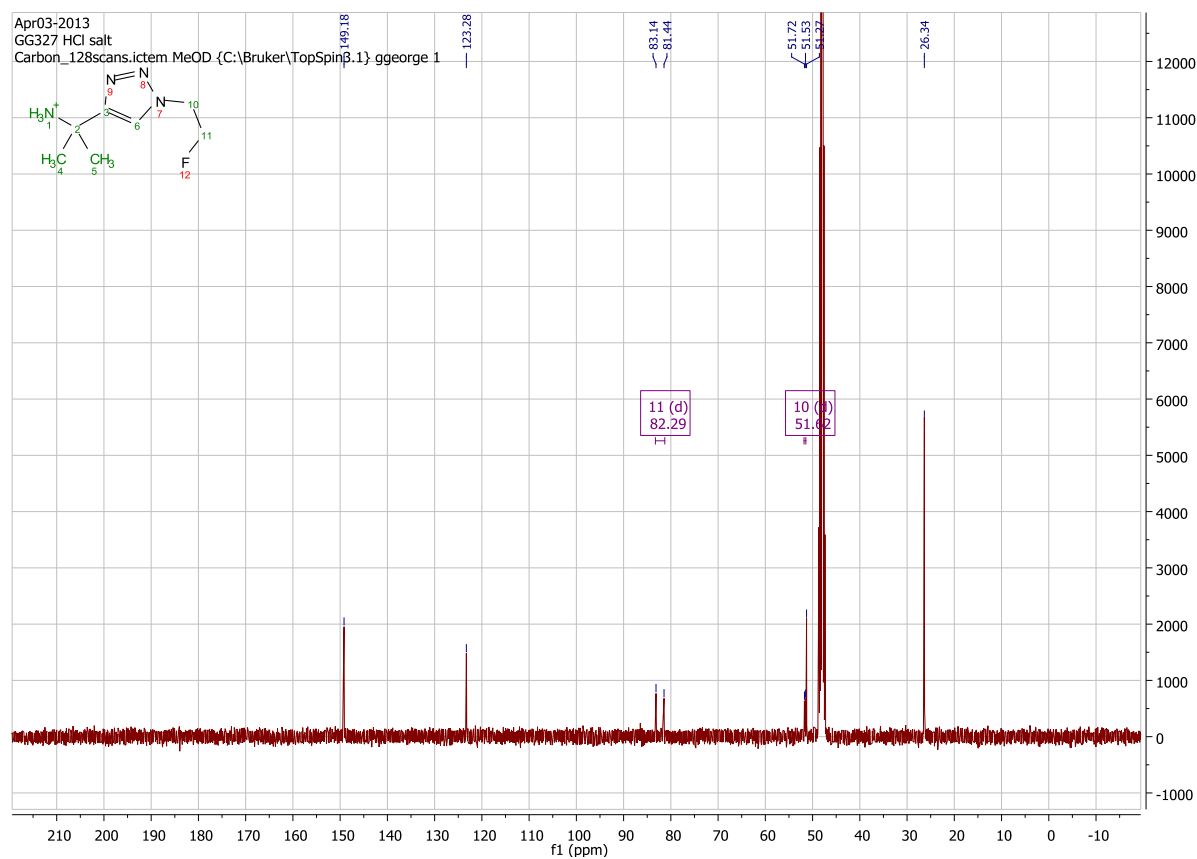
Infrared spectrum



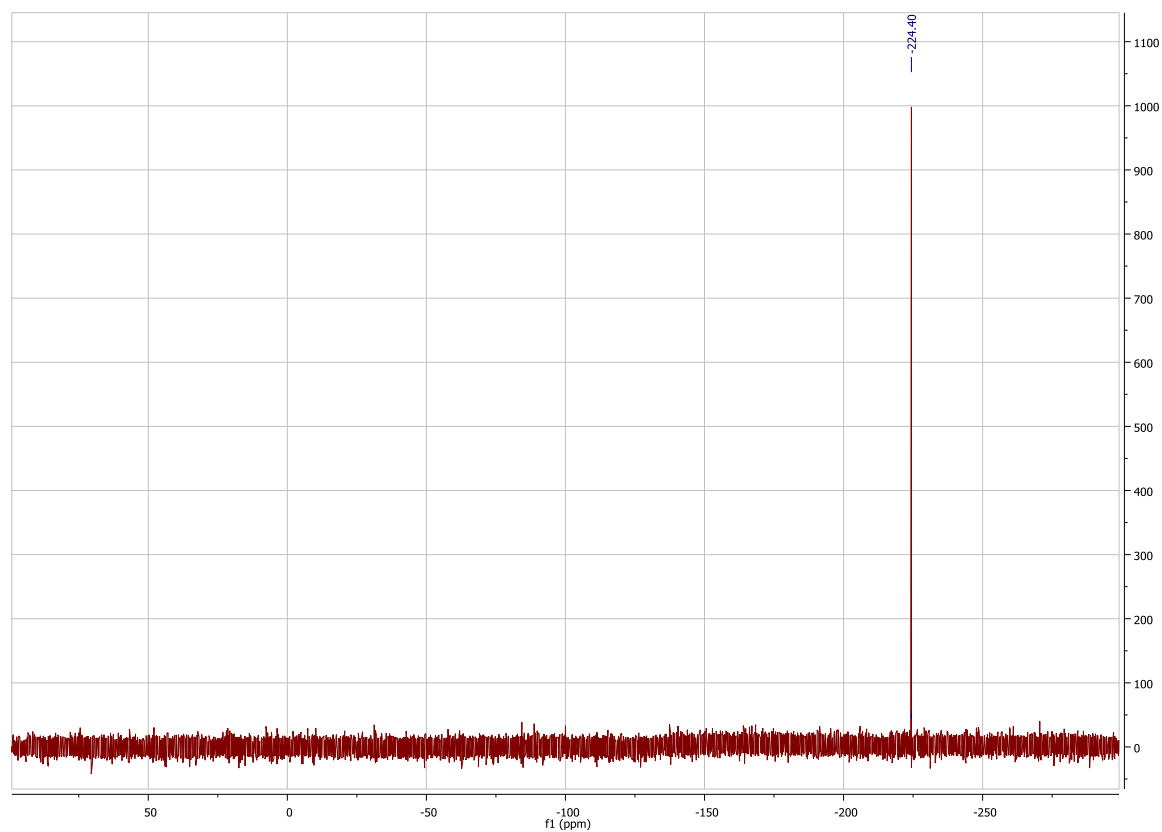
^1H NMR spectrum

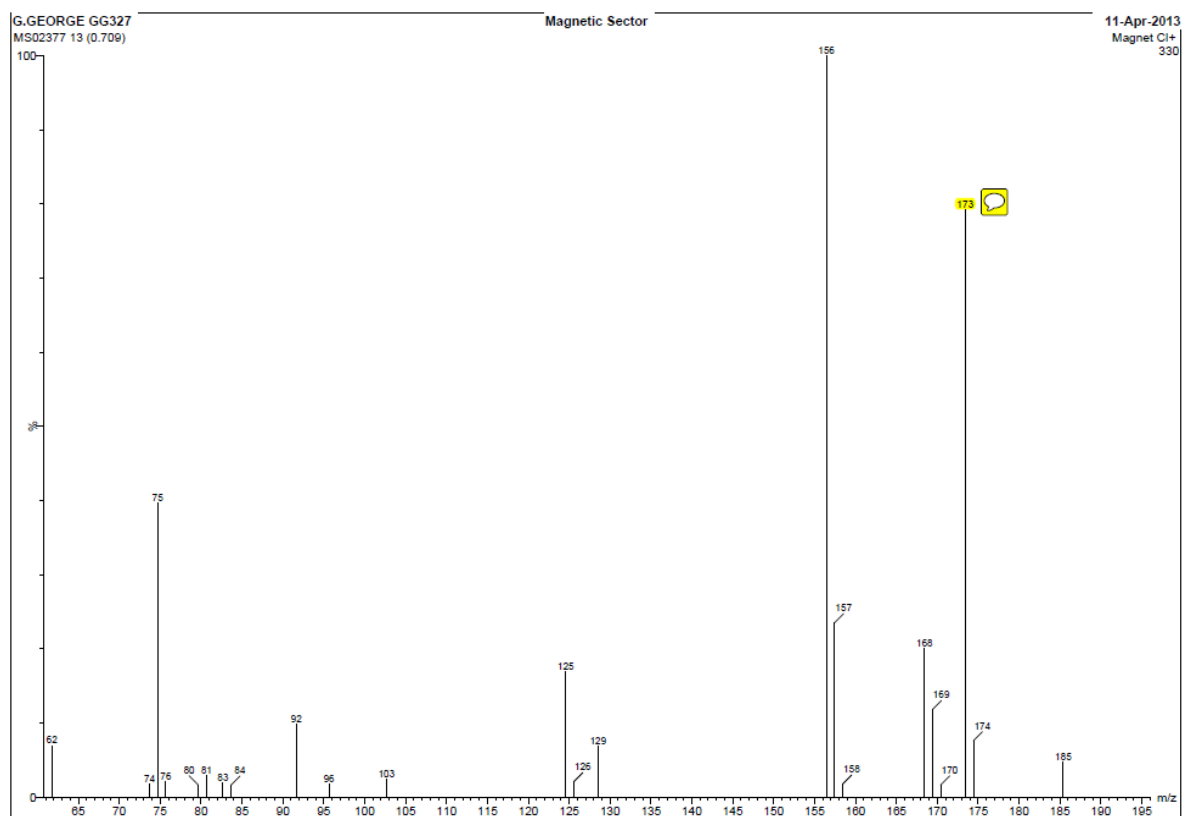


^{13}C NMR spectrum



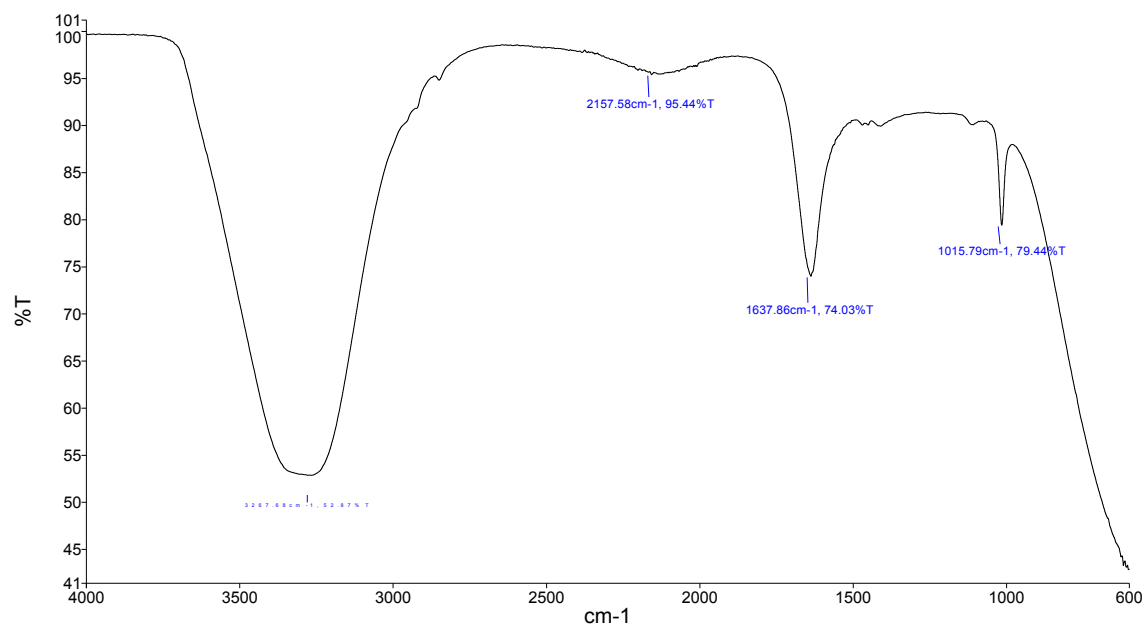
^{19}F NMR spectrum



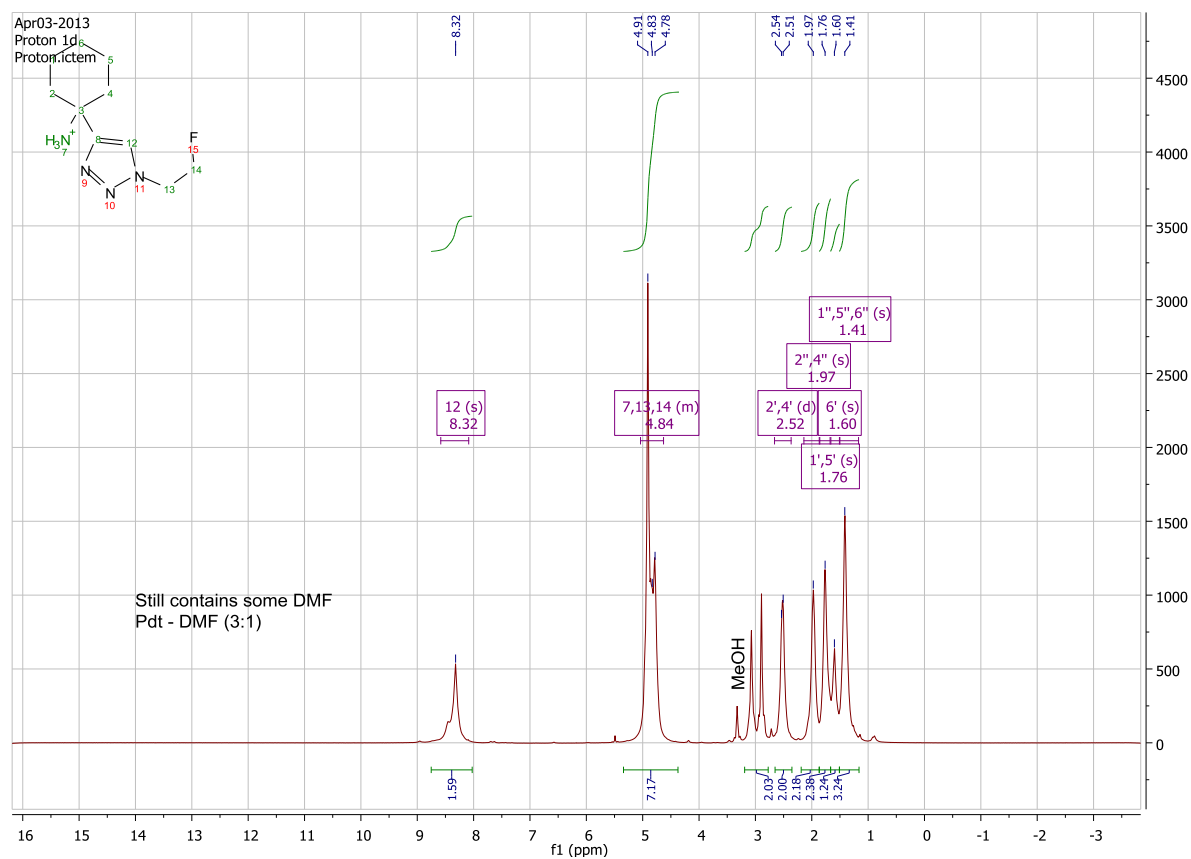


65. 1-[1-(2-fluoroethyl)-1*H*-1,2,3-triazol-4-yl]cyclohexan-1-aminium chloride

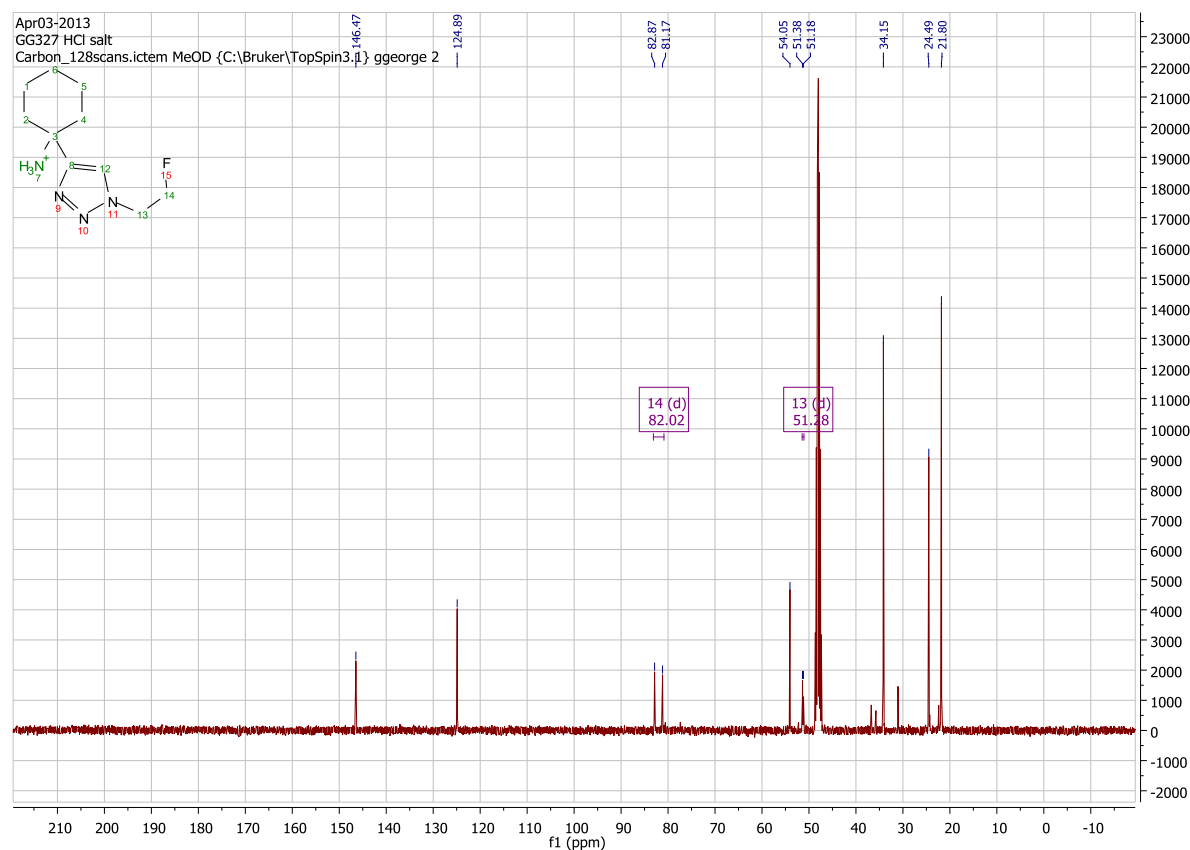
Infrared spectrum



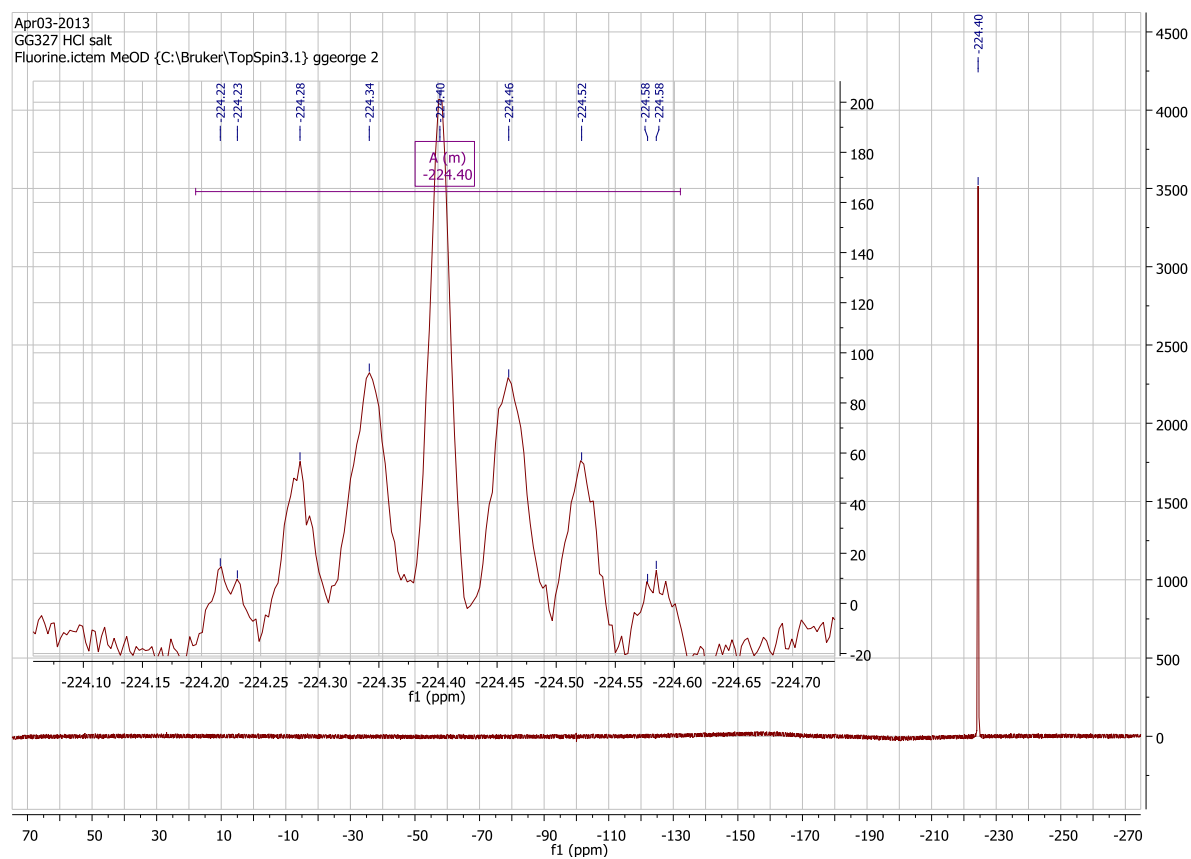
^1H NMR spectrum

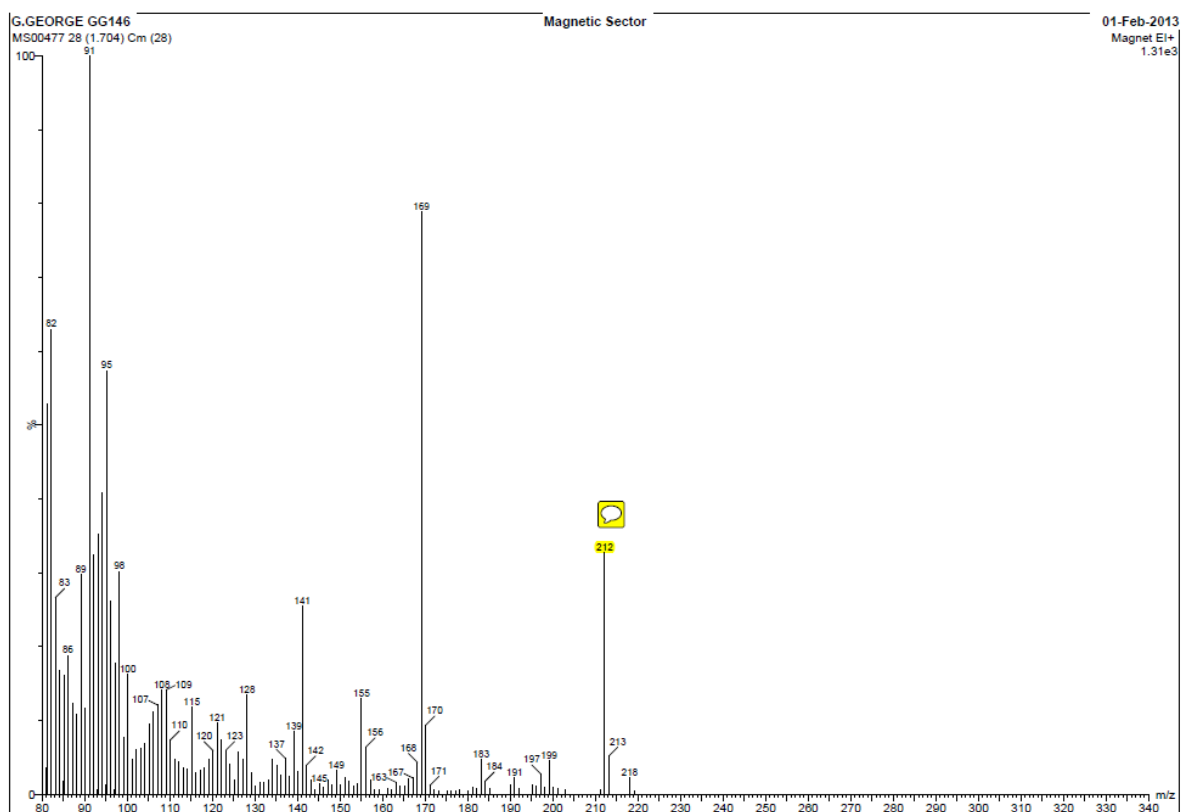


^{13}C NMR spectrum



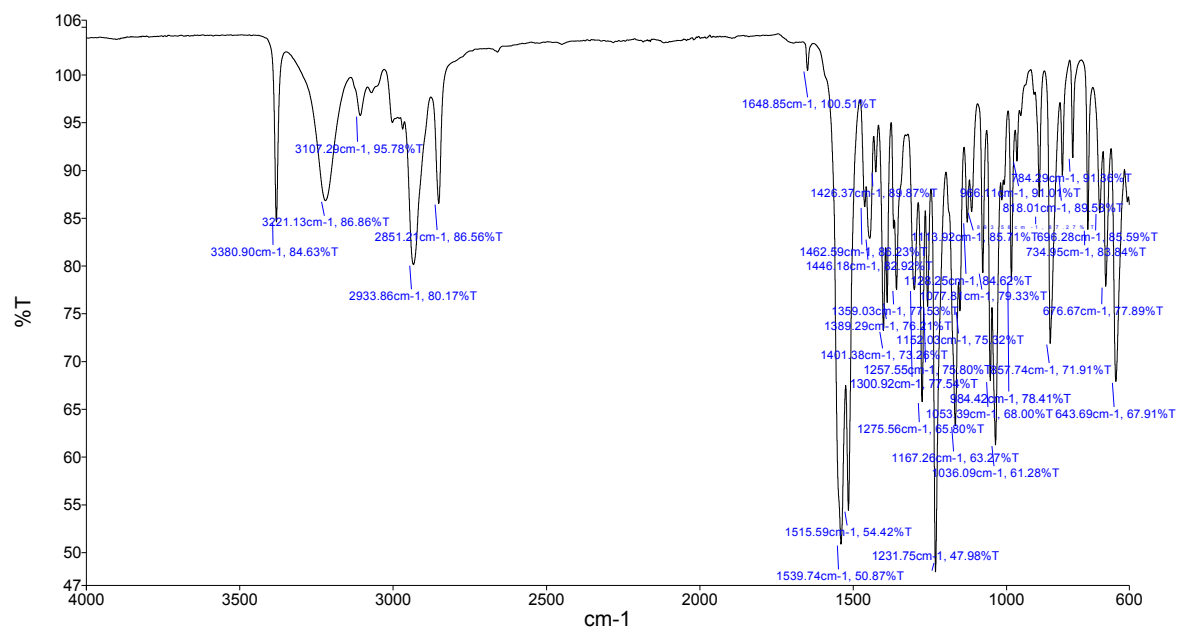
^{19}F NMR spectrum



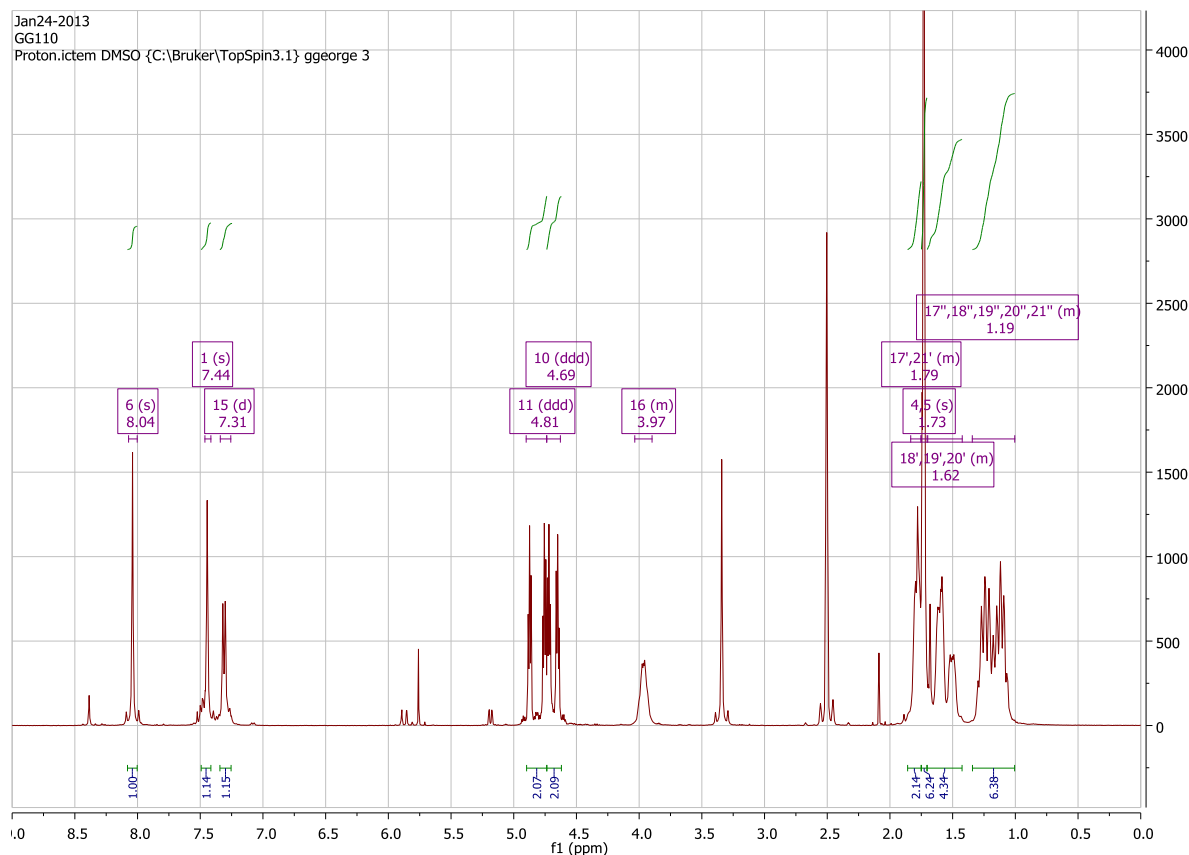


66. 1-Cyclohexyl-3-{2-[1-(2-fluoroethyl)-1H-1,2,3-triazol-4-yl]propan-2-yl}thiourea

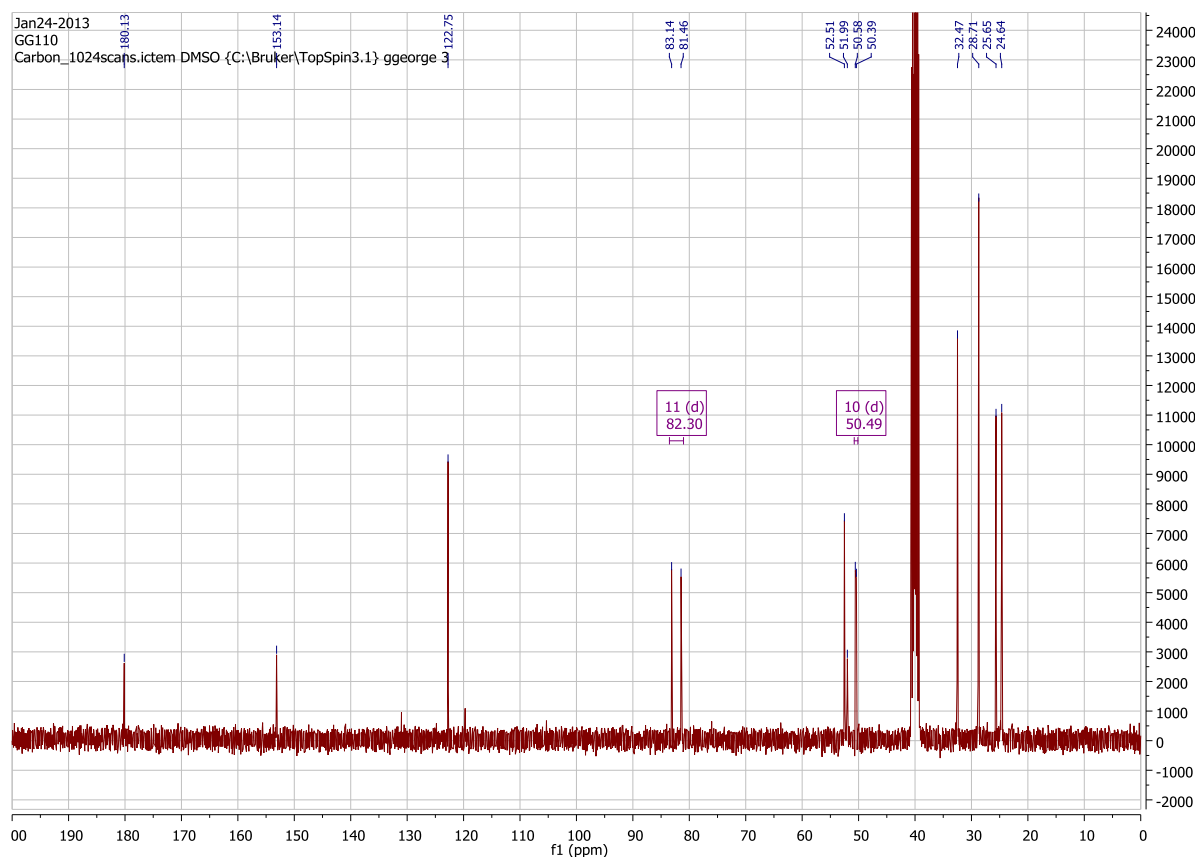
Infrared spectrum



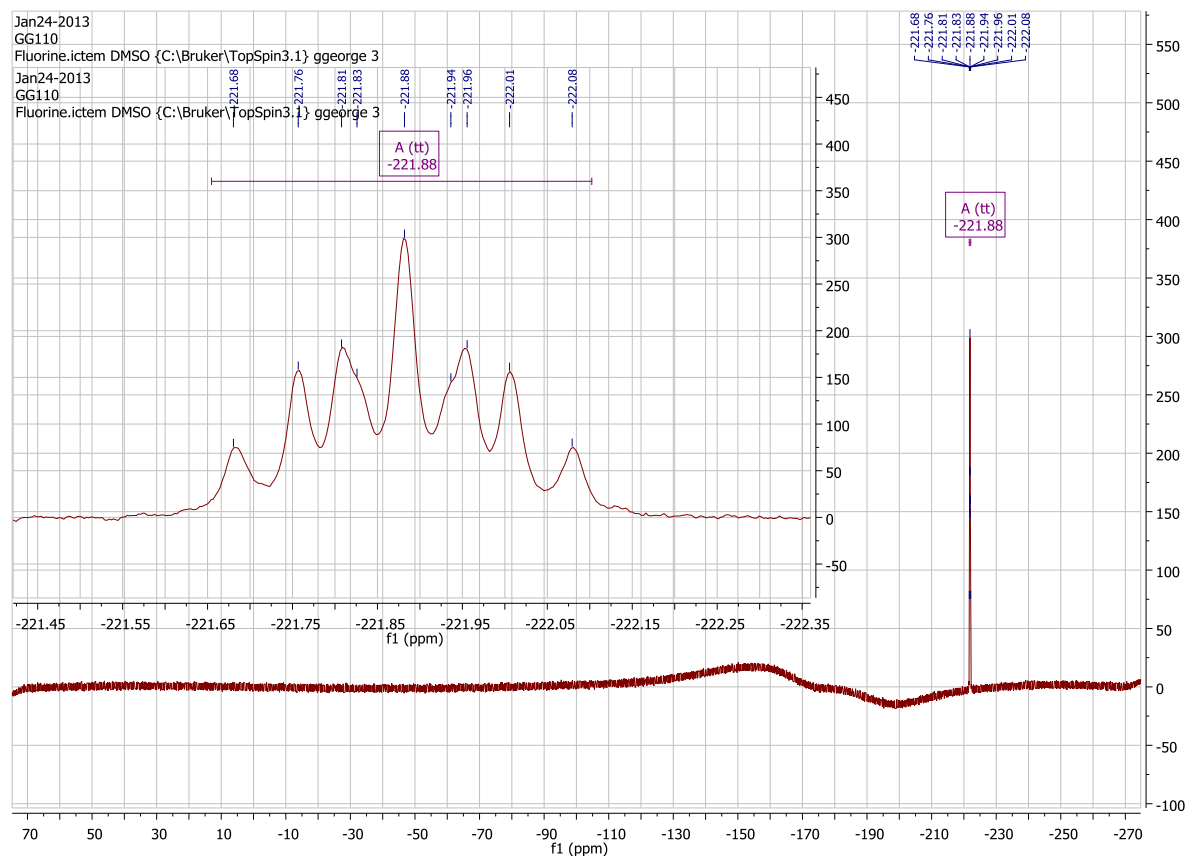
¹H NMR spectrum

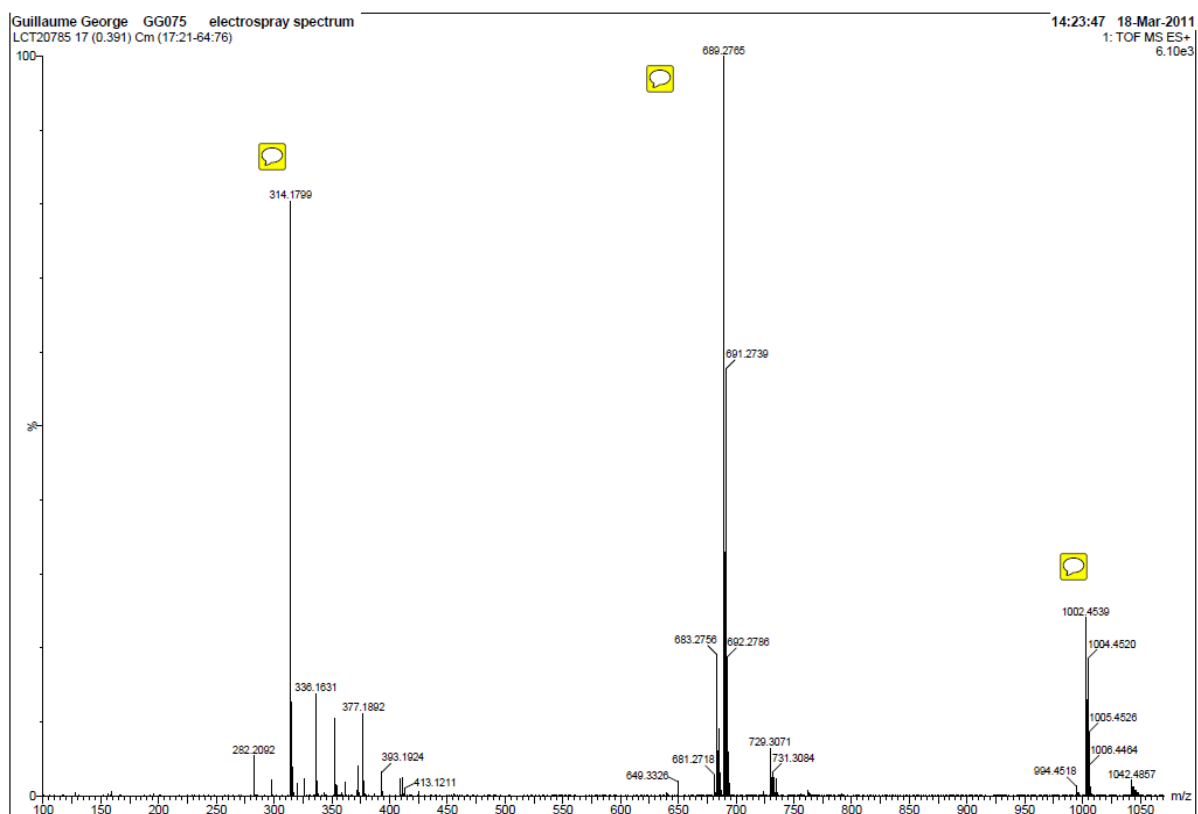


^{13}C NMR spectrum



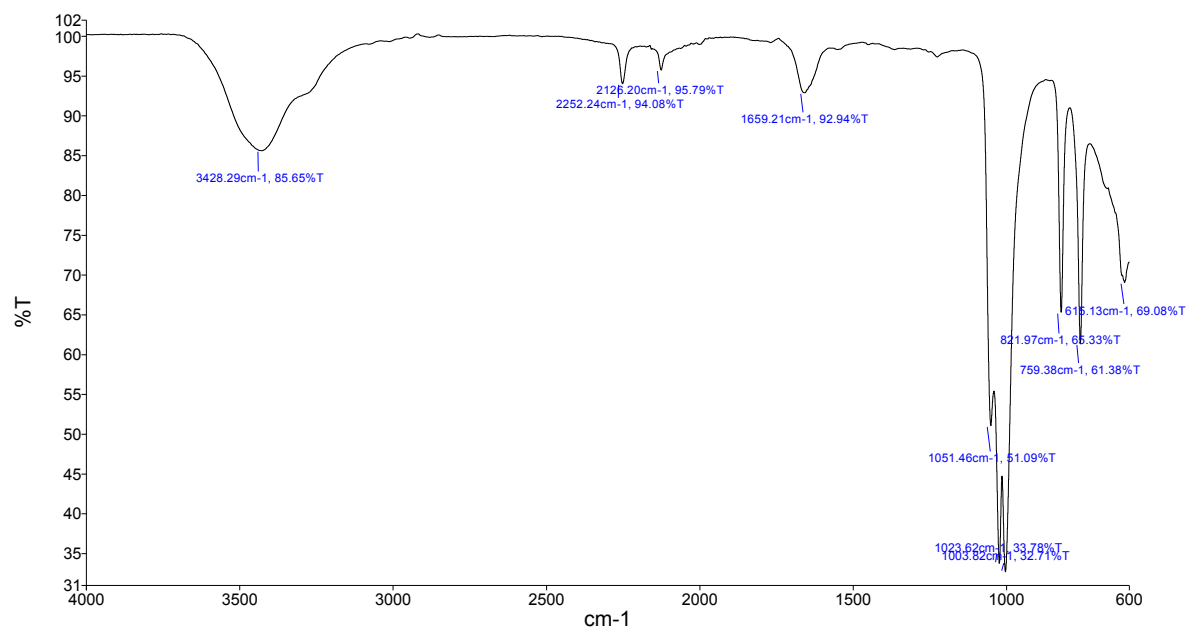
^{19}F NMR spectrum



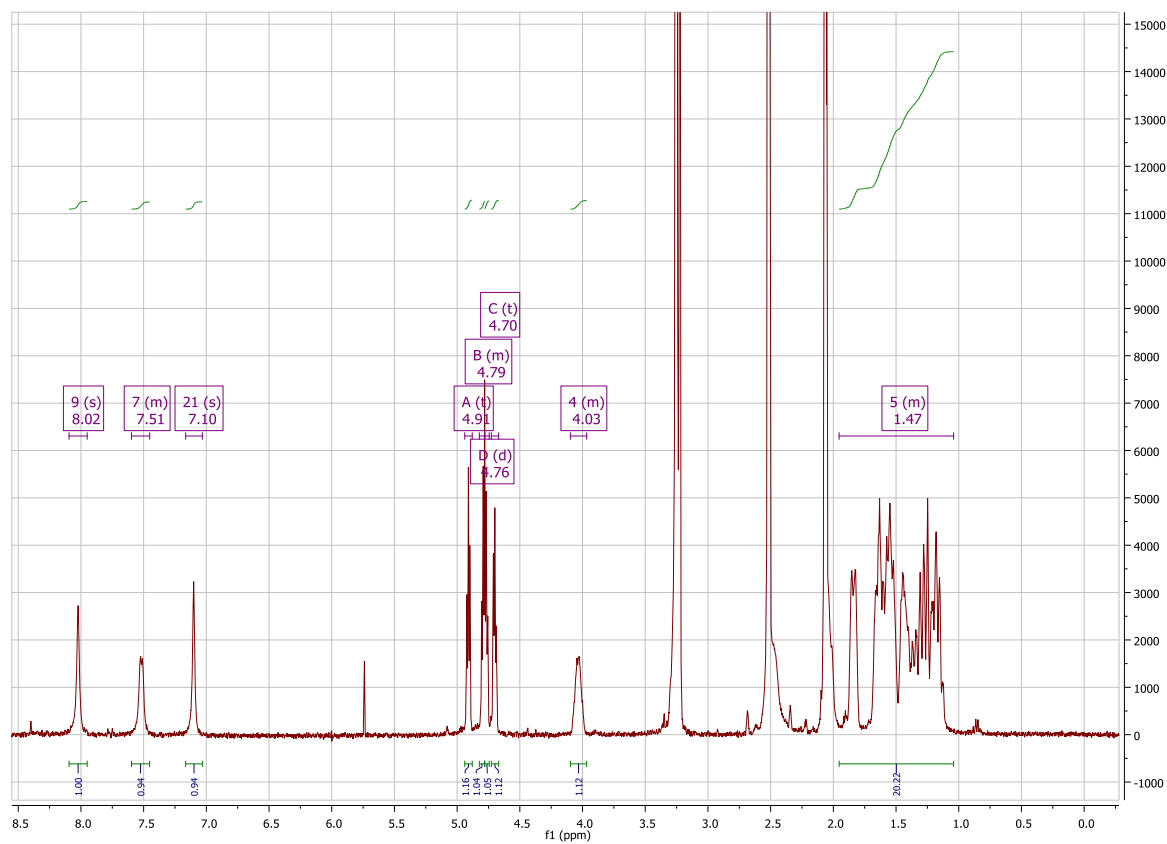


67. 3-Cyclohexyl-1-{1-[1-(2-fluoroethyl)-1H-1,2,3-triazol-4-yl]cyclohexyl}thiourea

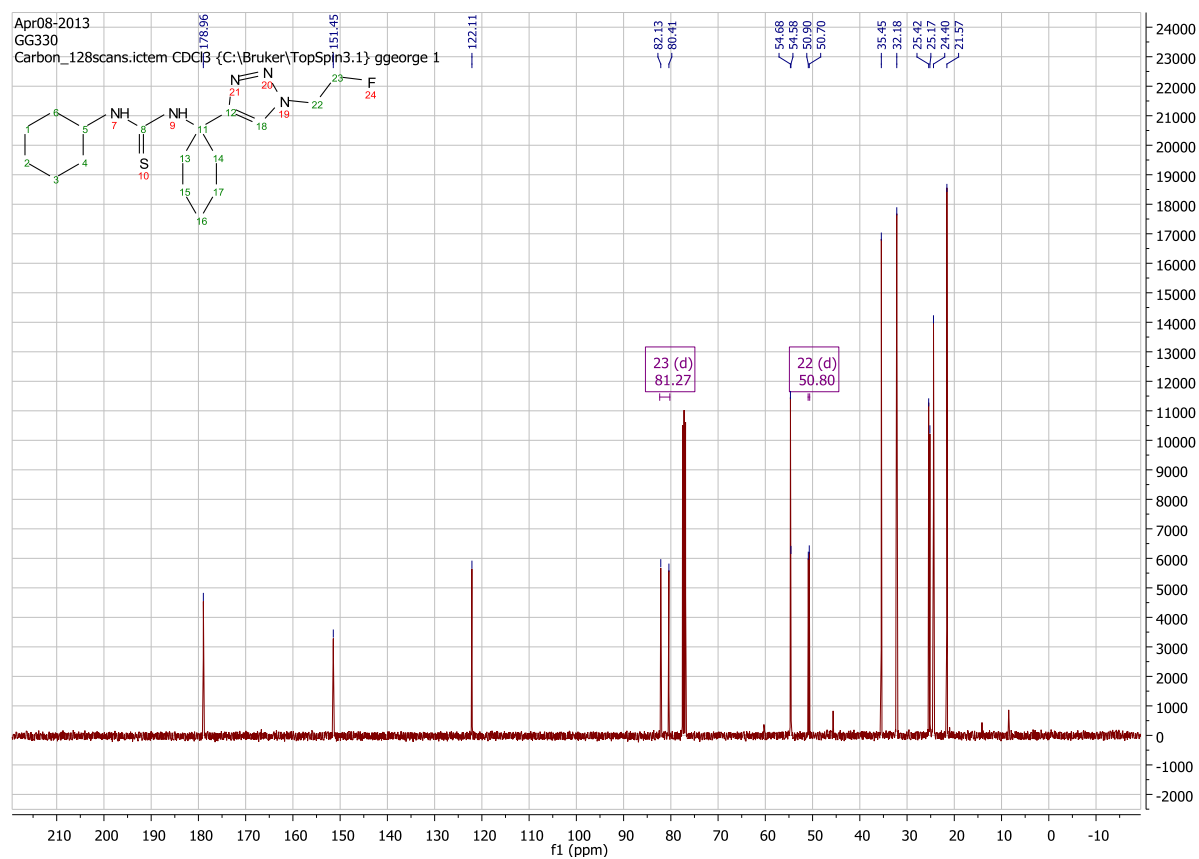
Infrared spectrum



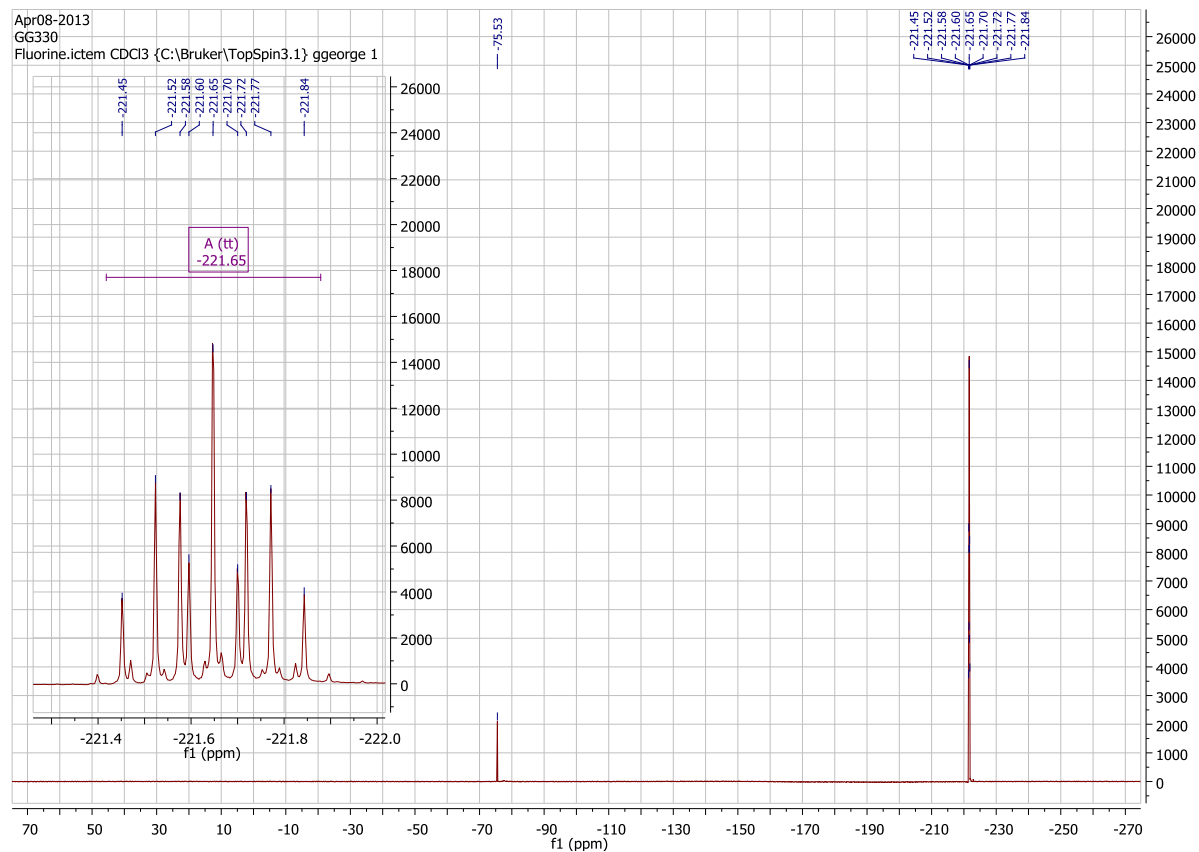
¹H NMR spectrum

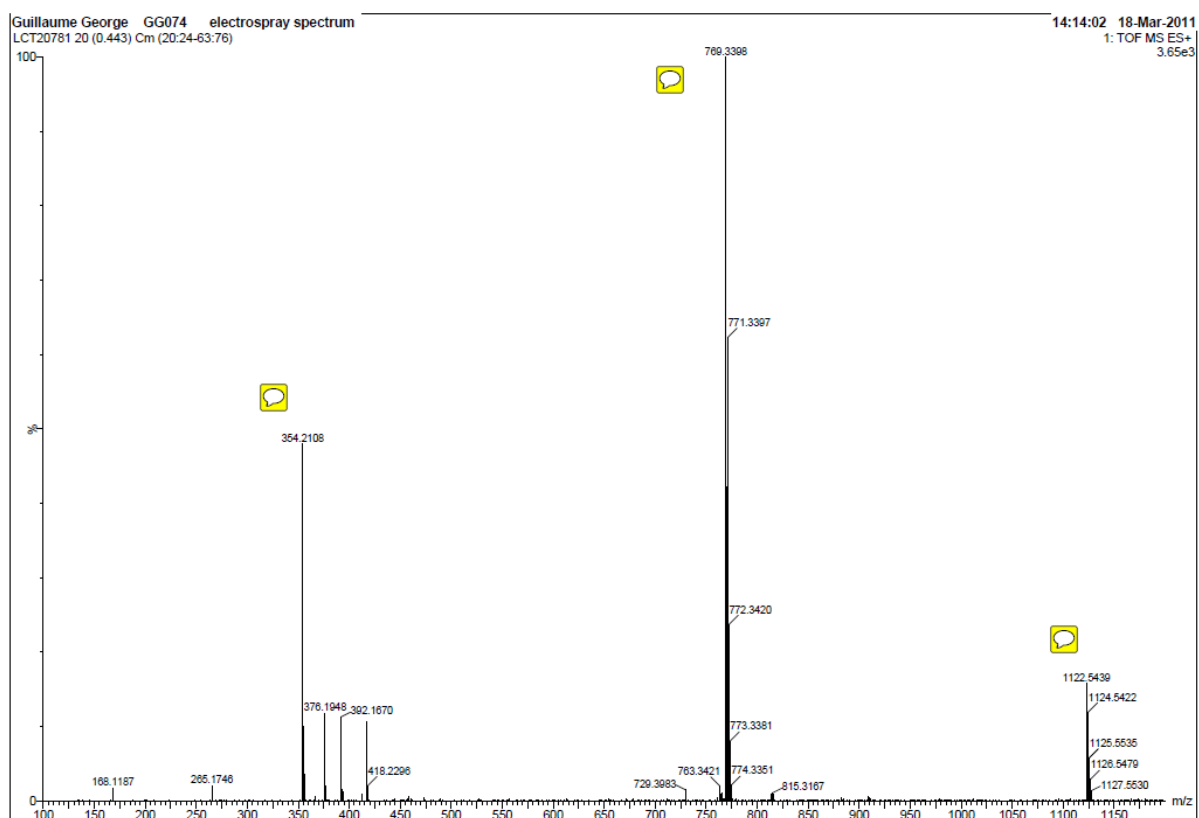


^{13}C NMR spectrum



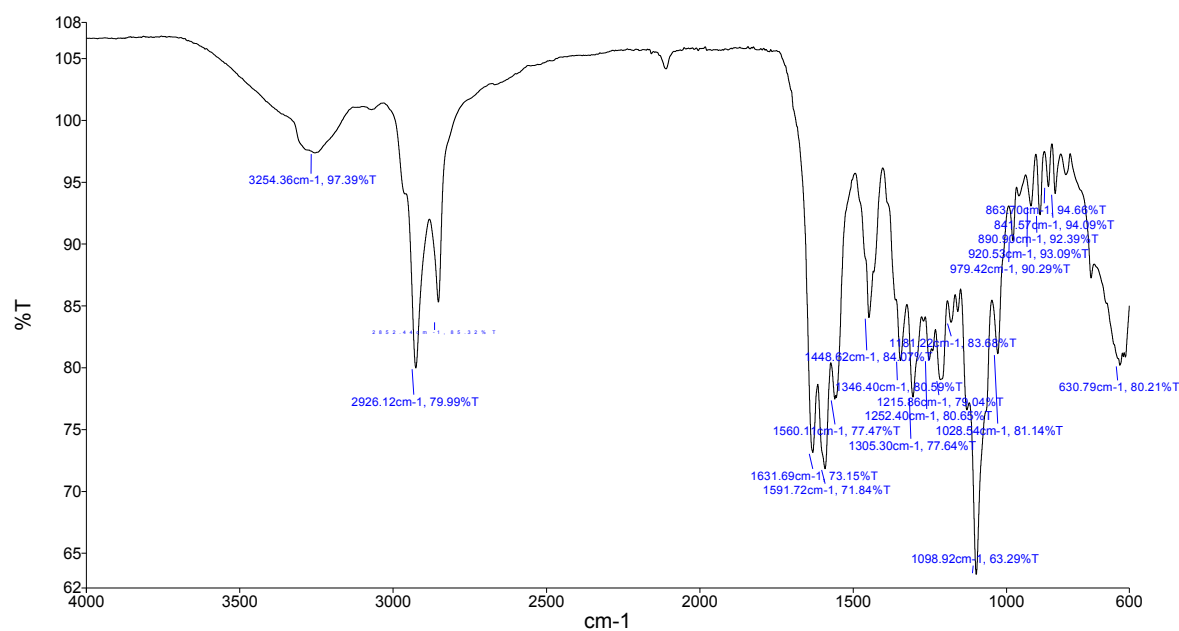
^{19}F NMR spectrum



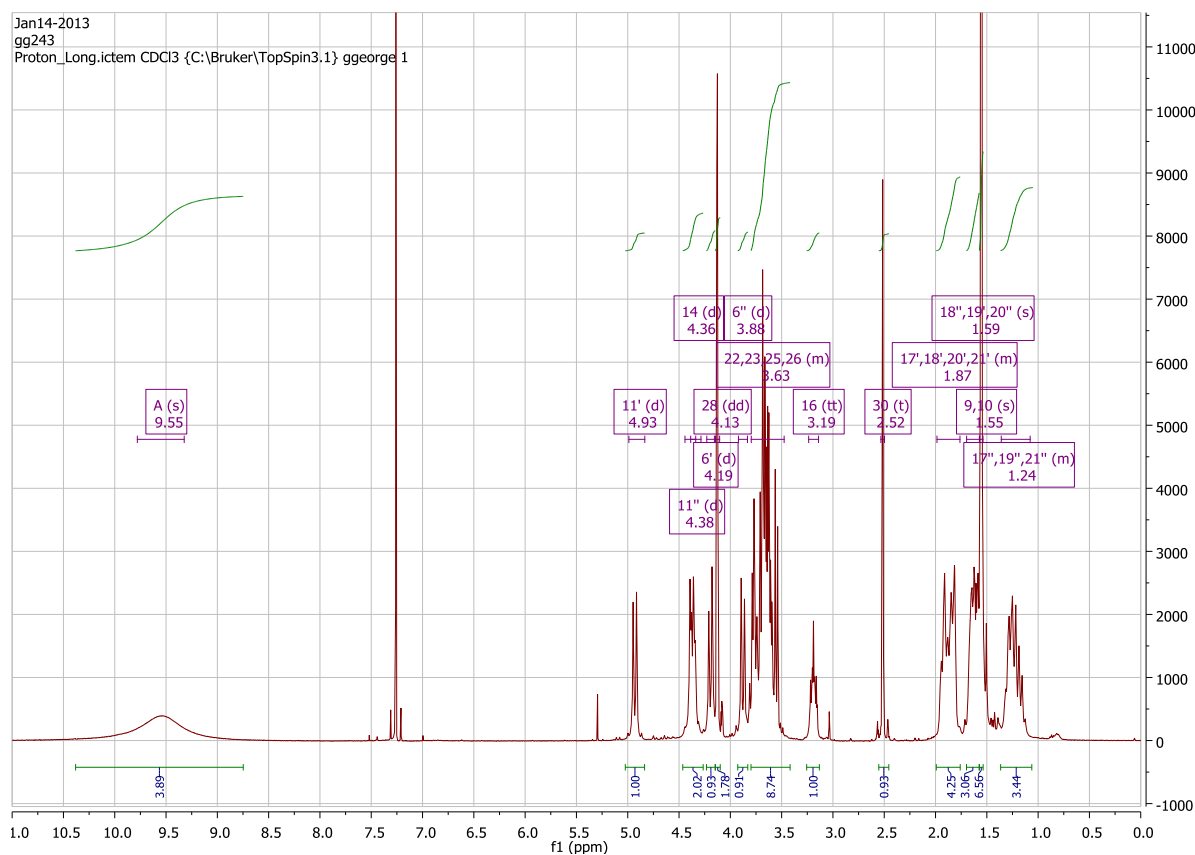


68. *N*-[(Cyclohexylimino)]({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl)methyl]-2-[2-(prop-2-yn-1-yloxy)ethoxy]ethan-1-amine .3TFA

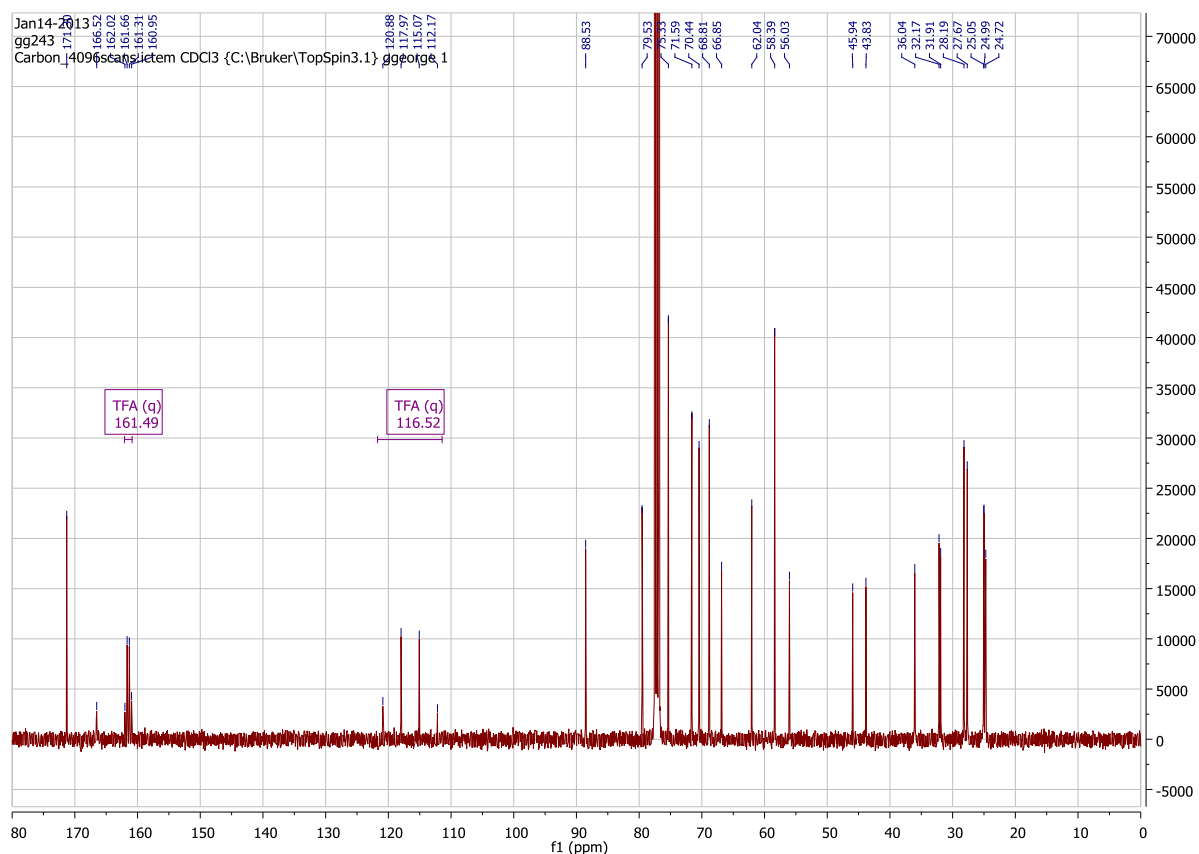
Infrared spectrum



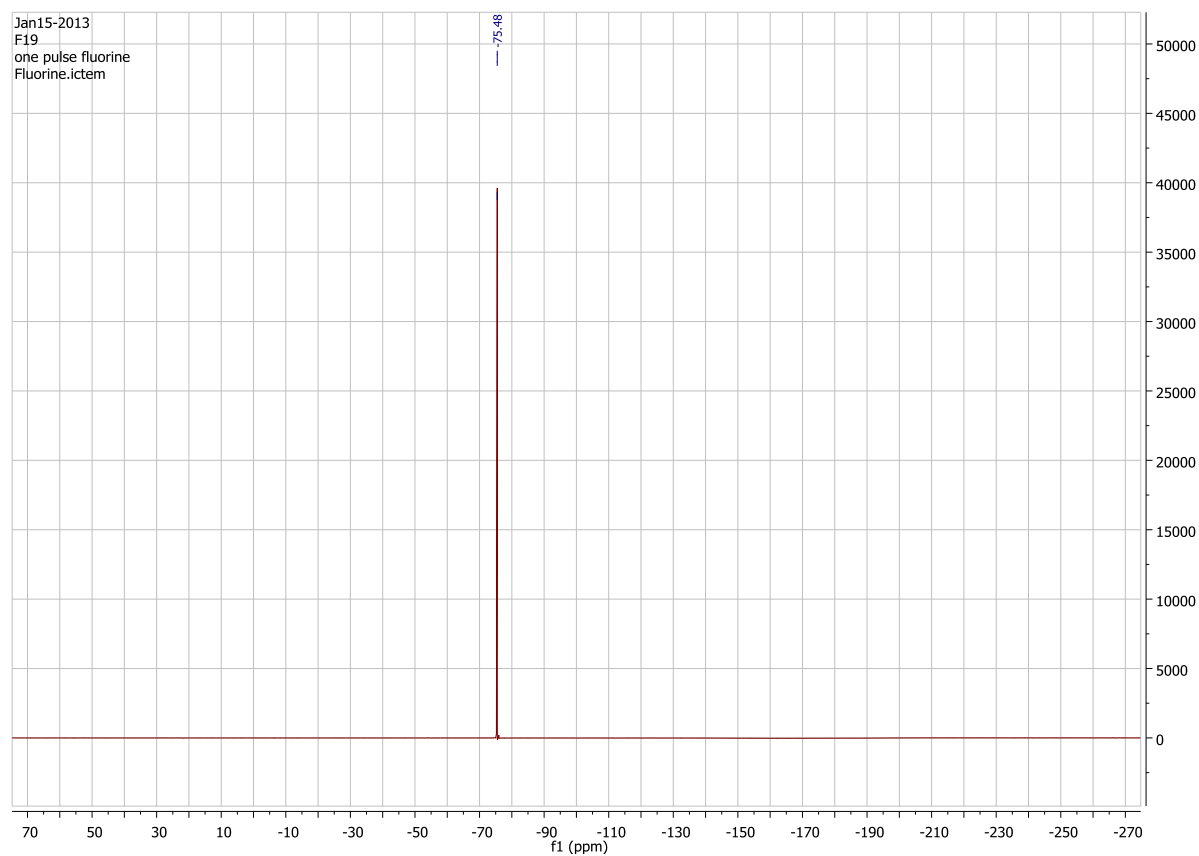
¹H NMR spectrum



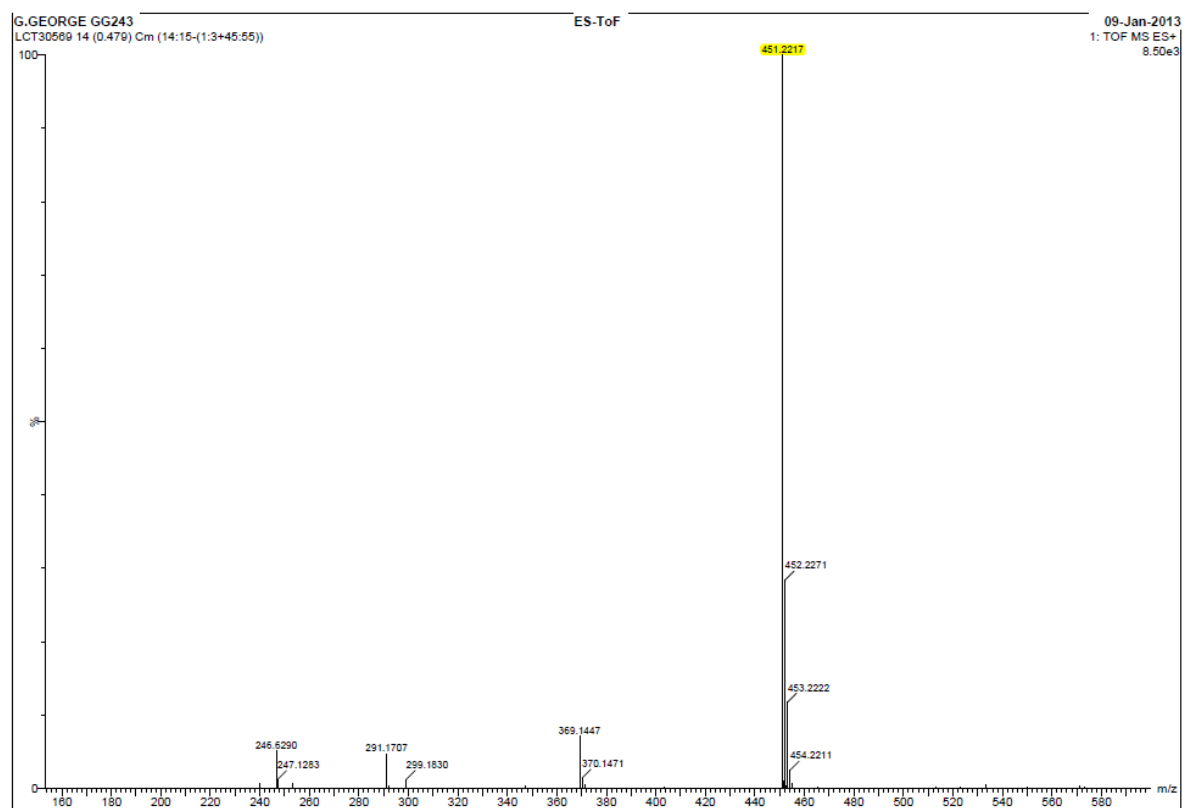
^{13}C NMR spectrum



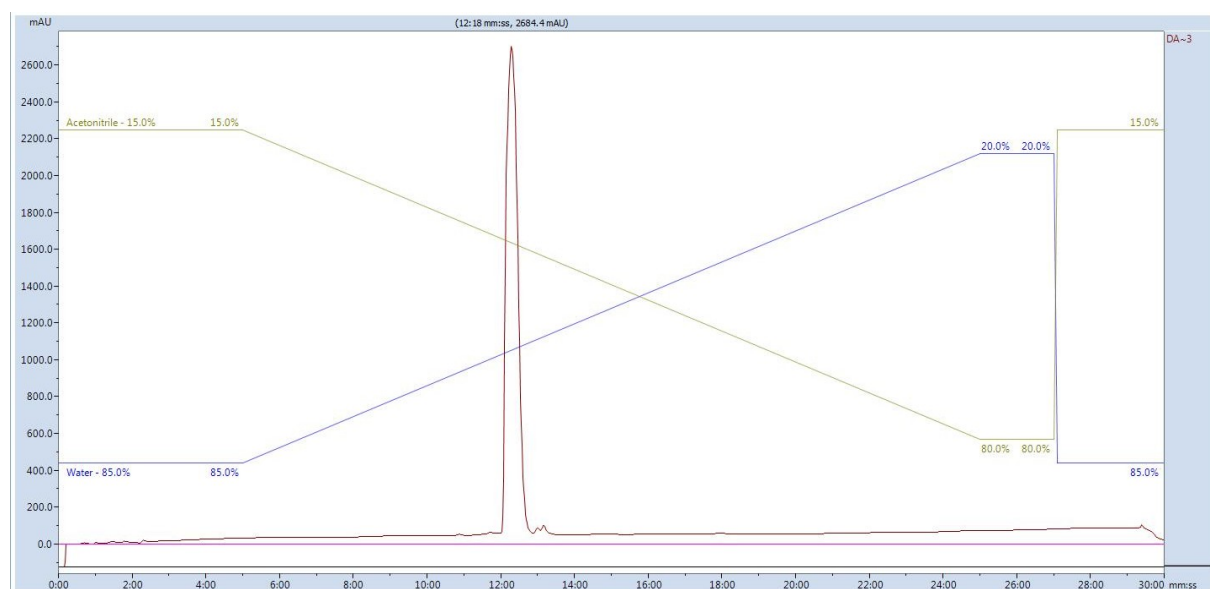
^{19}F NMR spectrum



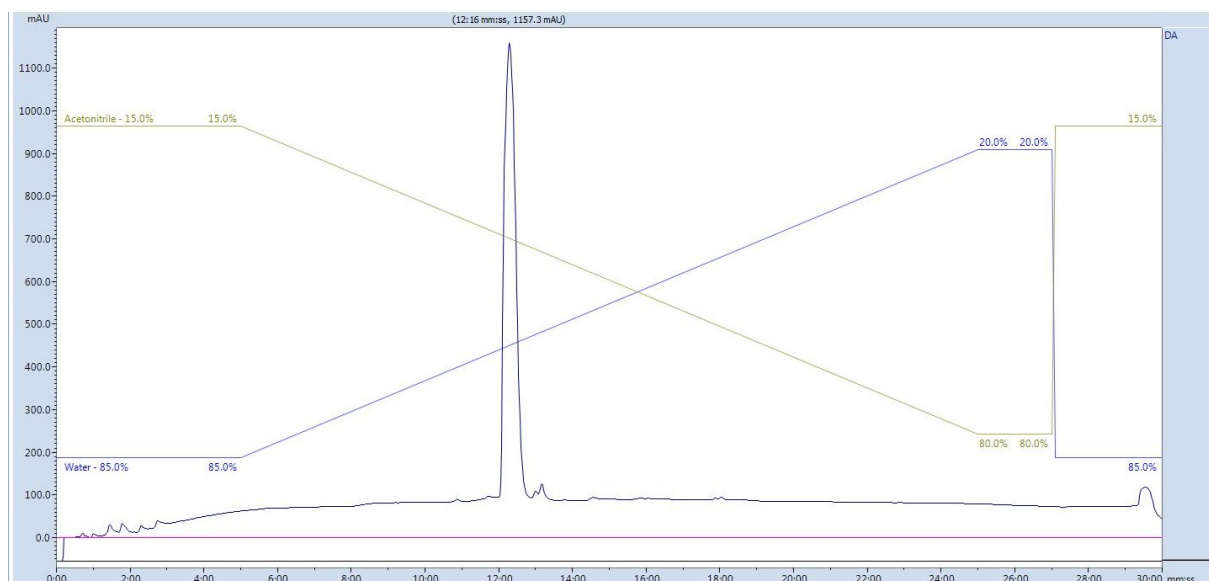
HRMS



HPLC trace at 210 nm

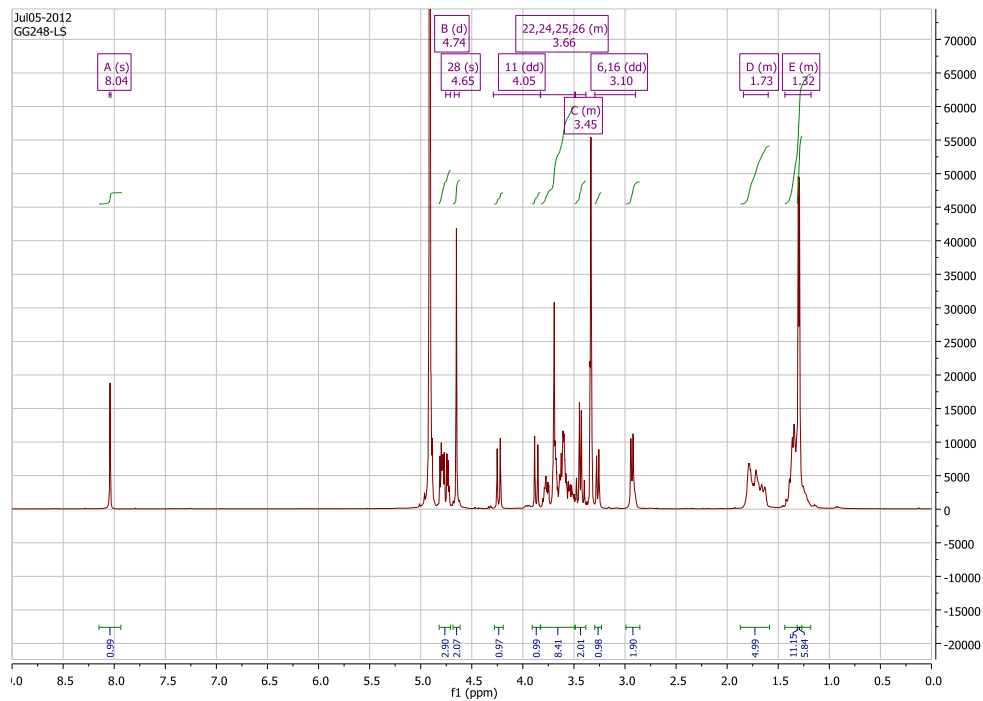


HPLC trace at 254 nm

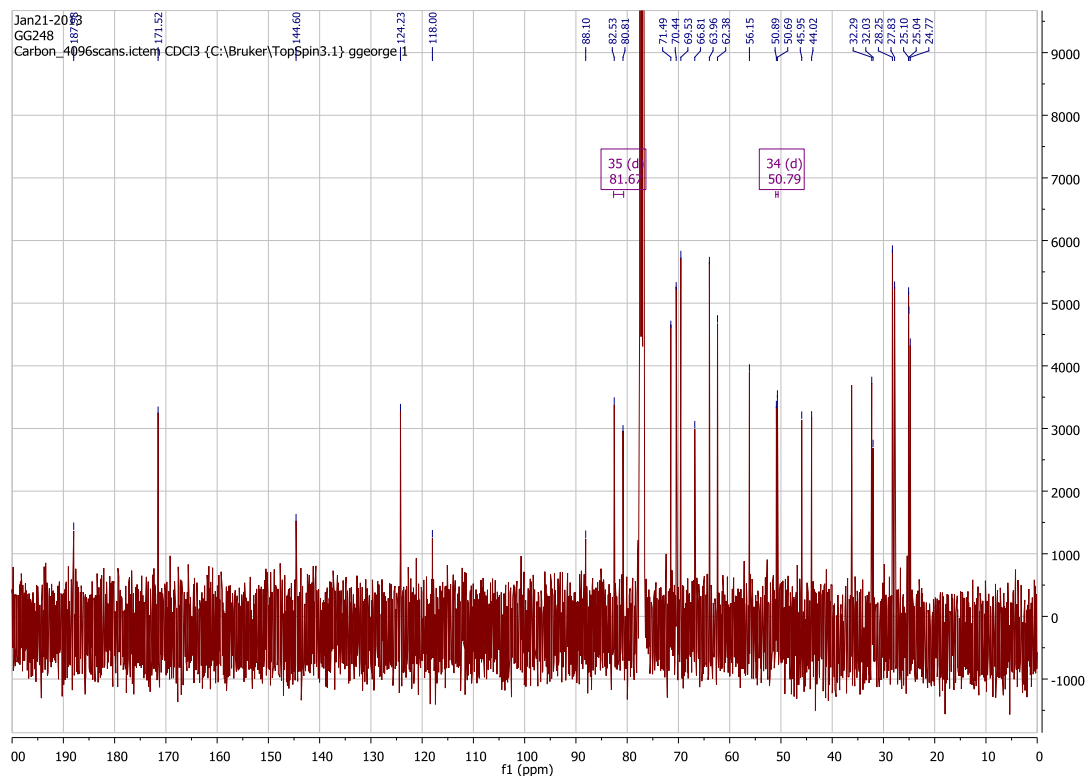


69. ***N*-[(Cyclohexylimino)[(6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl)methyl]-2-(2-{[1-(2-fluoroethyl)-1*H*-1,2,3-triazol-4-yl]methoxy}ethoxy)ethane-1-amine .3TFA**

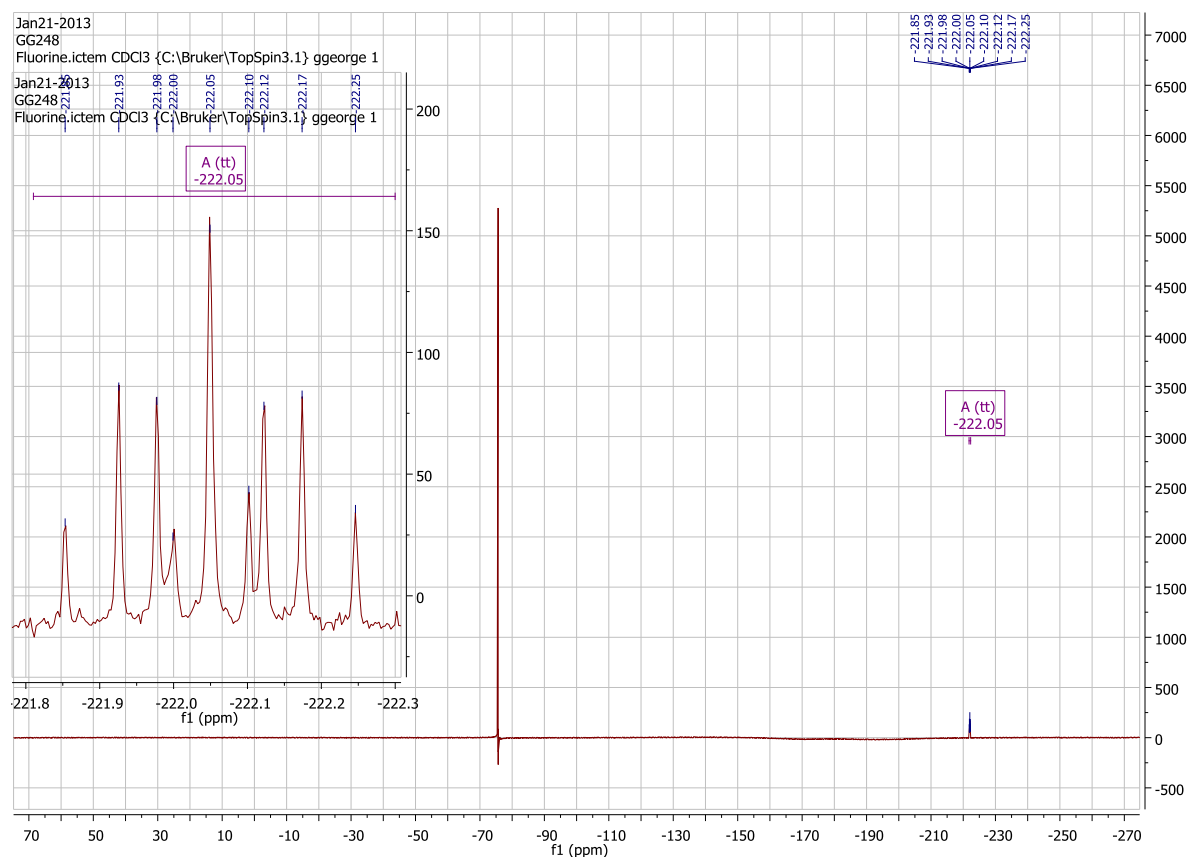
¹H NMR spectrum



¹³C NMR spectrum



¹⁹F NMR spectrum



HRMS

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 8.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

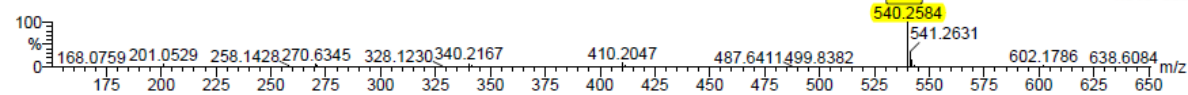
1392 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 24-24 H: 5-300 N: 0-10 O: 0-10 Na: 0-1 S: 0-2 I: 0-1 F: 1-1

G.GEORGE GG248
LCT28625 12 (0.394) Cm (12:14-43:50)

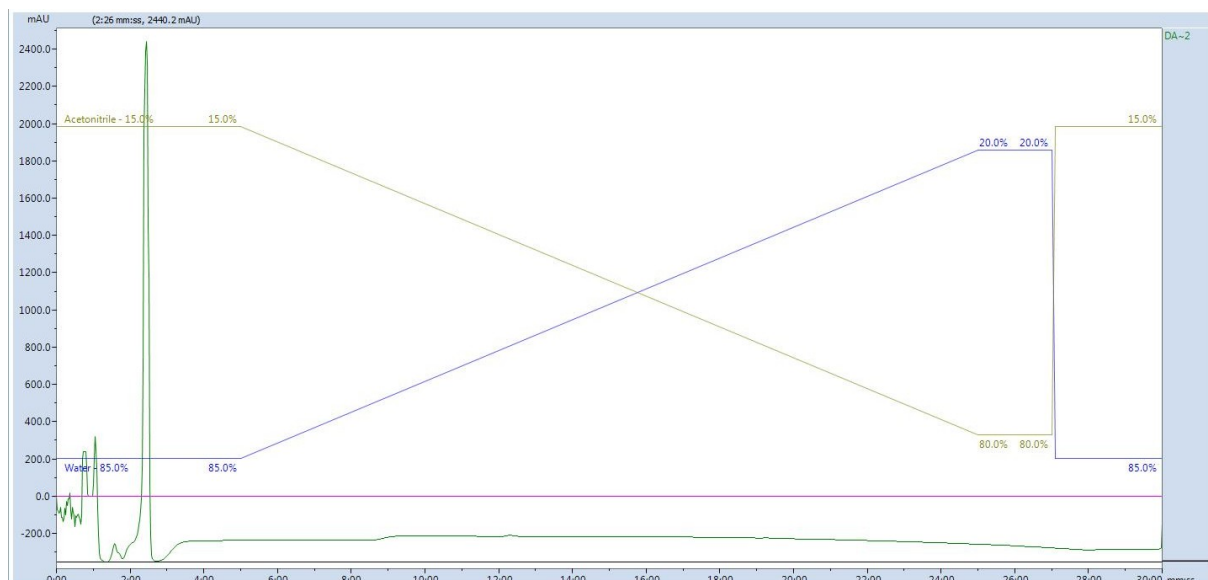
09:26:29 20-Aug-2012
1: TOF MS ES+
4.04e+003



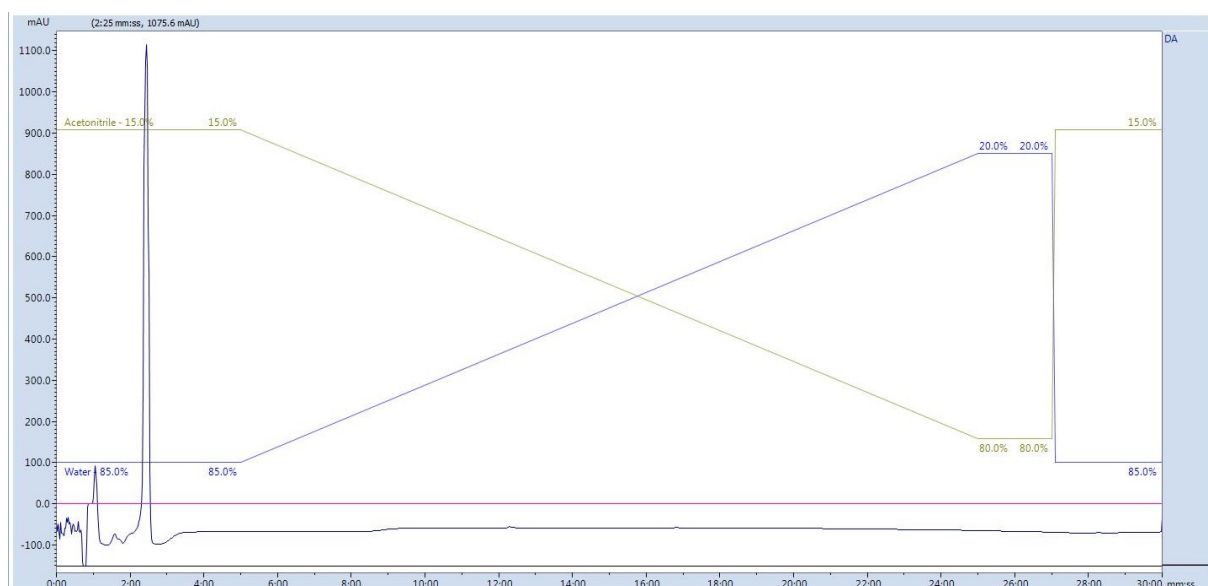
Minimum: -1.5
Maximum: 5.0 8.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
540.2584	540.2591	-0.7	-1.3	8.5	58.5	0.0	C24 H39 N7 O2 S2 F

HPLC trace at 210 nm

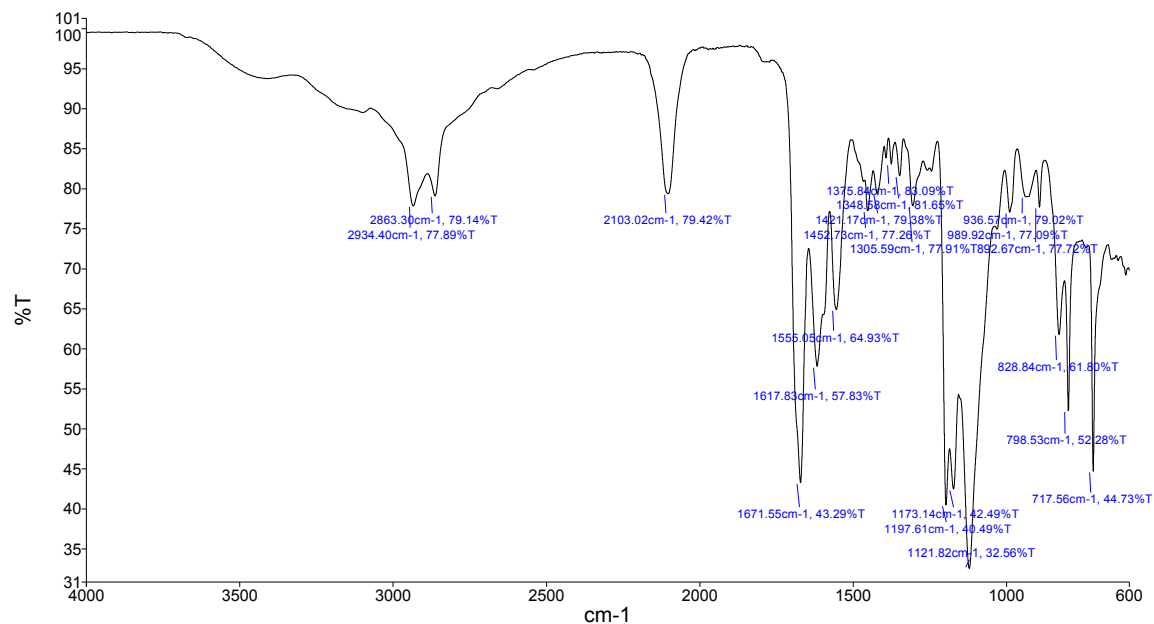


HPLC trace at 254 nm

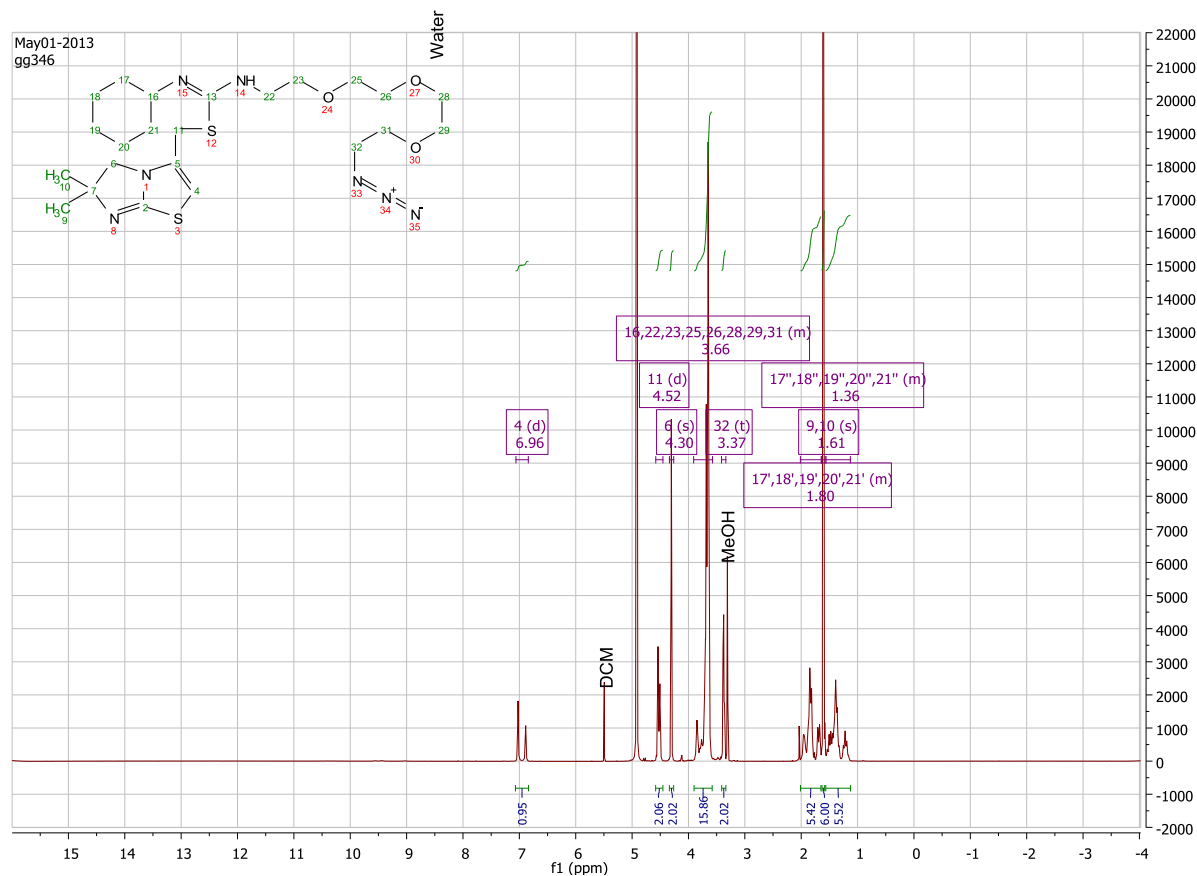


70. 2-{2-[2-(2-Azidoethoxy)ethoxy]ethoxy}-N-[(cyclohexylimino)]({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl)methyl]ethan-1-amine .3TFA

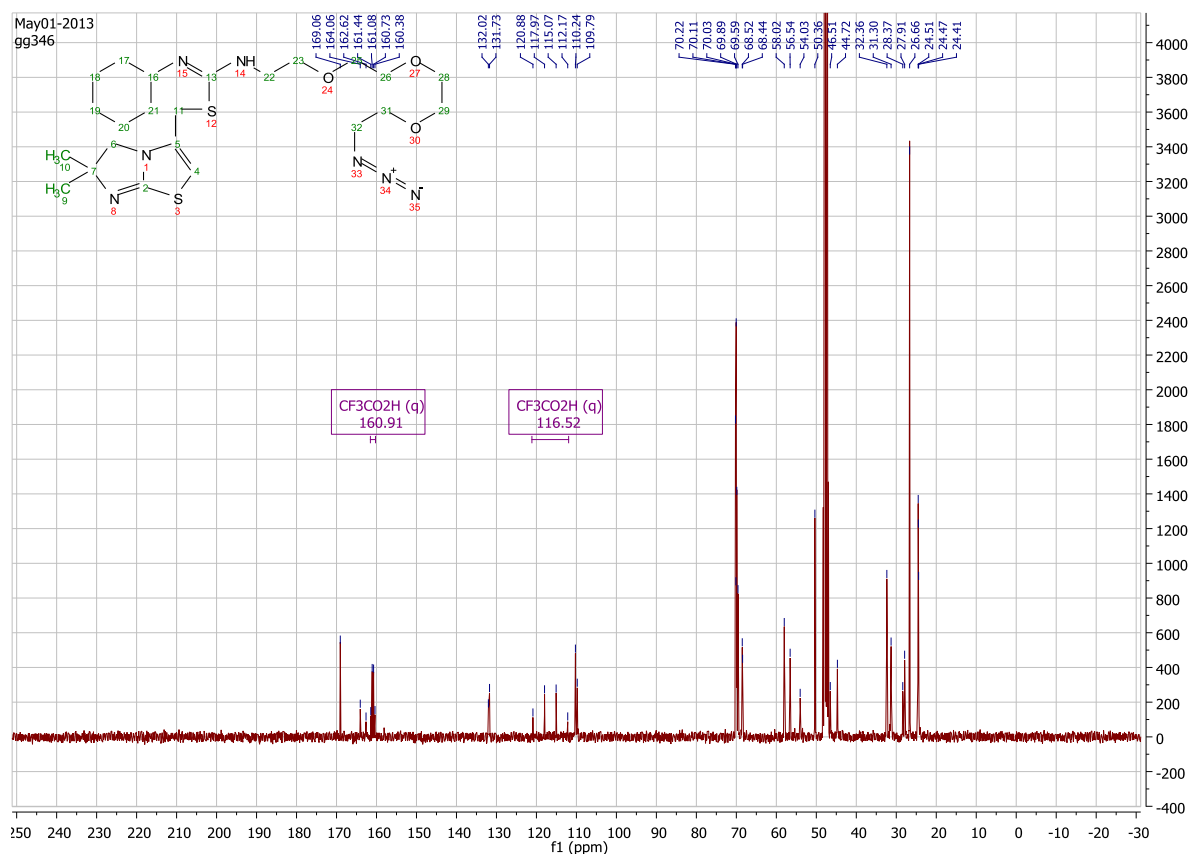
Infrared spectrum



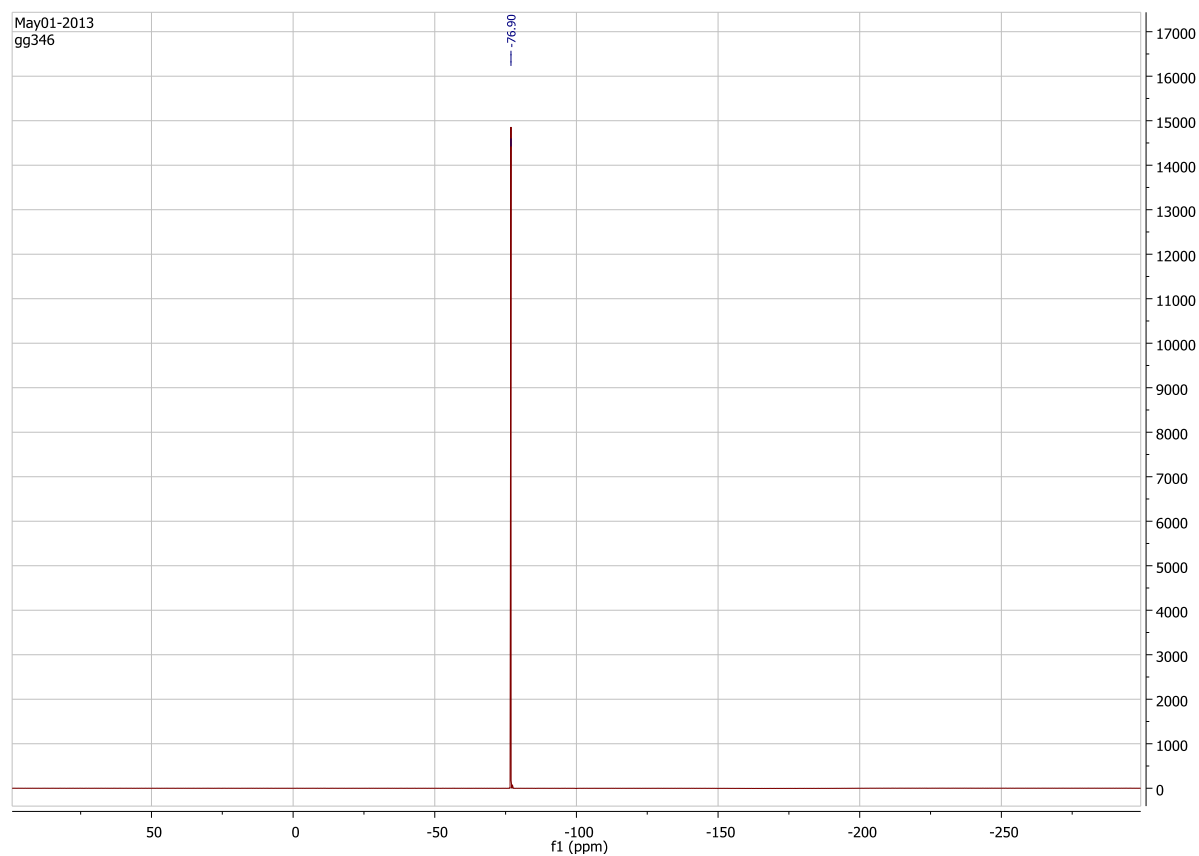
¹H NMR spectrum



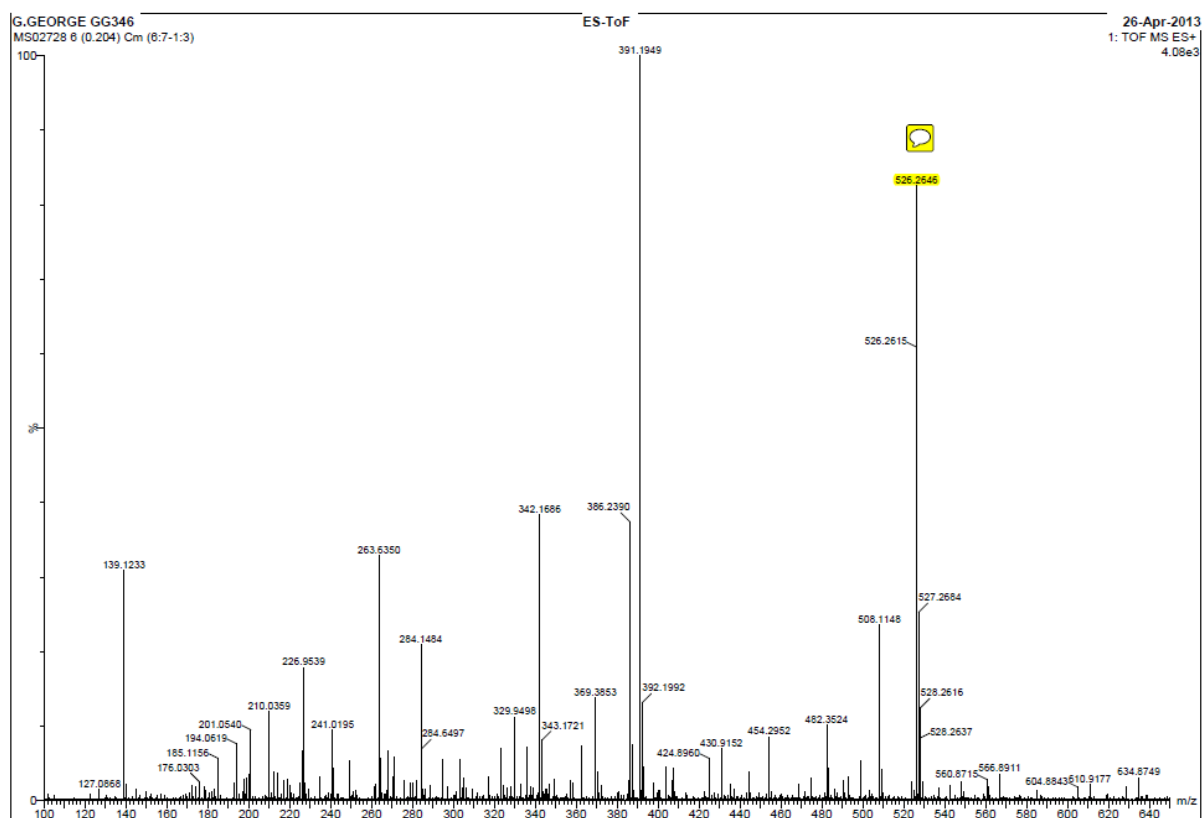
^{13}C NMR spectrum



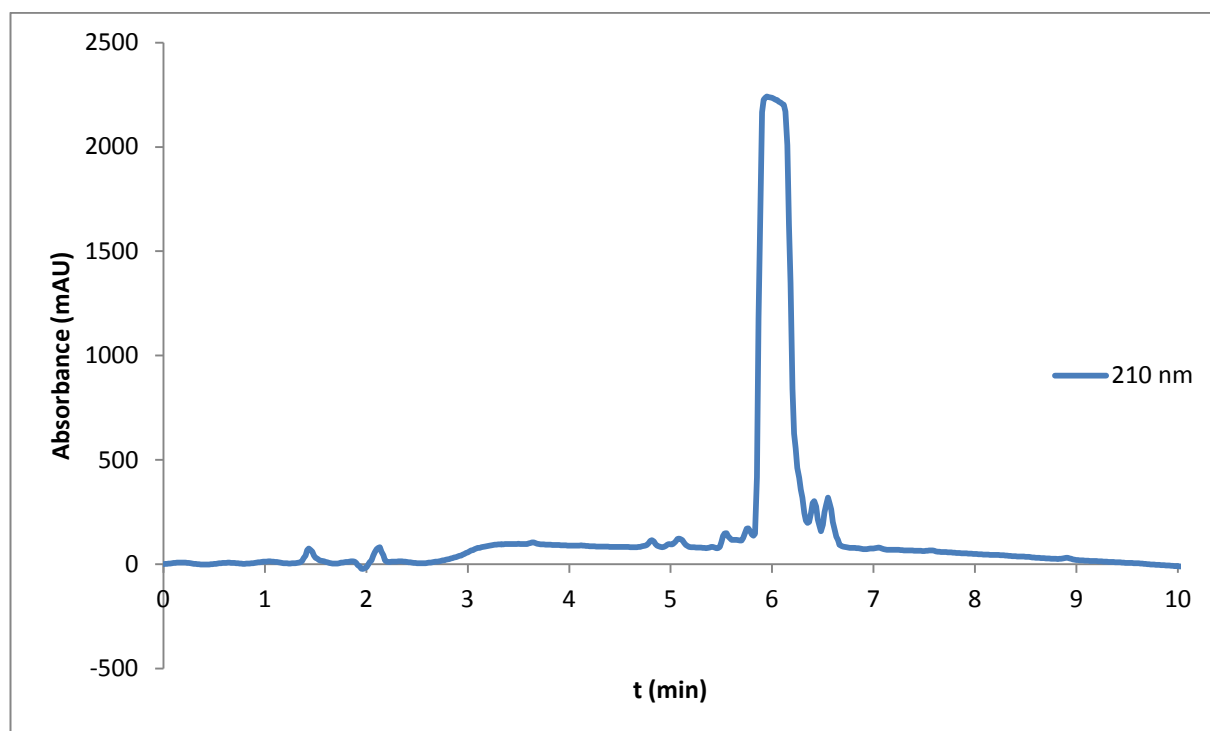
^{19}F NMR spectrum



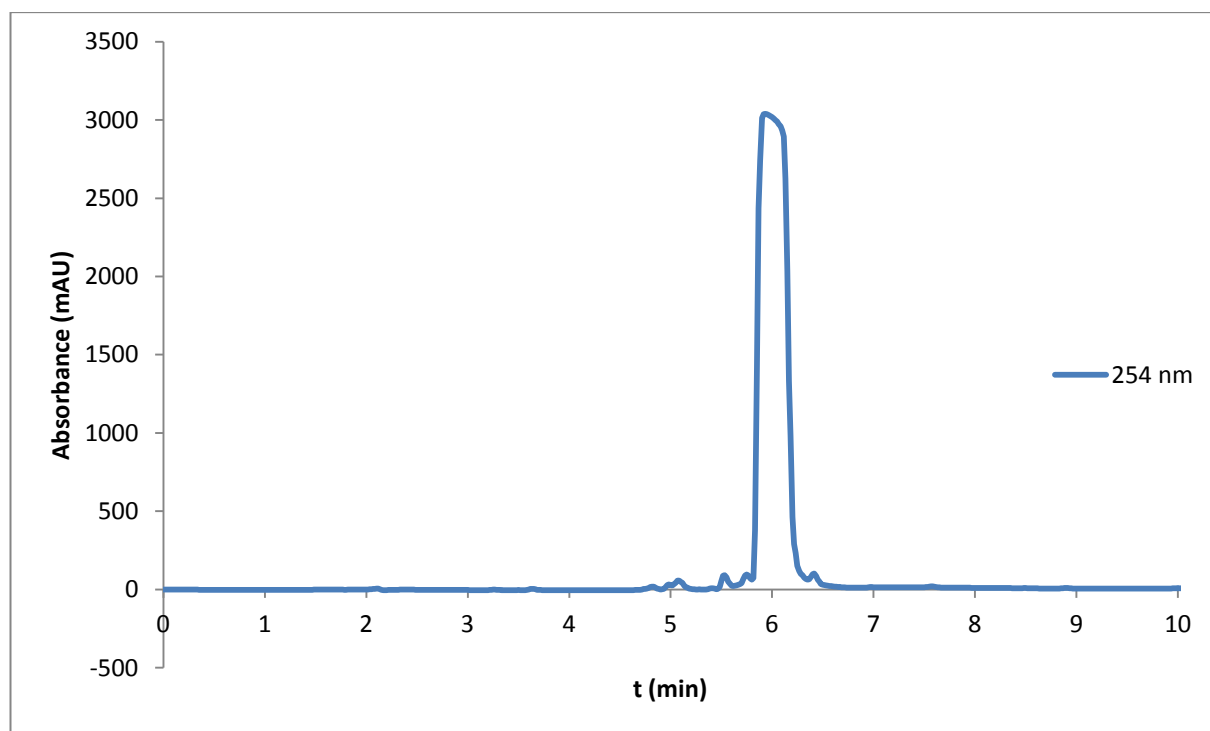
HRMS



HPLC trace at 210 nm

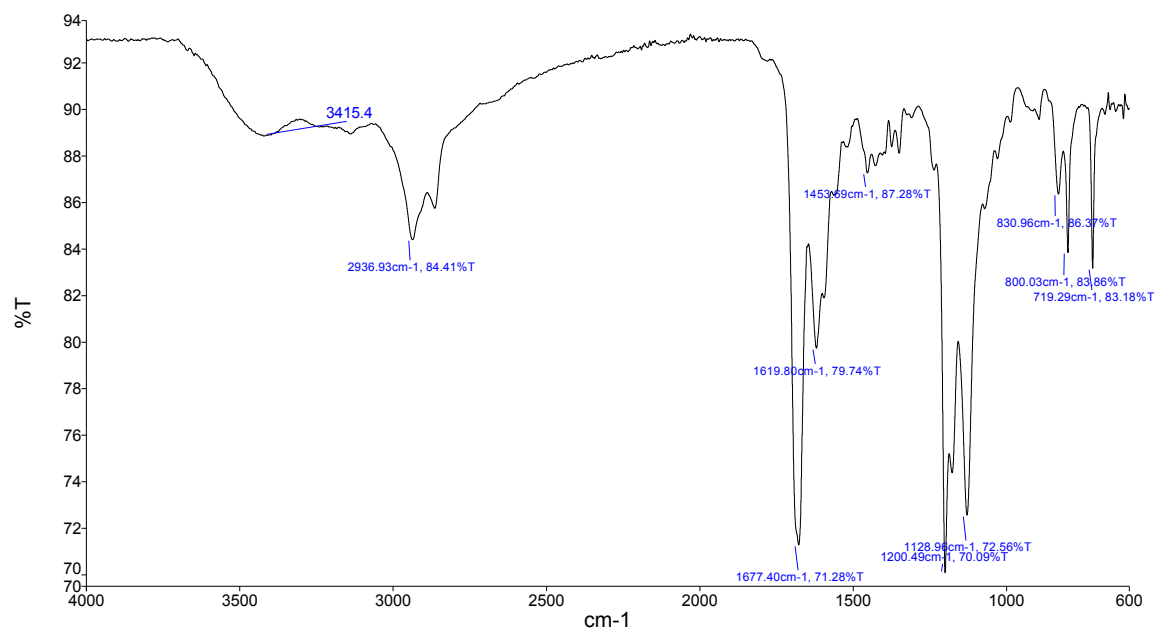


HPLC trace at 254 nm

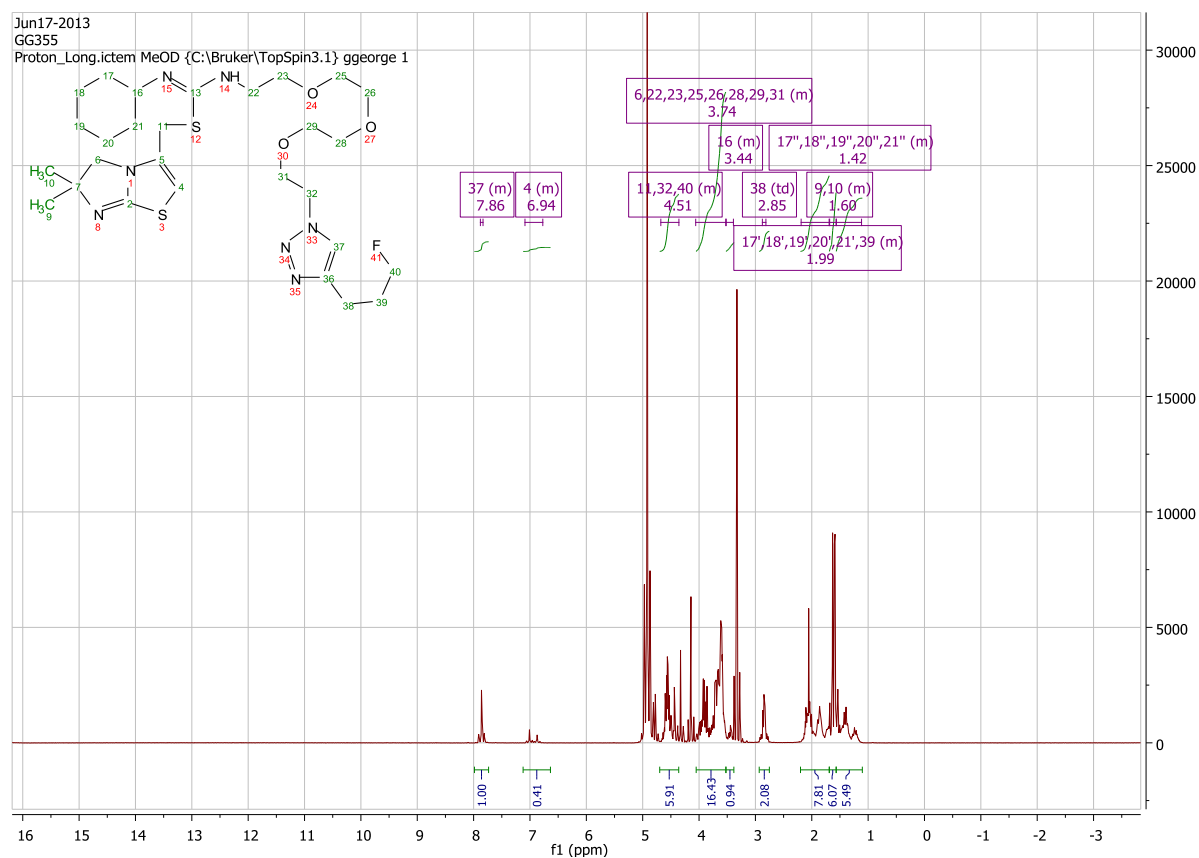


71. ***N*-[(cyclohexylimino)[(6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*]thiazol-3-yl)methyl)sulfanyl]methyl]-2-[2-(2-{2-[4-(3-fluoropropyl)-1*H*-1,2,3-triazol-1-yl]ethoxy}ethoxy)ethoxy]ethan-1-amine .3TFA**

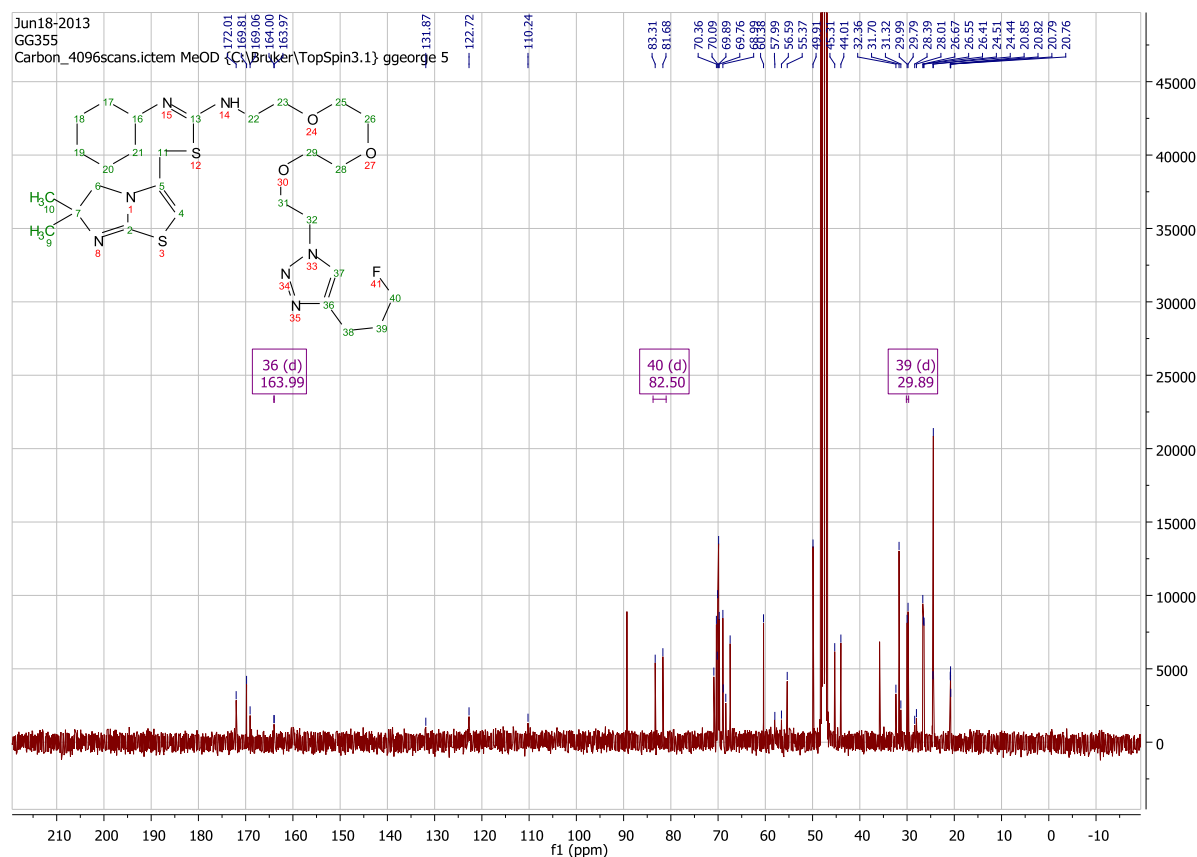
Infrared spectrum



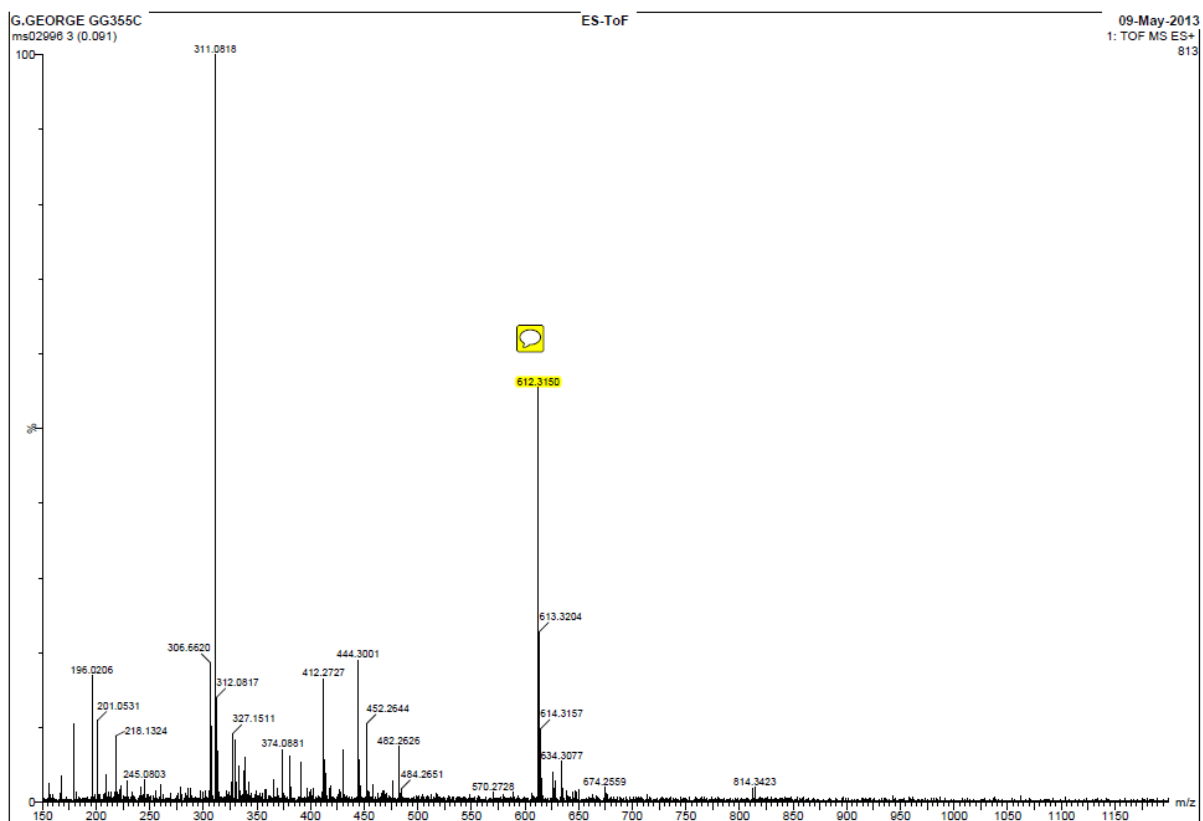
¹H NMR spectrum



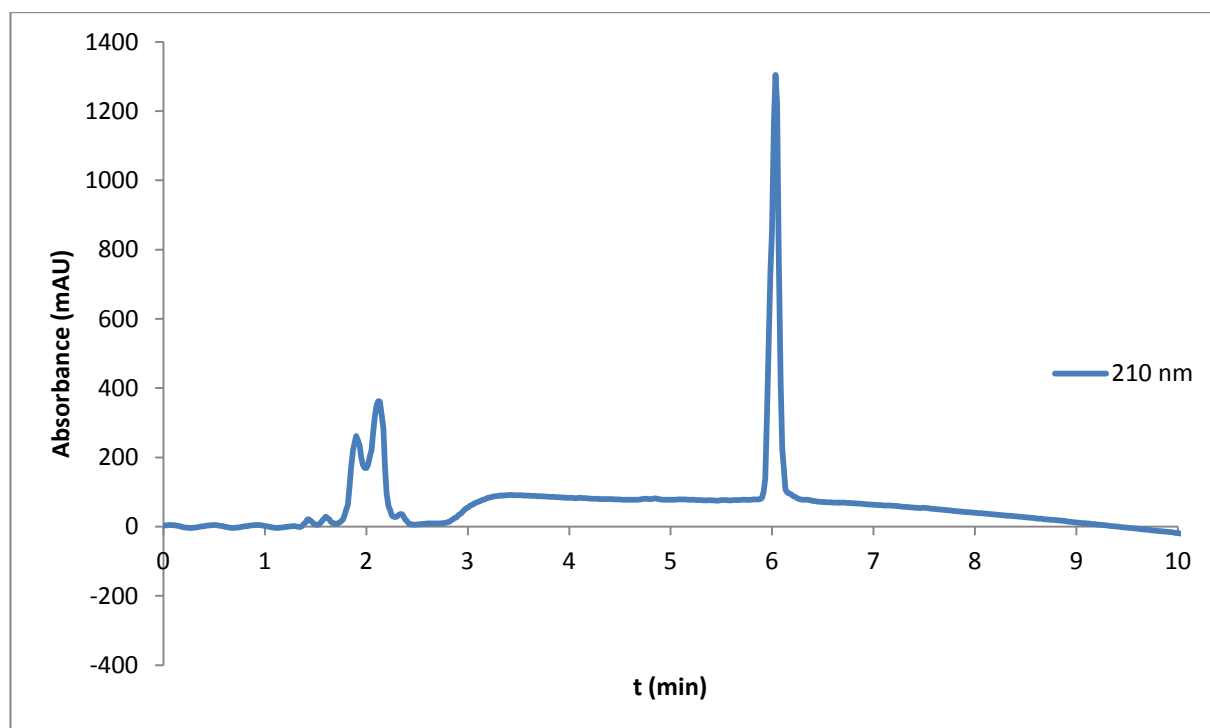
^{13}C NMR spectrum



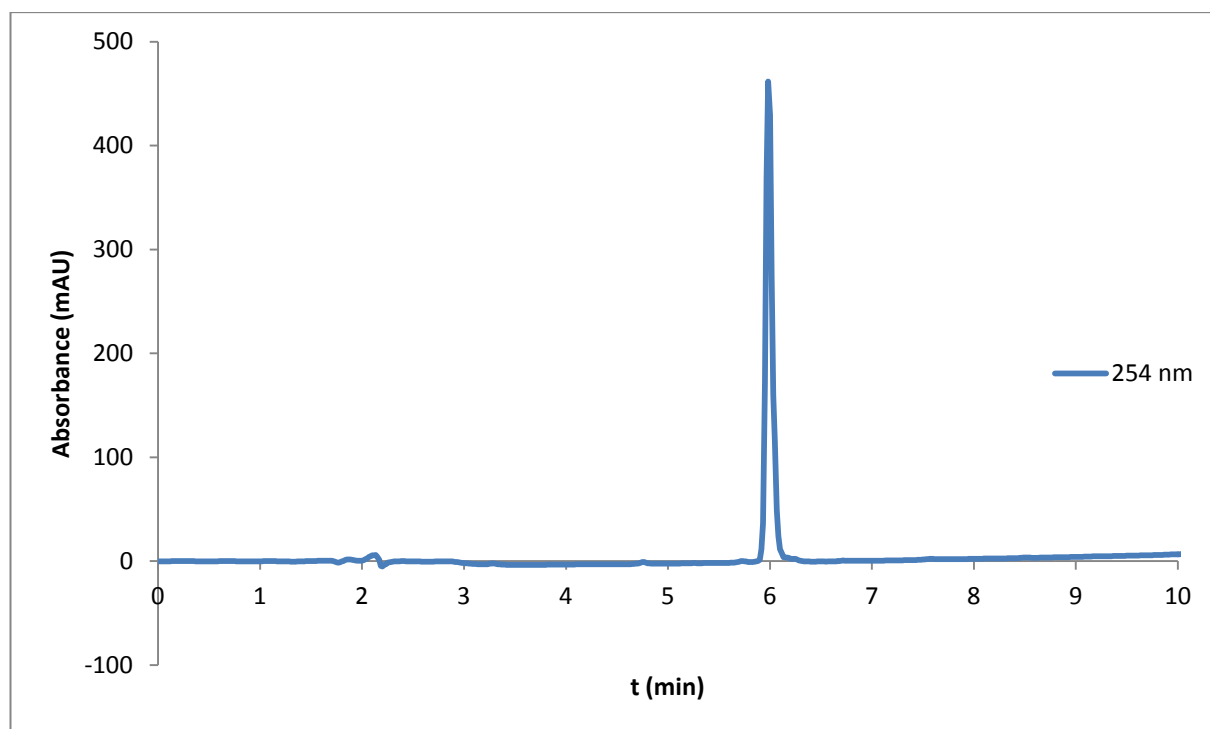
HRMS



HPLC trace at 210 nm

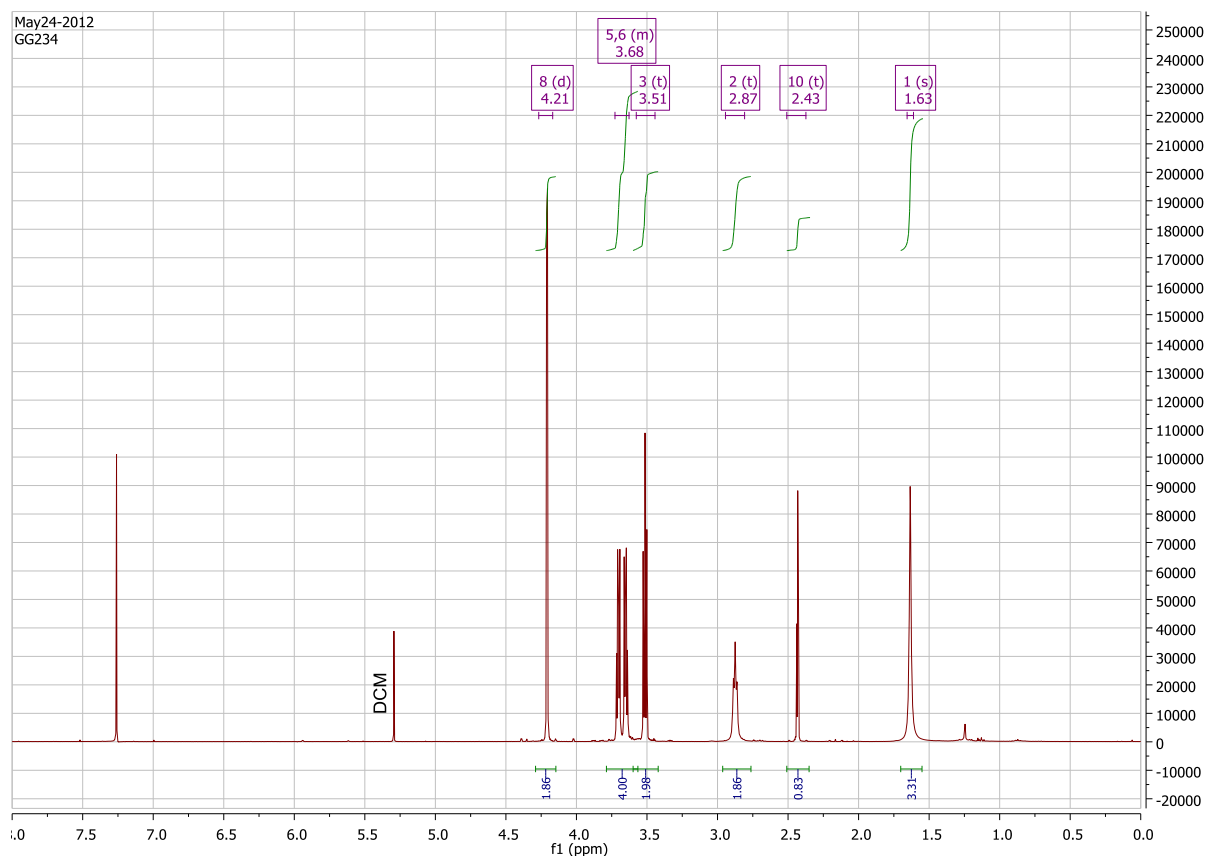


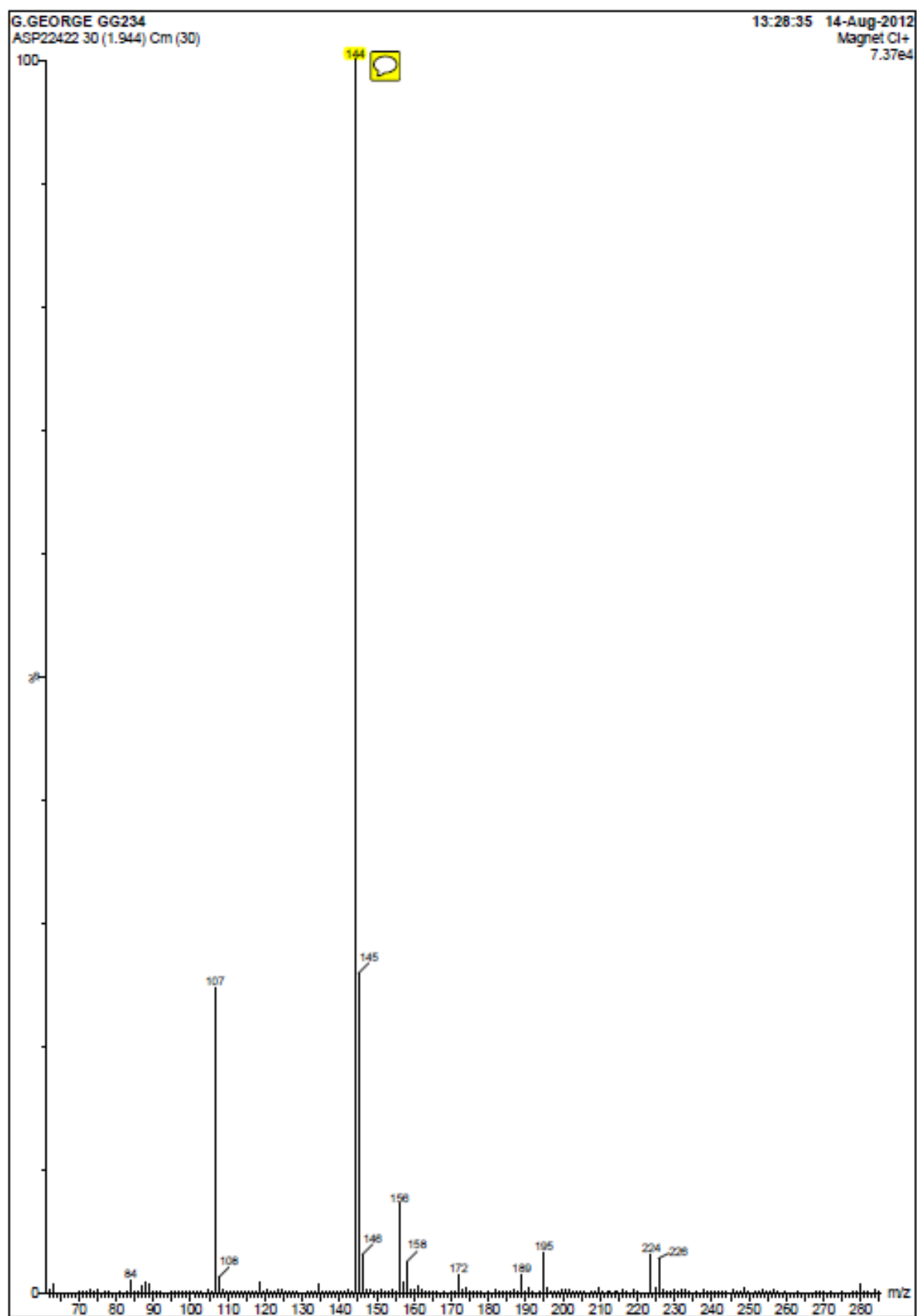
HPLC trace at 254 nm



72. 3-[2-(2-aminoethoxy)ethoxy]prop-1-yne

^1H NMR spectrum





Single Mass Analysis (displaying only valid results)

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

55 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 7-7 H: 0-1000 N: 0-8 O: 0-8 I: 0-2

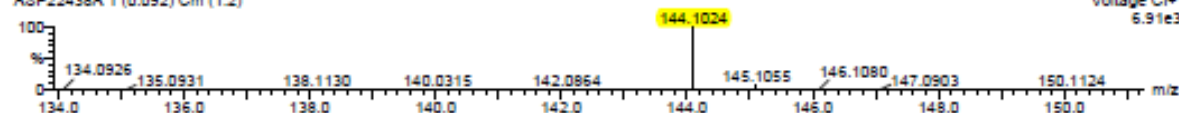
G. GEORGE GG234 AM M+H

ASP22438A 1 (0.092) Cm (1:2)

10:14:31 15-Aug-2012

Voltage Cl+

6.91e3



Minimum:

-1.5

Maximum:

7.0

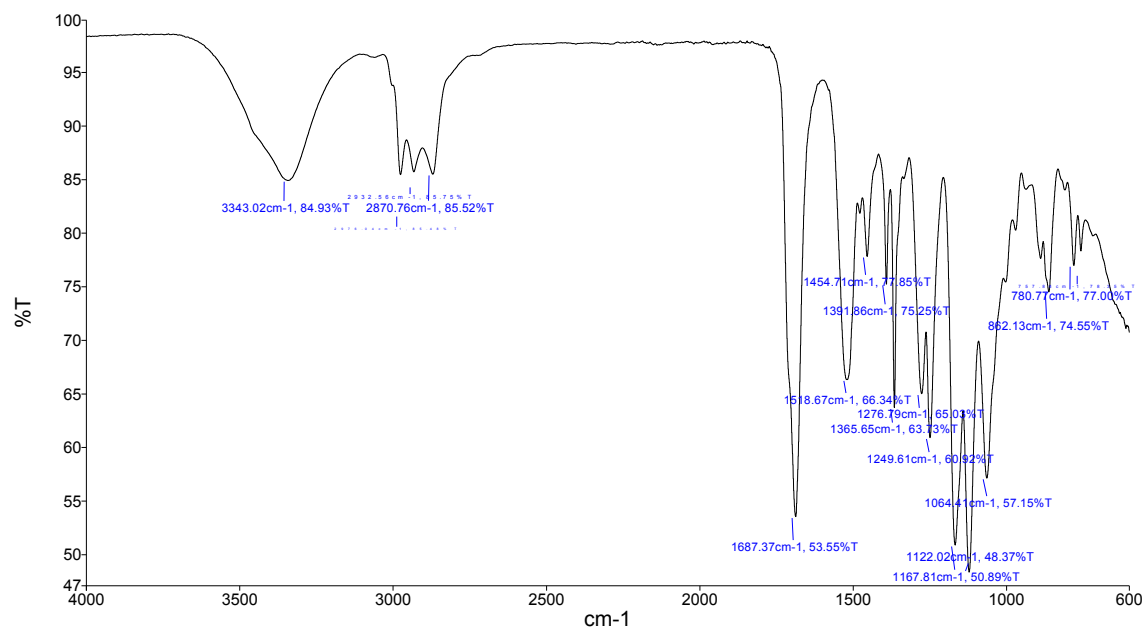
10.0

50.0

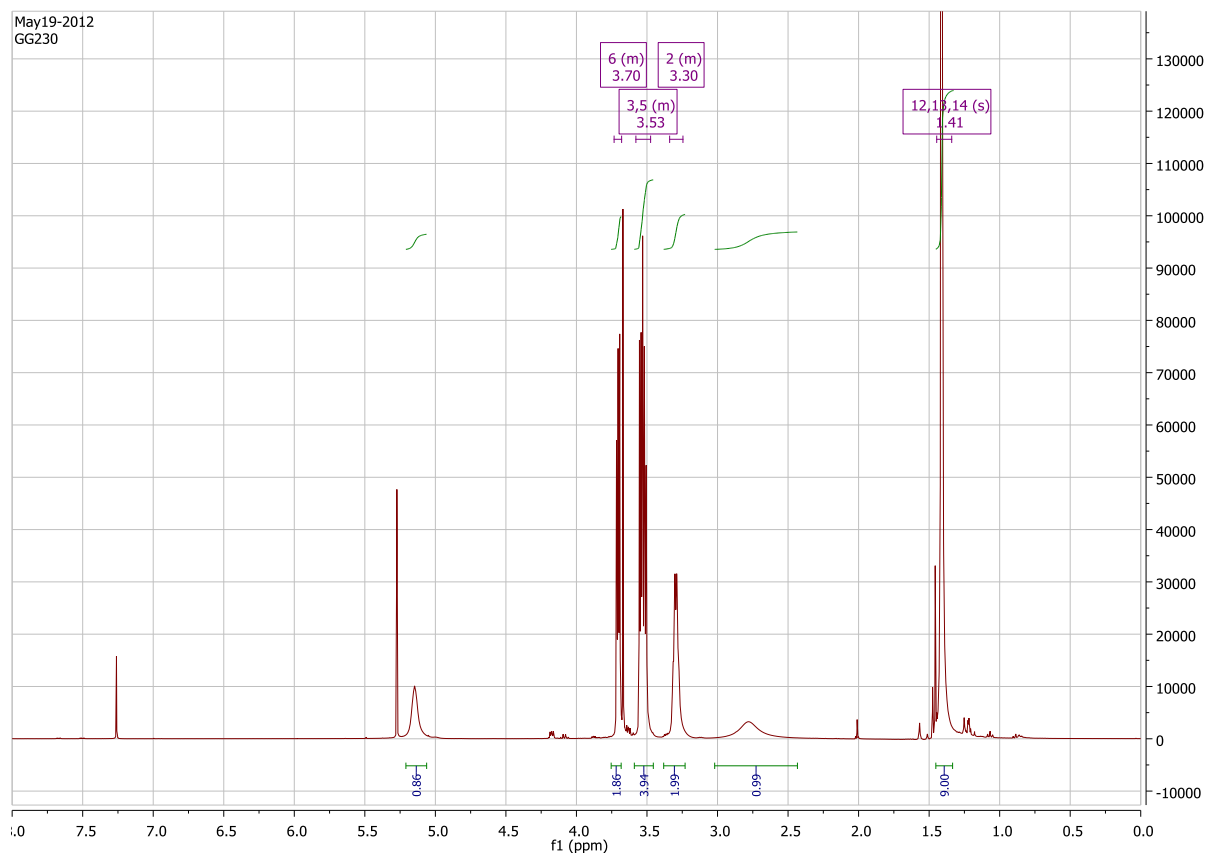
Mass	Calc. Mass	mDa	PPM	DBE	1-FIT	Formula
144.1024	144.1025	-0.1	-0.7	1.5	0.2	C7 H14 N O2

74. *tert*-Butyl *N*-[2-(2-hydroxyethoxy)ethyl]carbamate

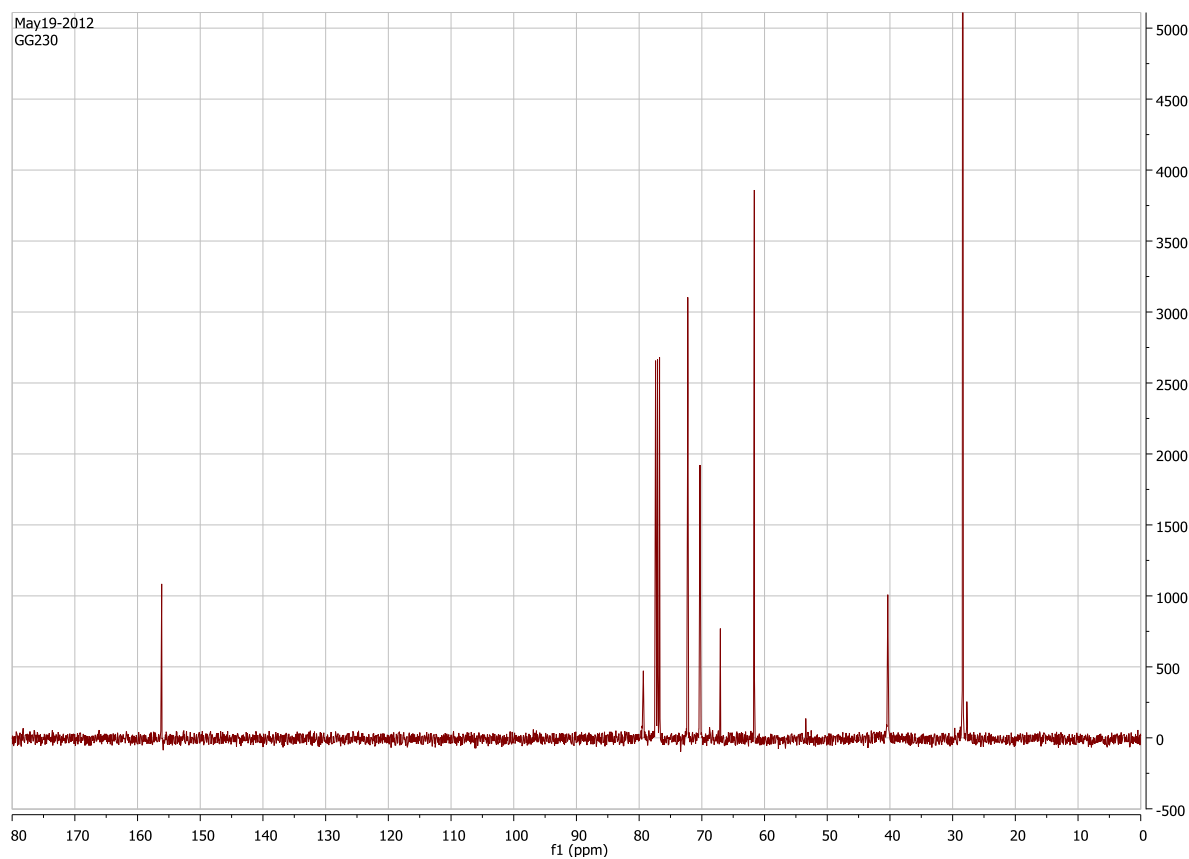
Infrared spectrum



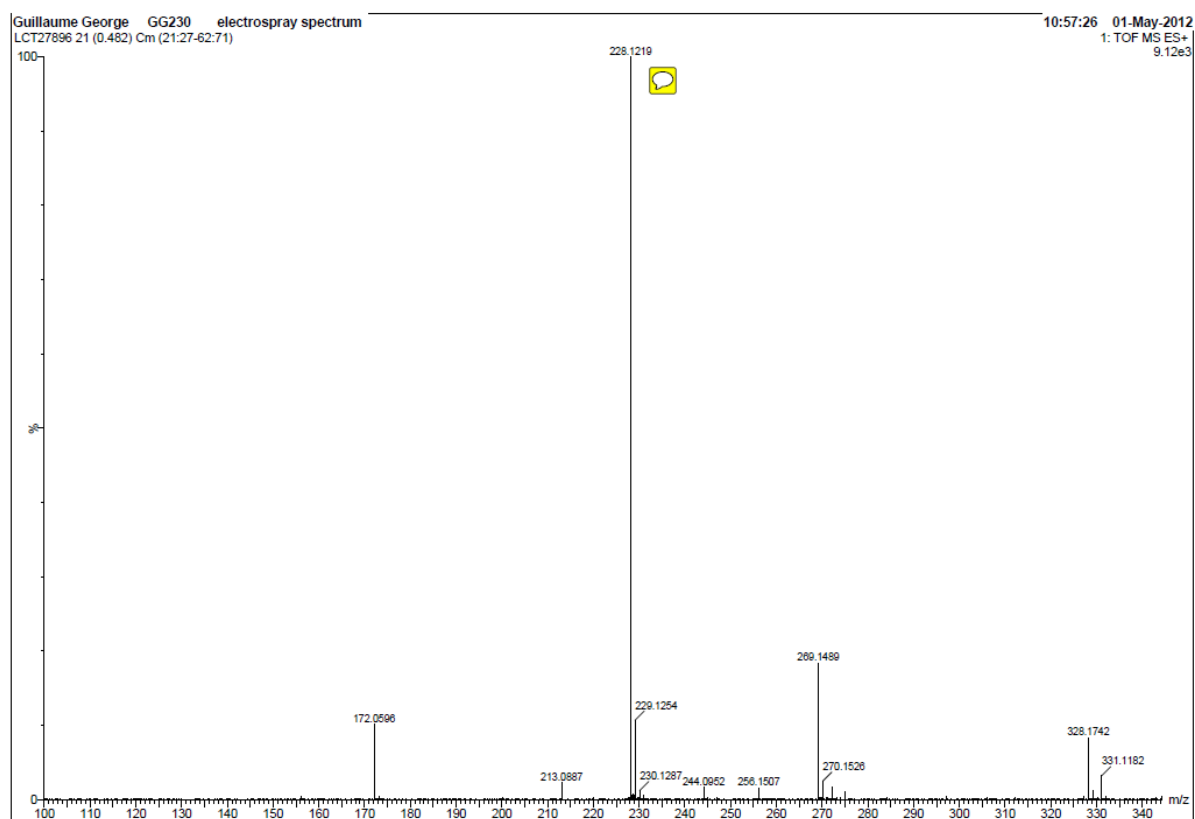
^1H NMR spectrum



^{13}C NMR spectrum

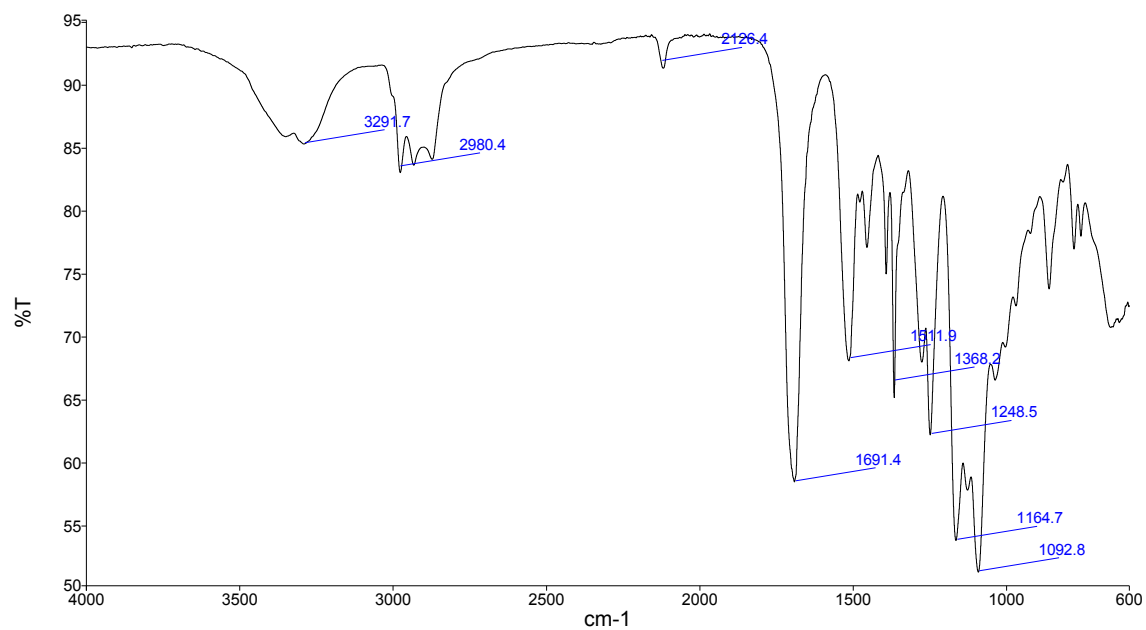


HRMS

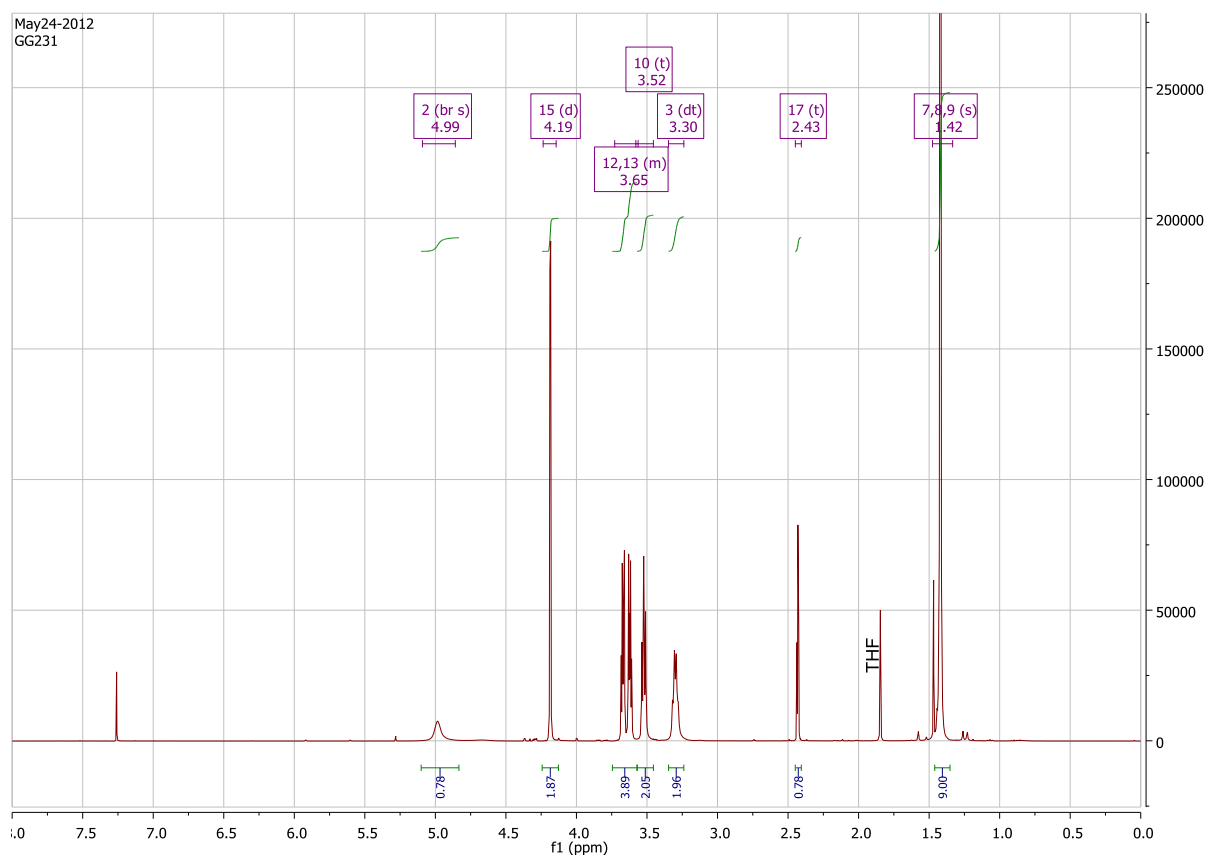


75. *tert*-Butyl *N*-{2-[2-(prop-2-yn-1-yloxy)ethoxy]ethyl}carbamate

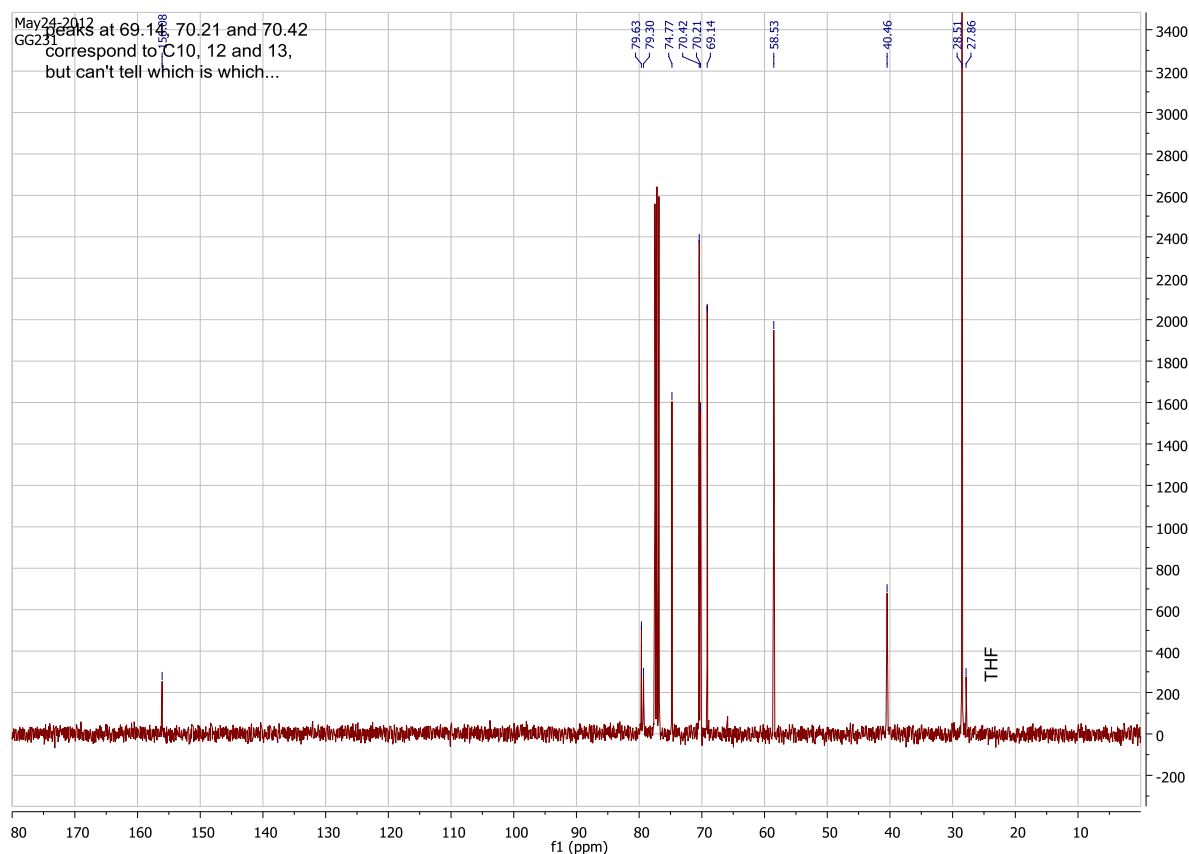
Infrared spectrum



^1H NMR spectrum



^{13}C NMR spectrum



HRMS

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

212 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 12-12 H: 5-300 N: 0-10 O: 0-10 Na: 0-1

G.GEORGE GG231

LCT28598 9 (0.312) Cm (9:11-37:40)

13:56:55 17-Aug-2012

1: TOF MS ES+

9.25e+003



Minimum:

Maximum:

5.0

10.0

-1.5

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

266.1379

266.1368

1.1

4.1

2.5

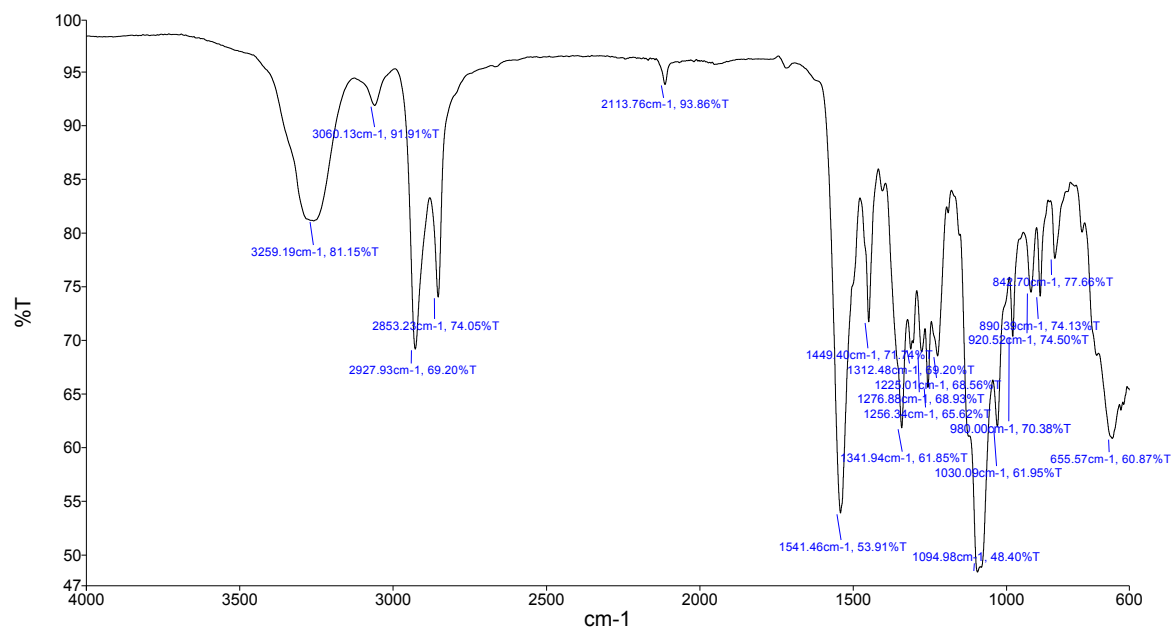
184.1

0.0

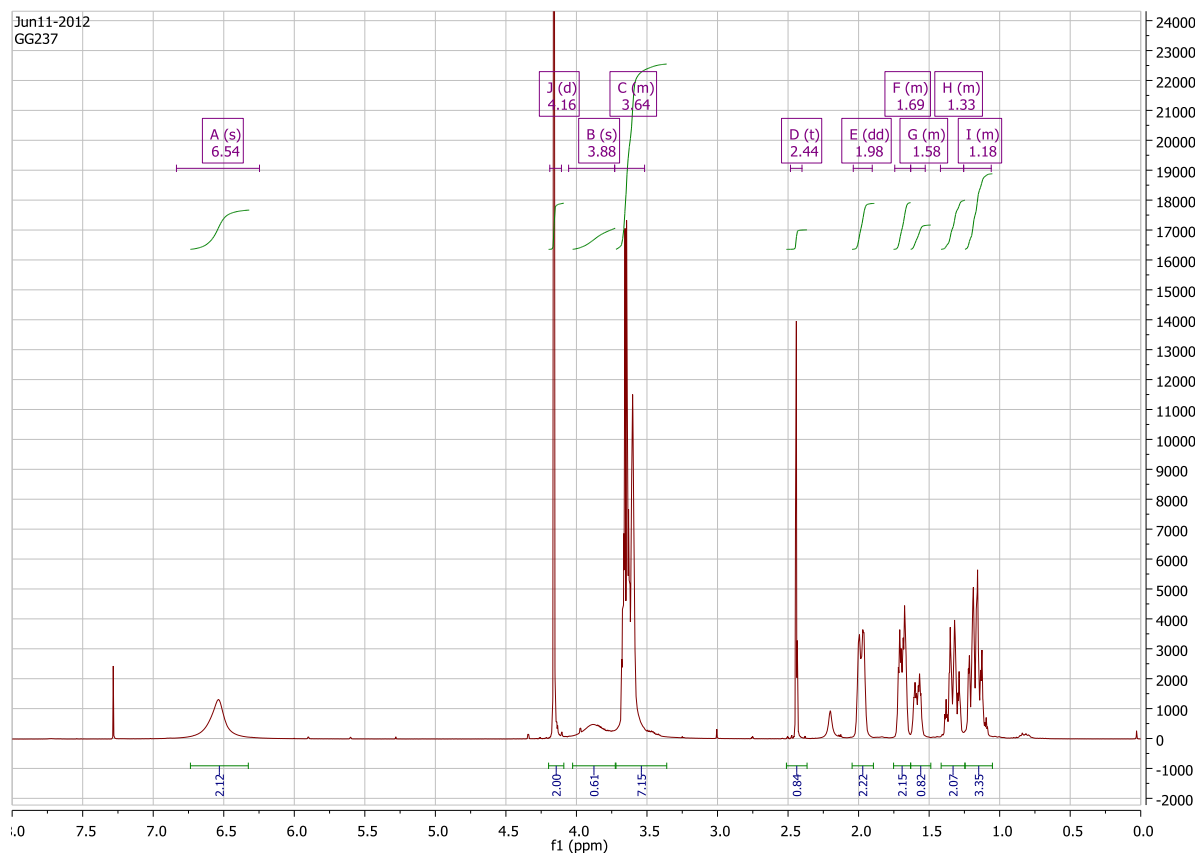
C12 H21 N O4 Na

76. 1-Cyclohexyl-3-{2-[2-(prop-2-yn-1-yloxy)ethoxy]ethyl}thiourea

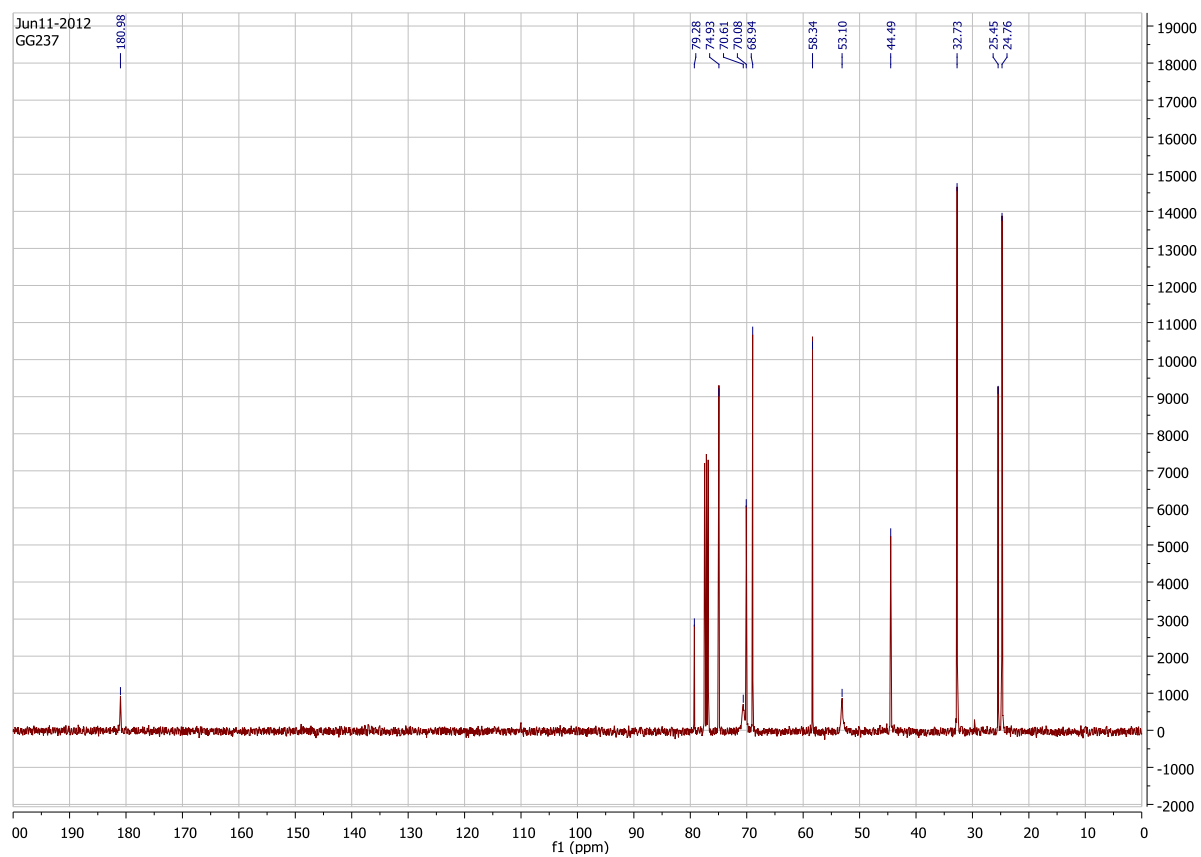
Infrared spectrum



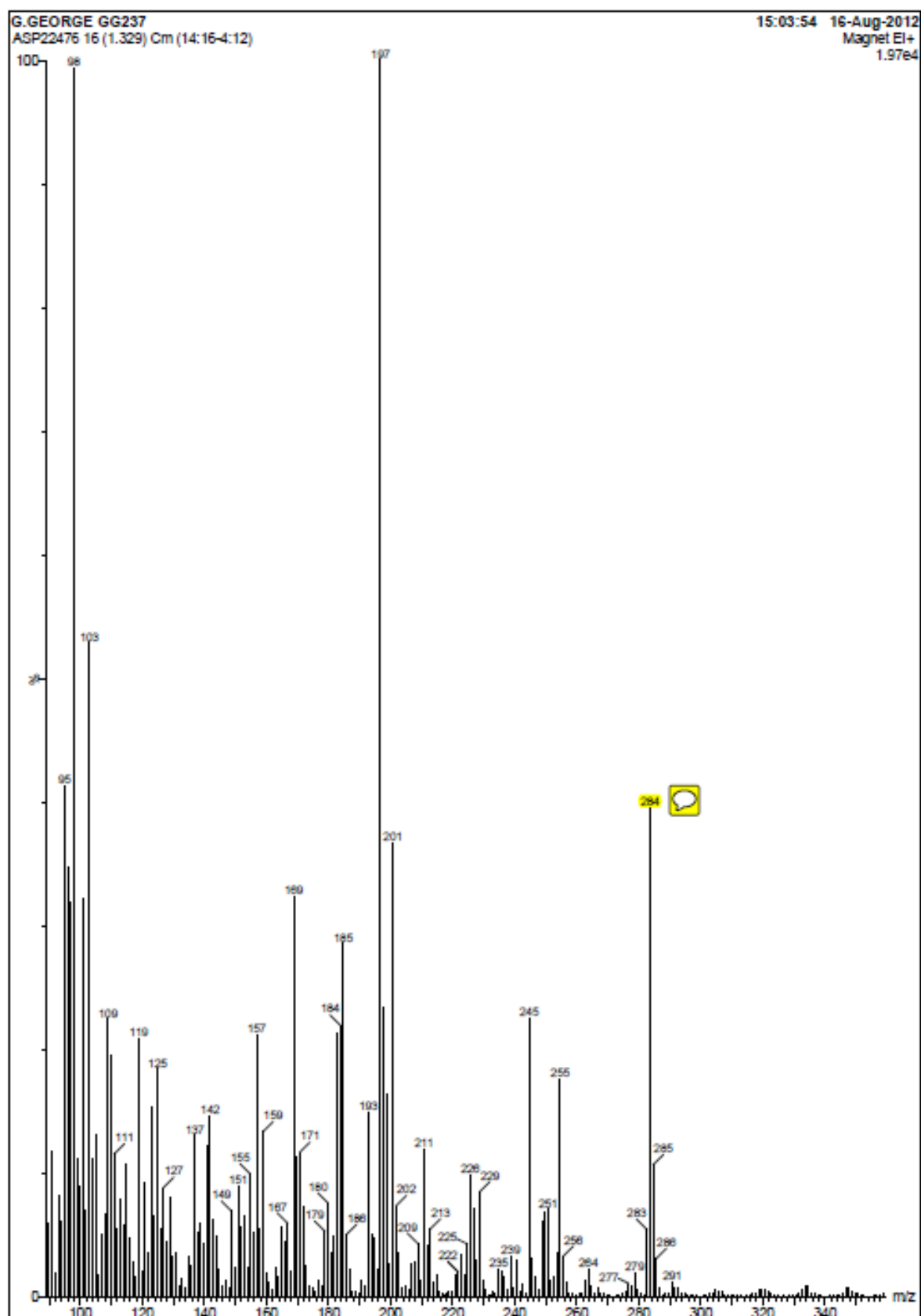
^1H NMR spectrum



^{13}C NMR spectrum



HRMS



Single Mass Analysis (displaying only valid results)

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

277 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 14-14 H: 0-1000 N: 0-10 O: 0-10 S: 1-2 I: 0-2

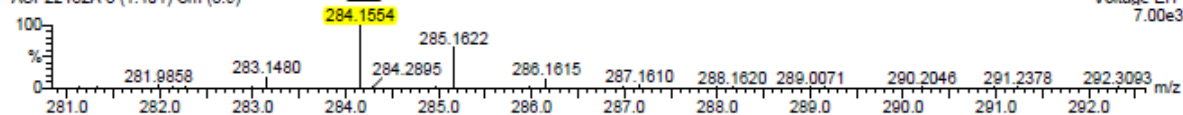
G.GEORGE GG237 AM

ASP22482A 8 (1.401) Cm (8:9)

15:37:21 16-Aug-2012

Voltage EI+

7.00e3



Minimum:

Maximum:

7.0

10.0

-1.5

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

Formula

284.1554

284.1559

-0.5

-1.8

4.0

1247.0

C14

H24

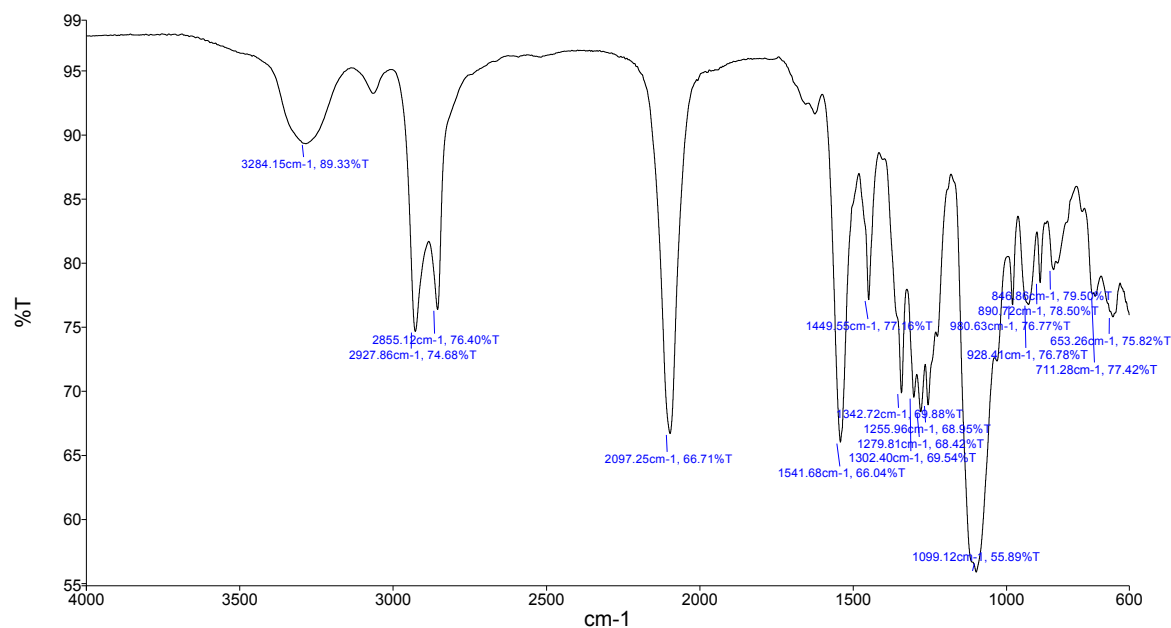
N2

O2

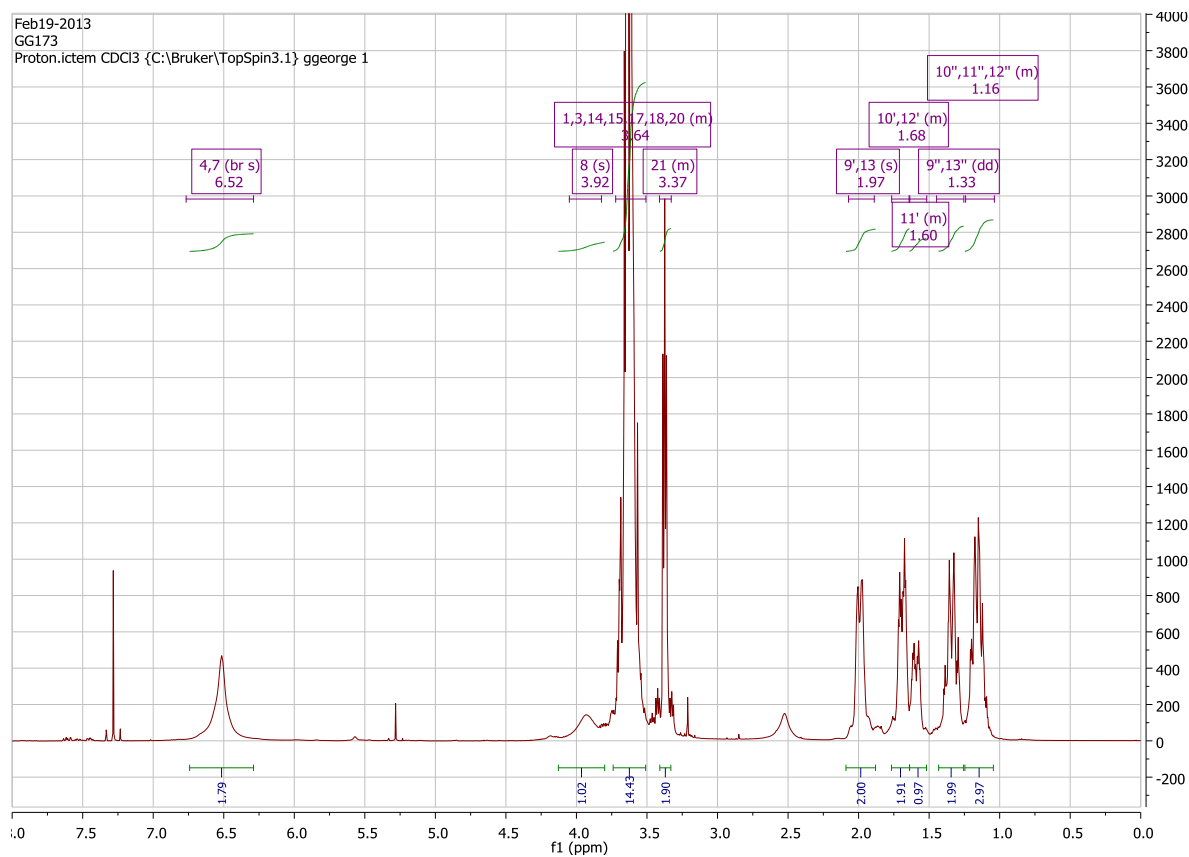
S

79. 3-(2-{2-[2-(2-Azidoethoxy)ethoxy]ethoxy}ethyl)-1-cyclohexylthiourea

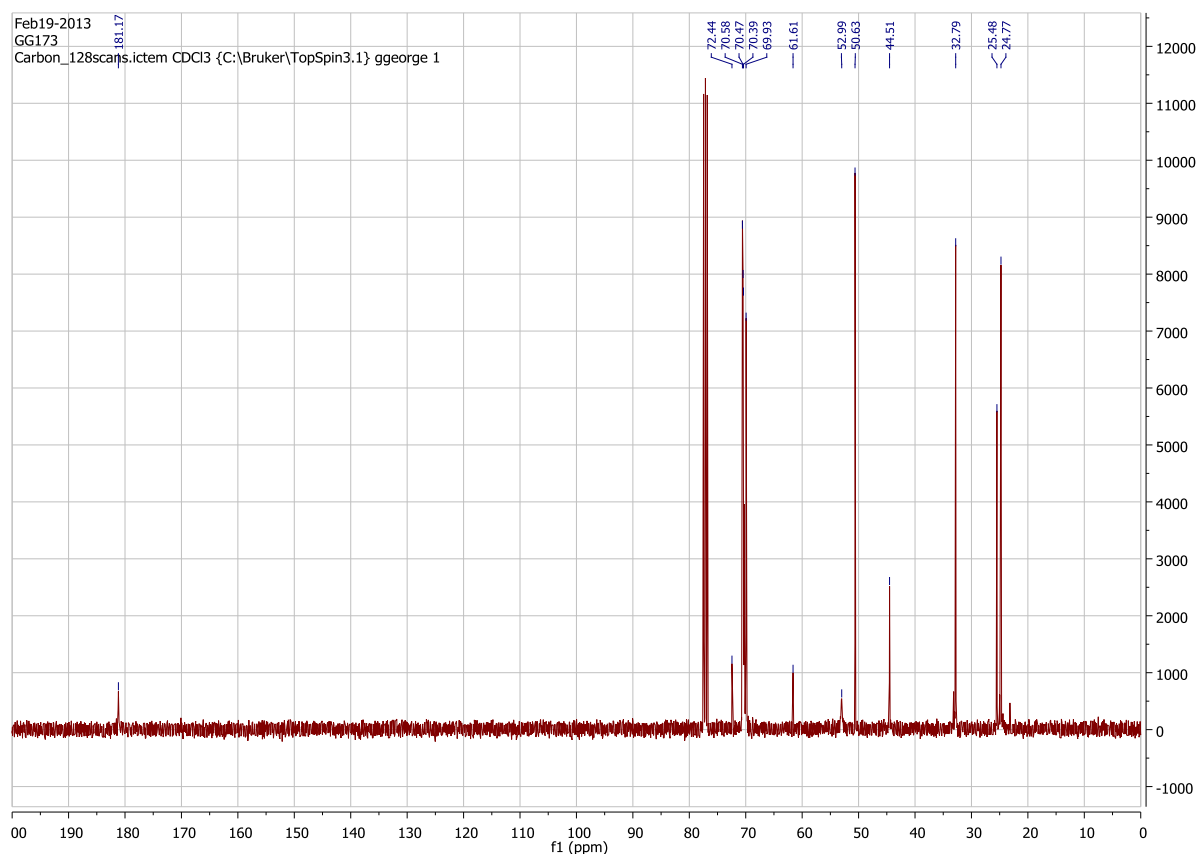
Infrared spectrum



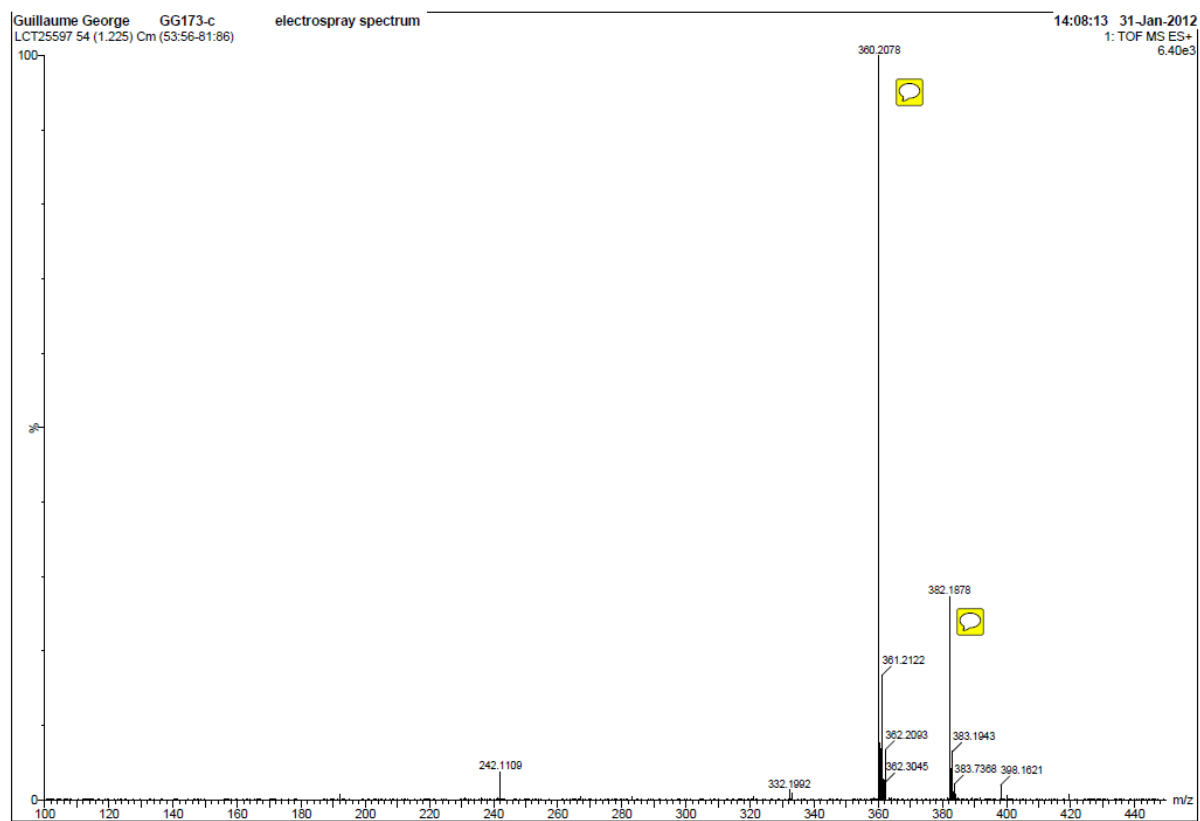
¹H NMR spectrum



^{13}C NMR spectrum

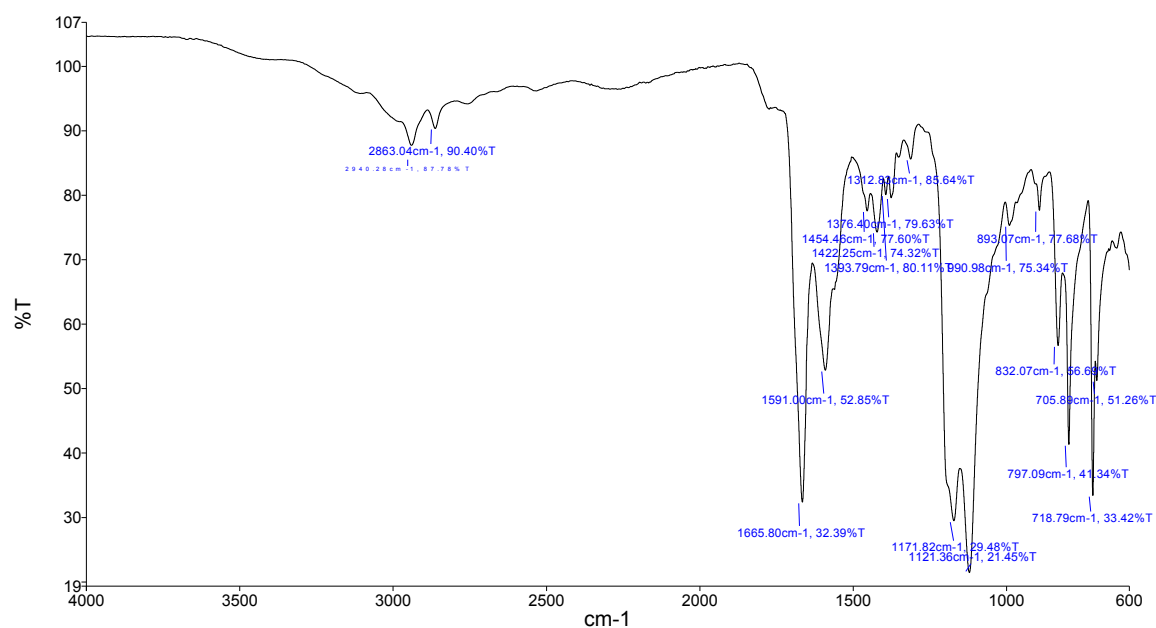


HRMS

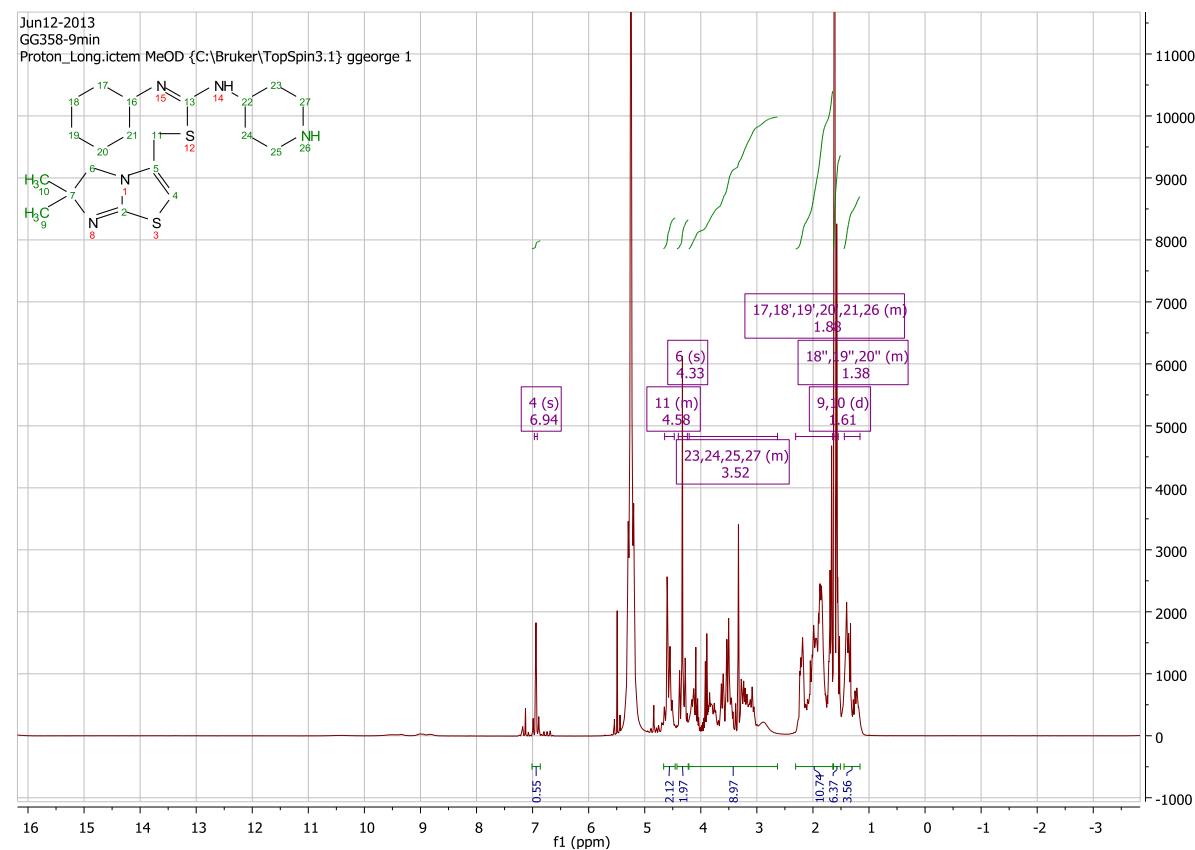


80. *N*'-Cyclohexyl-*N*-(piperidine-4-yl)[({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidamide .3TFA

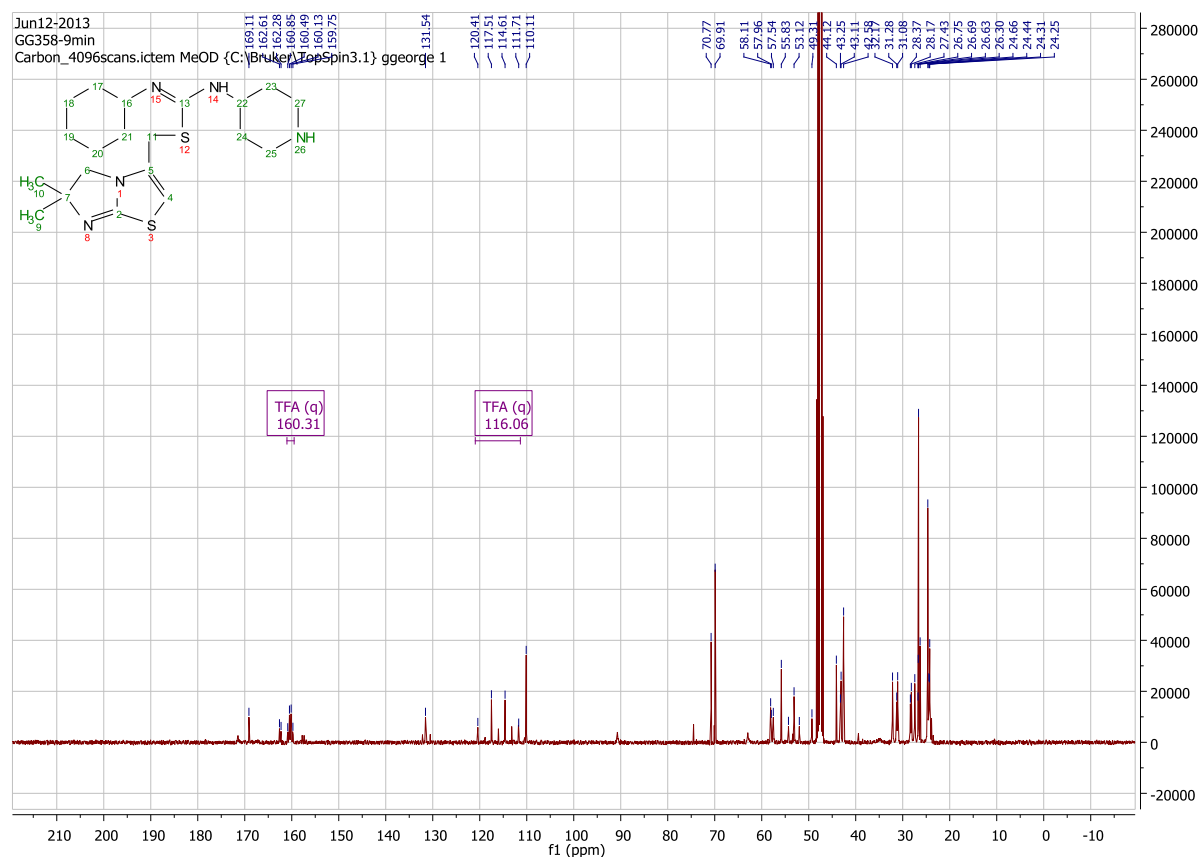
Infrared spectrum



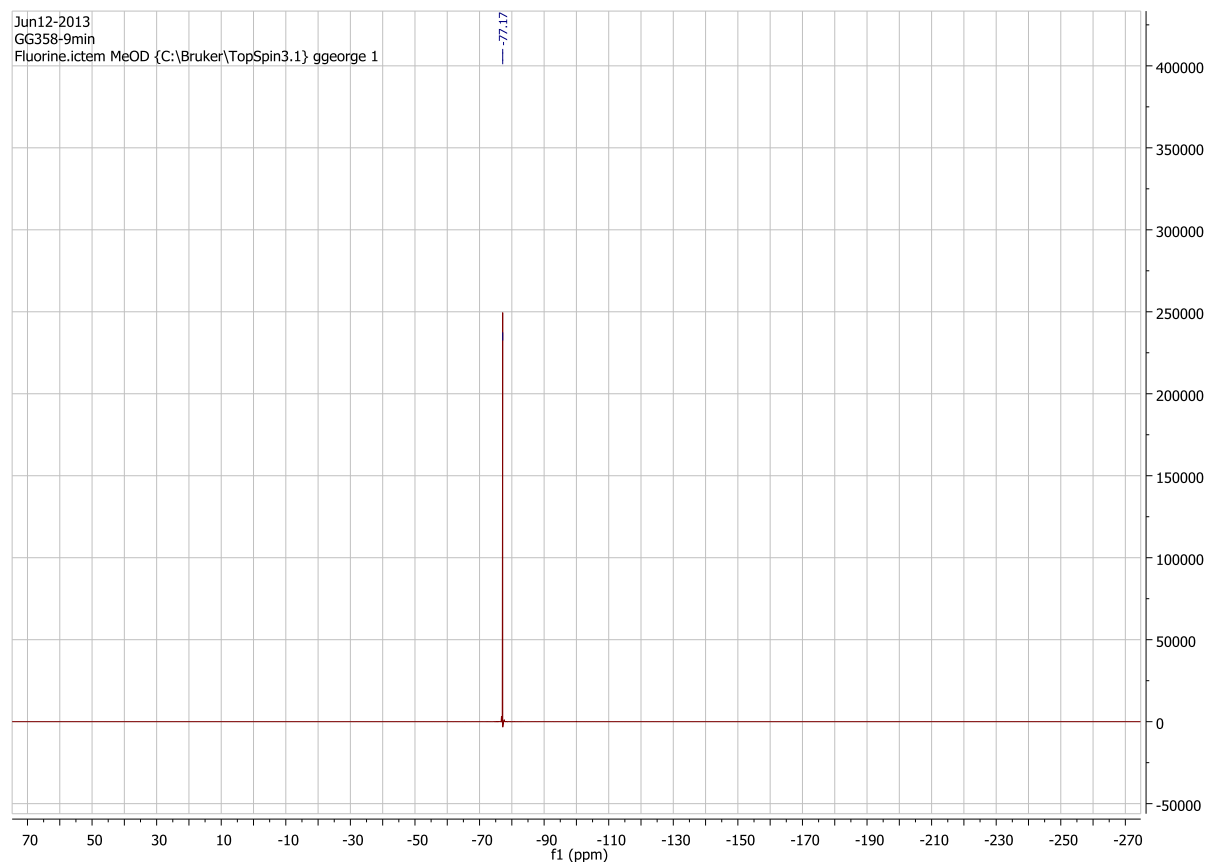
¹H NMR spectrum



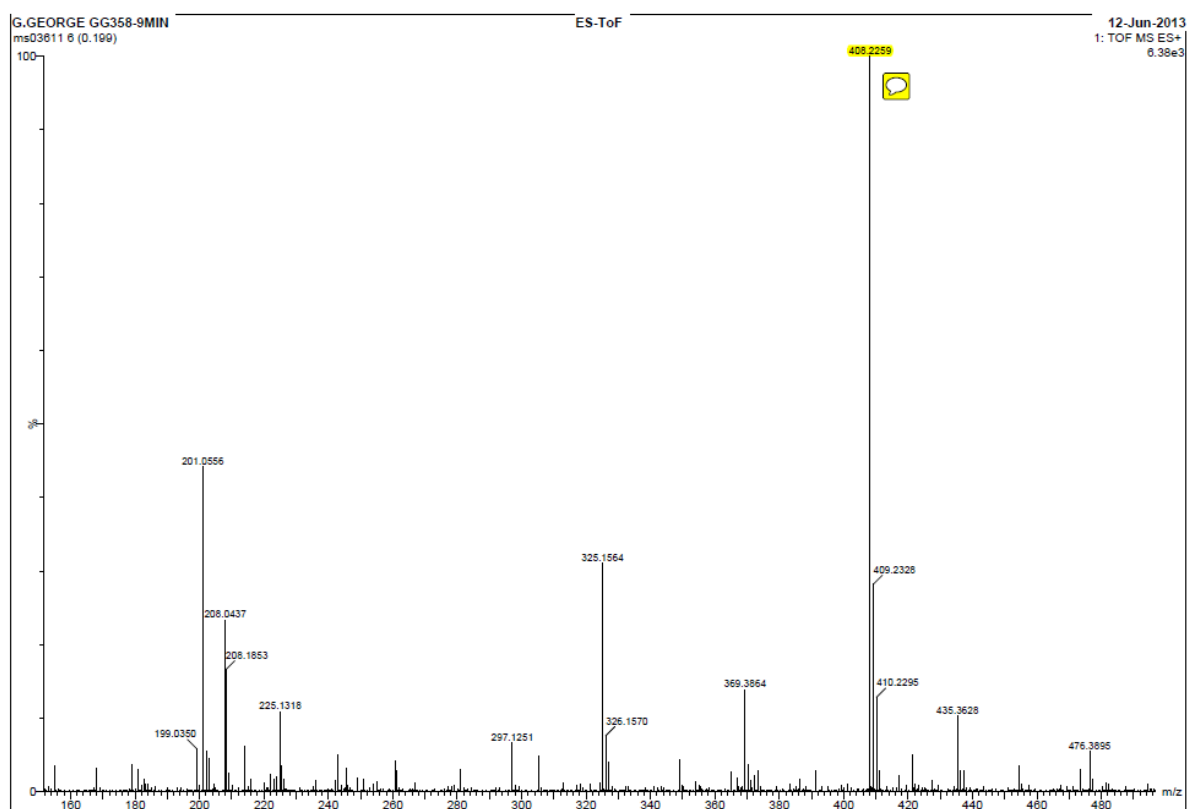
^{13}C NMR spectrum



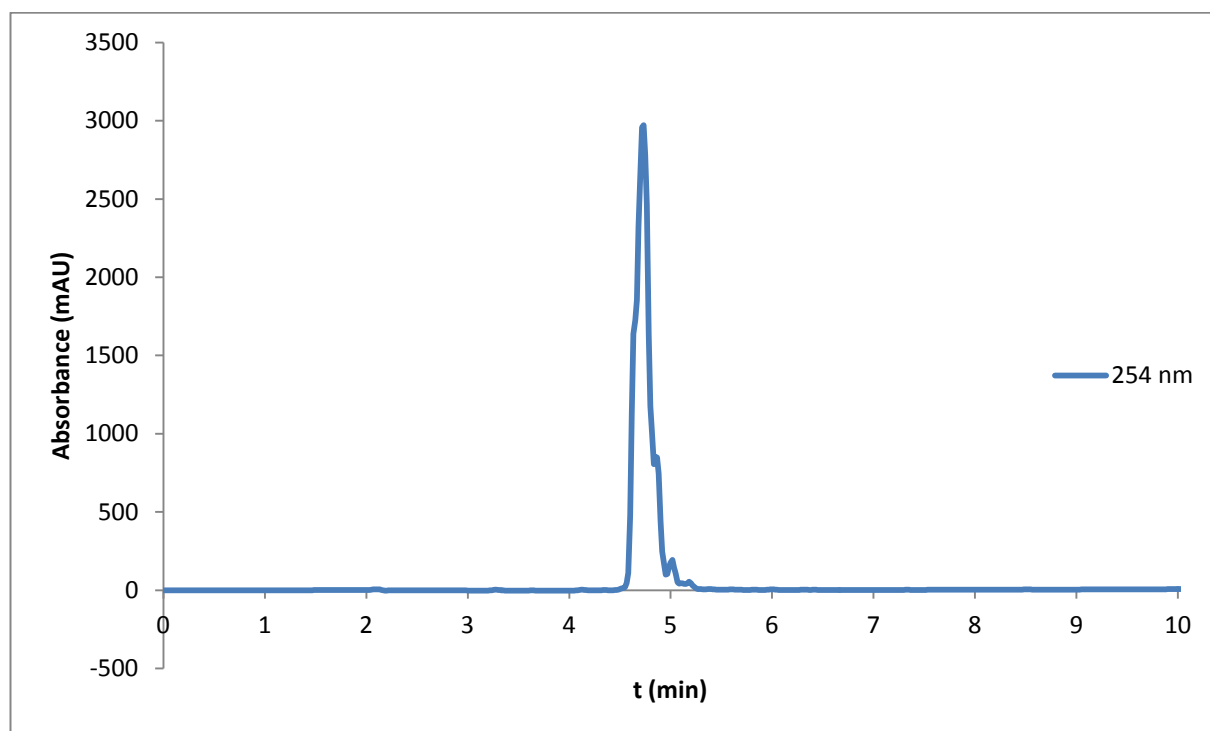
^{19}F NMR spectrum



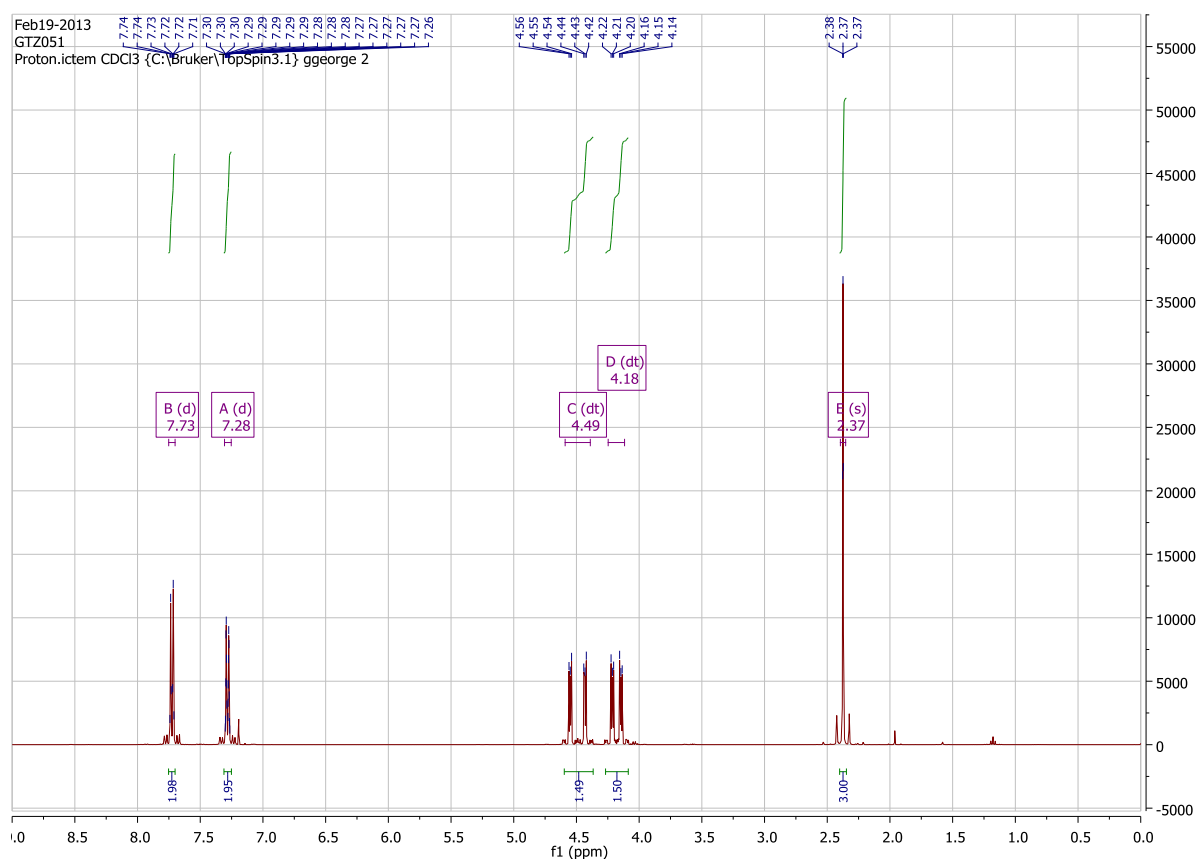
HRMS



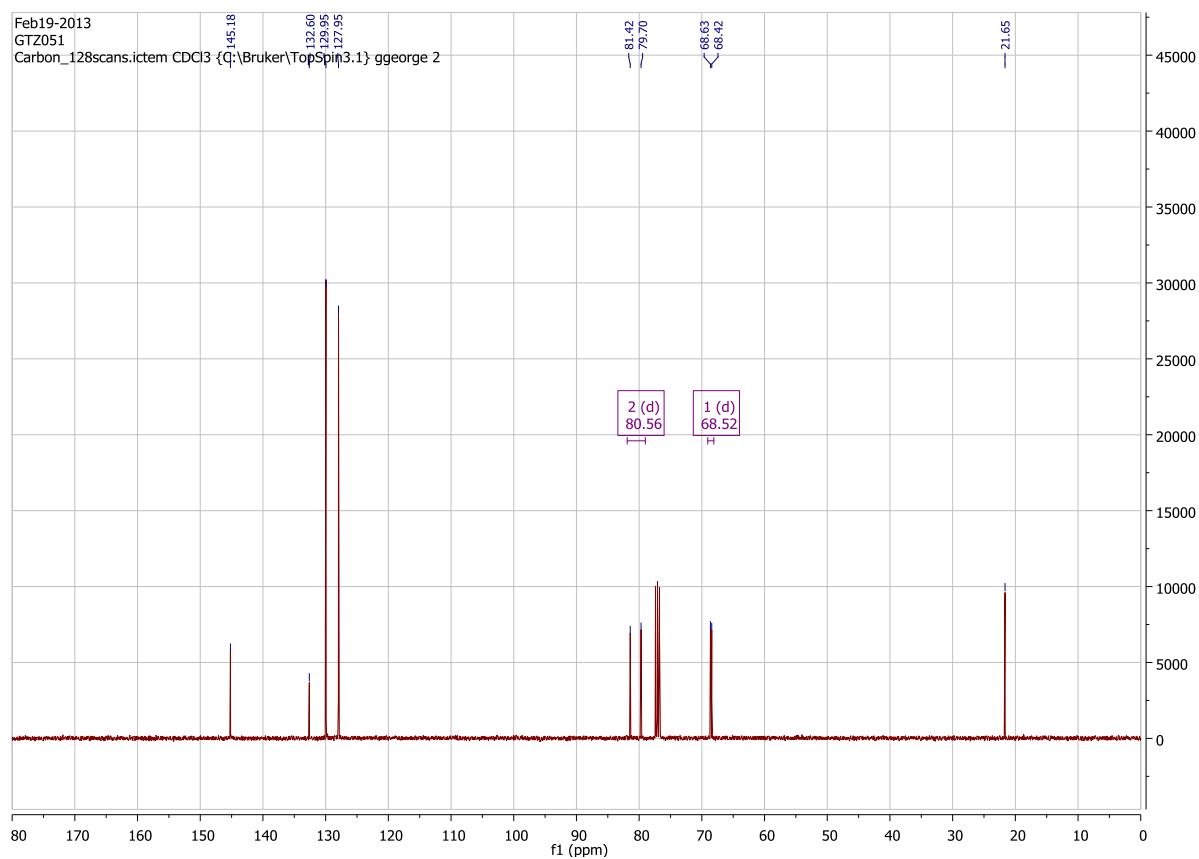
HPLC trace at 254 nm



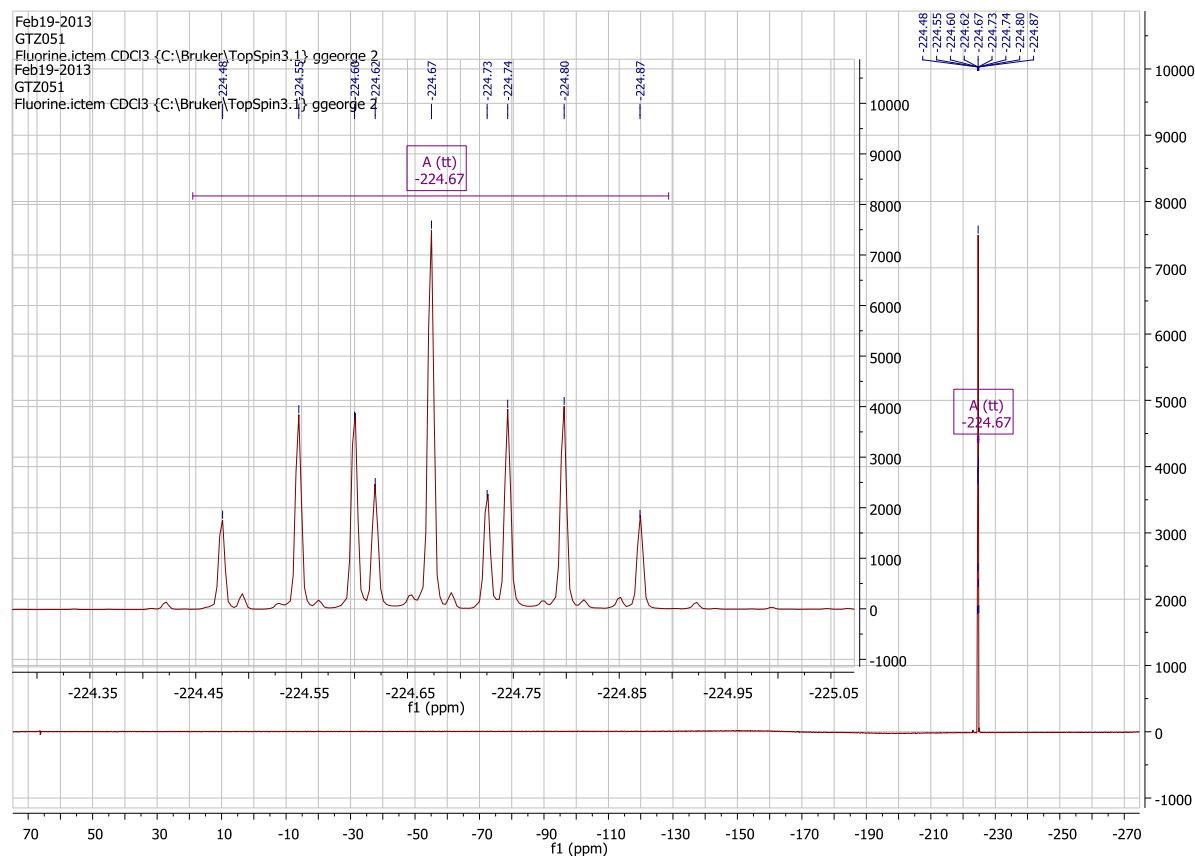
Infrared spectrum

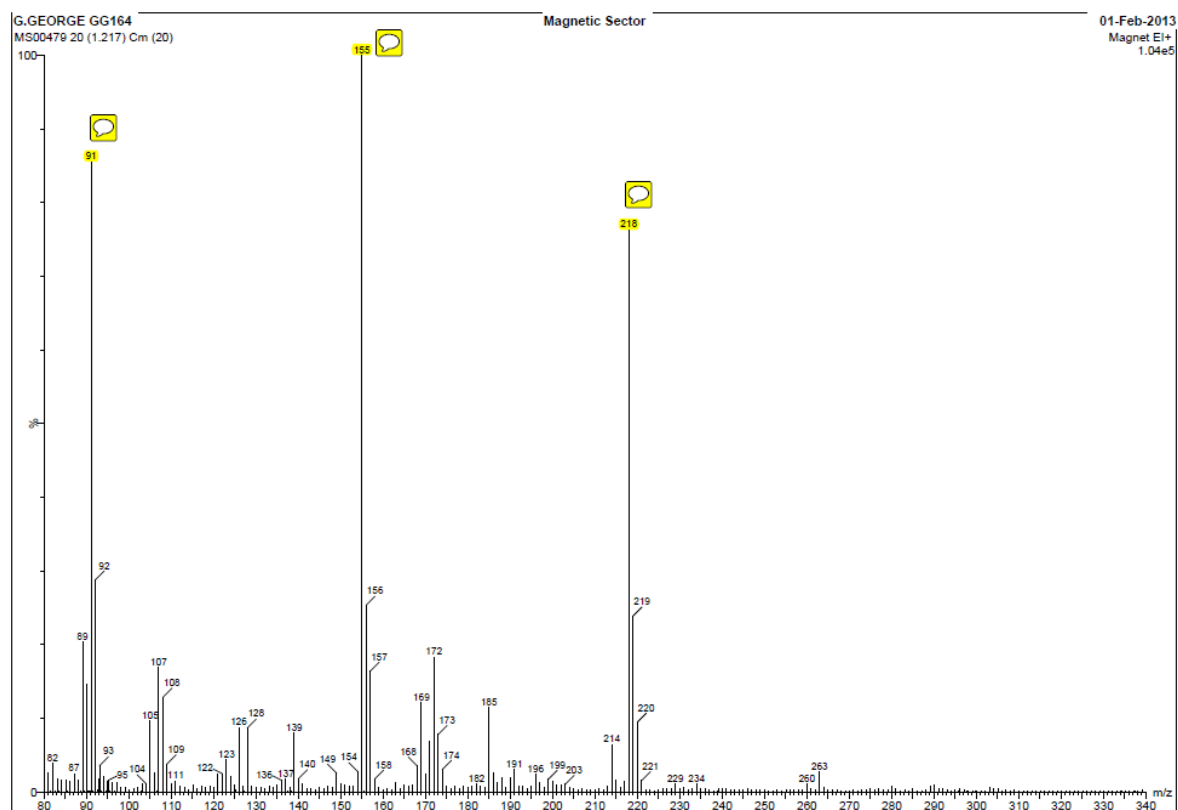


^{13}C NMR spectrum



^{19}F NMR spectrum





Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

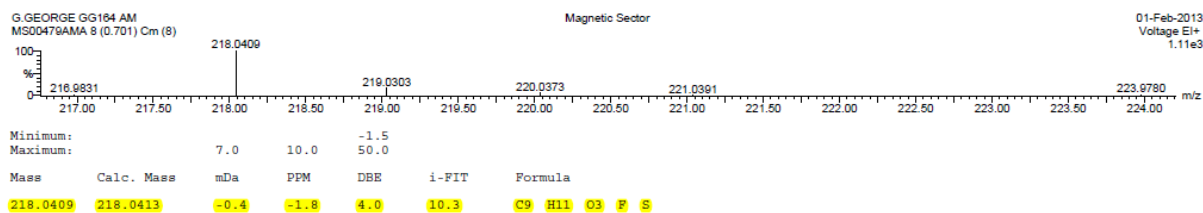
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

111 formula(e) evaluated with 1 results within limits (up to 10 closest results for each mass)

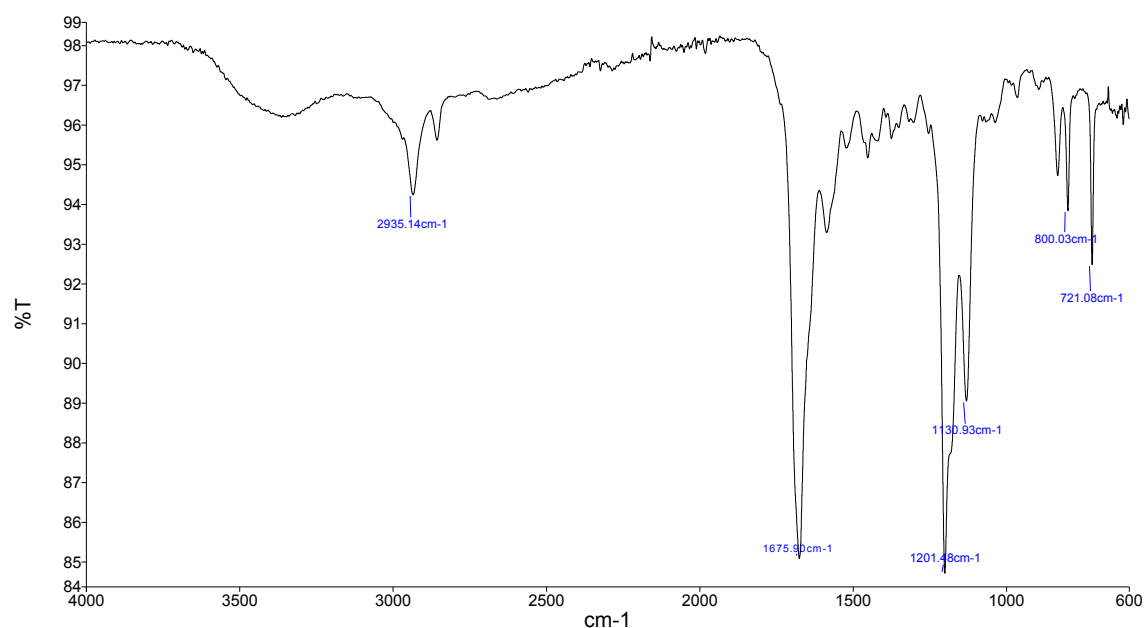
Elements Used:

C: 9-9 H: 0-500 N: 0-10 O: 0-10 F: 1-1 S: 1-1 Fe: 0-1 I: 0-2

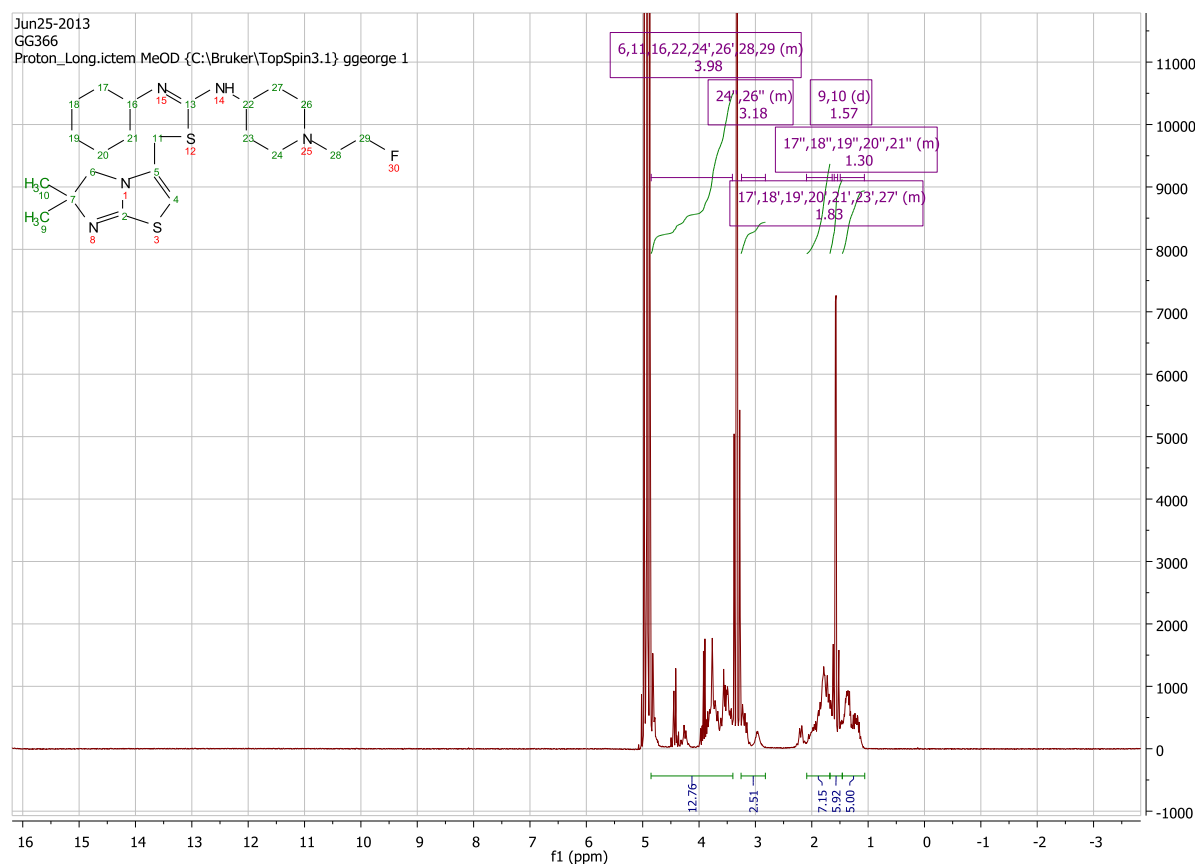


82. *N*'-Cyclohexyl-*N*-[1-(2-fluoroethyl)piperidine-4-yl][({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl}methyl)sulfanyl]methanimidamide

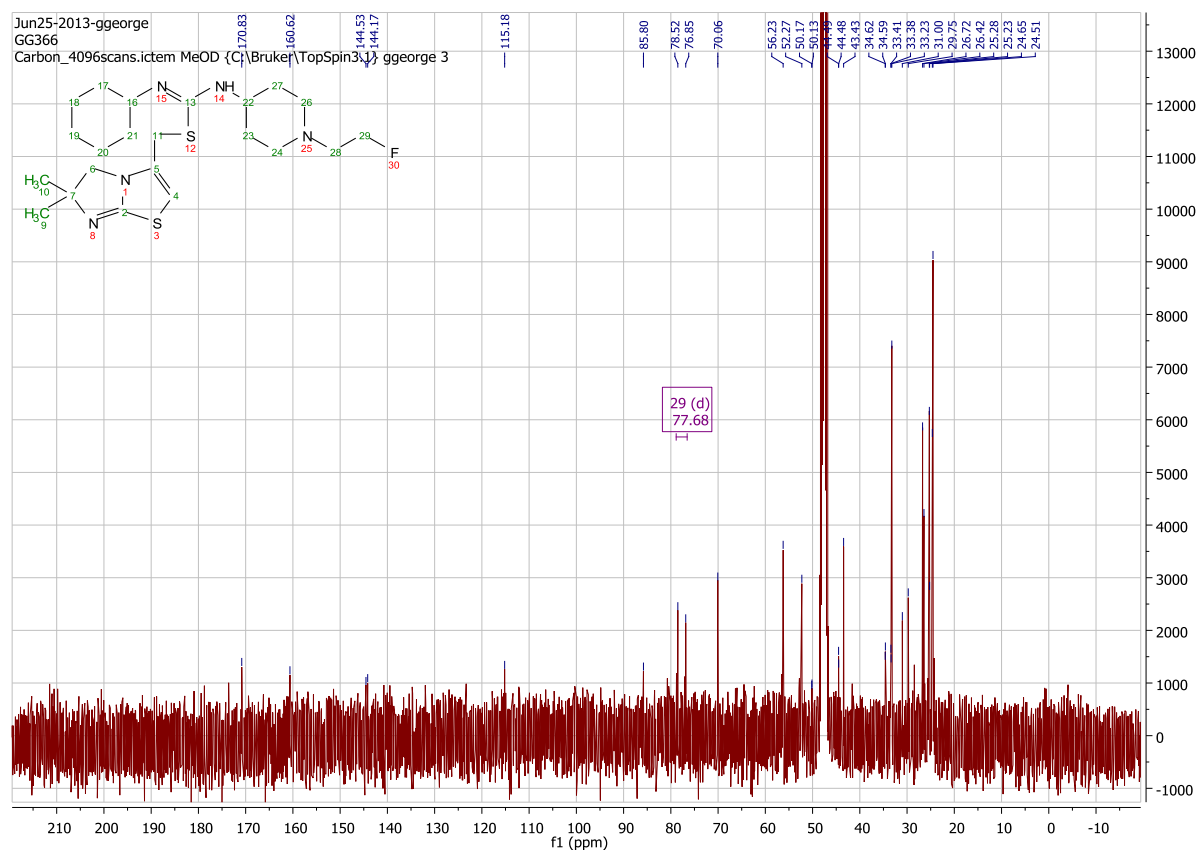
Infrared spectrum



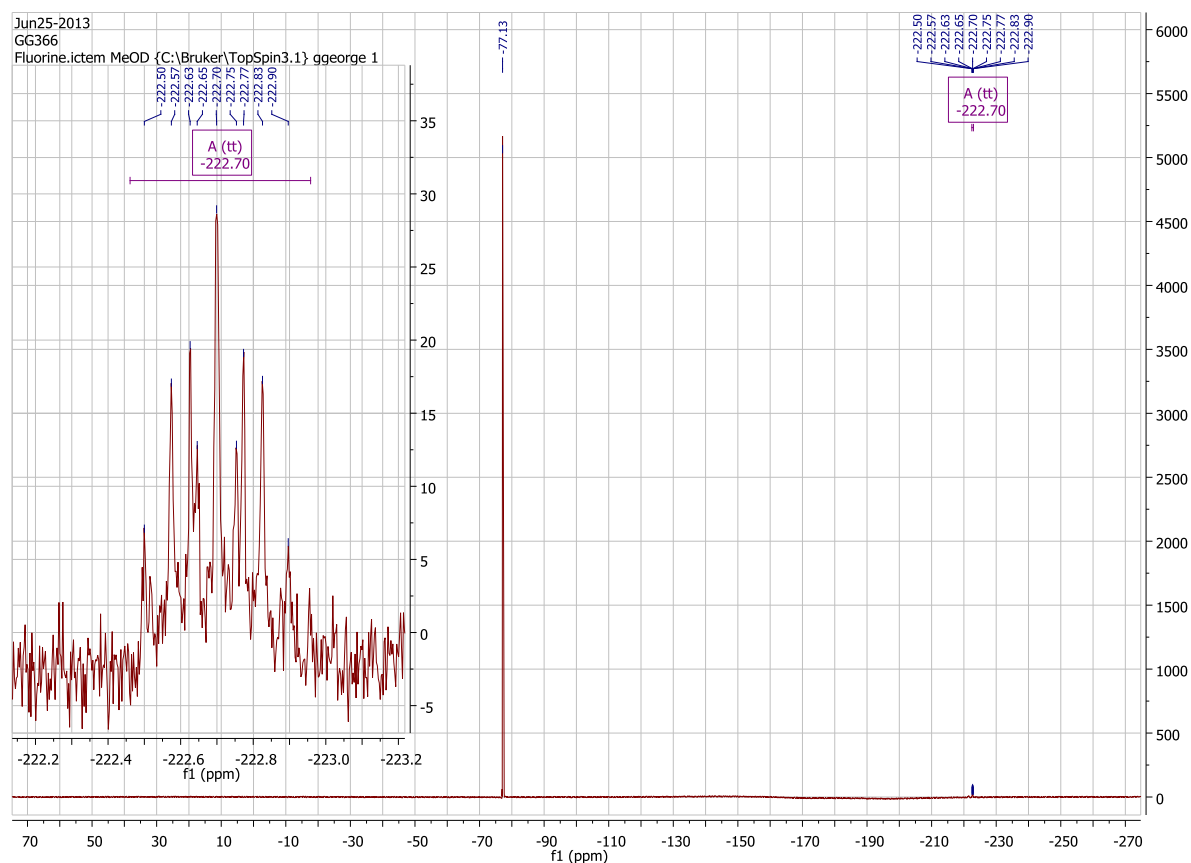
¹H NMR spectrum



^{13}C NMR spectrum

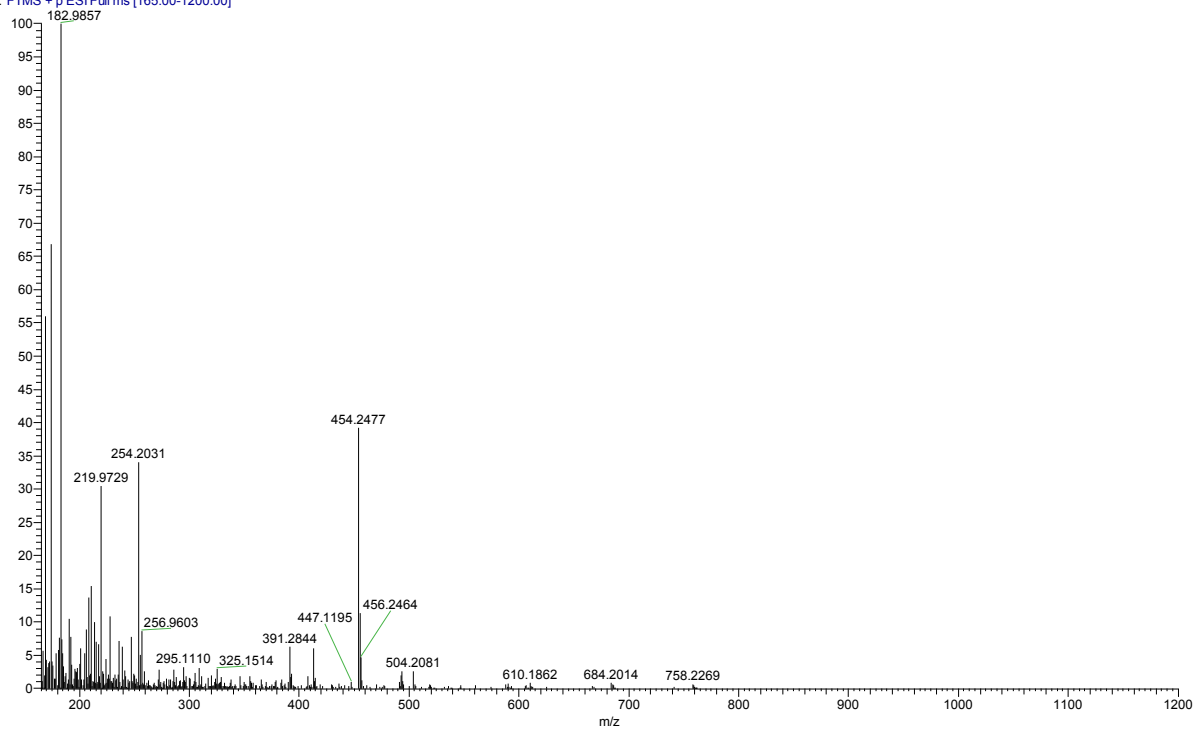


^{19}F NMR spectrum

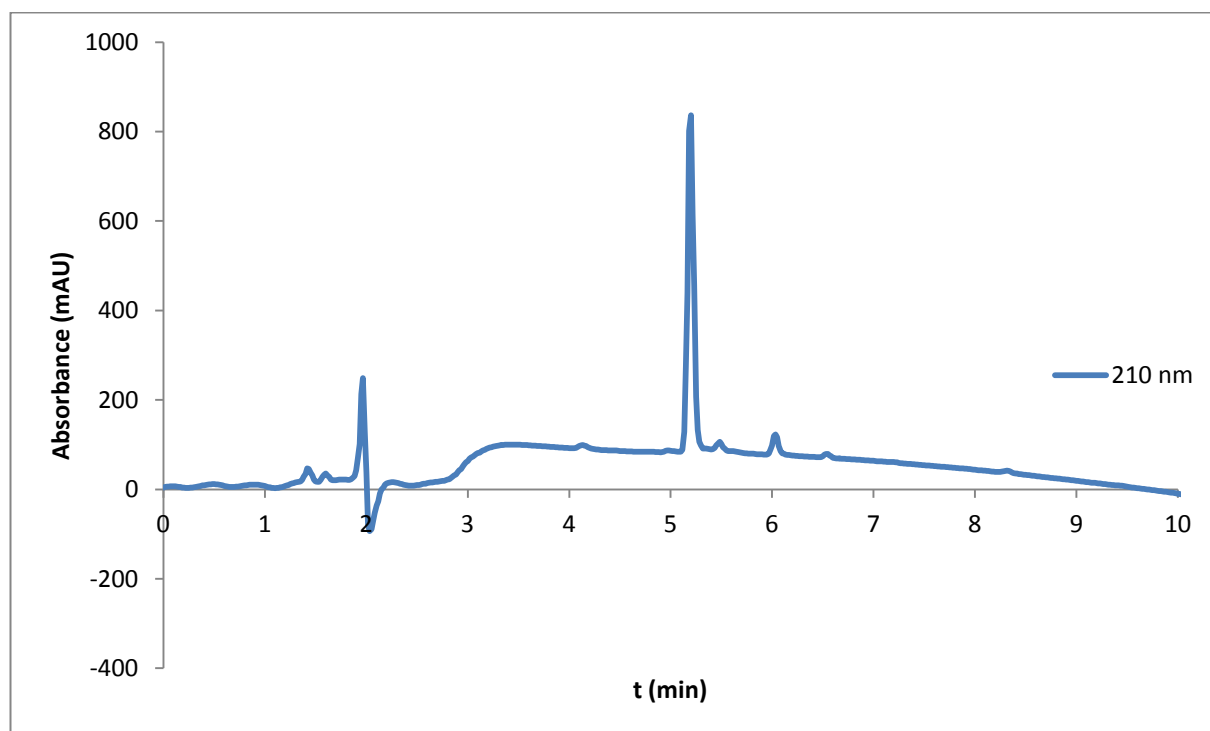


HRMS

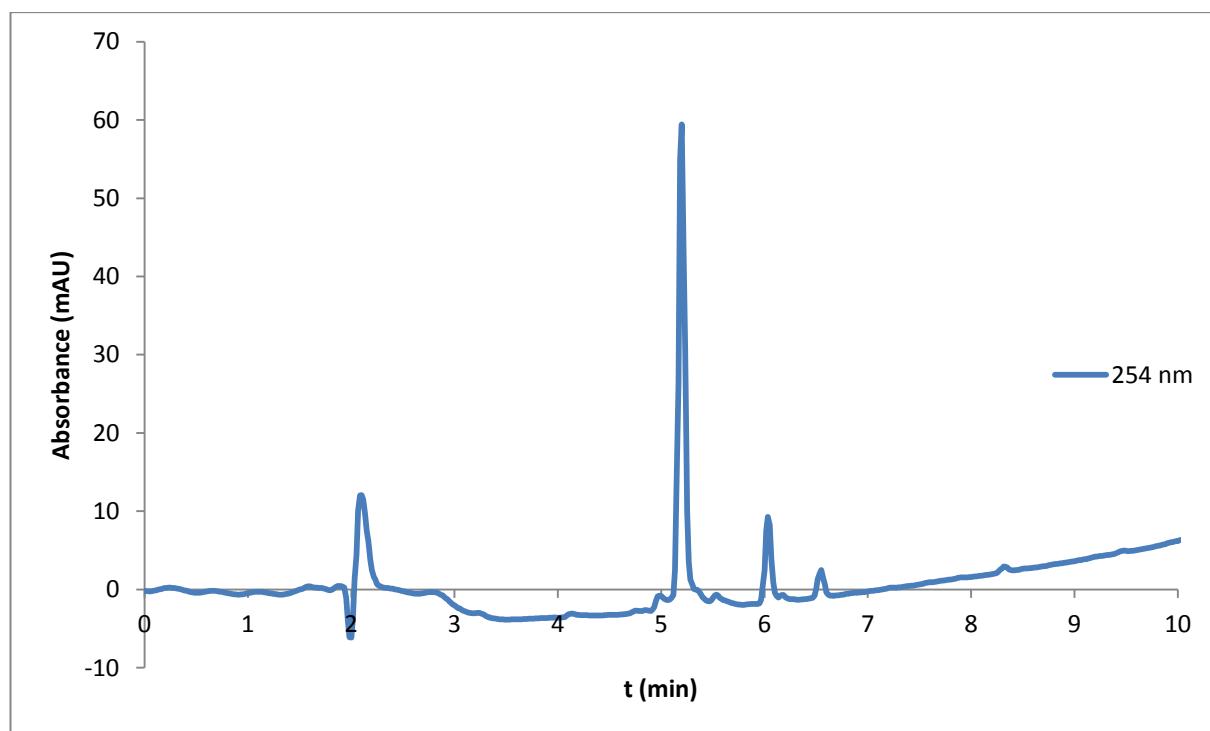
MS03874#291 RT: 1.11 AV: 1 NL: 1.22E8
T: FTMS +pESI Full ms [165.00-1200.00]



HPLC trace at 210 nm

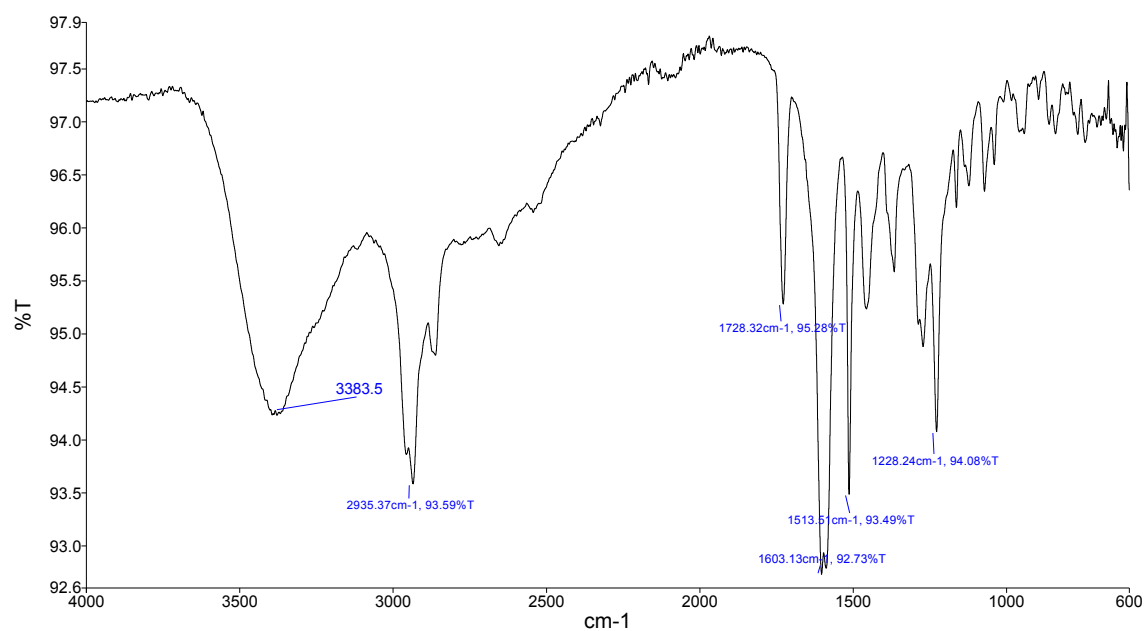


HPLC trace at 254 nm

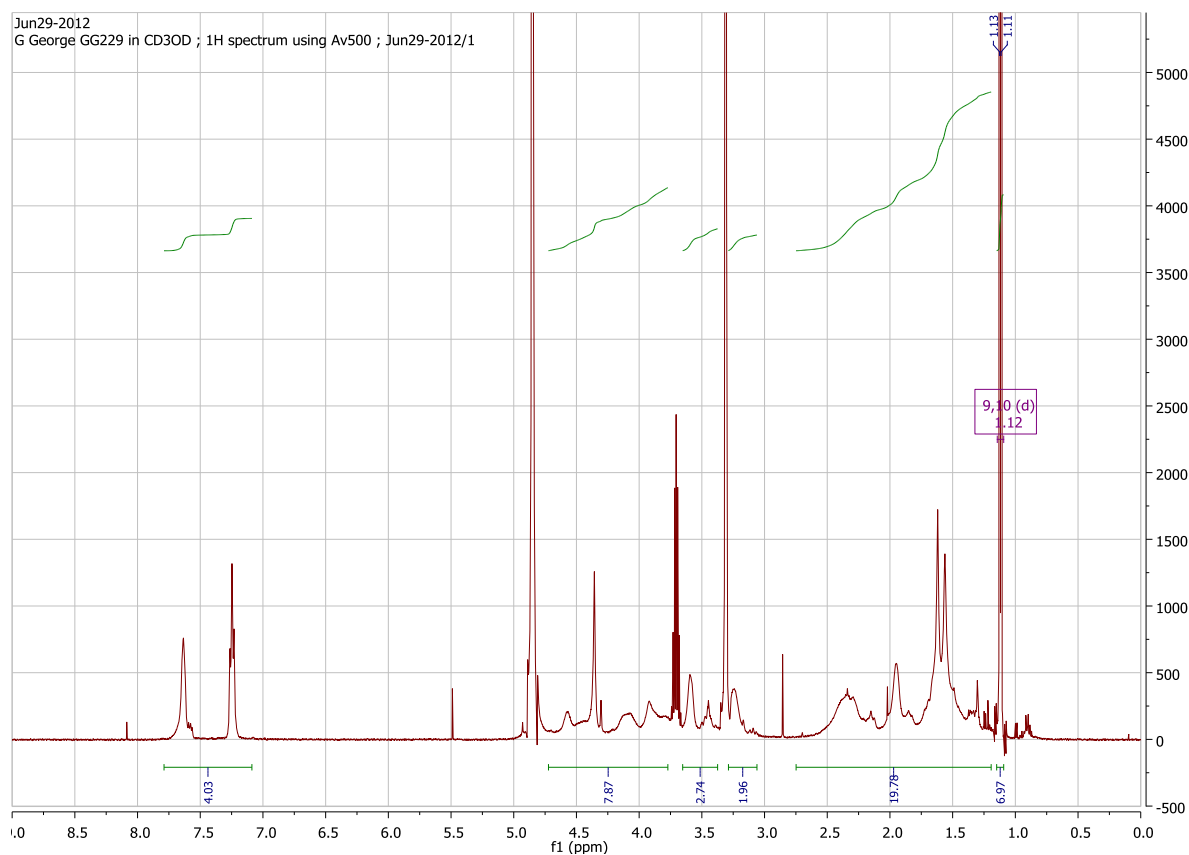


83. *N*'-Cyclohexyl-*N*-{1-[(4-fluorophenyl)methyl]piperidin-4-yl}[(6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidamide trihydrochloride

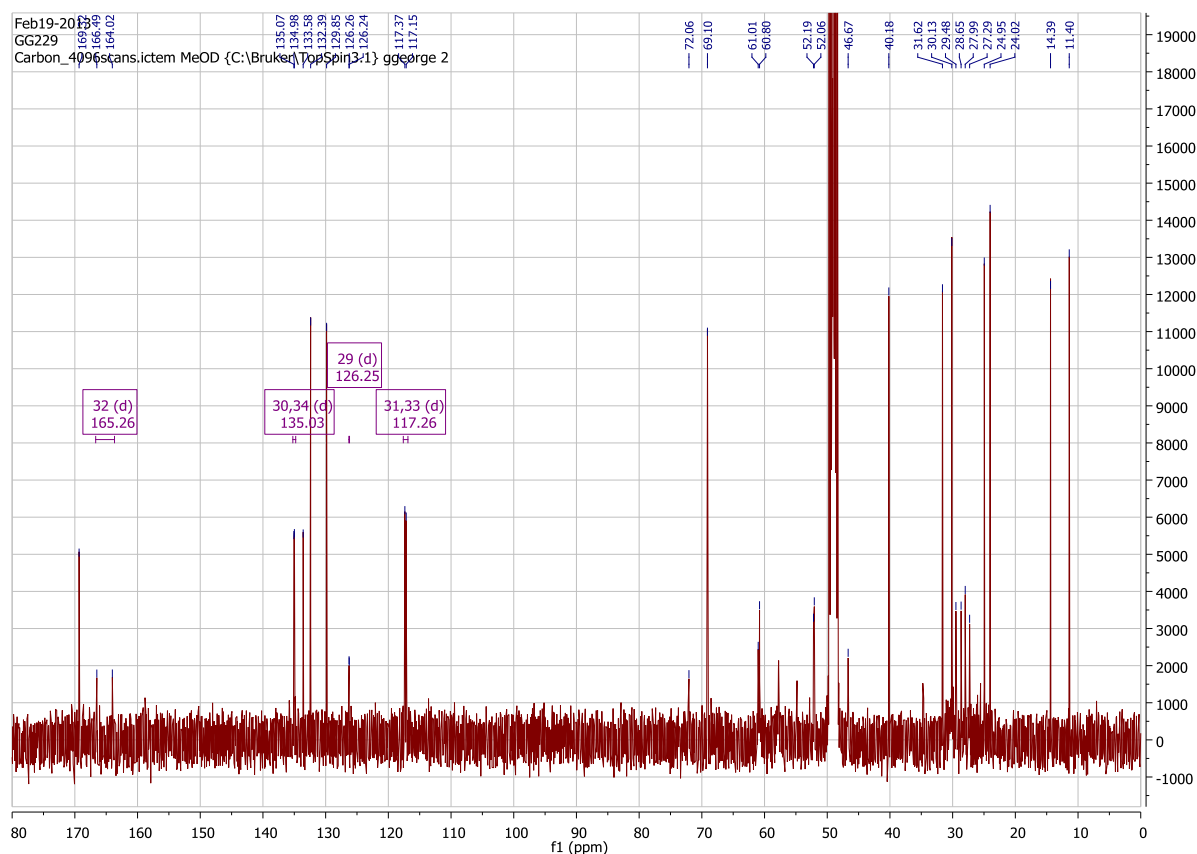
Infrared spectrum



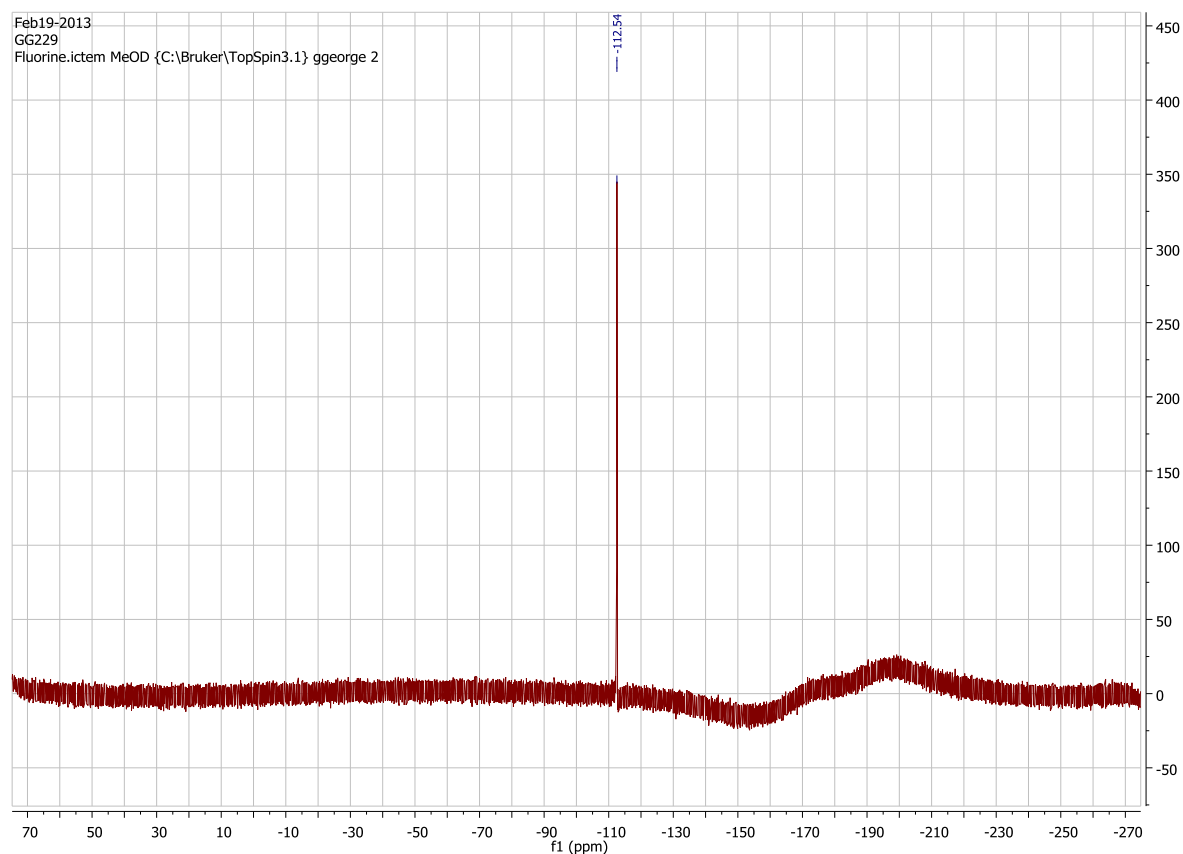
¹H NMR spectrum



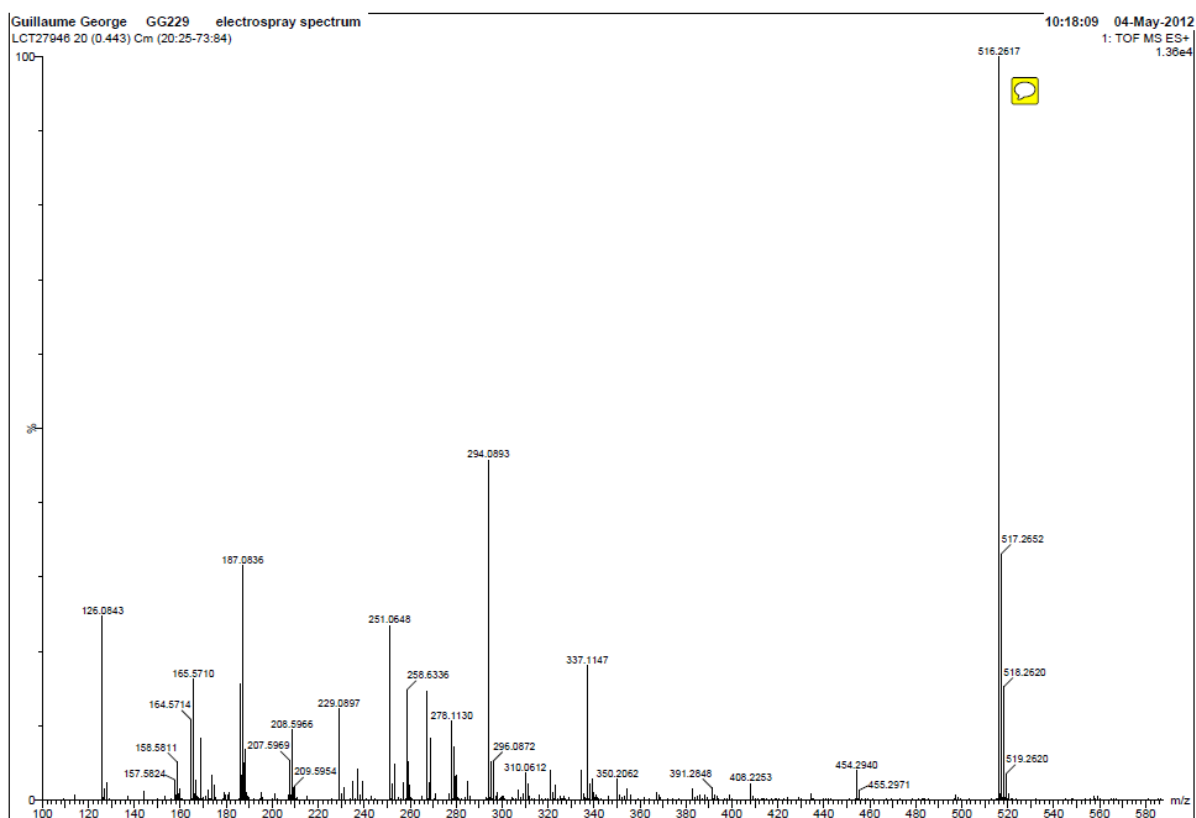
^{13}C NMR spectrum



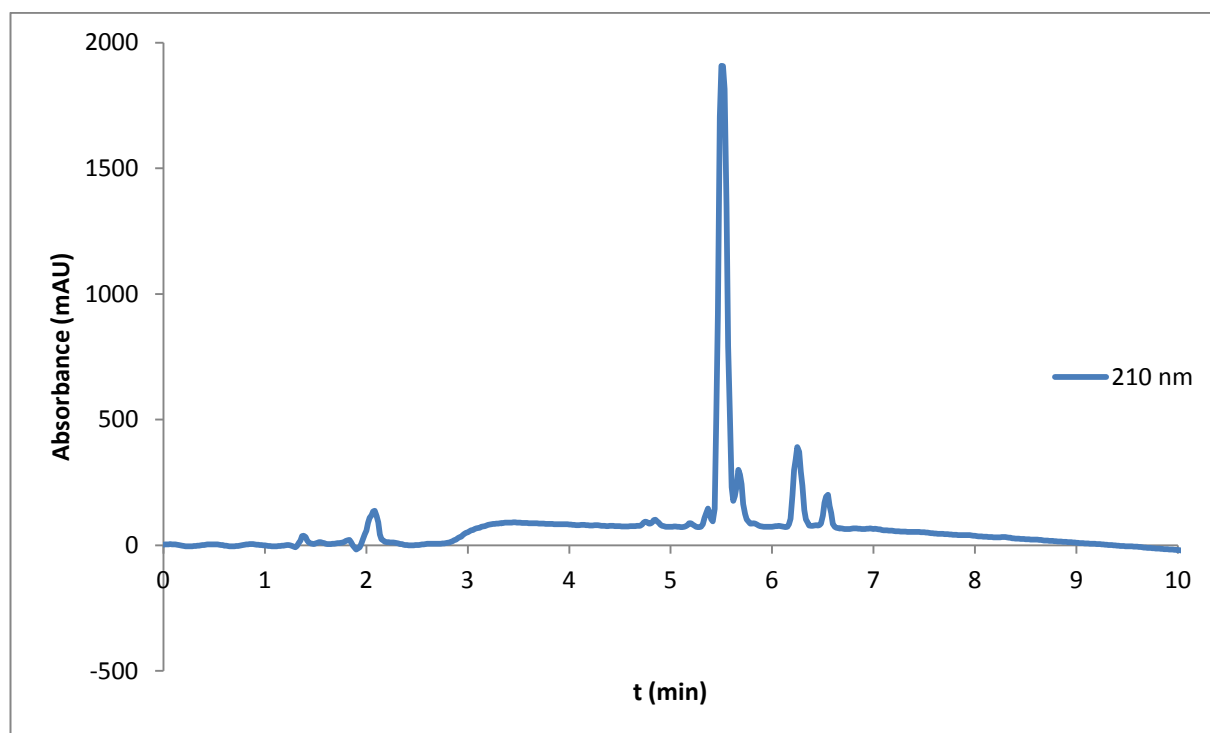
^{19}F NMR spectrum



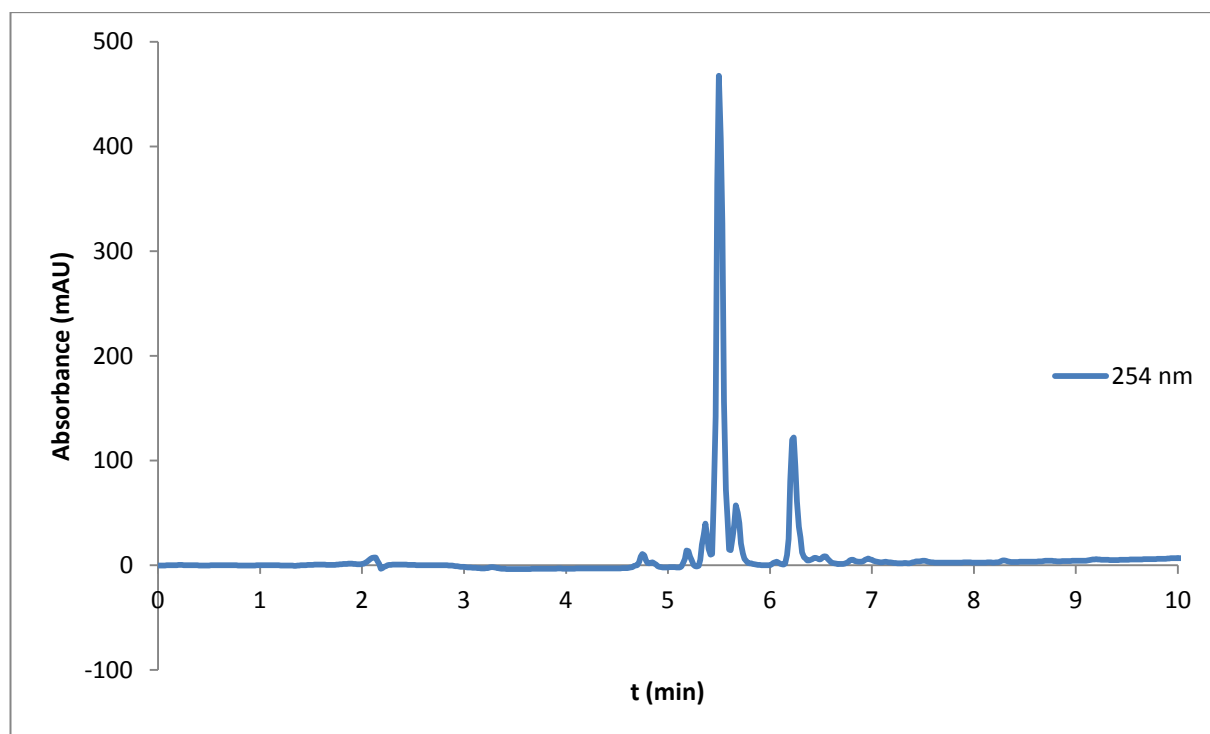
HRMS



HPLC trace at 210 nm

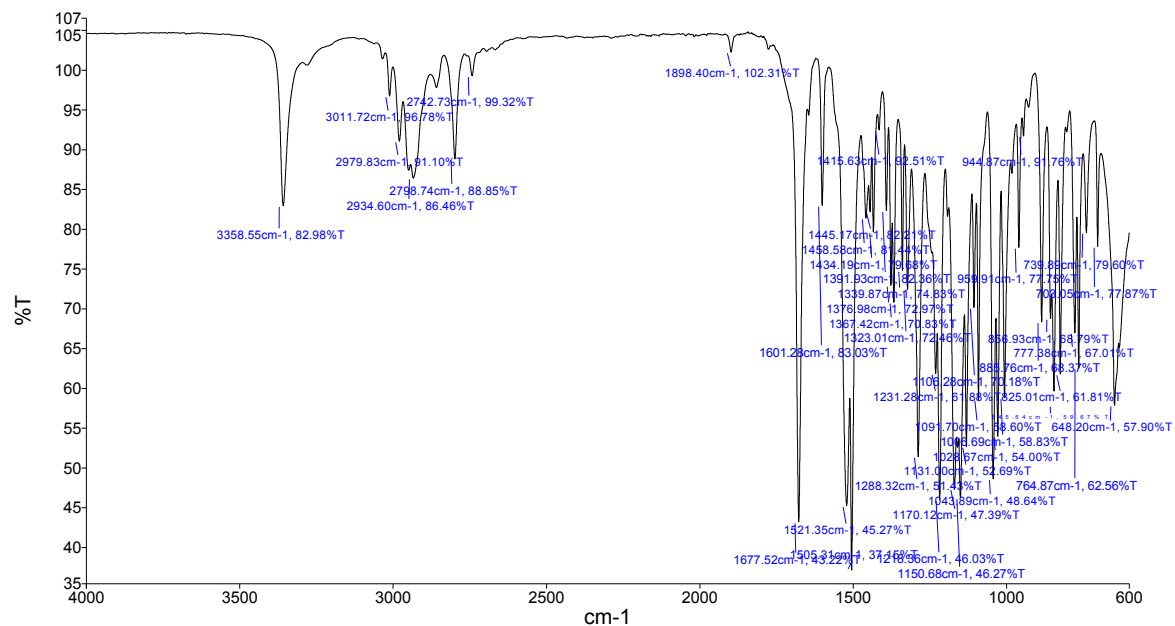


HPLC trace at 254 nm

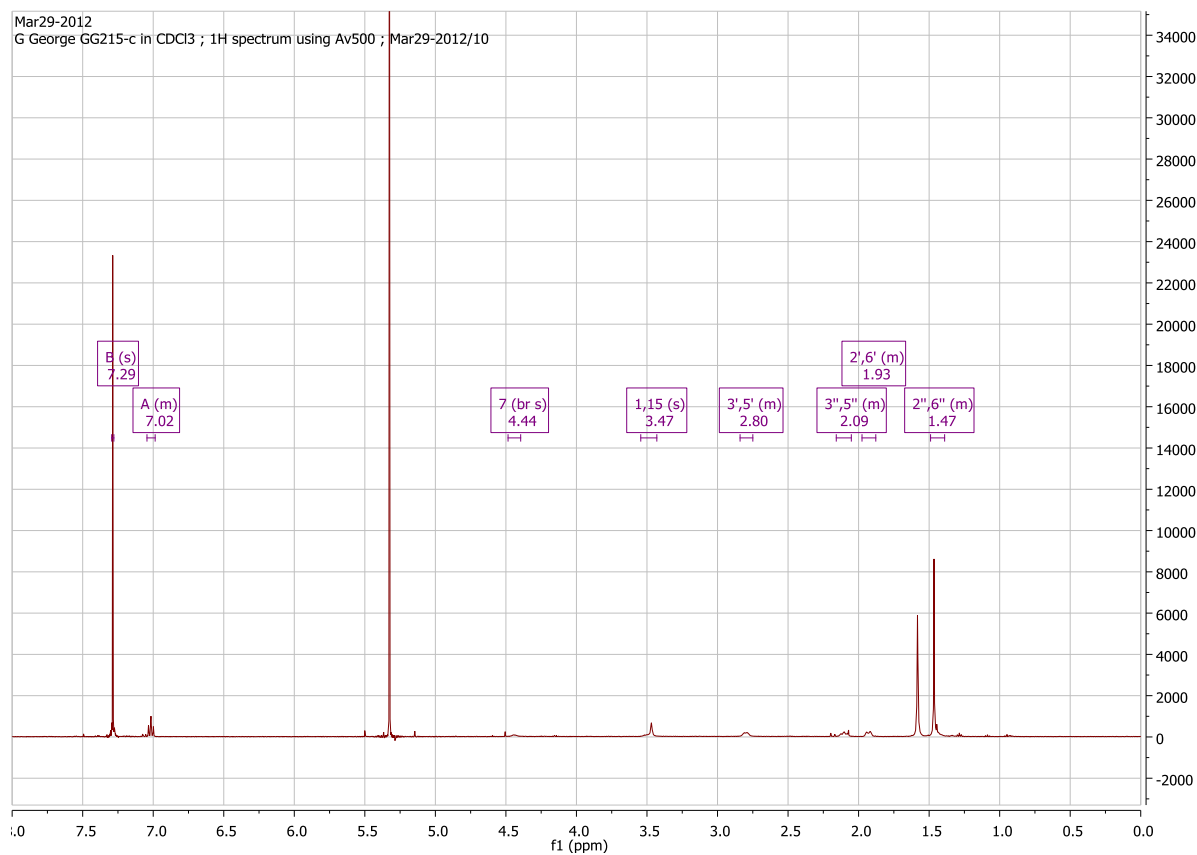


86. *tert*-Butyl *N*-{1-[(4-fluorophenyl)methyl]piperidine-4-yl}carbamate

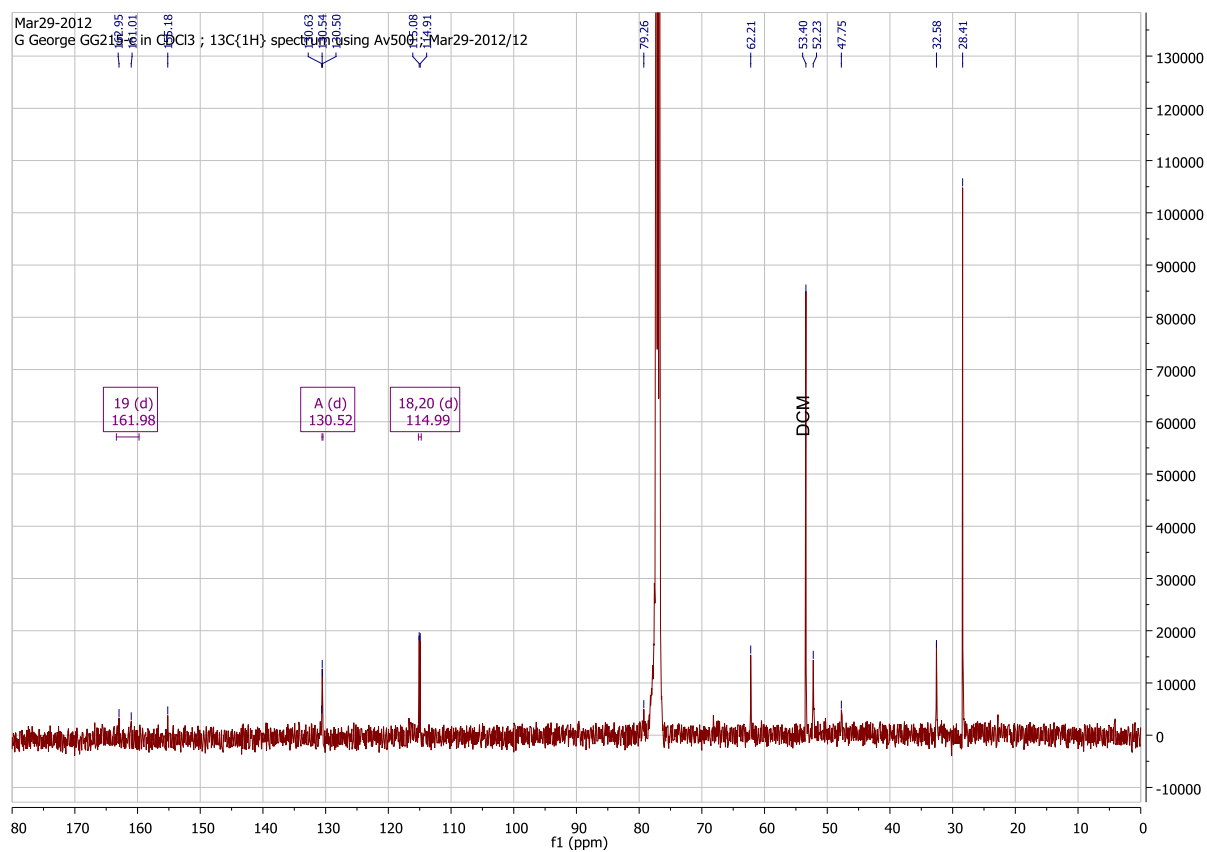
Infrared spectrum



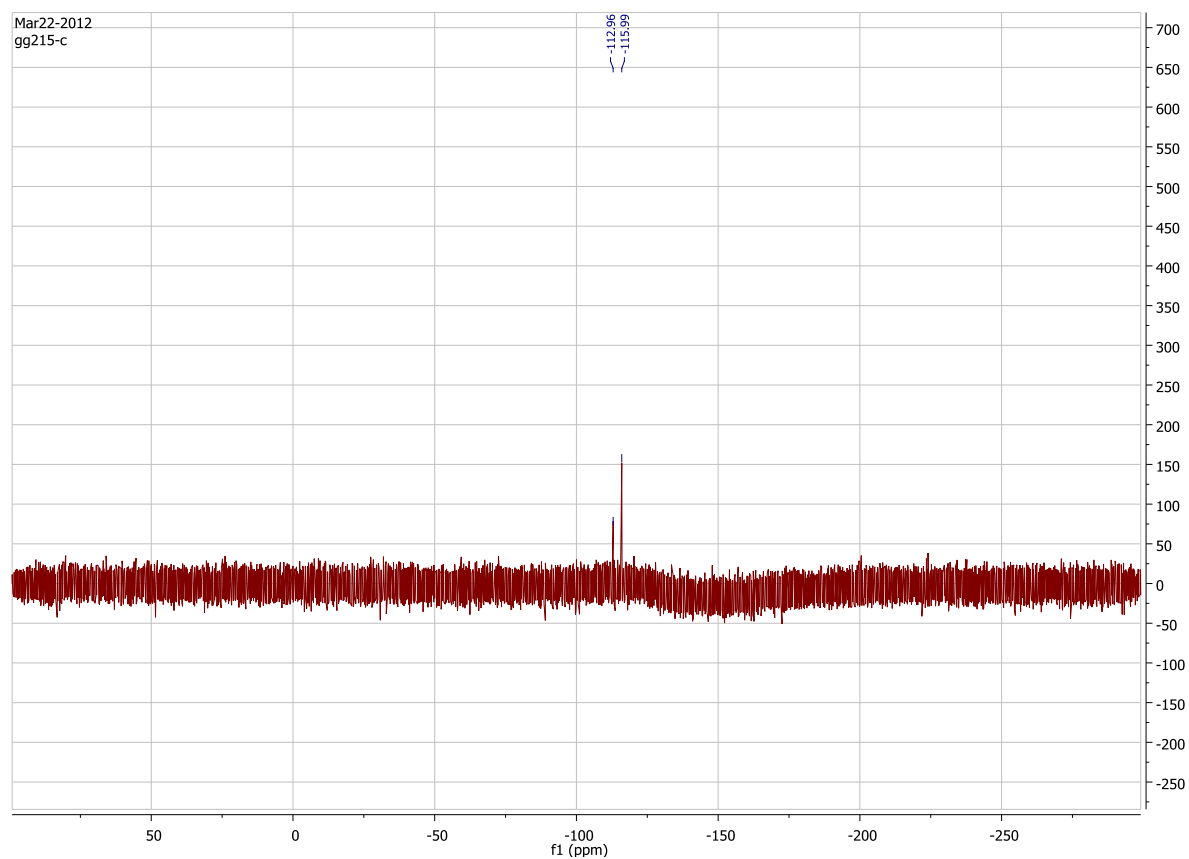
¹H NMR spectrum

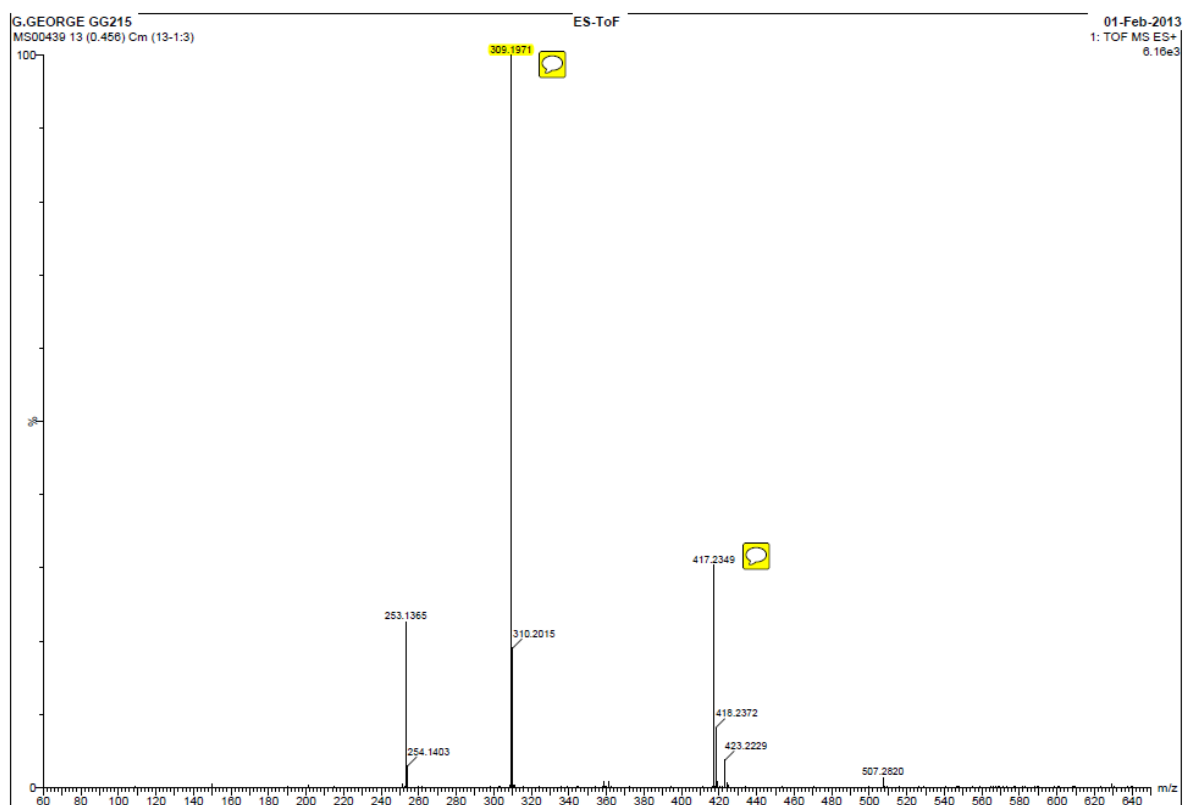


^{13}C NMR spectrum



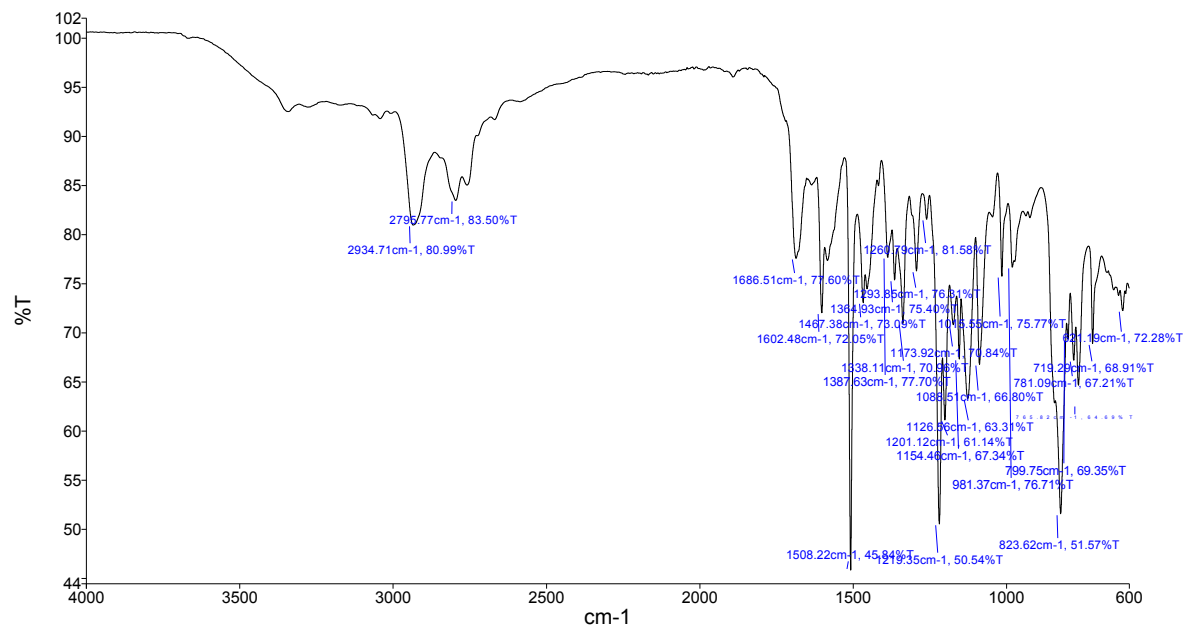
^{19}F NMR spectrum



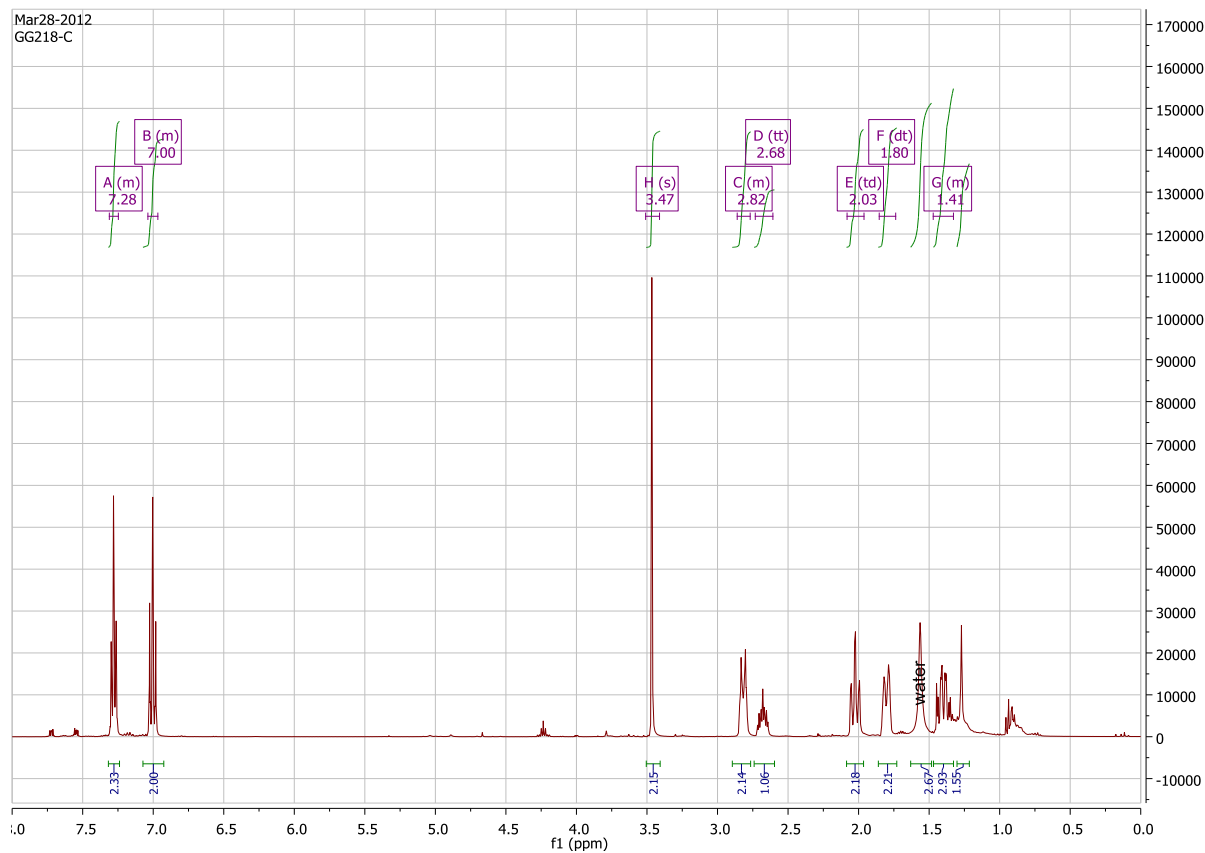


87. 1-[(4-Fluorophenyl)methyl]piperidin-4-amine

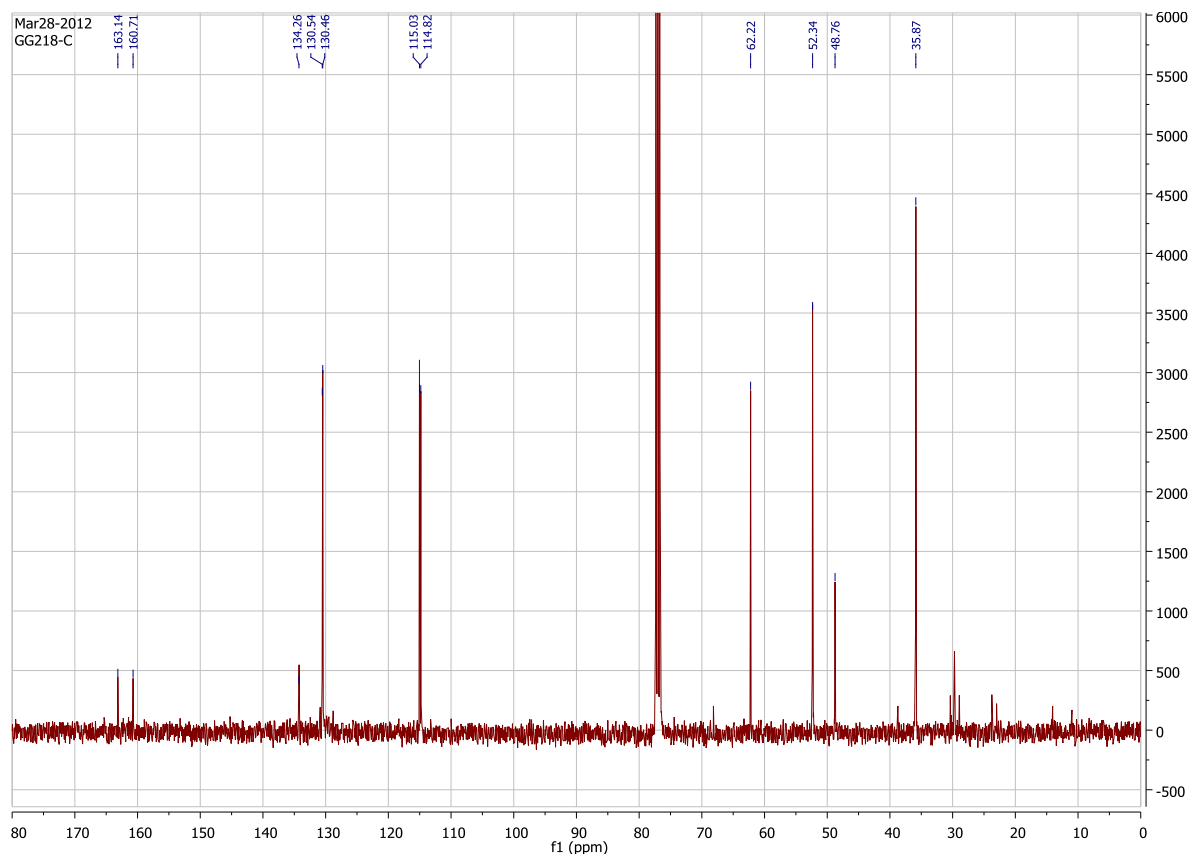
Infrared spectrum



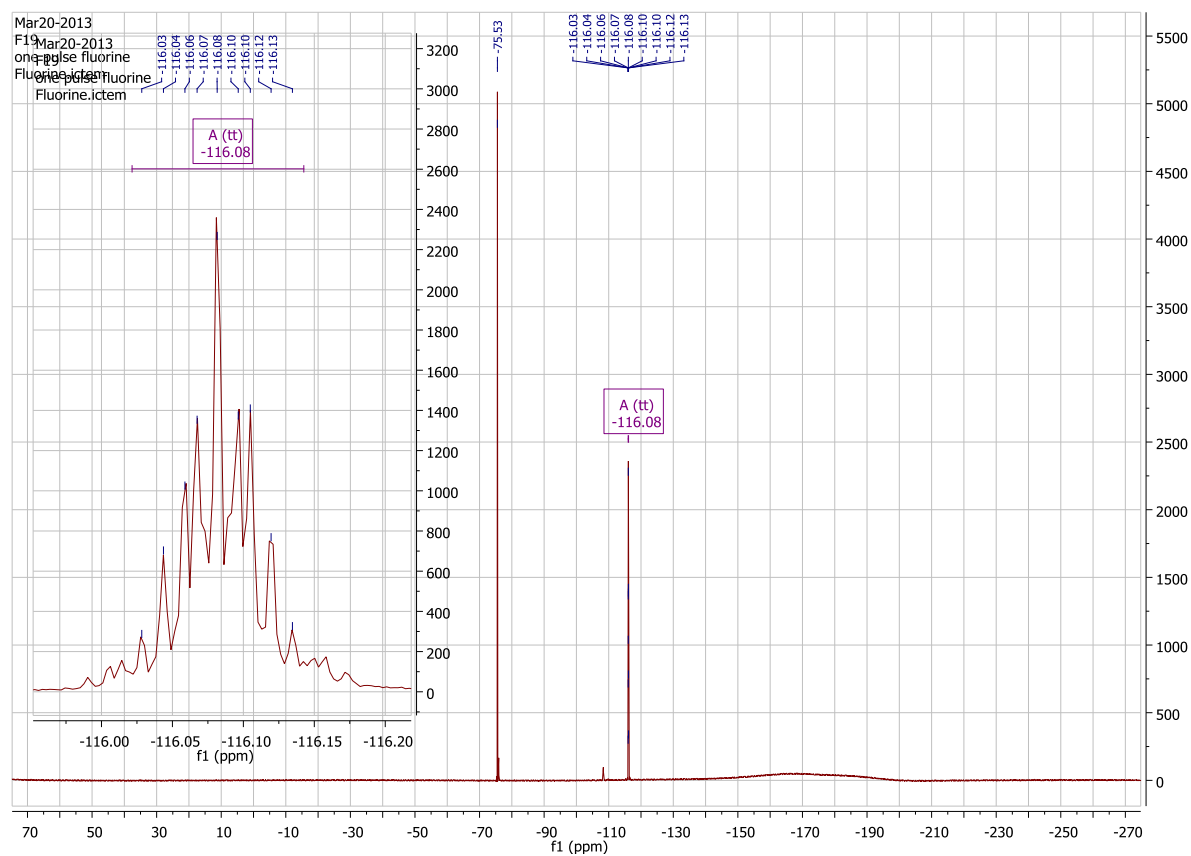
^1H NMR spectrum

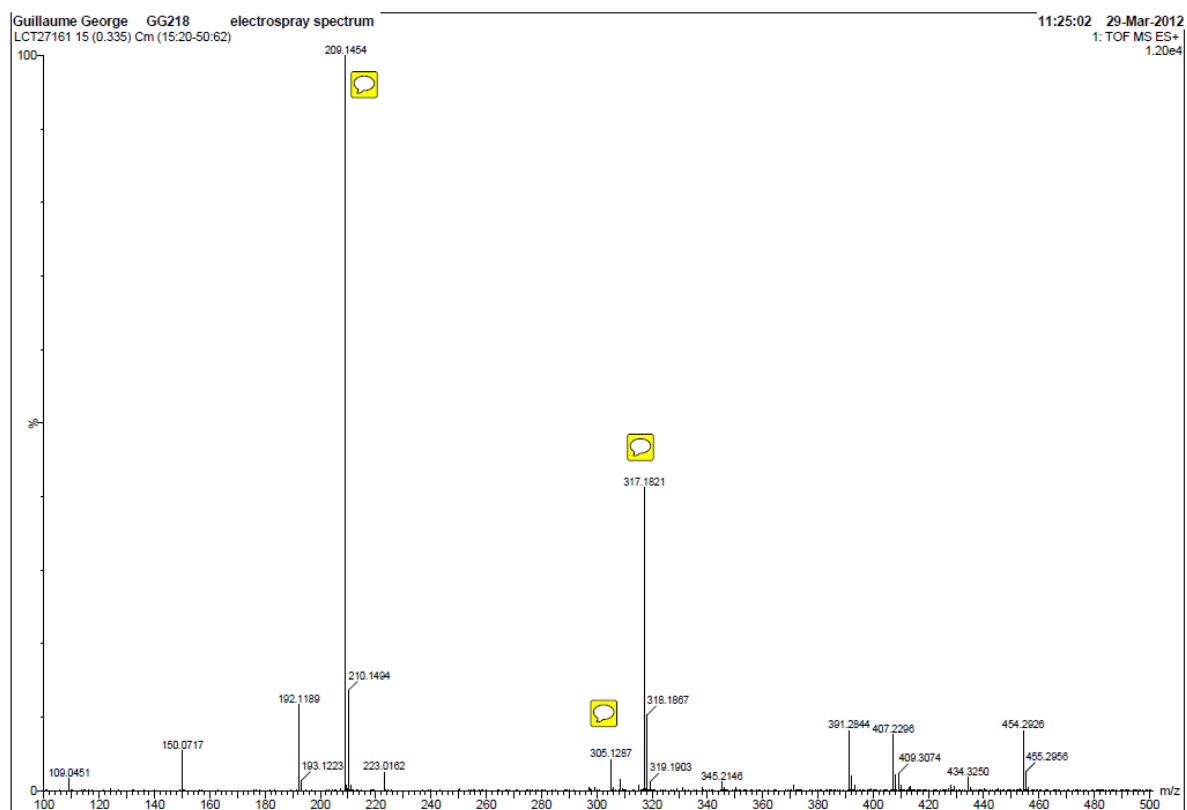


^{13}C NMR spectrum



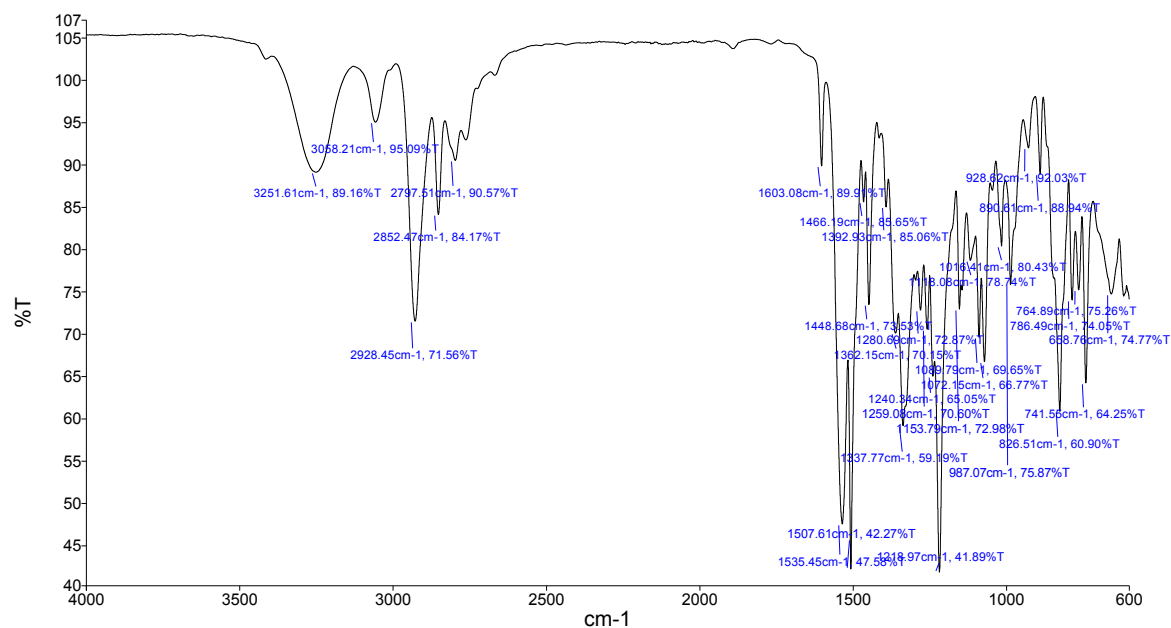
^{19}F NMR spectrum



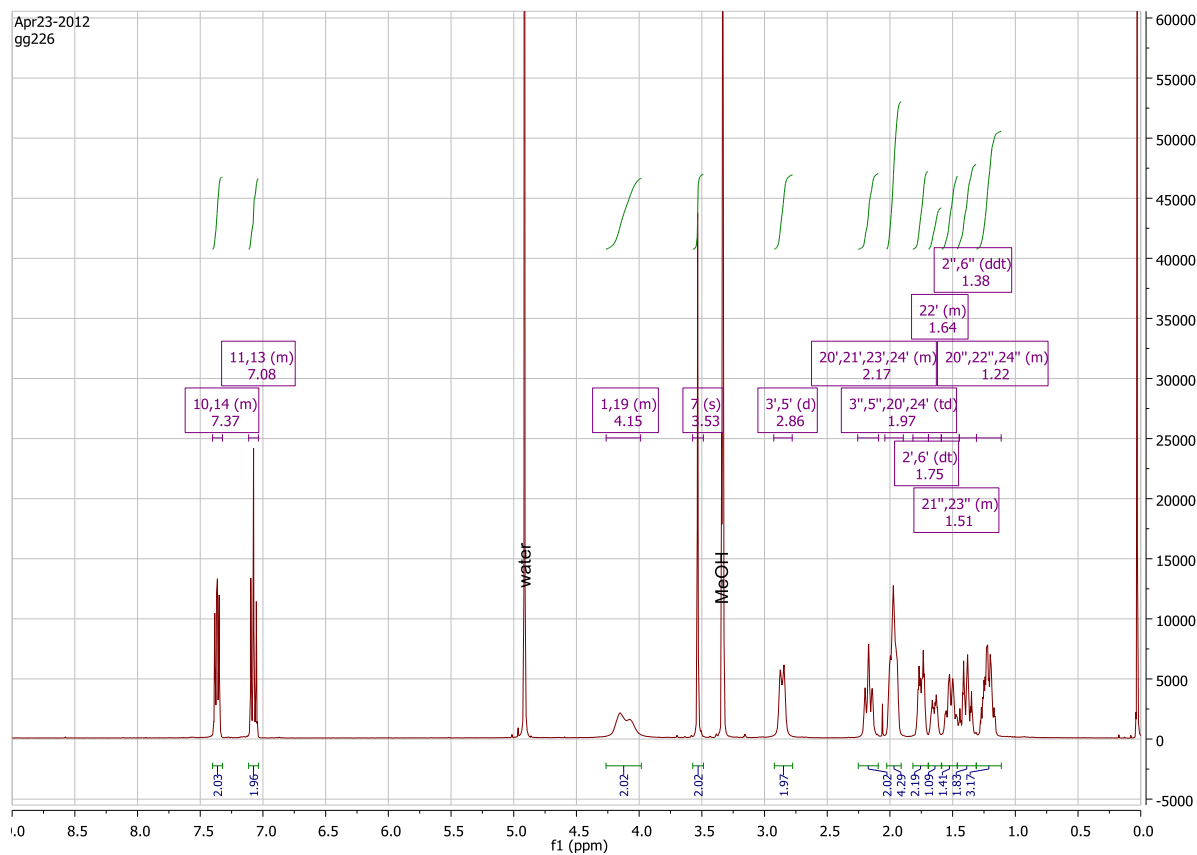


88. 1-(Cyclohexyl)-3-{1-[(4-fluorophenyl)methyl]piperidin-4-yl}thiourea

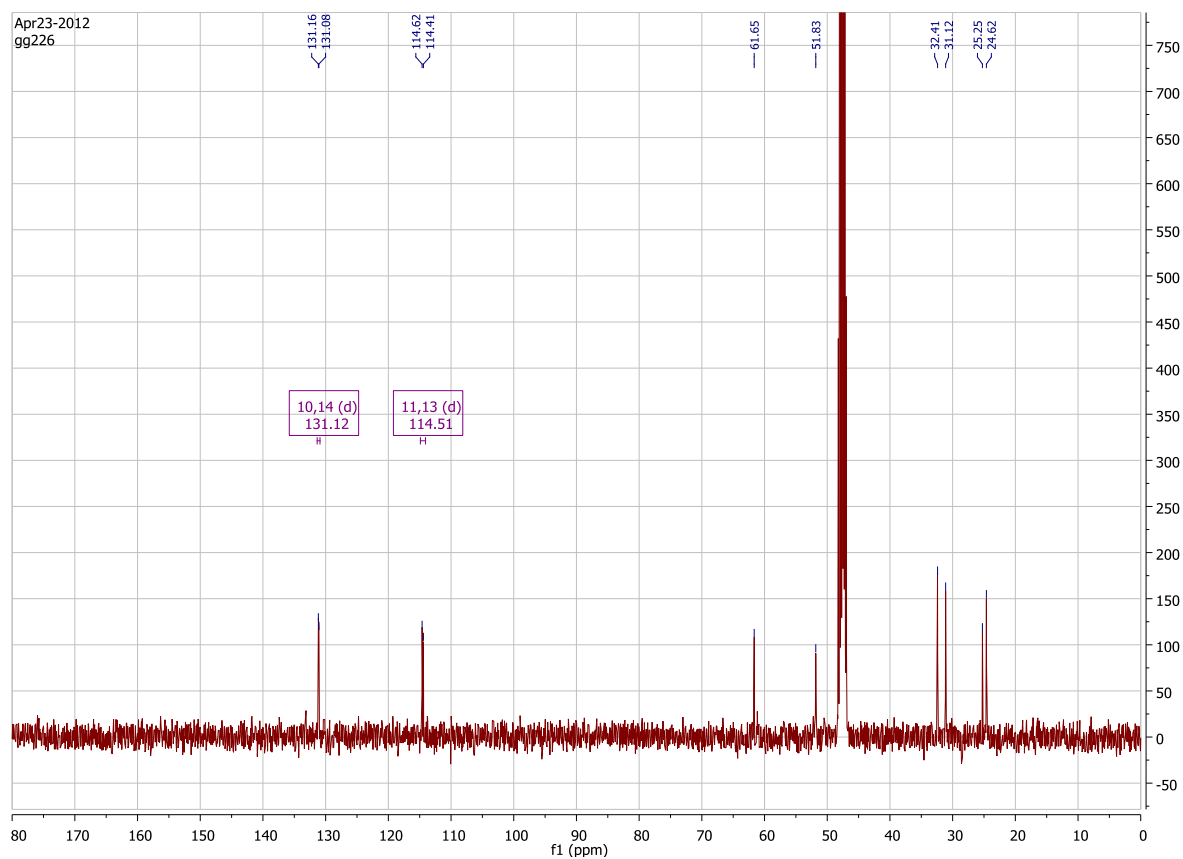
Infrared spectrum



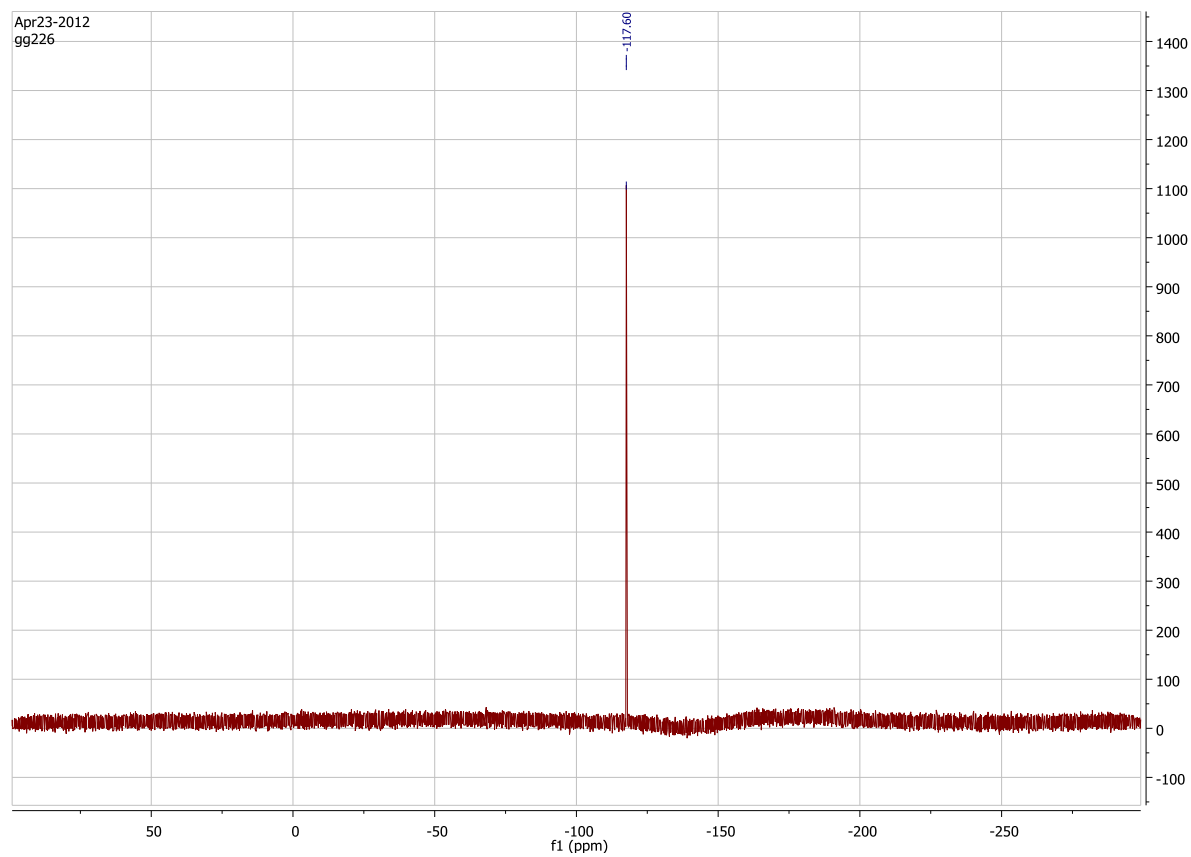
¹H NMR spectrum

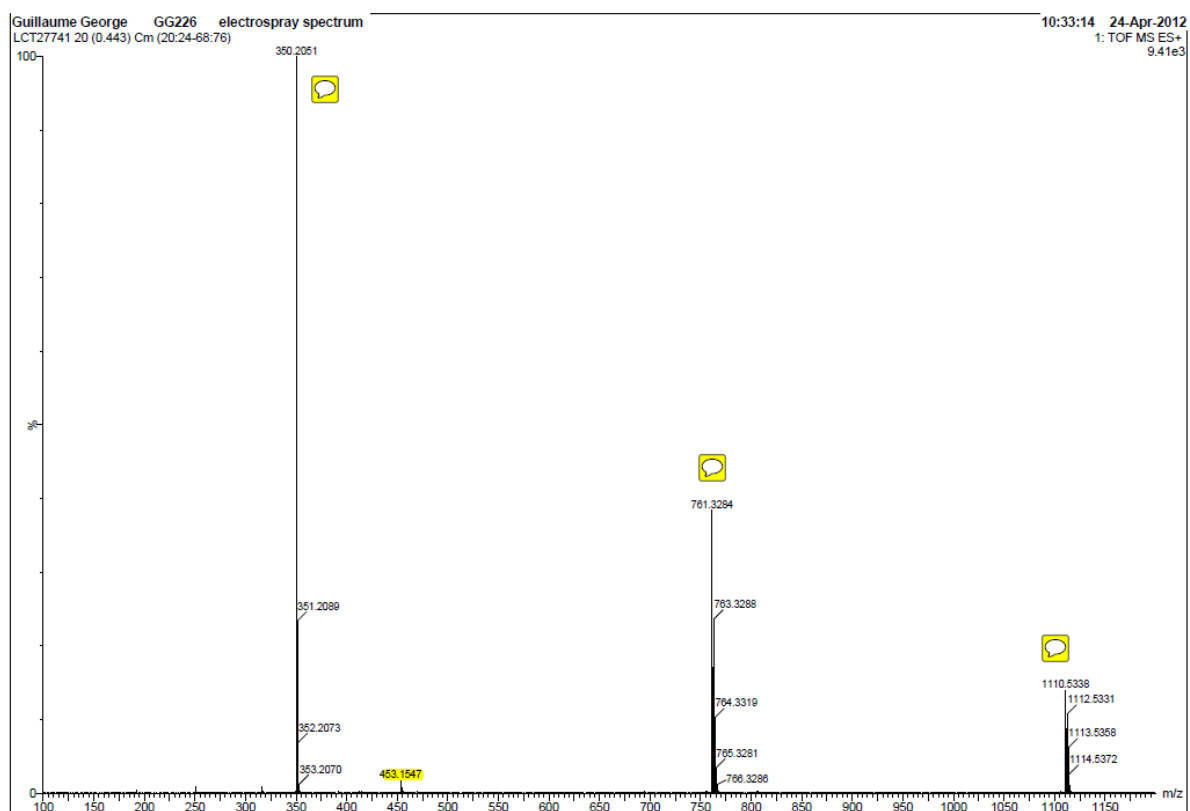


^{13}C NMR spectrum



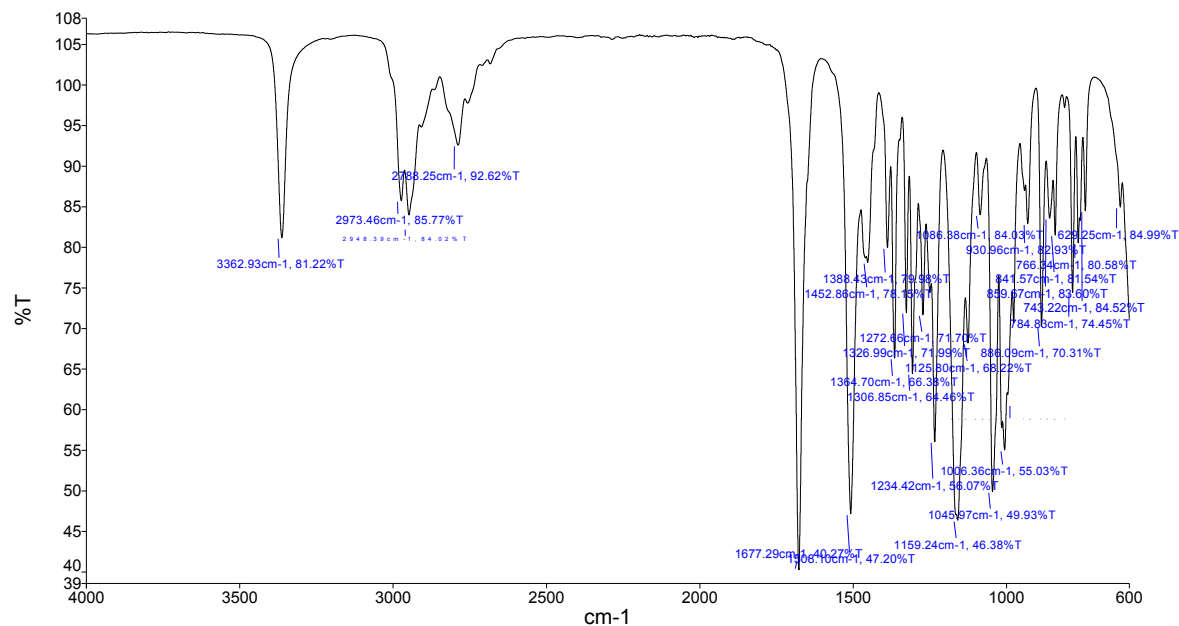
^{19}F NMR spectrum



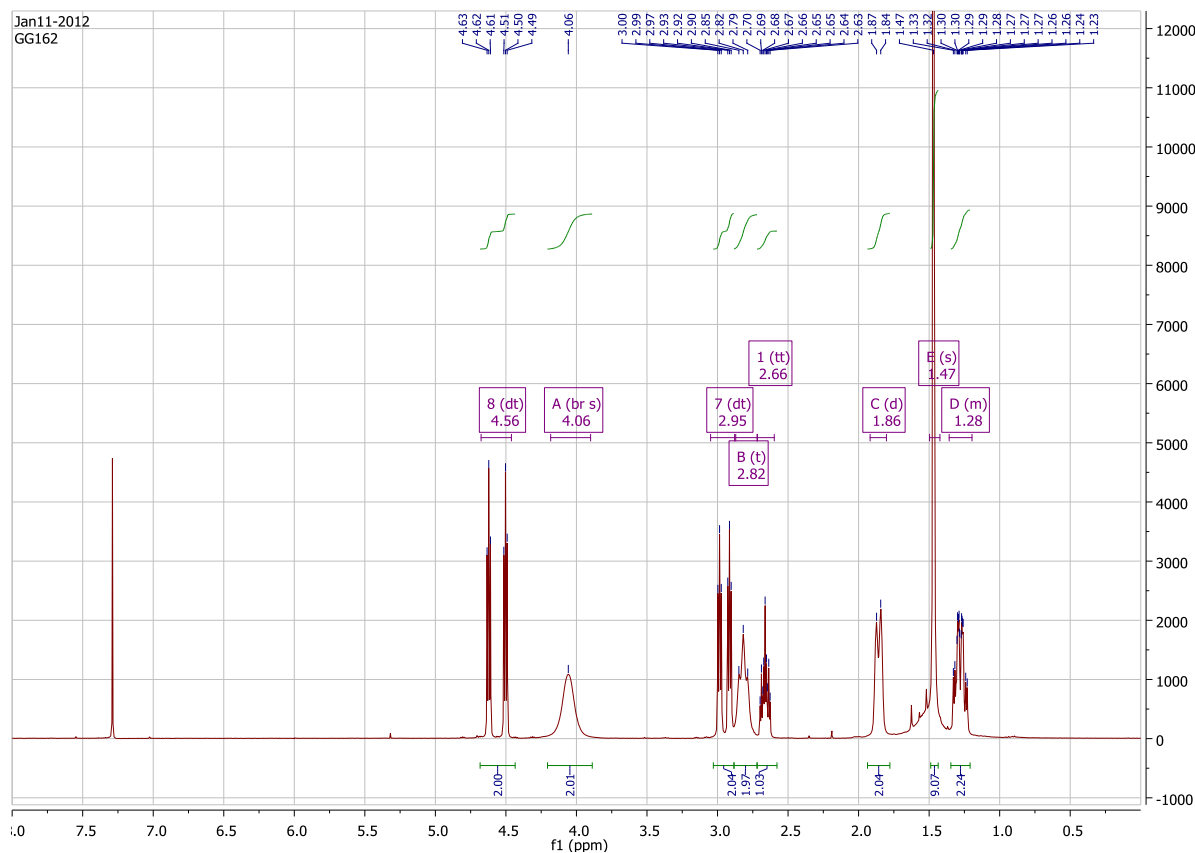


89. *tert*-Butyl-*N*-[1-(2-fluoroethyl)piperidine-4-yl]carbamate

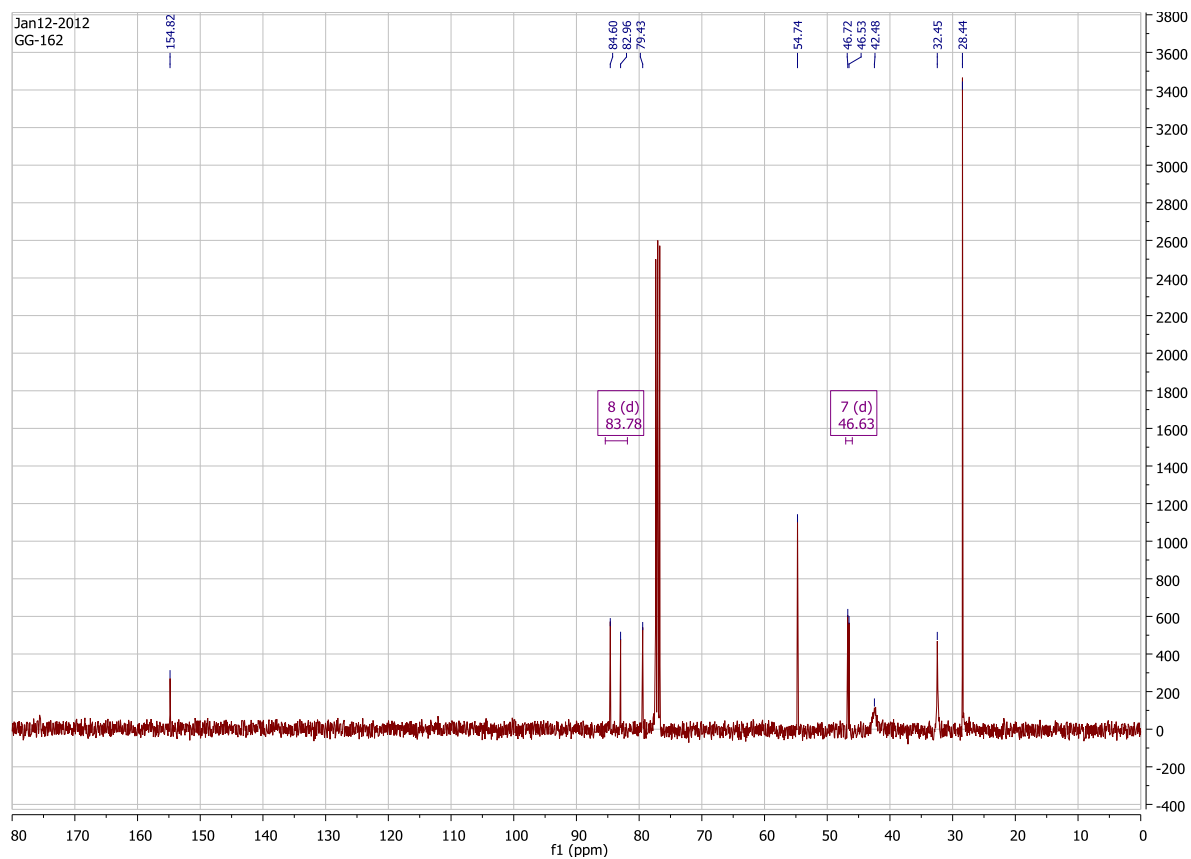
Infrared spectrum



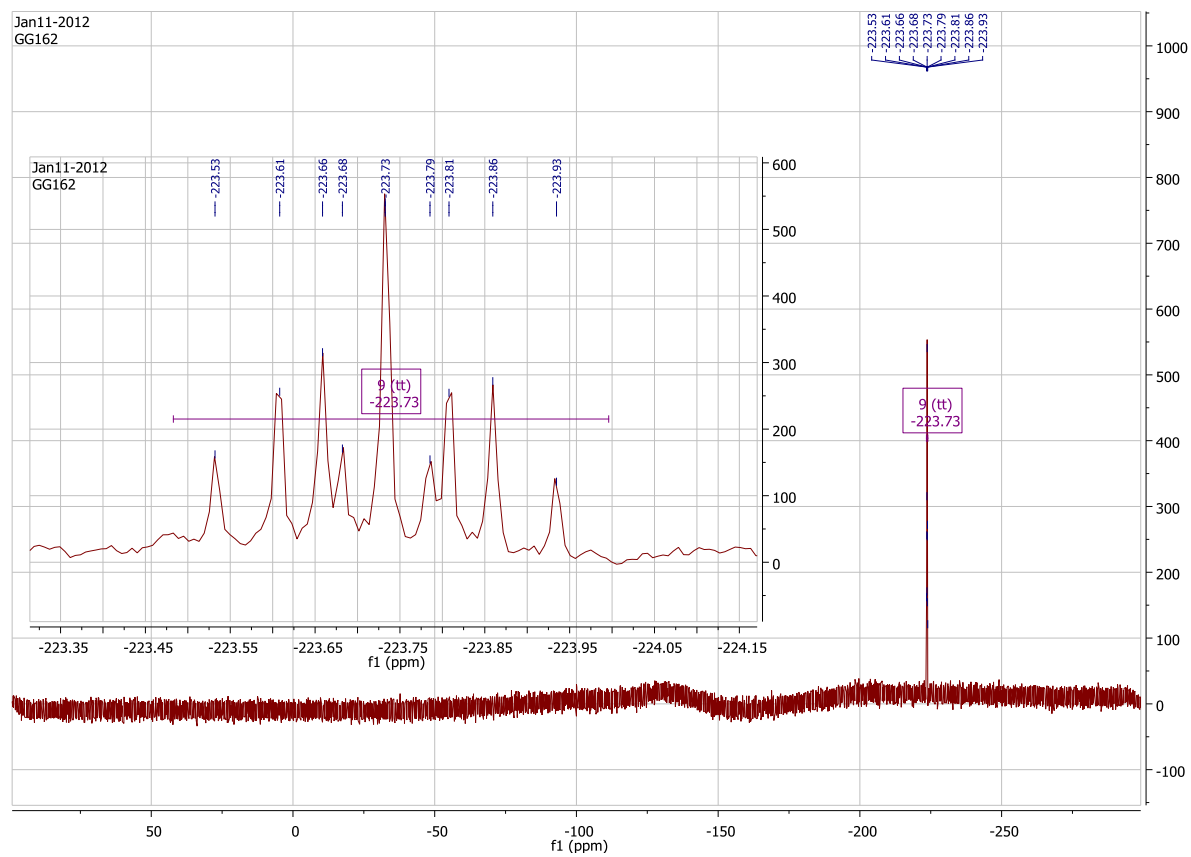
¹H NMR spectrum

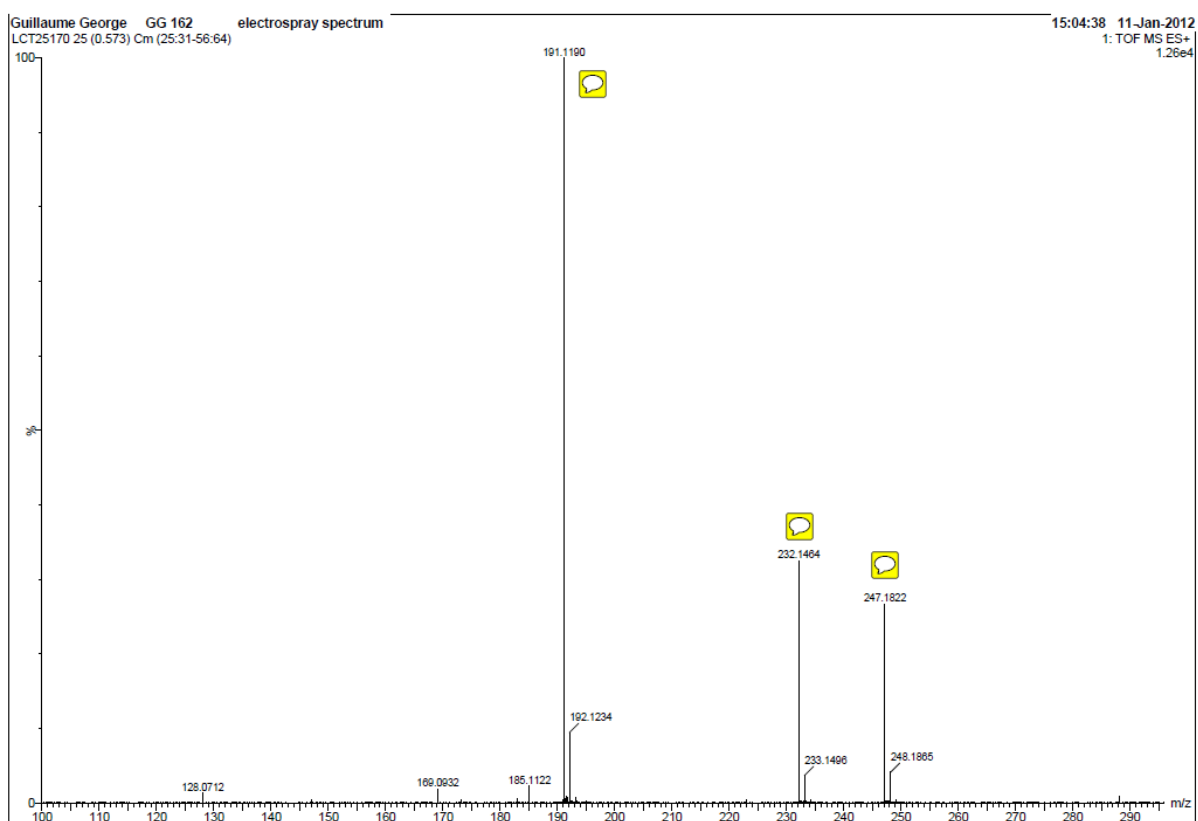


^{13}C NMR spectrum



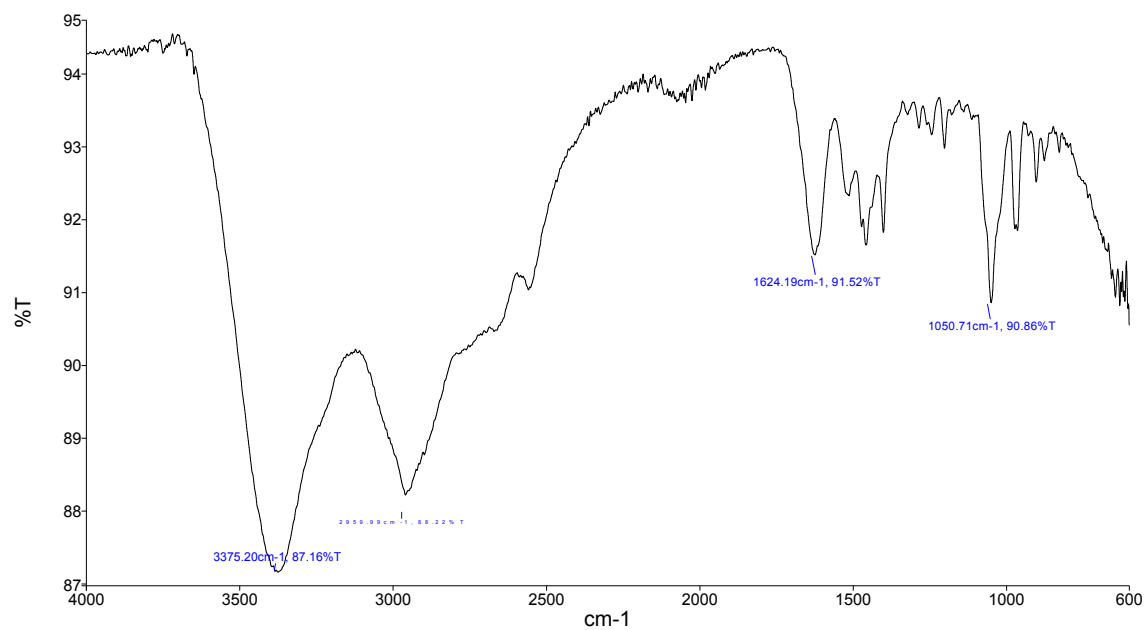
^{19}F NMR spectrum



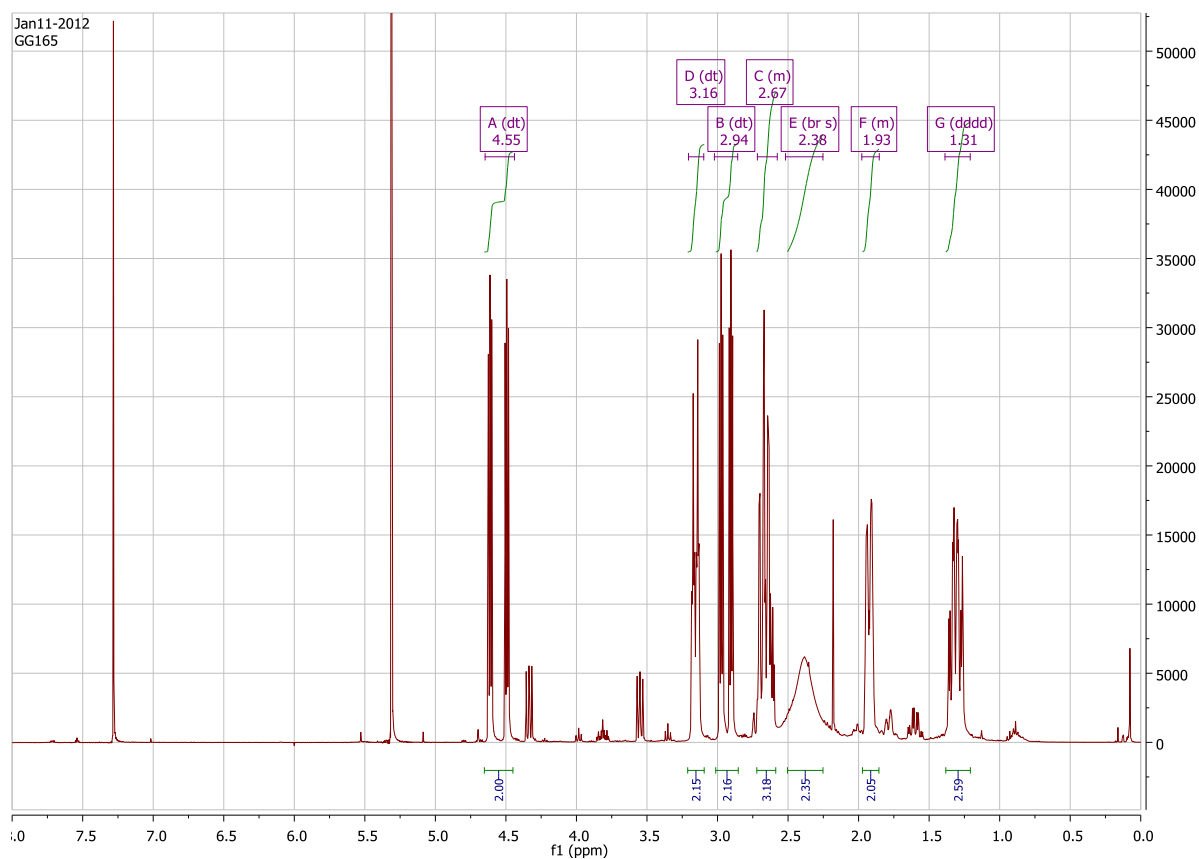


90. 4-Ammonio-1-(2-fluoroethyl)piperidinium dichloride

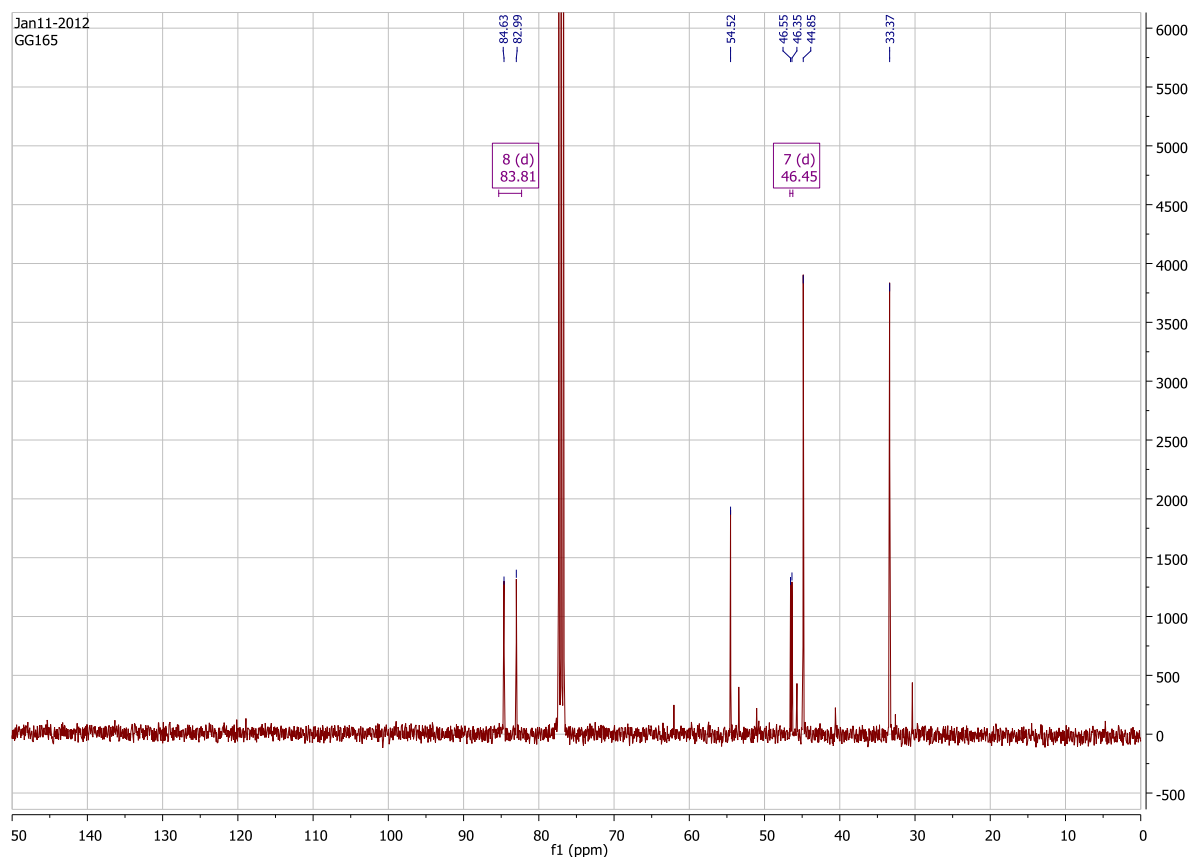
Infrared spectrum



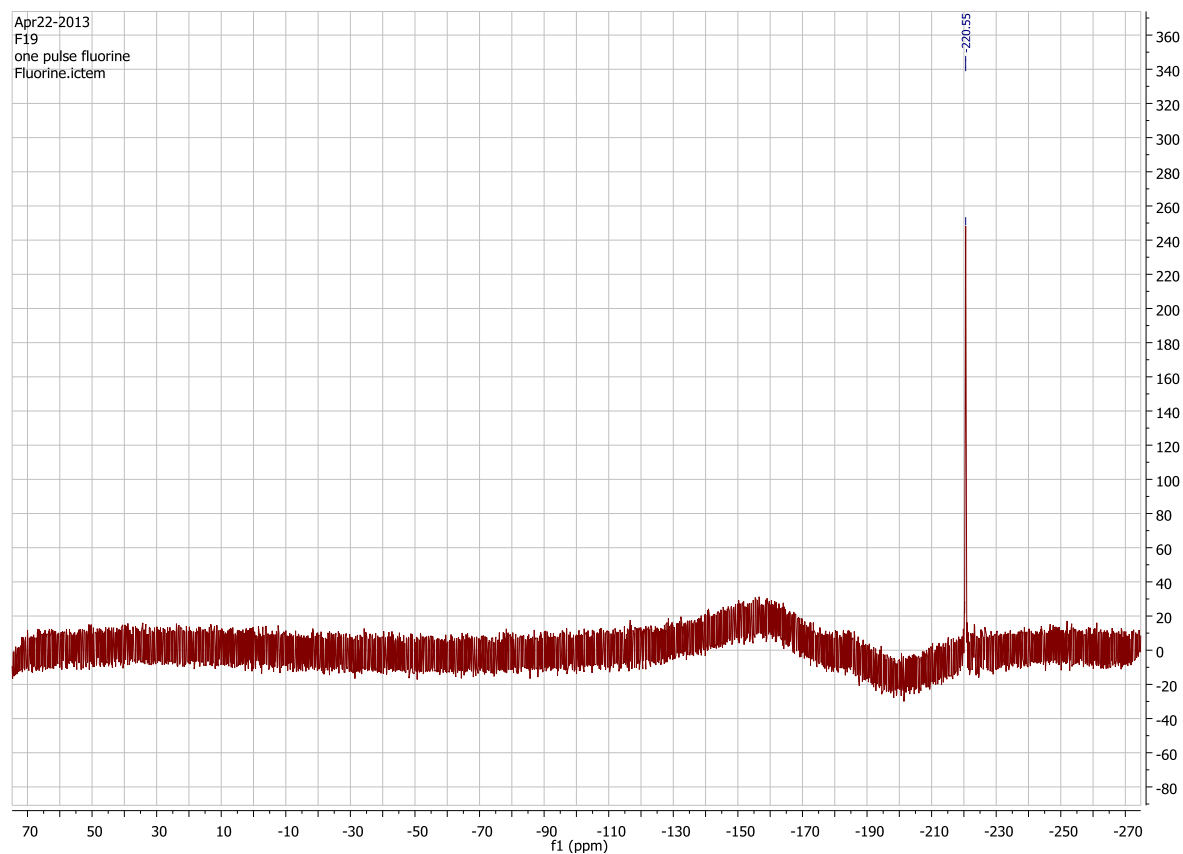
¹H NMR spectrum

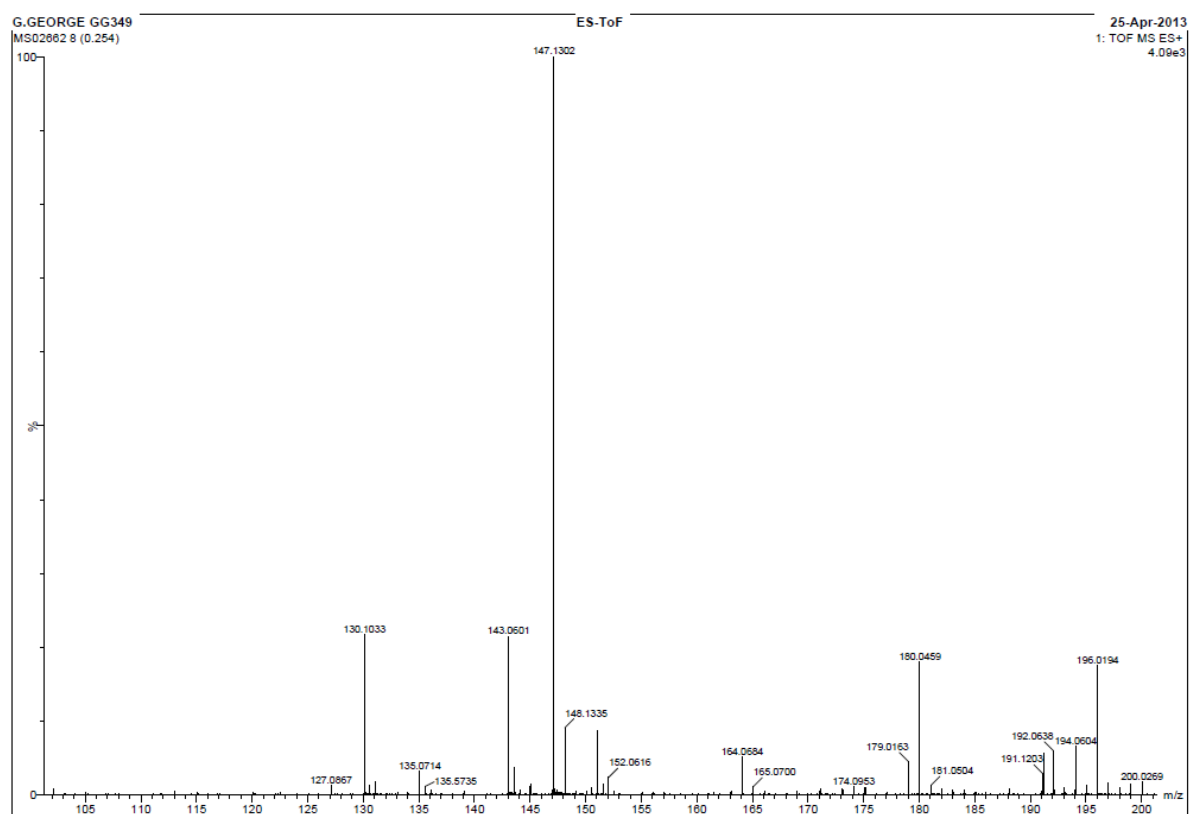


^{13}C NMR spectrum

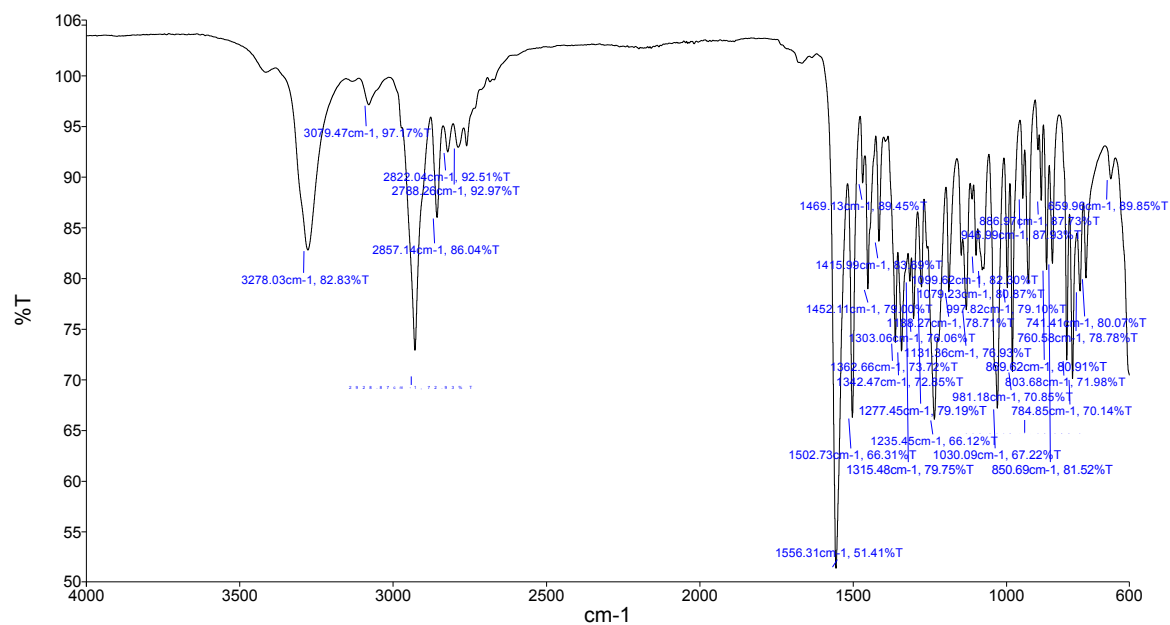
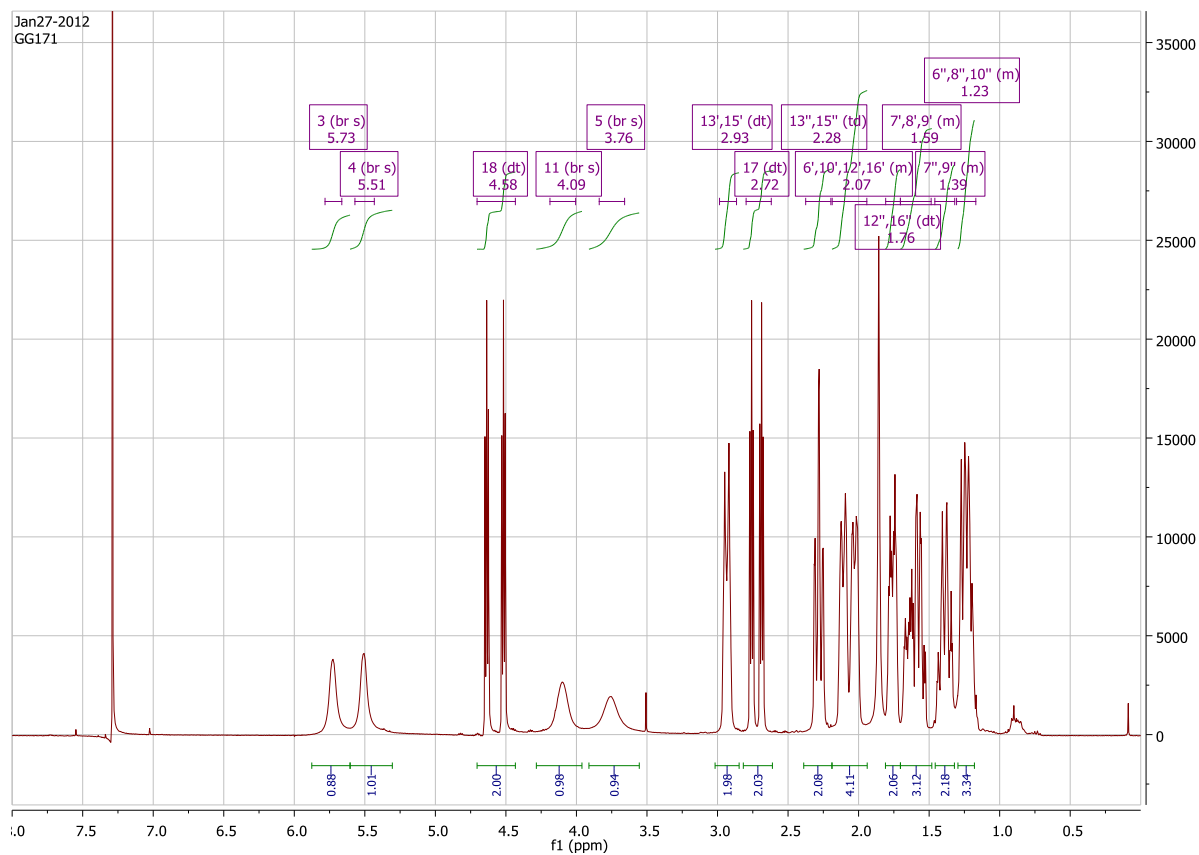


^{19}F NMR spectrum

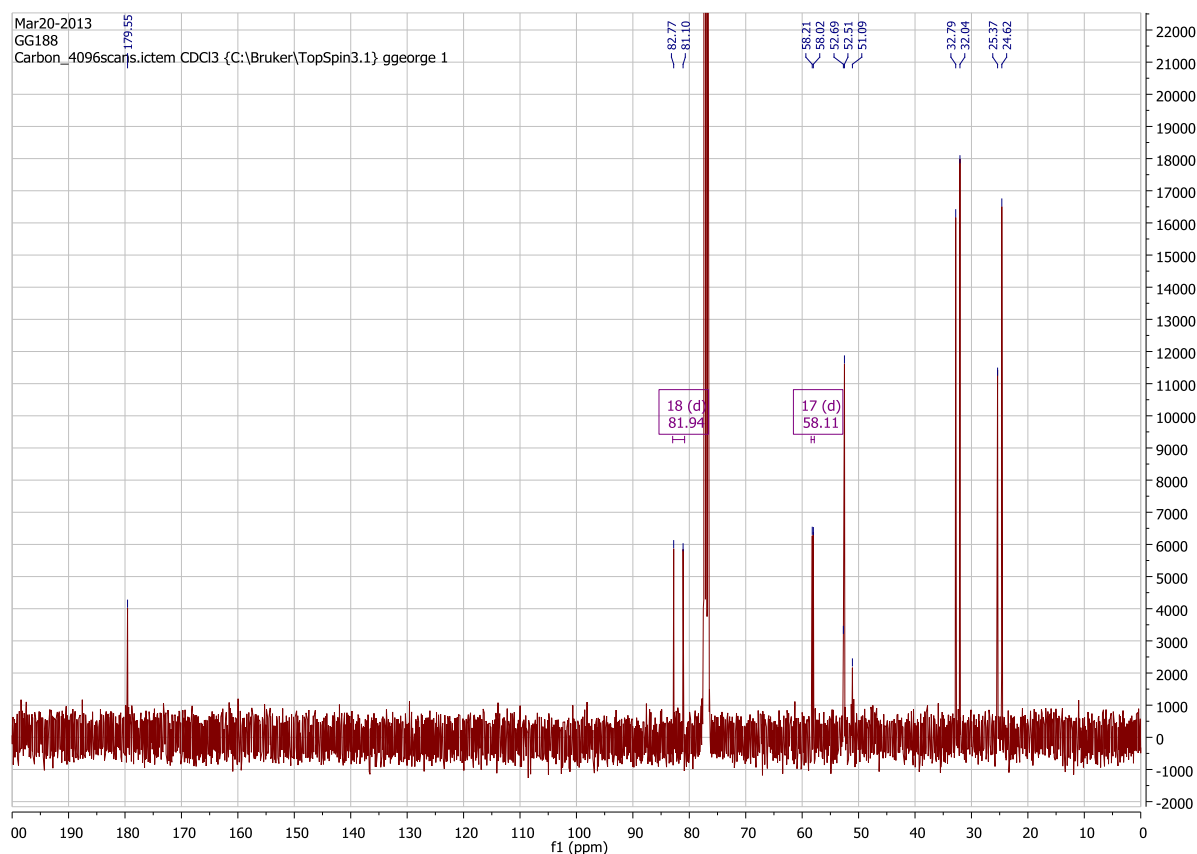




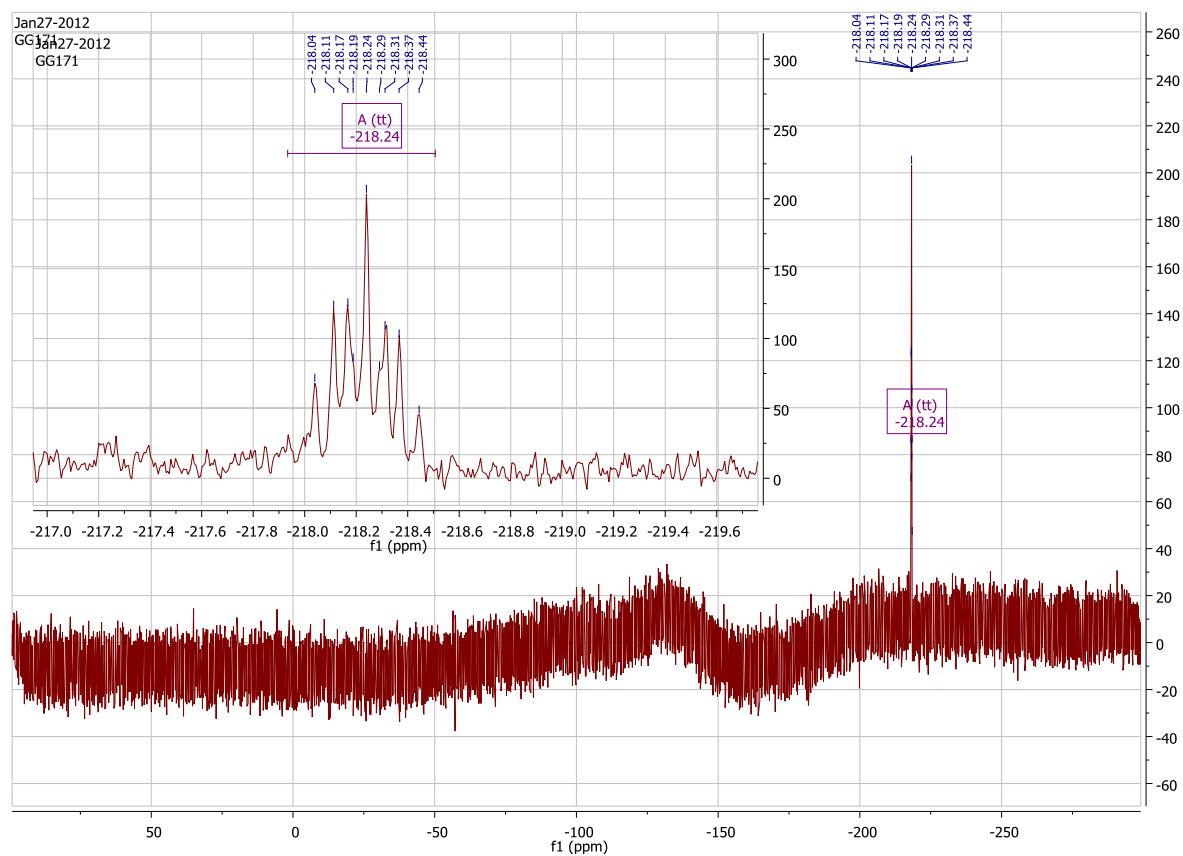
Infrared spectrum

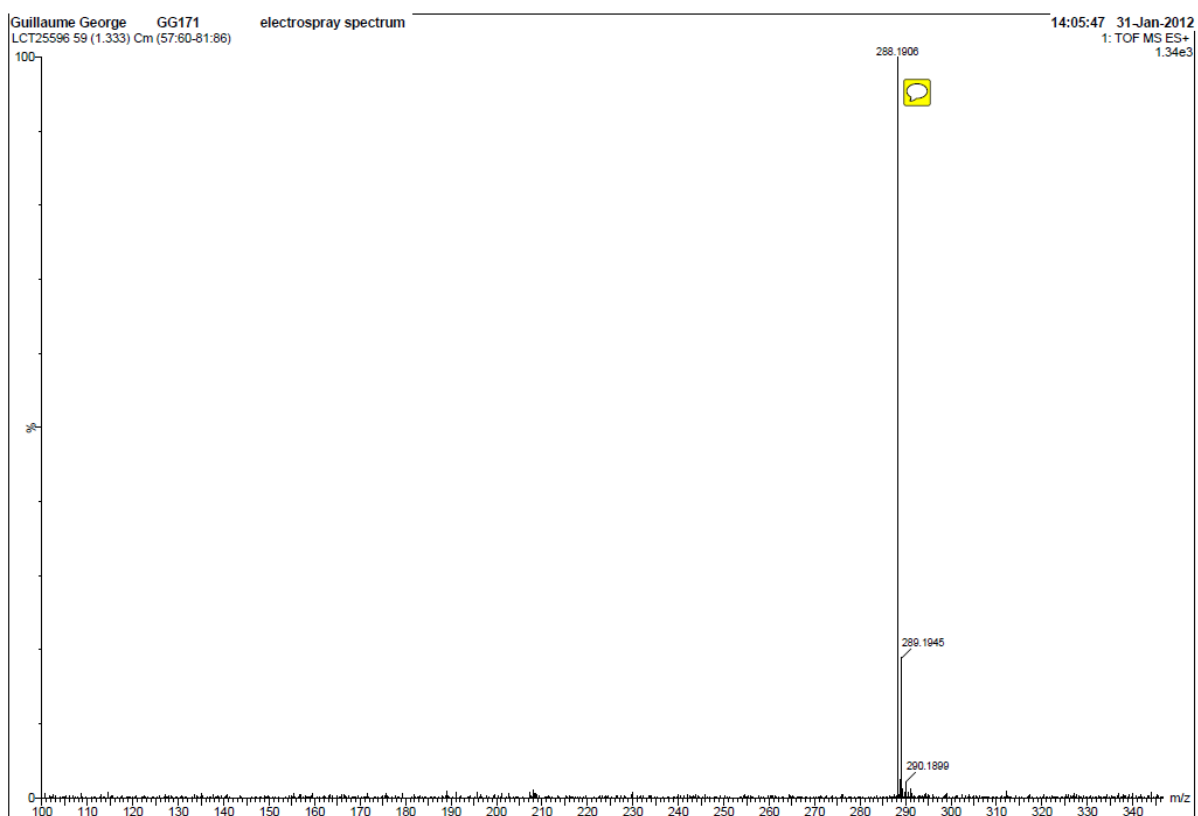
 ^1H NMR spectrum

^{13}C NMR spectrum



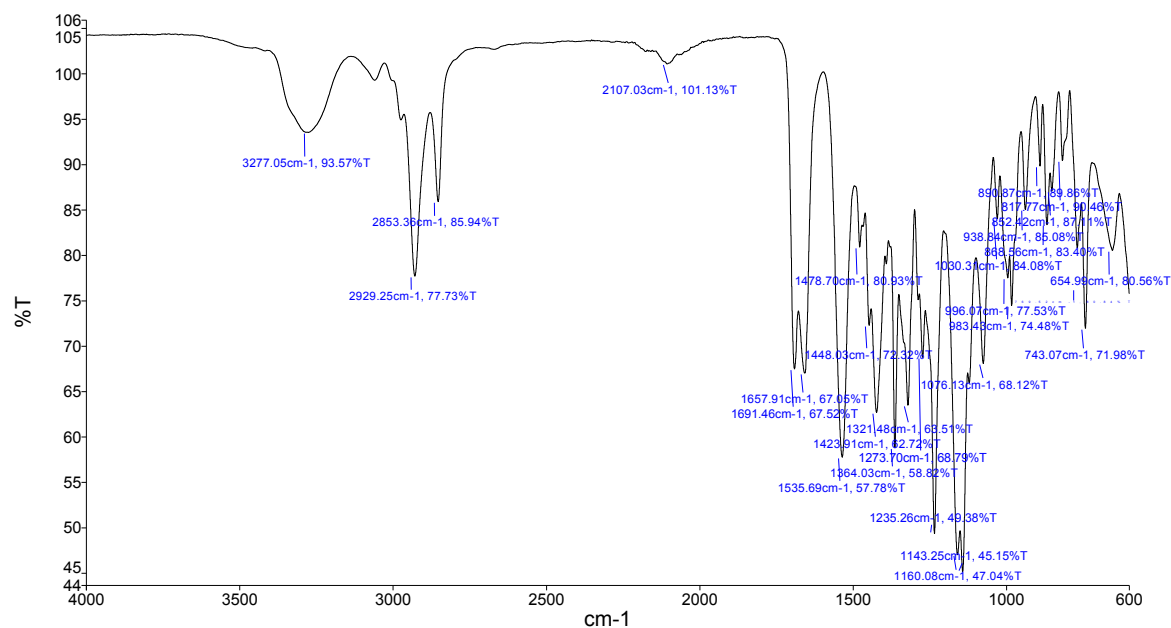
^{19}F NMR spectrum



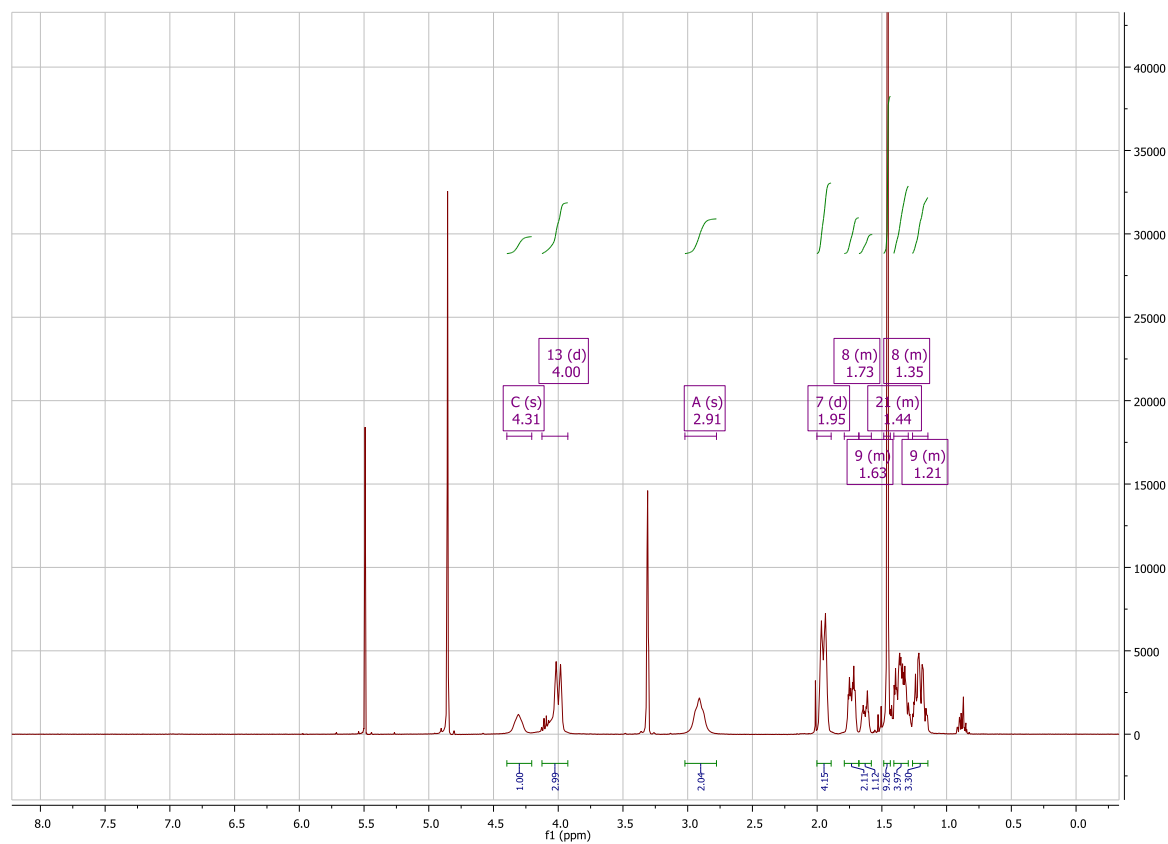


93. *tert*-Butyl 4-[(cyclohexylcarbamothioyl)amino]piperidine-1-carboxylate

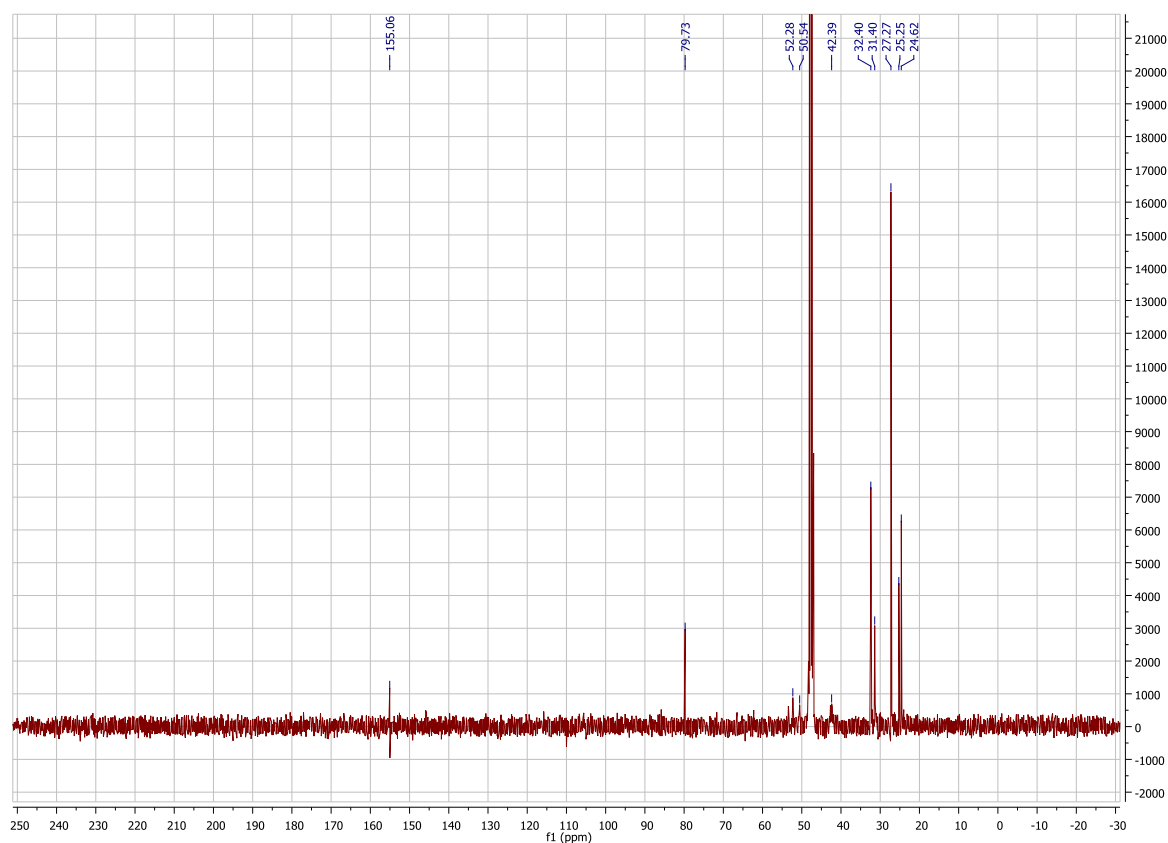
Infrared spectrum



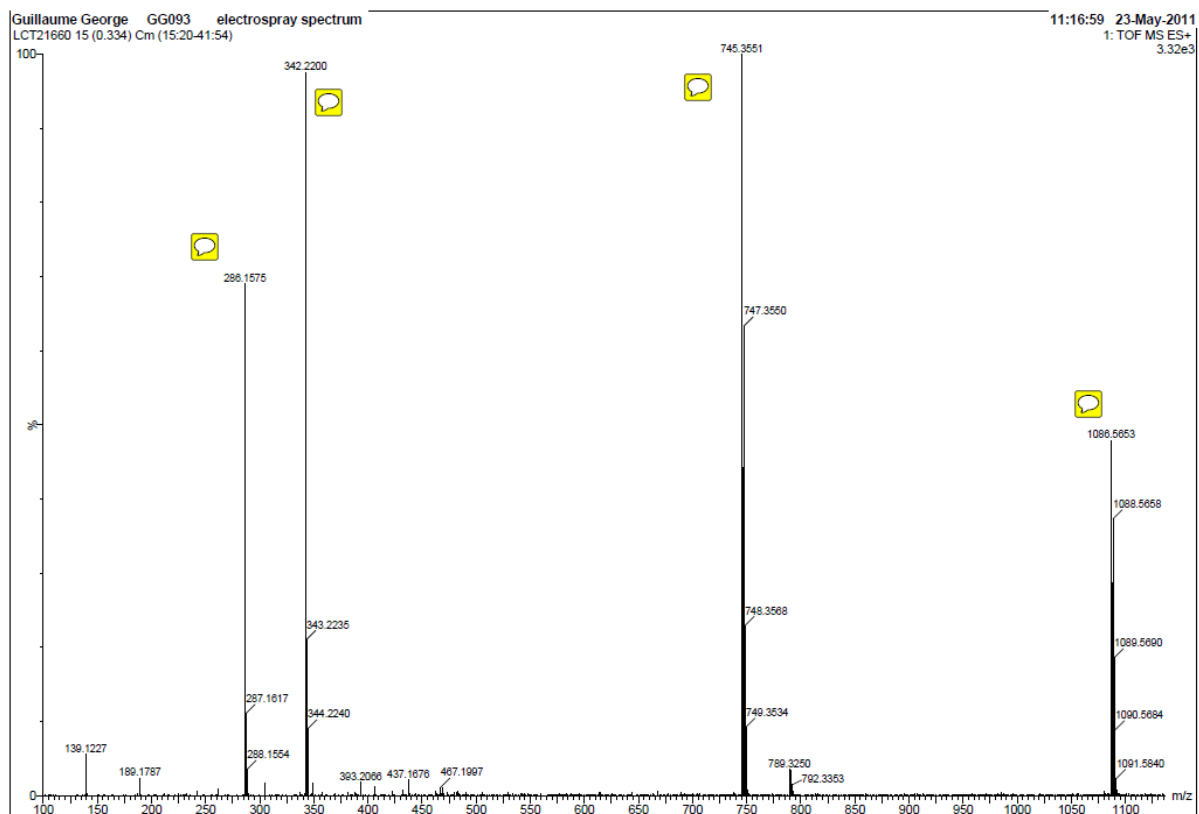
^1H NMR spectrum



^{13}C NMR spectrum

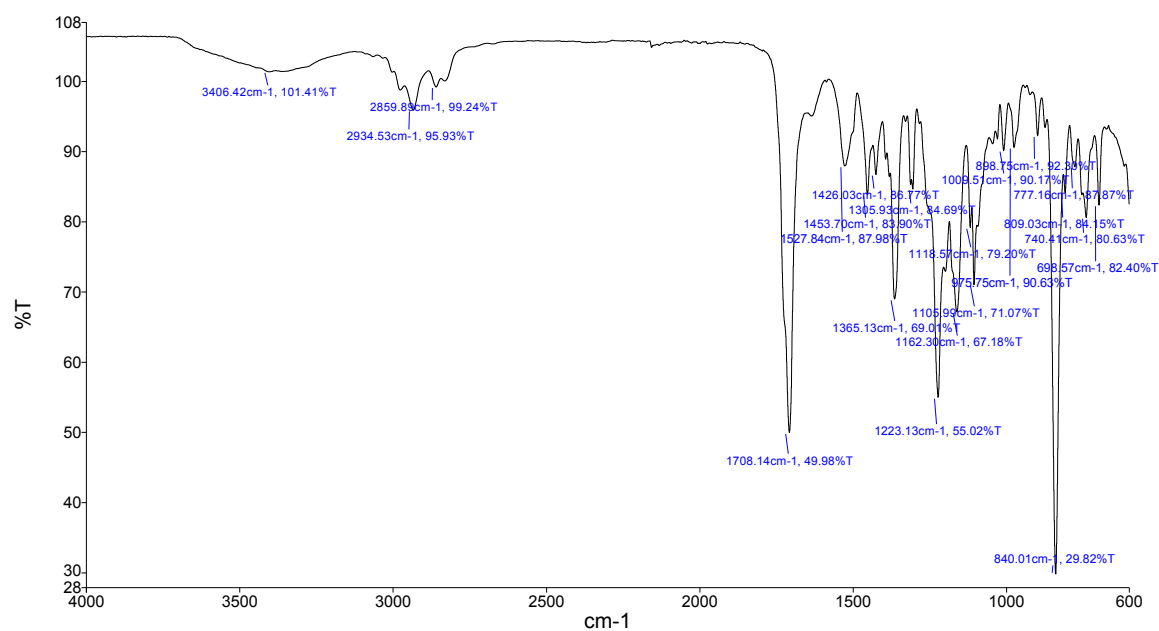


HRMS

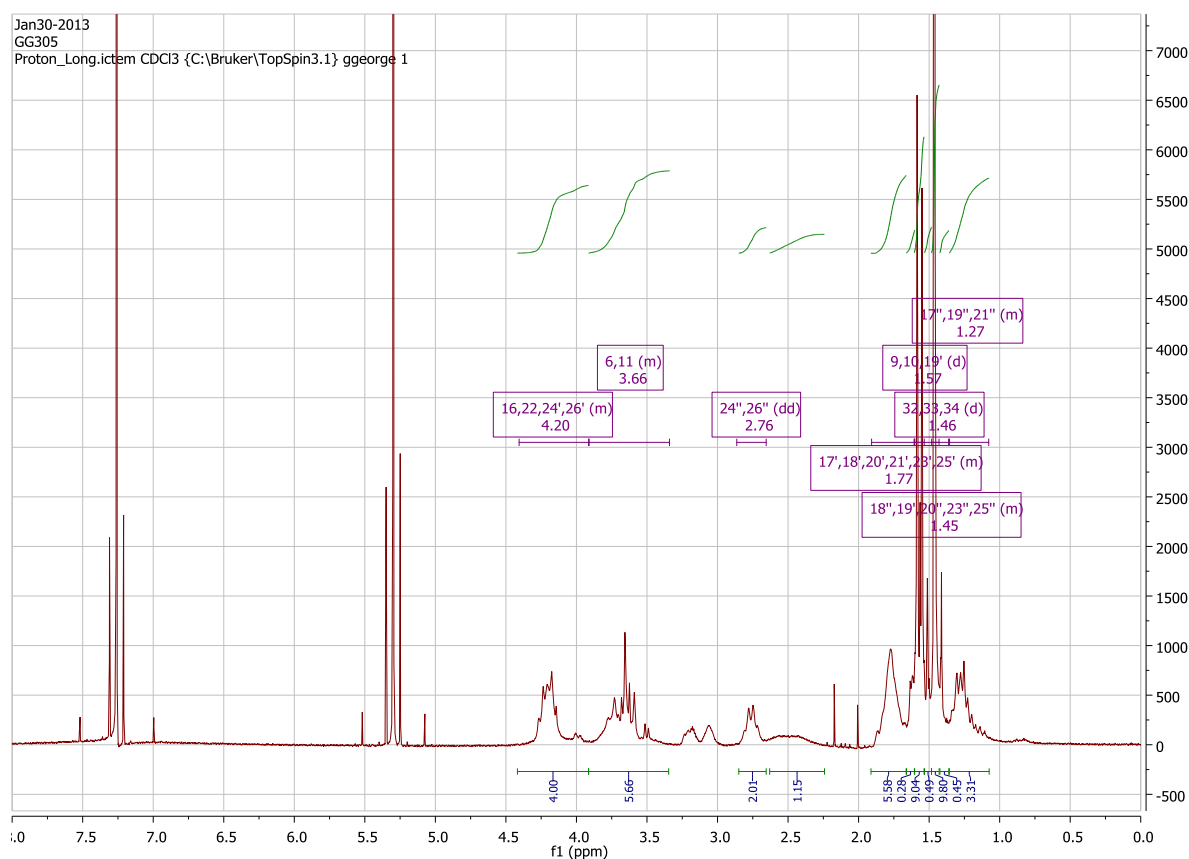


94. *tert*-Butyl 4- $\{[(N'$ -cyclohexylimino)] $\{6,6$ -dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl)methylamino}piperidine-1-carboxylate

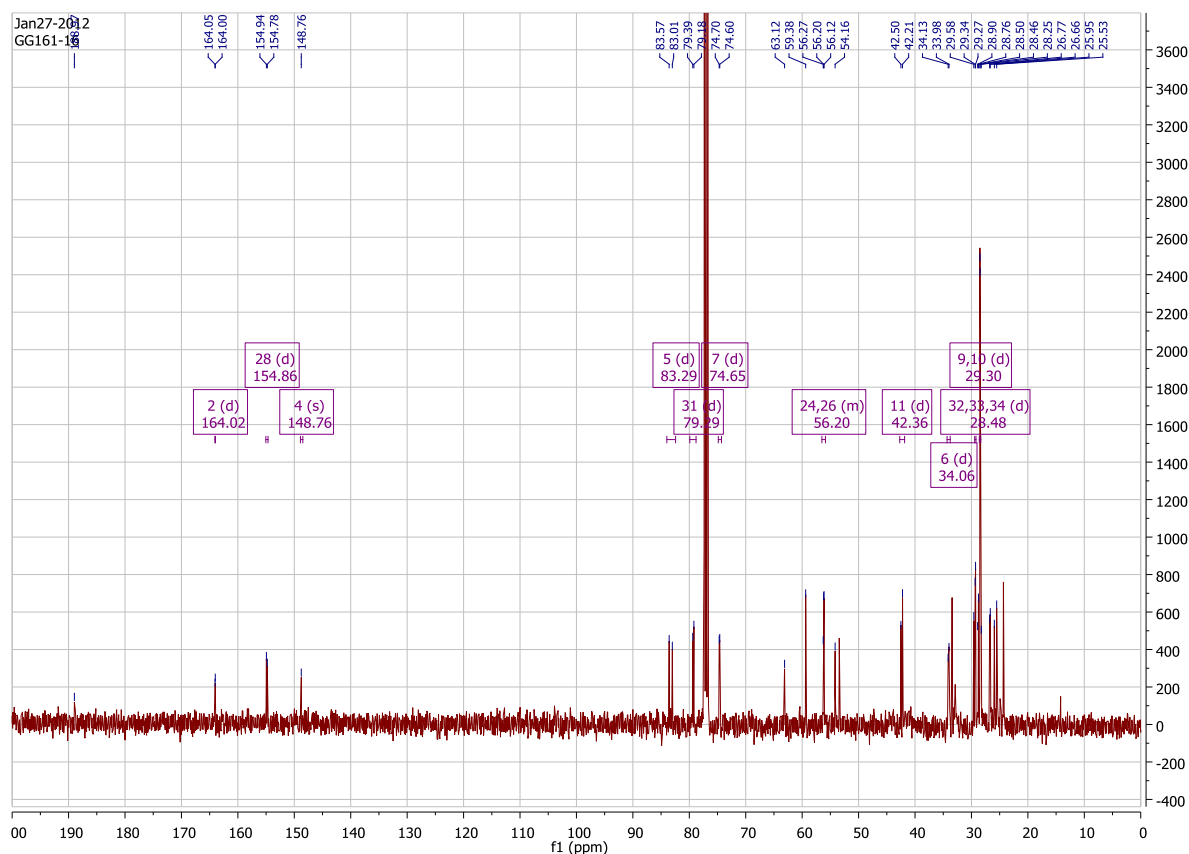
Infrared spectrum



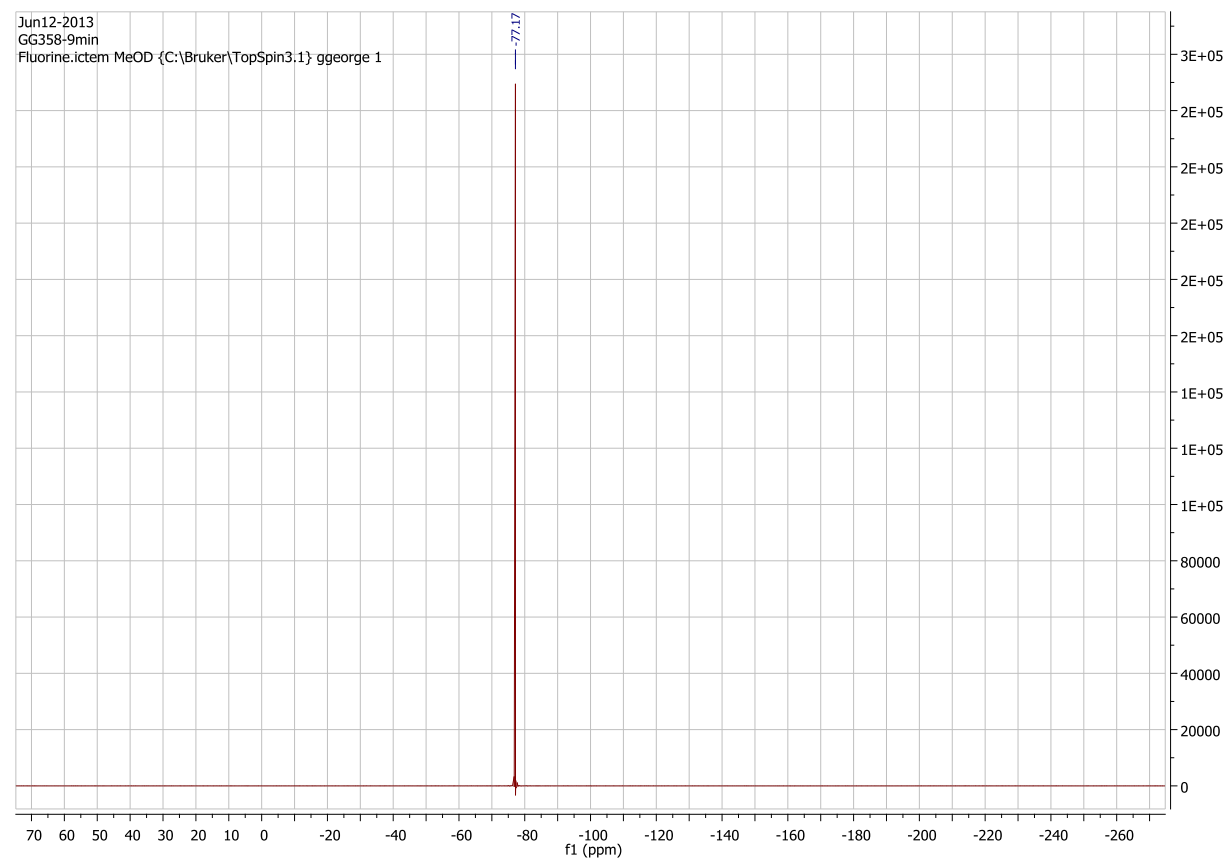
¹H NMR spectrum

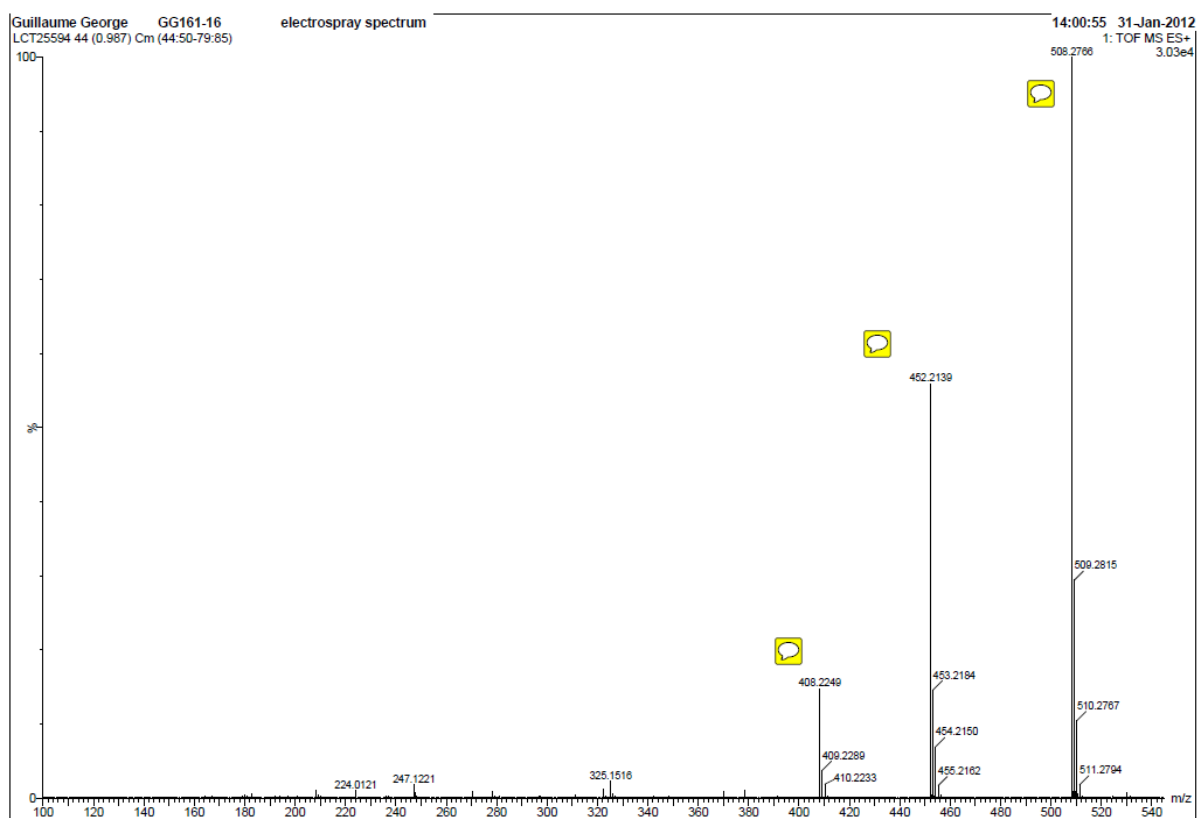


^{13}C NMR spectrum



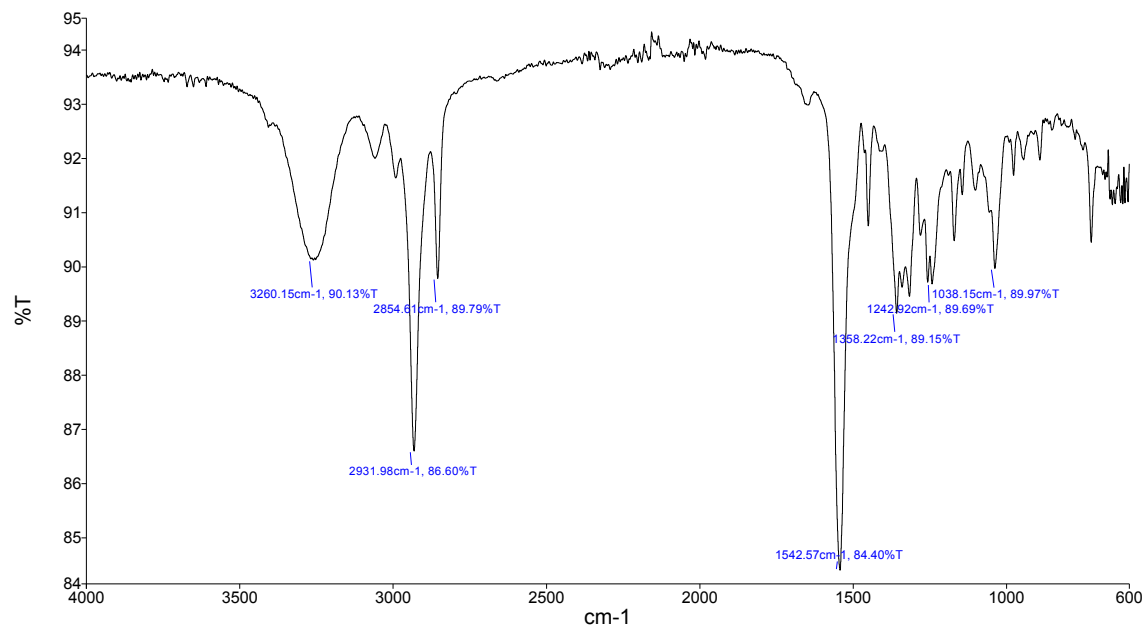
^{19}F NMR spectrum



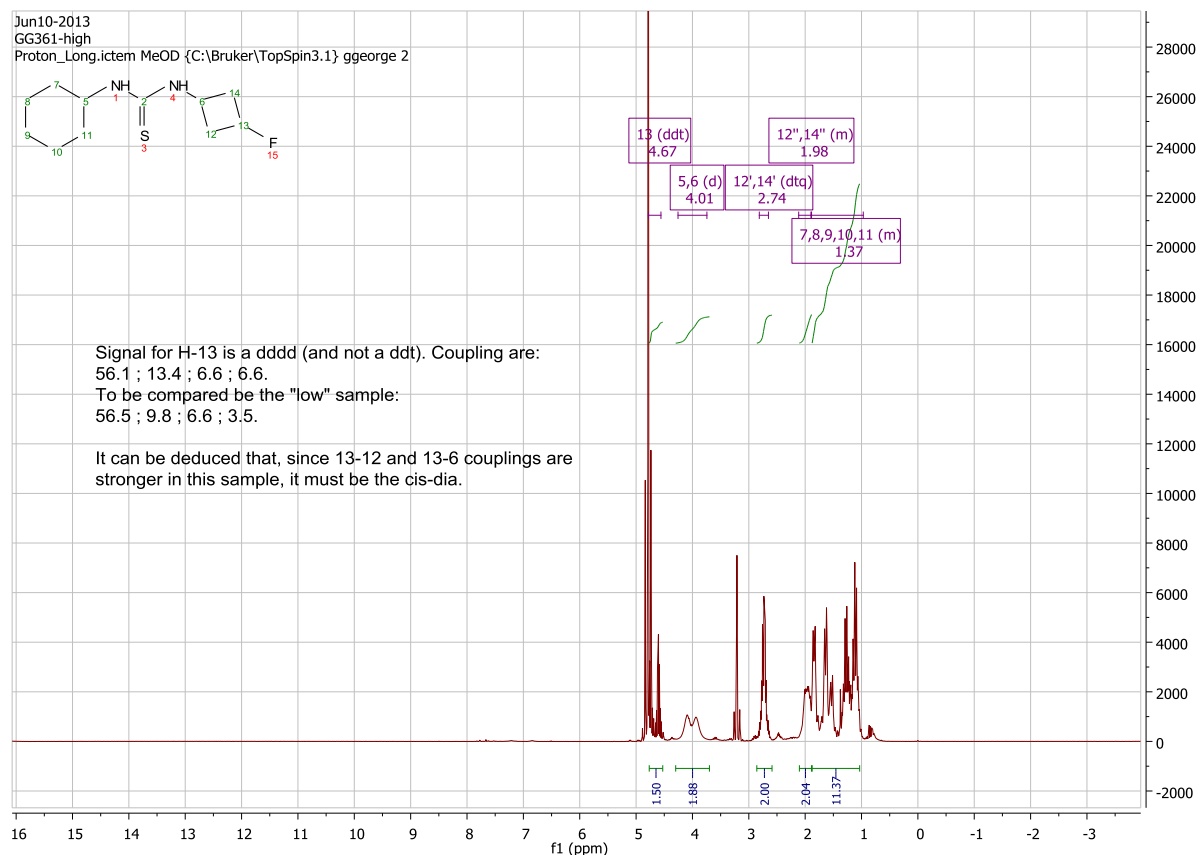


96. 1-Cyclohexyl-3-(*cis*-3-fluorocyclobutyl)thiourea

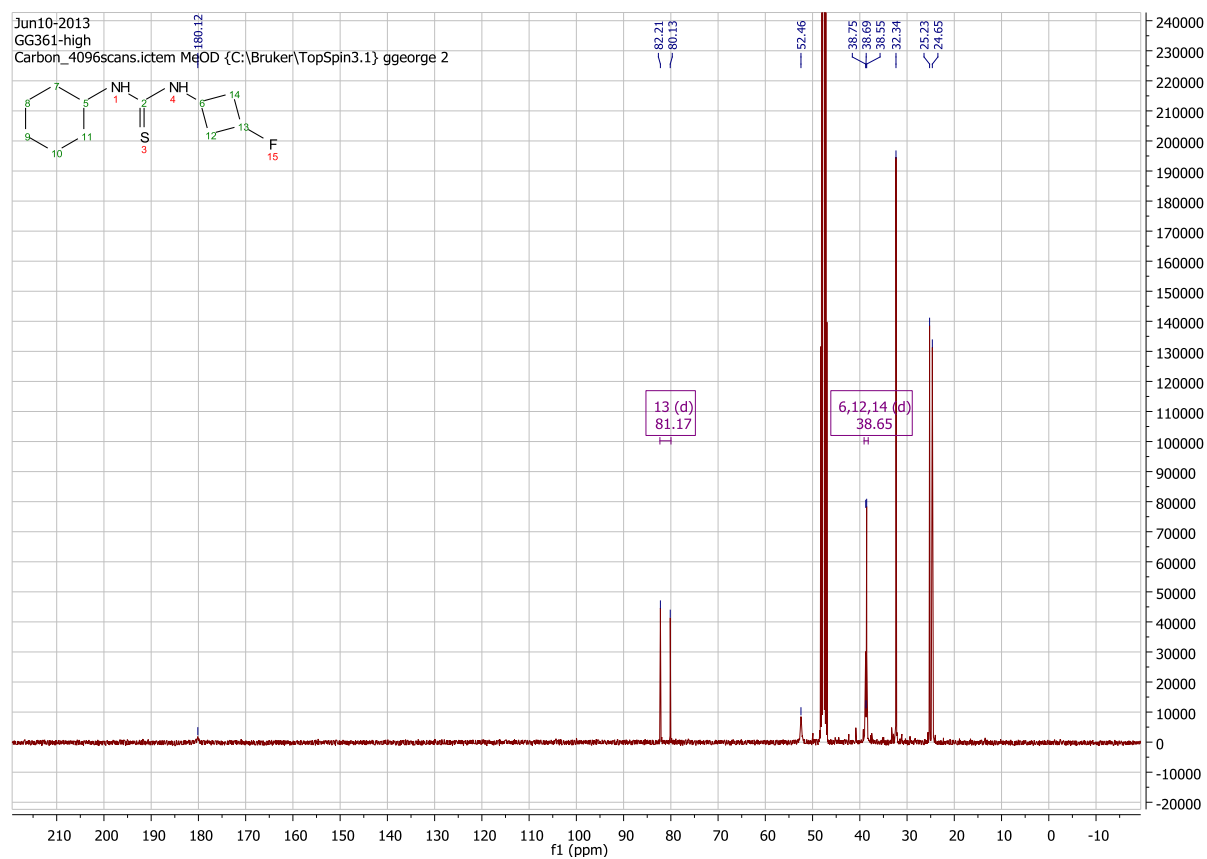
Infrared spectrum



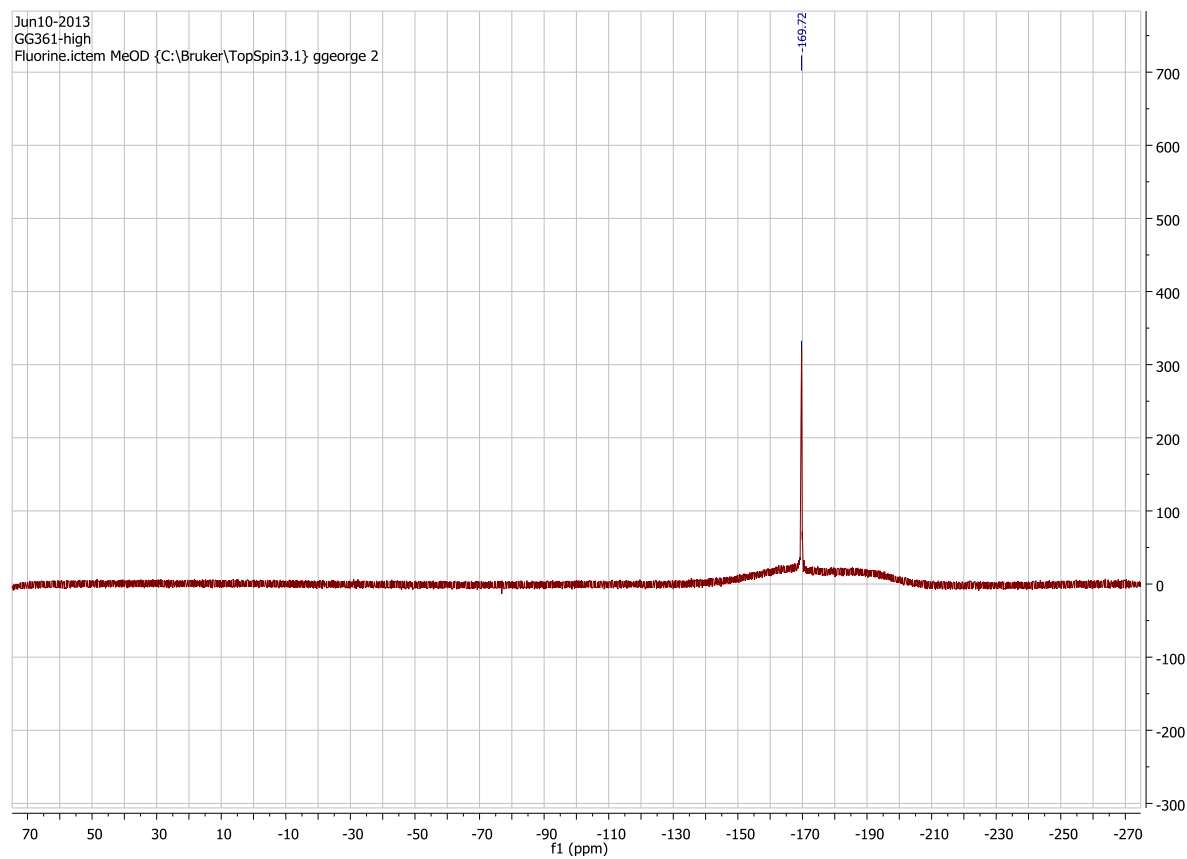
¹H NMR spectrum

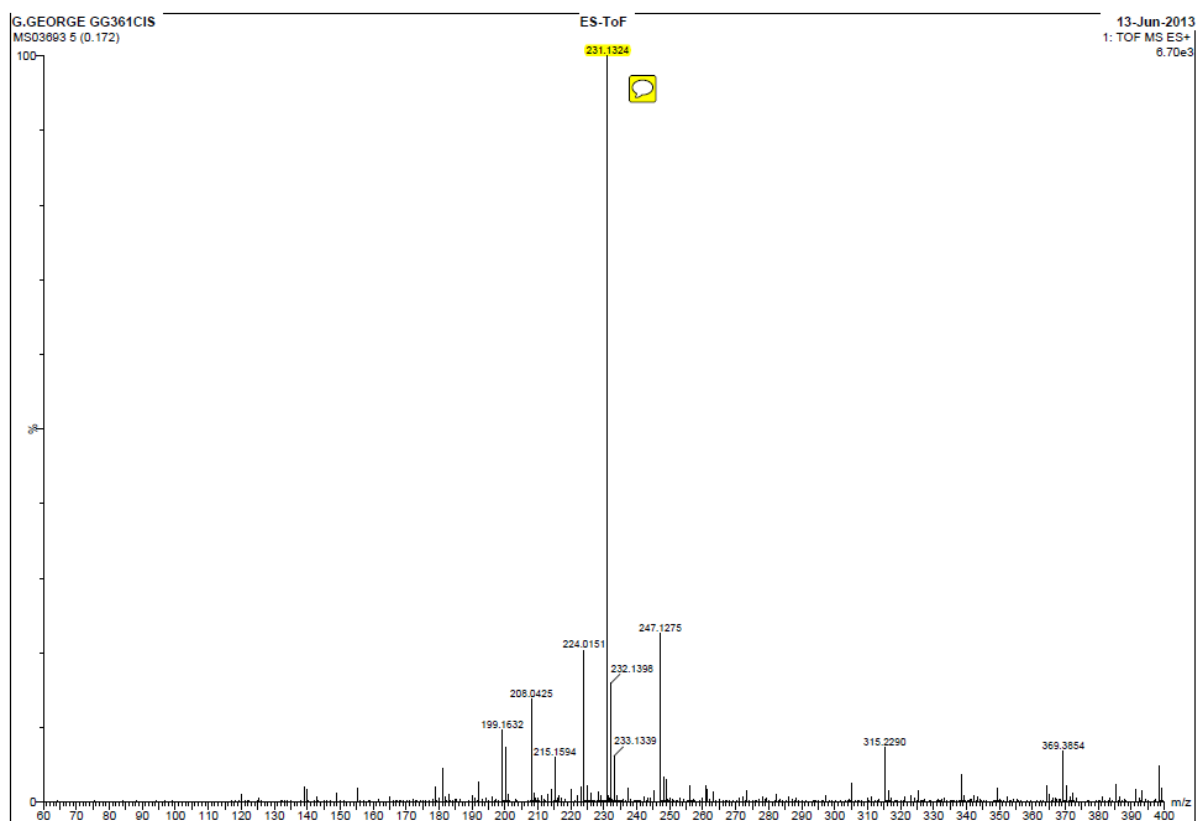


^{13}C NMR spectrum



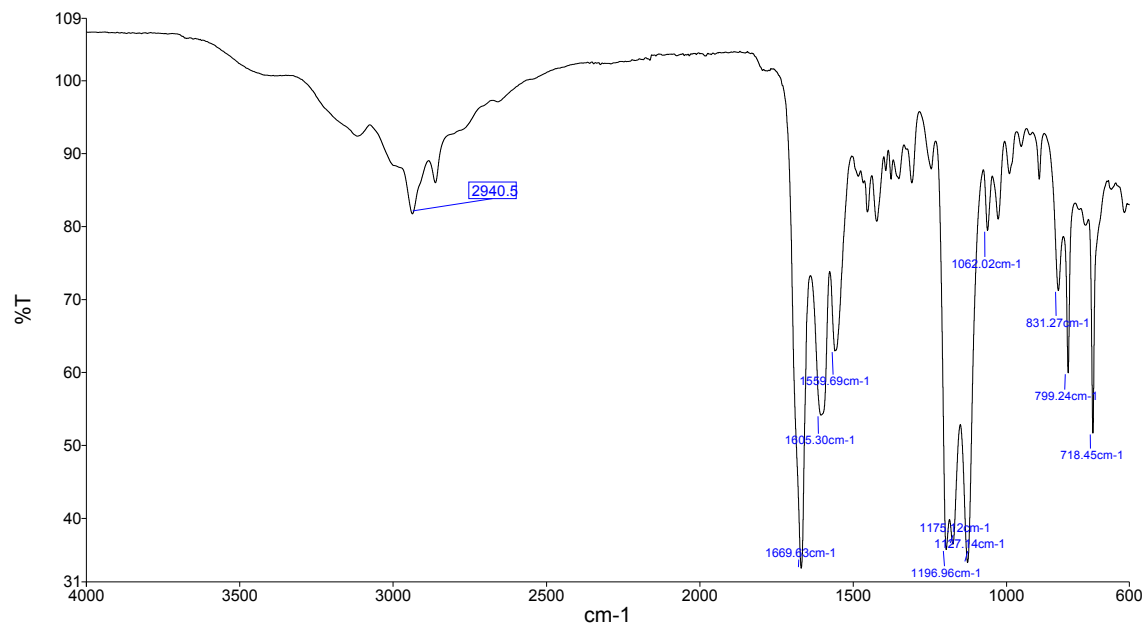
^{19}F NMR spectrum



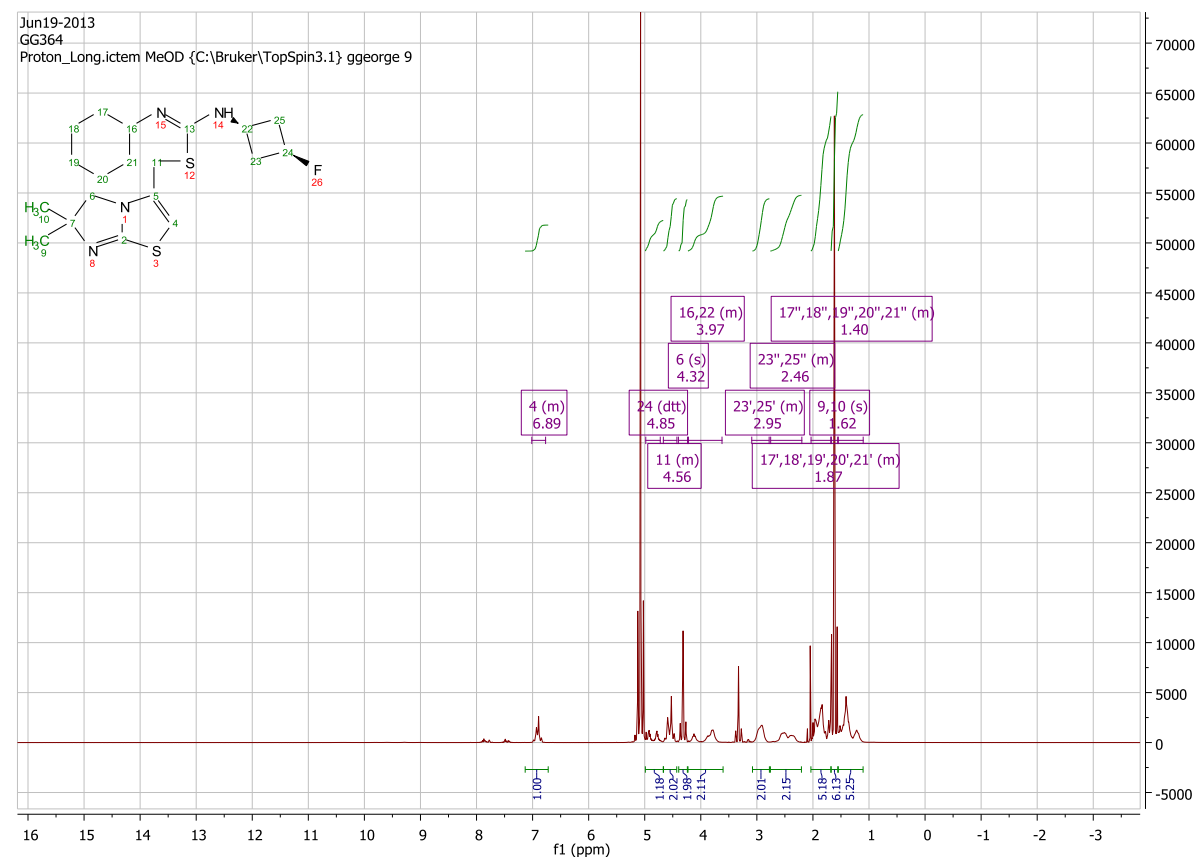


97. *N*'-Cyclohexyl-*N*-(*cis*-3-fluorocyclobutyl)(({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidamide .3TFA

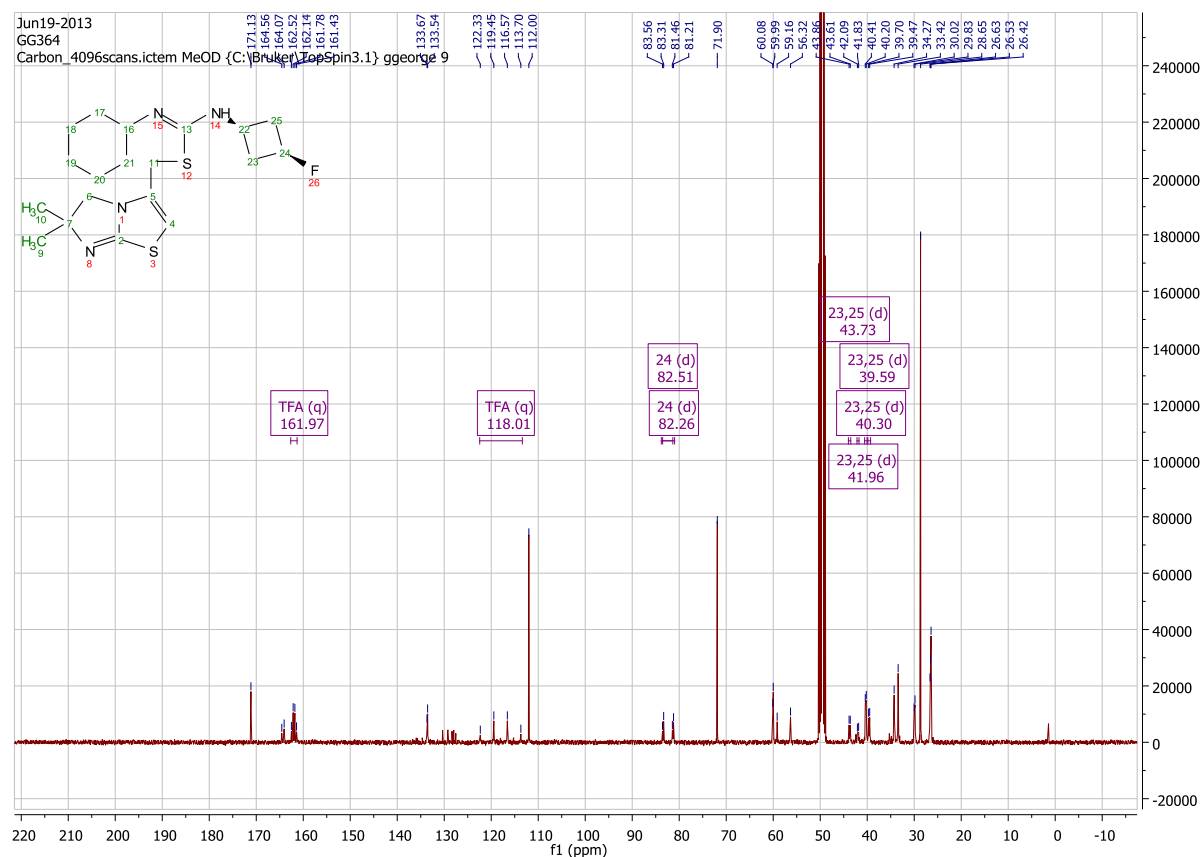
Infrared spectrum



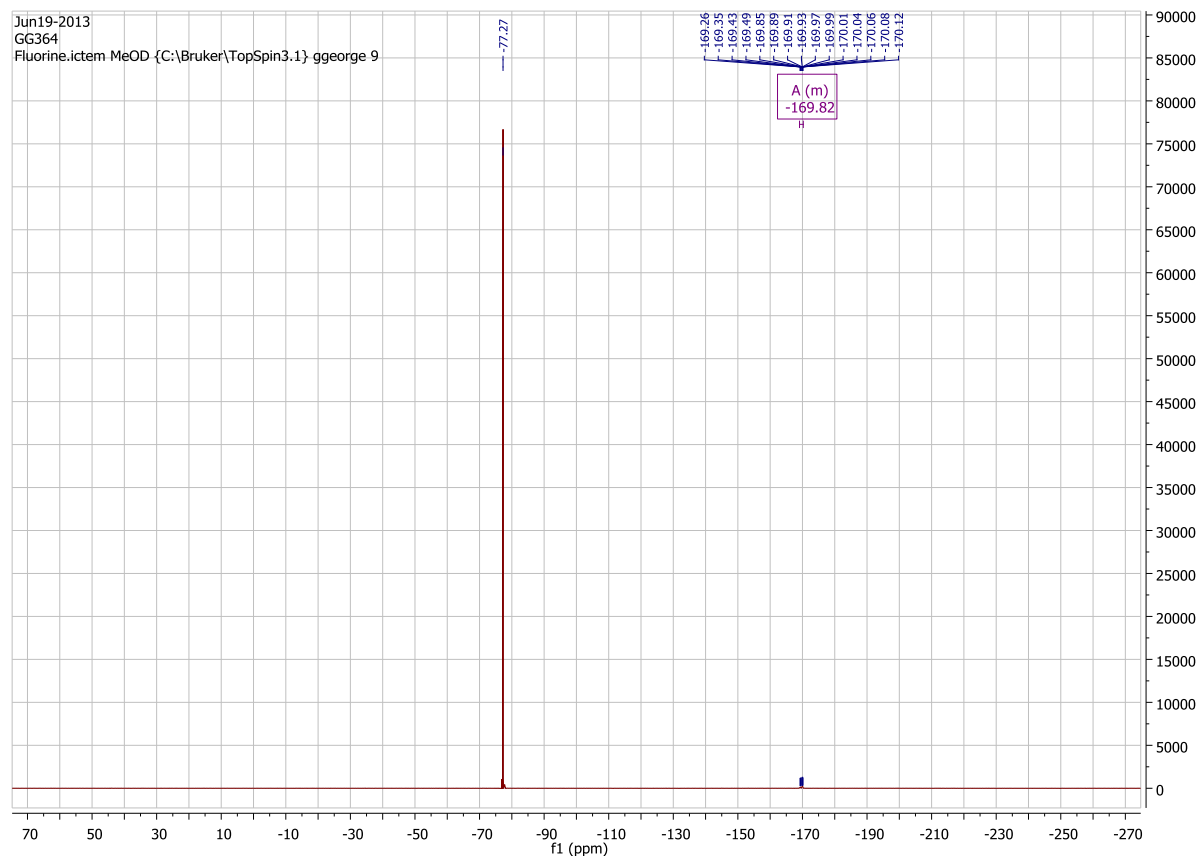
¹H NMR spectrum



^{13}C NMR spectrum

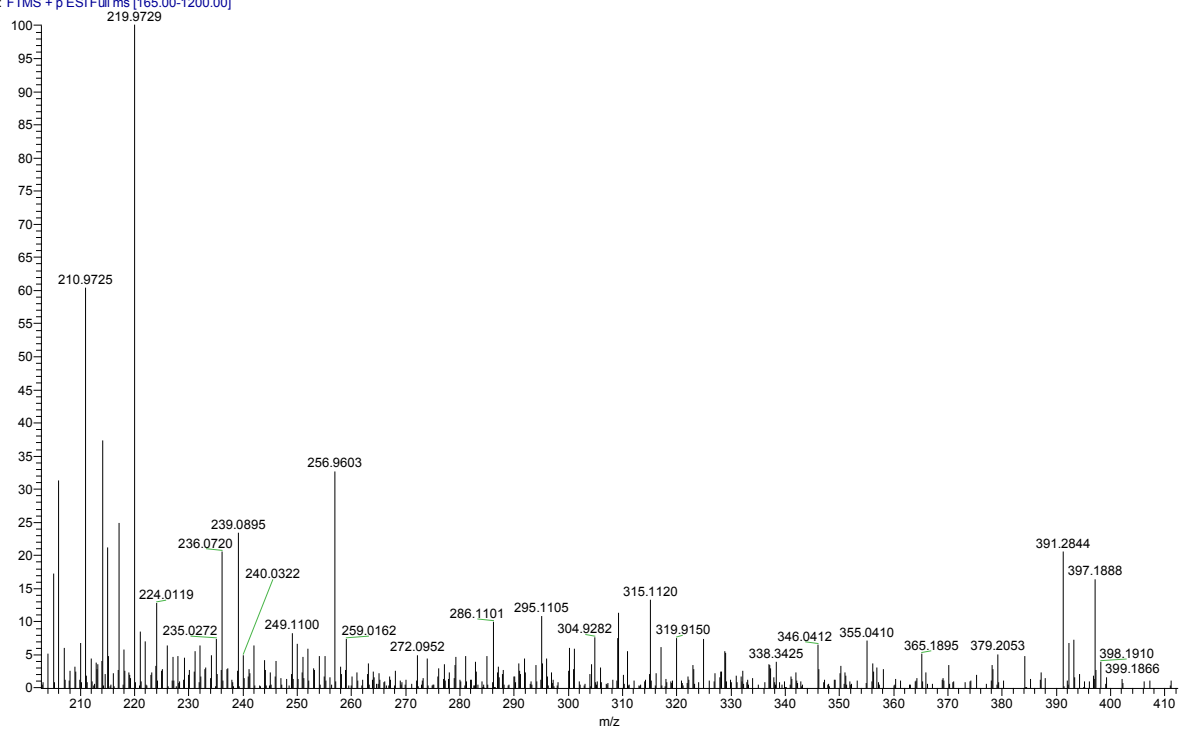


^{19}F NMR spectrum

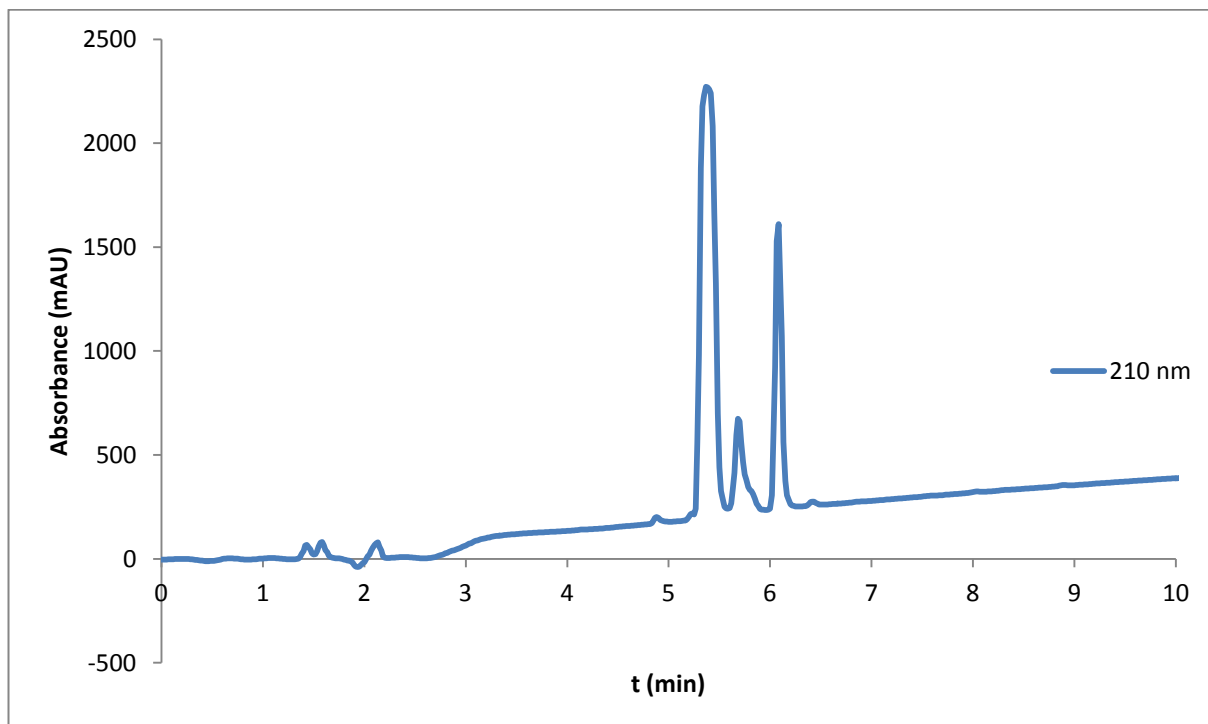


HRMS

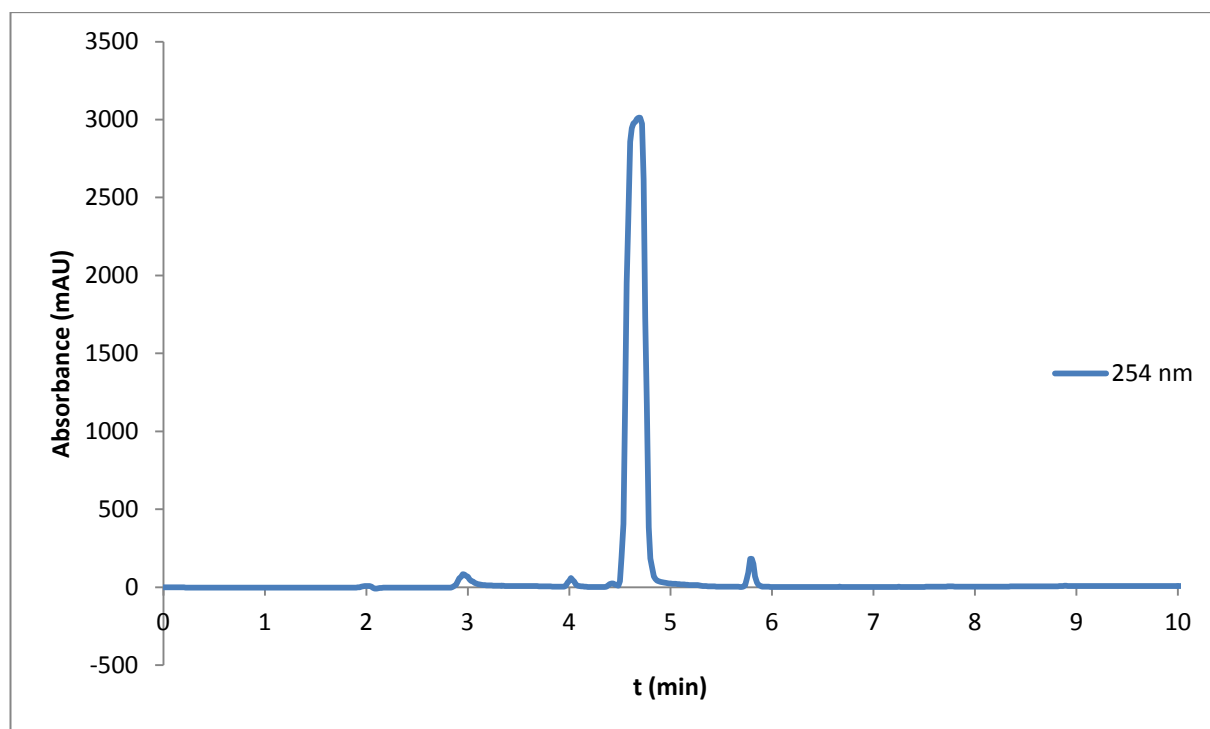
MS03872 #318 RT: 1.21 AV: 1 NL: 3.53E7
T: FTMS + p ESI Full ms [165.00-1200.00]



HPLC trace at 210 nm

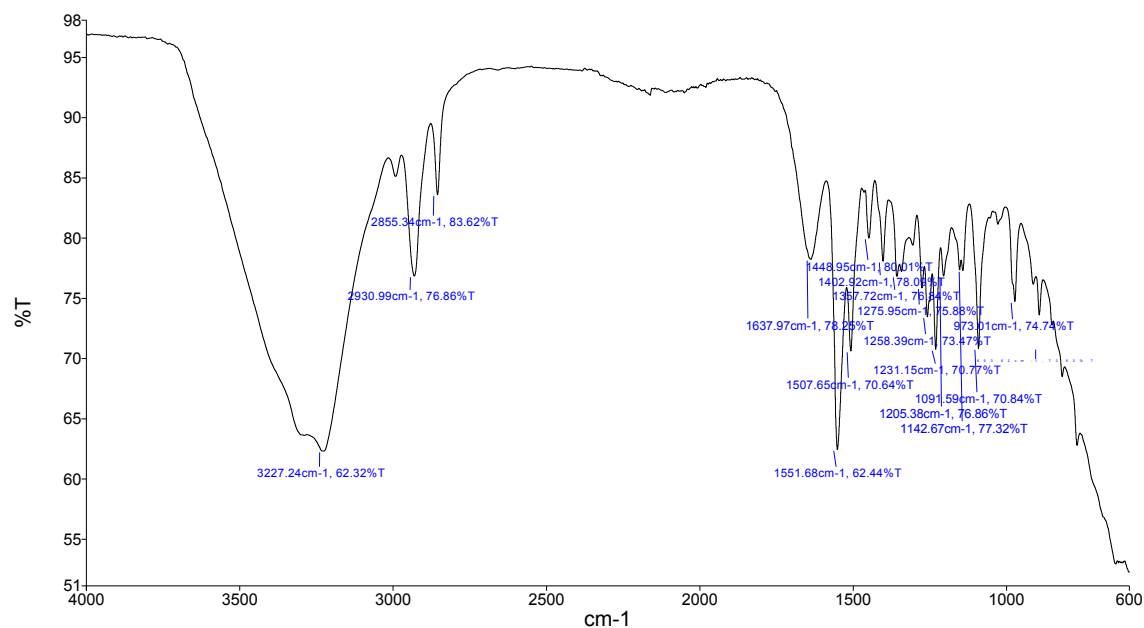


HPLC trace at 254 nm

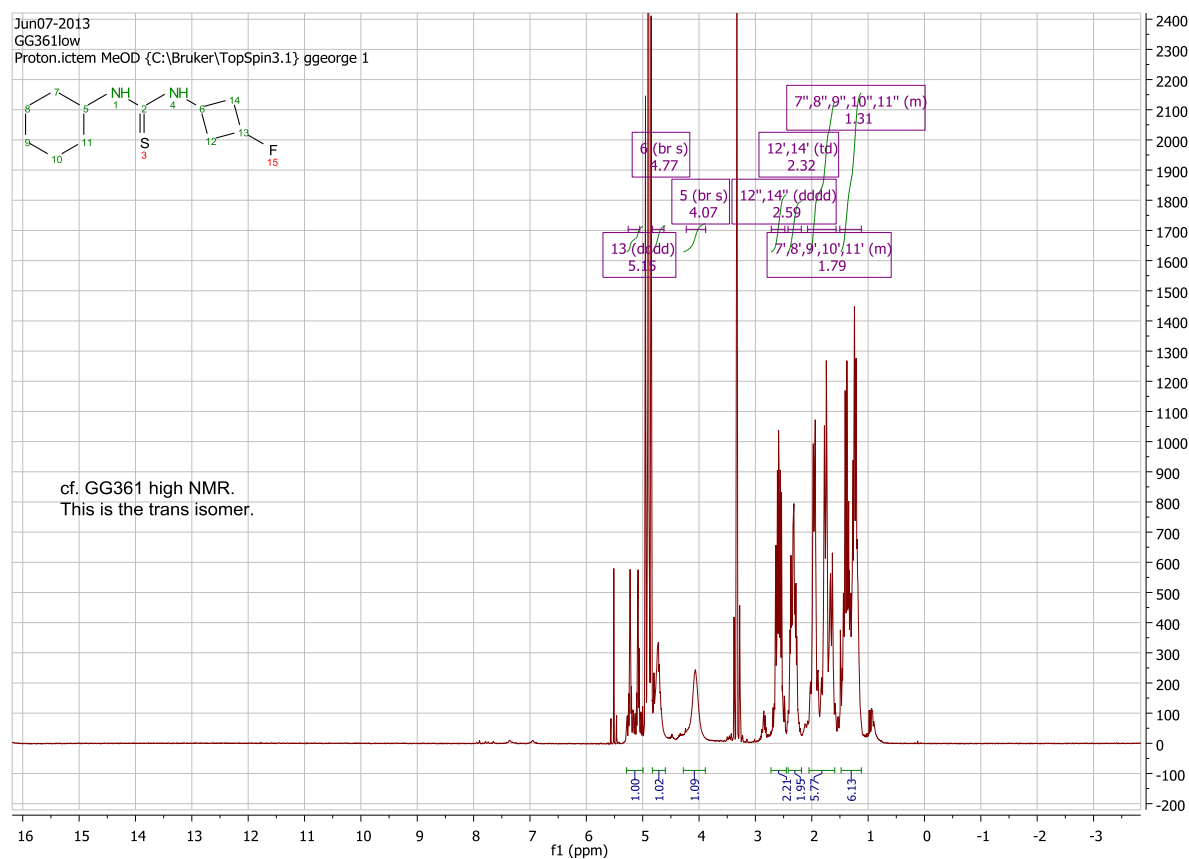


98. 1-Cyclohexyl-3-(*trans*-3-fluorocyclobutyl)thiourea

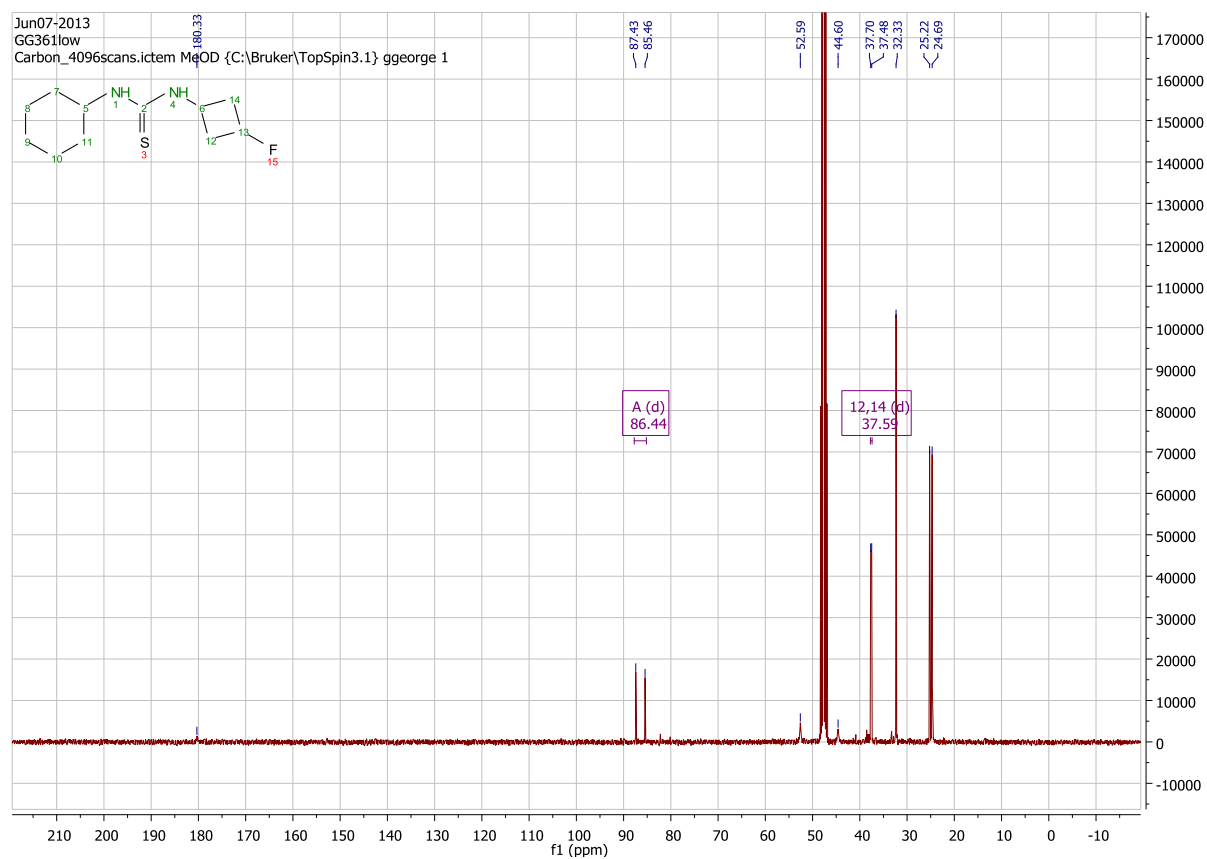
Infrared spectrum



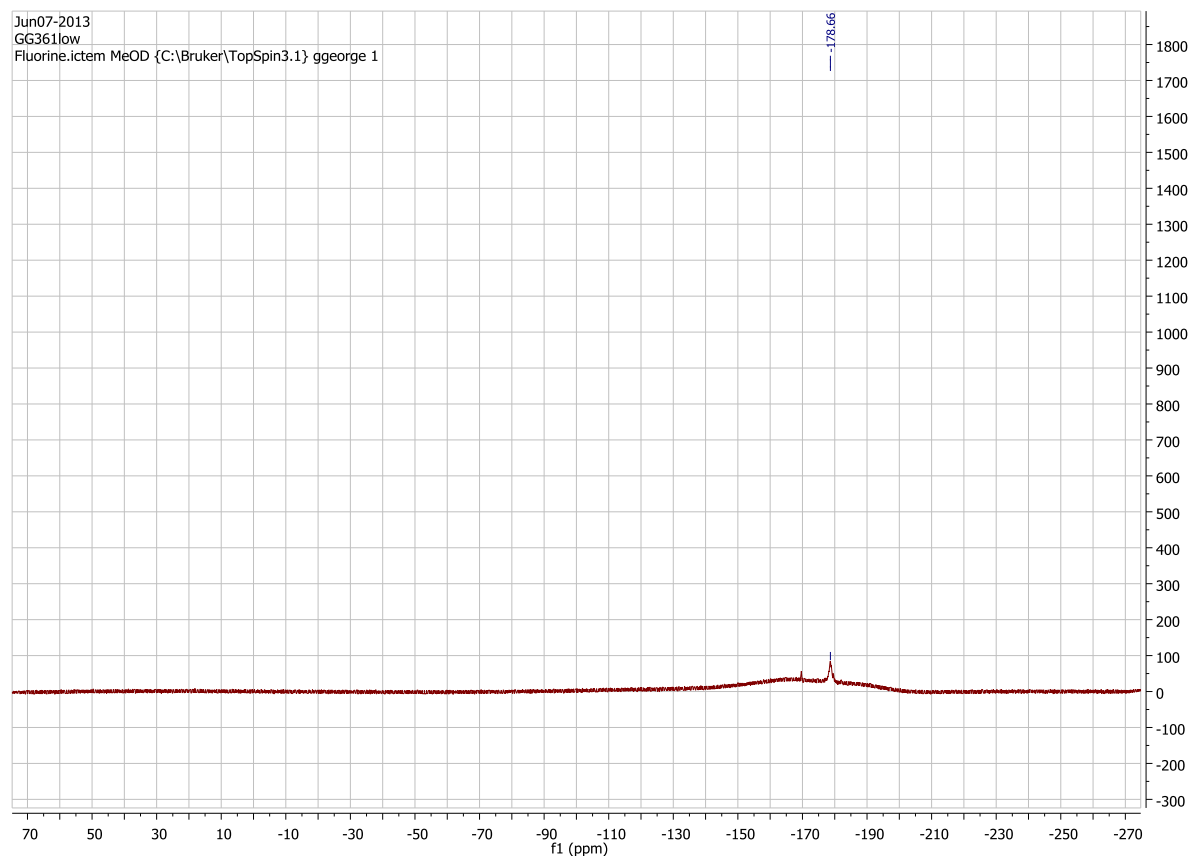
¹H NMR spectrum

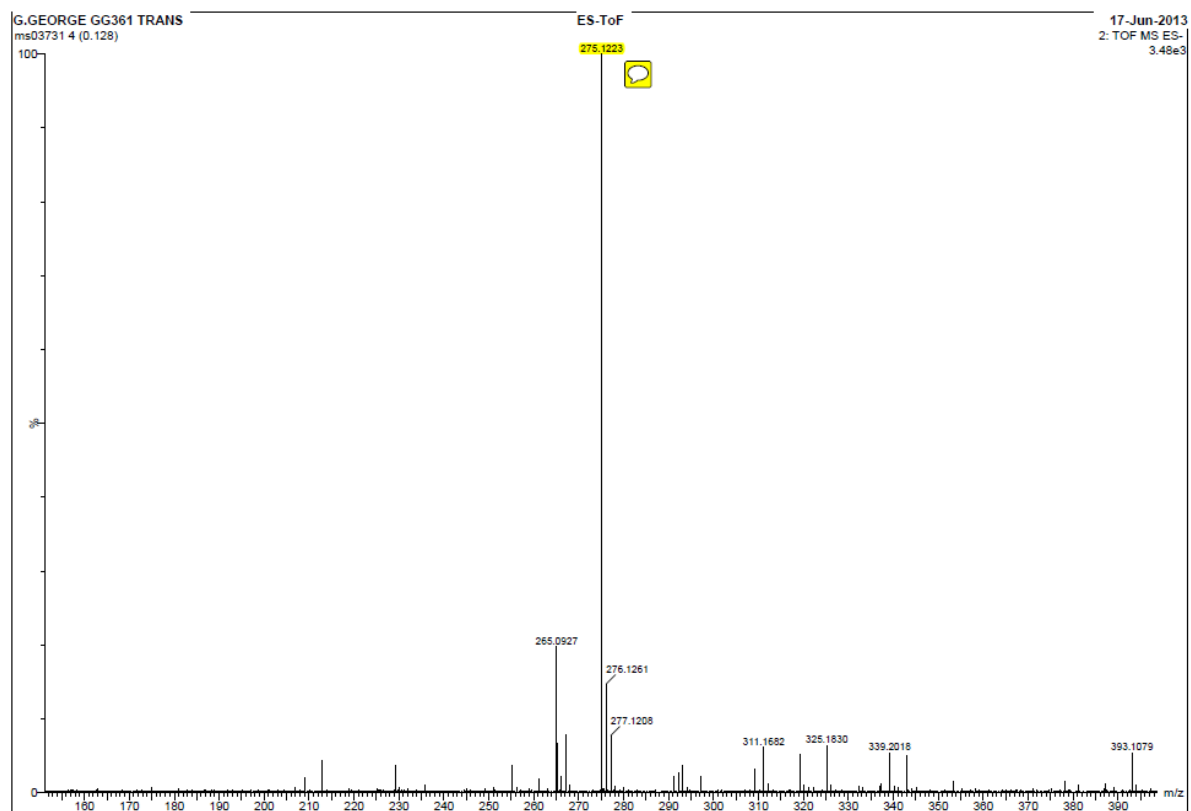


^{13}C NMR spectrum



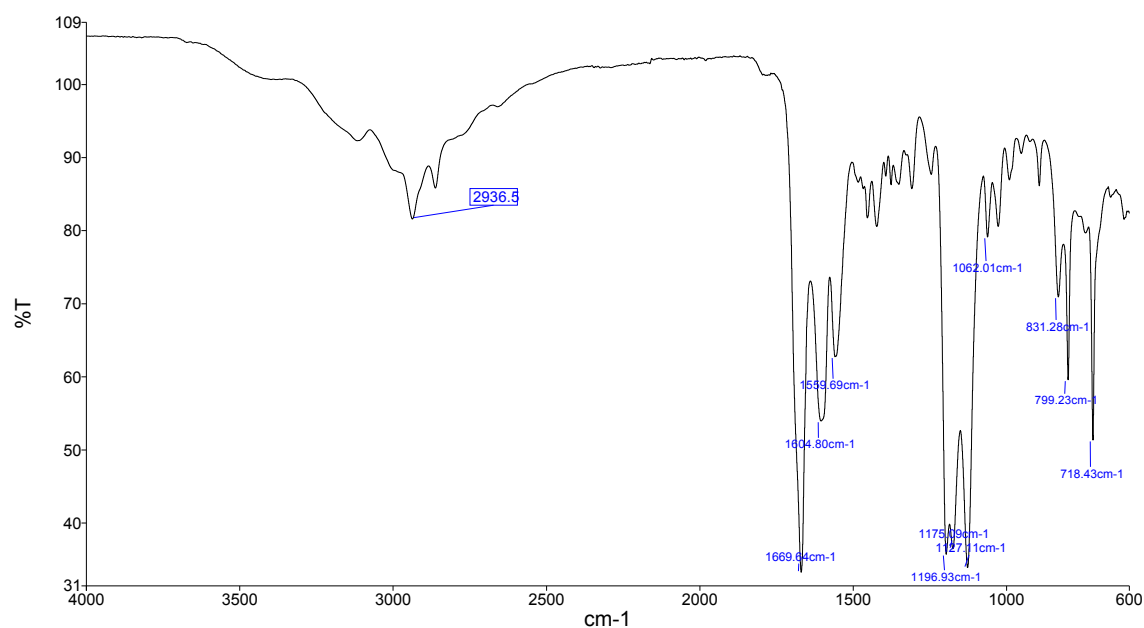
^{19}F NMR spectrum



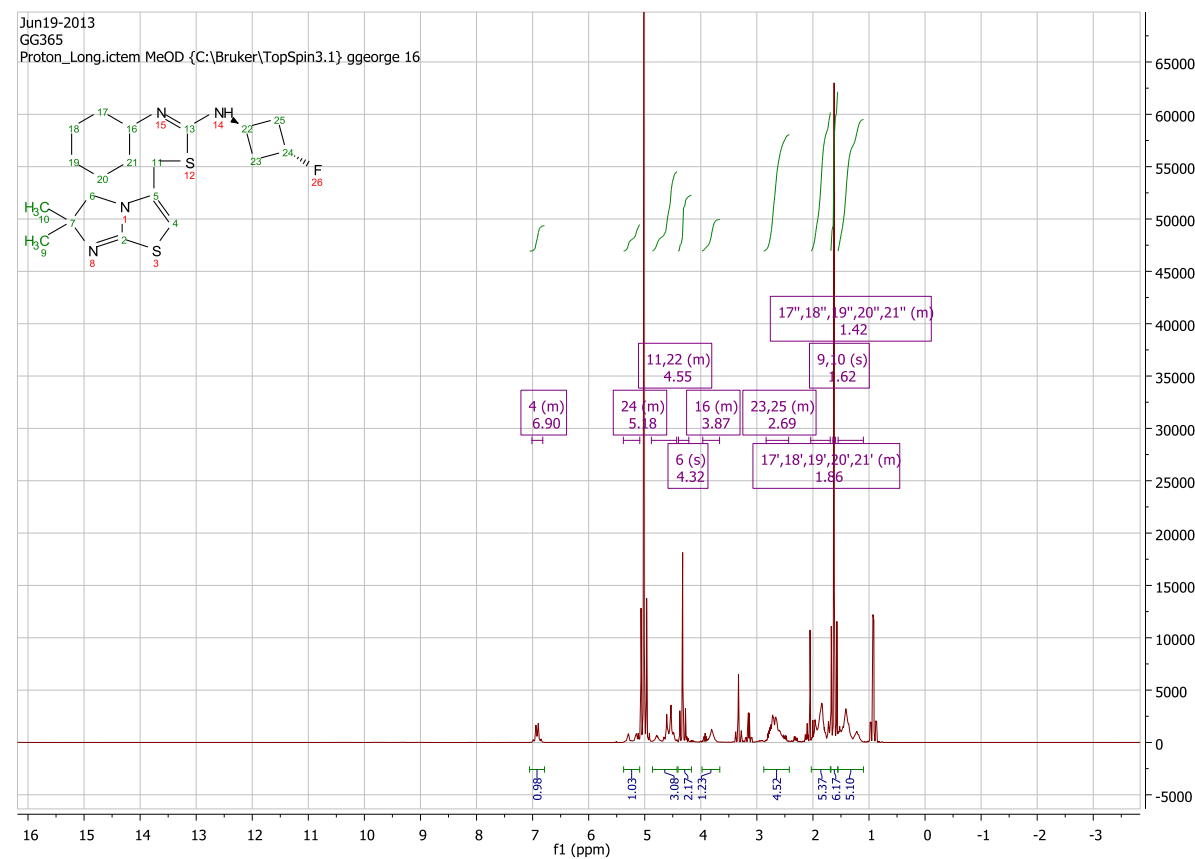


99. *N*'-Cyclohexyl-*N*-(*trans*-3-fluorocyclobutyl)[({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl}methyl)sulfanyl]methanimidamide .3TFA

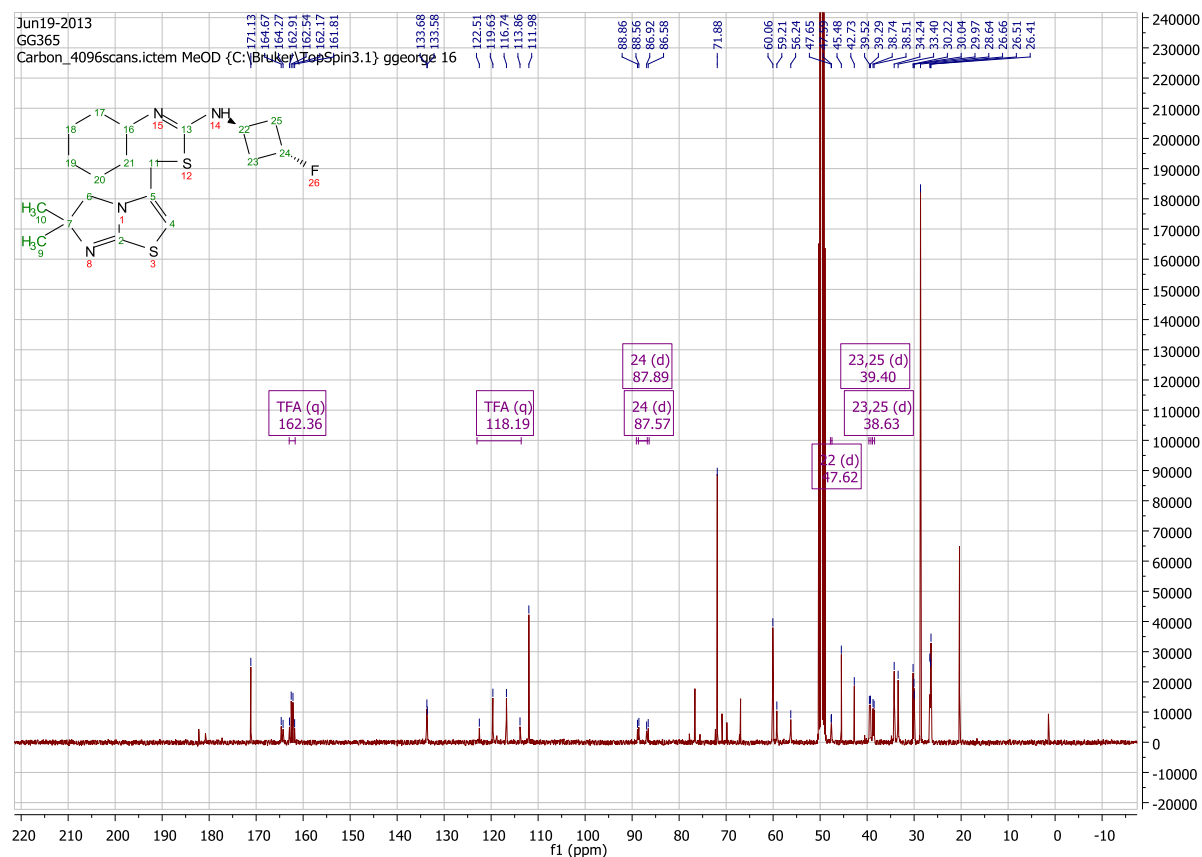
Infrared spectrum



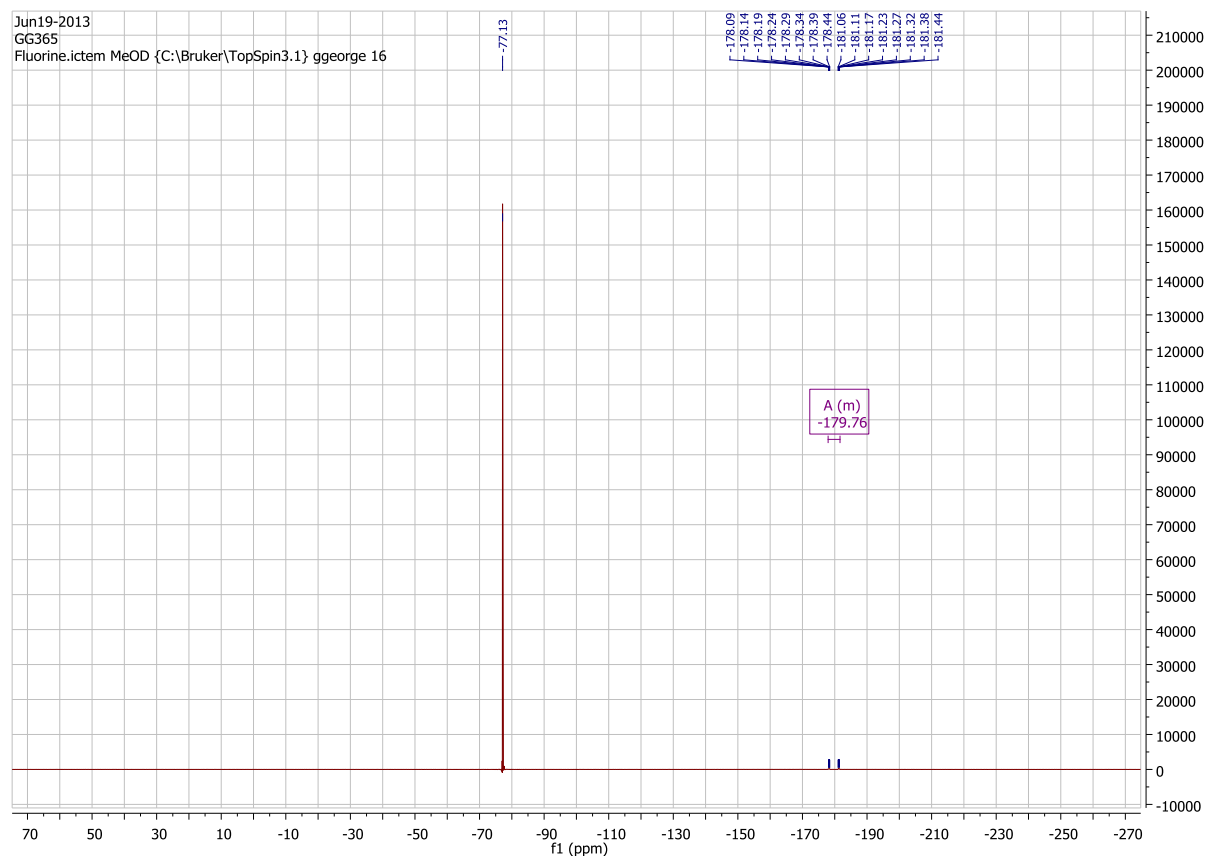
¹H NMR spectrum



^{13}C NMR spectrum

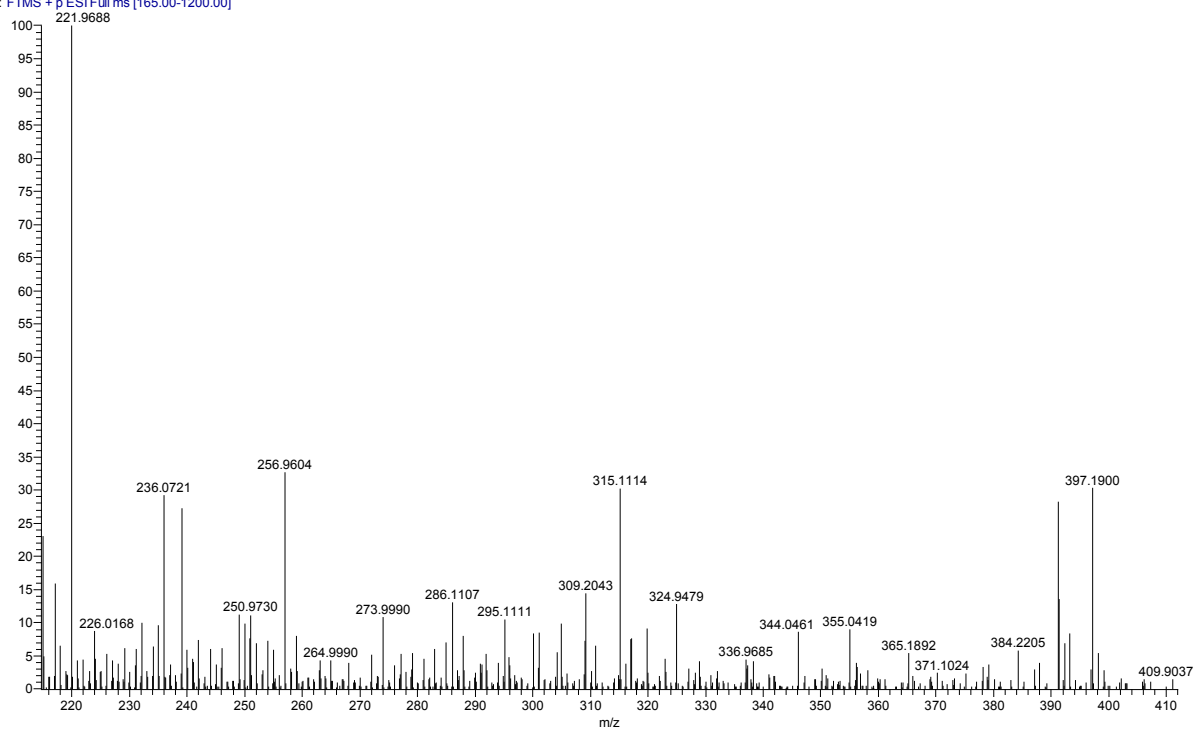


^{19}F NMR spectrum

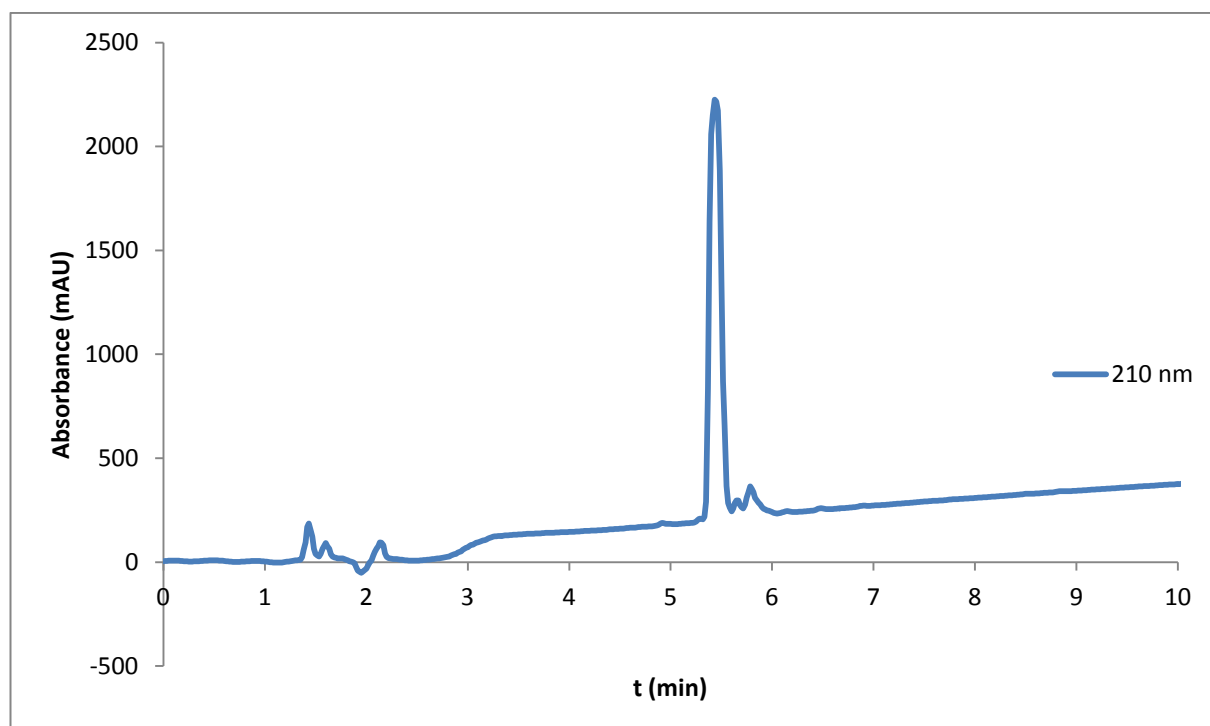


HRMS

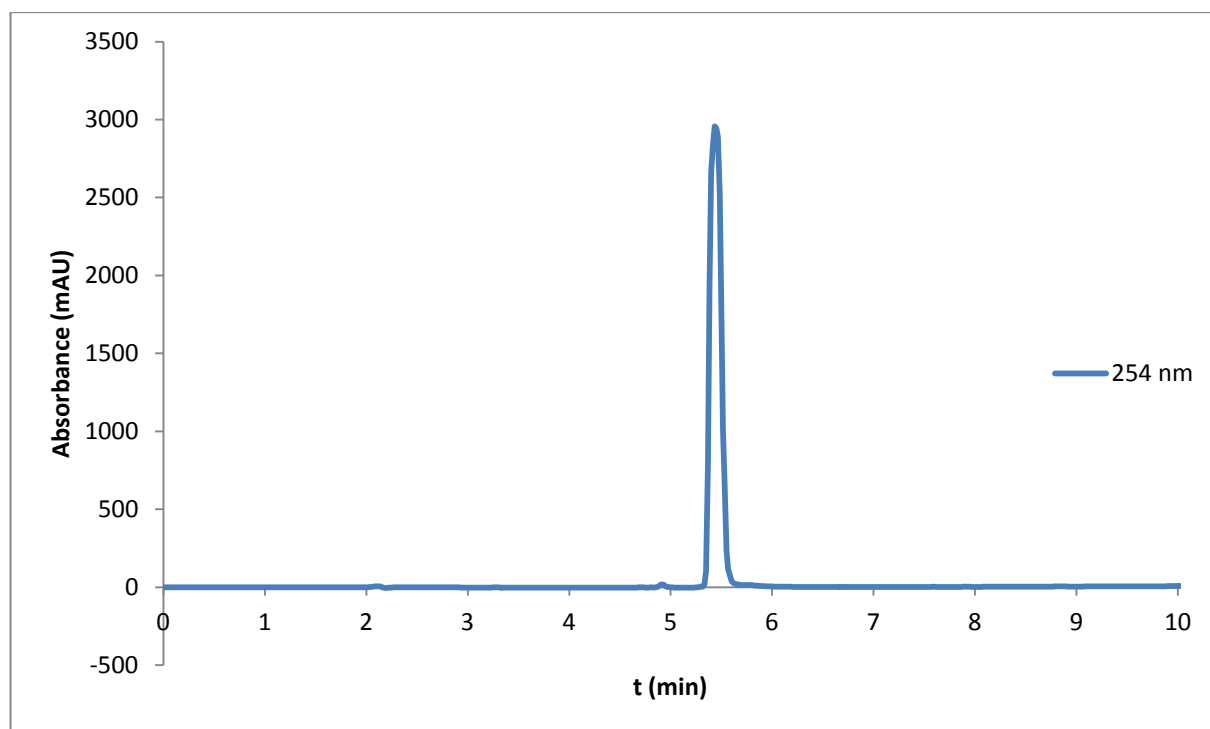
MS03873_130621110405#285 RT: 1.28 AV: 1 NL: 2.95E7
T: FTMS +pESI Full ms [165.00-1200.00]



HPLC trace at 210 nm

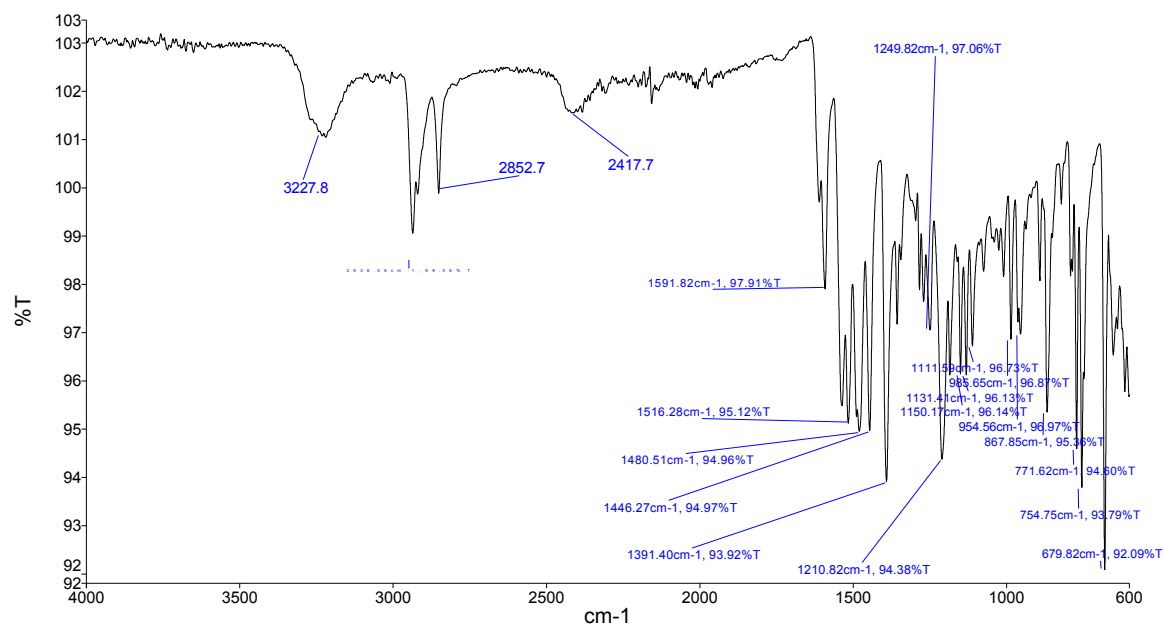


HPLC trace at 254 nm

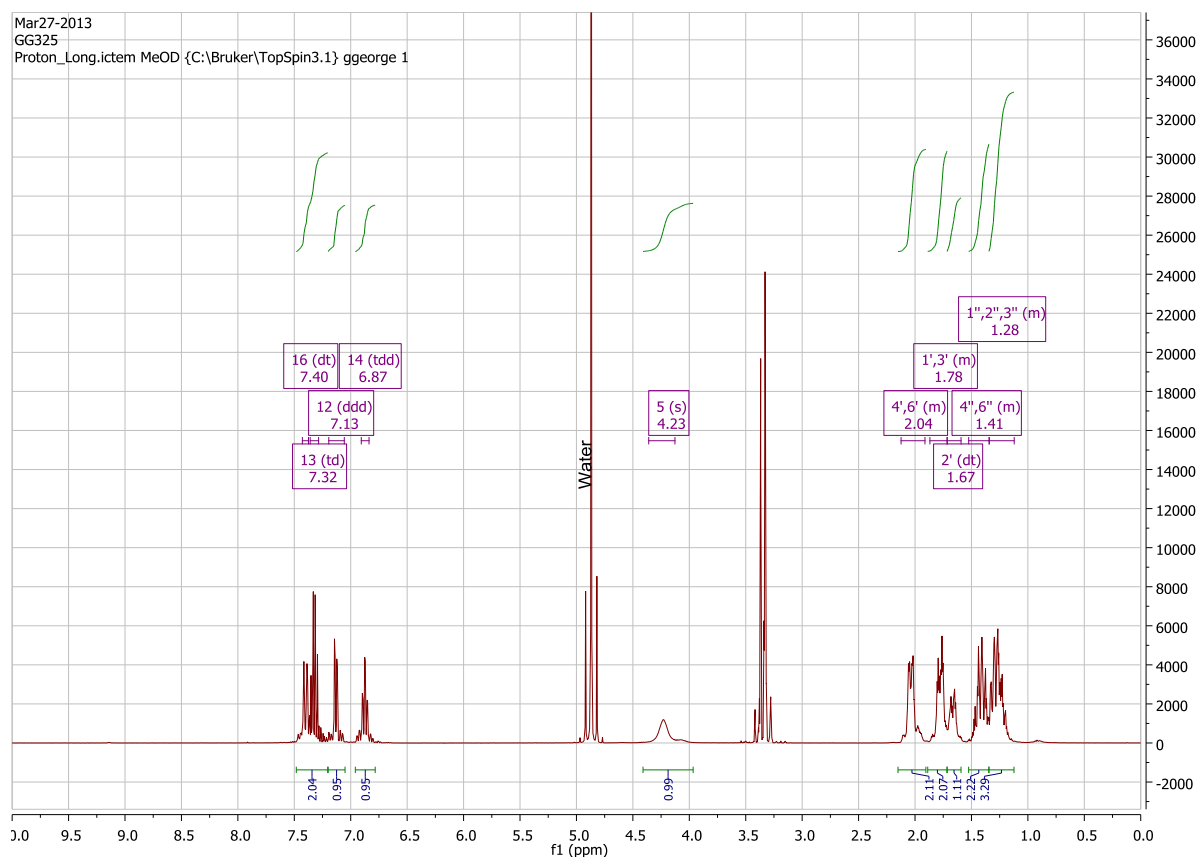


104. 3-(Cyclohexyl)-1-(3-fluorophenyl)thiourea

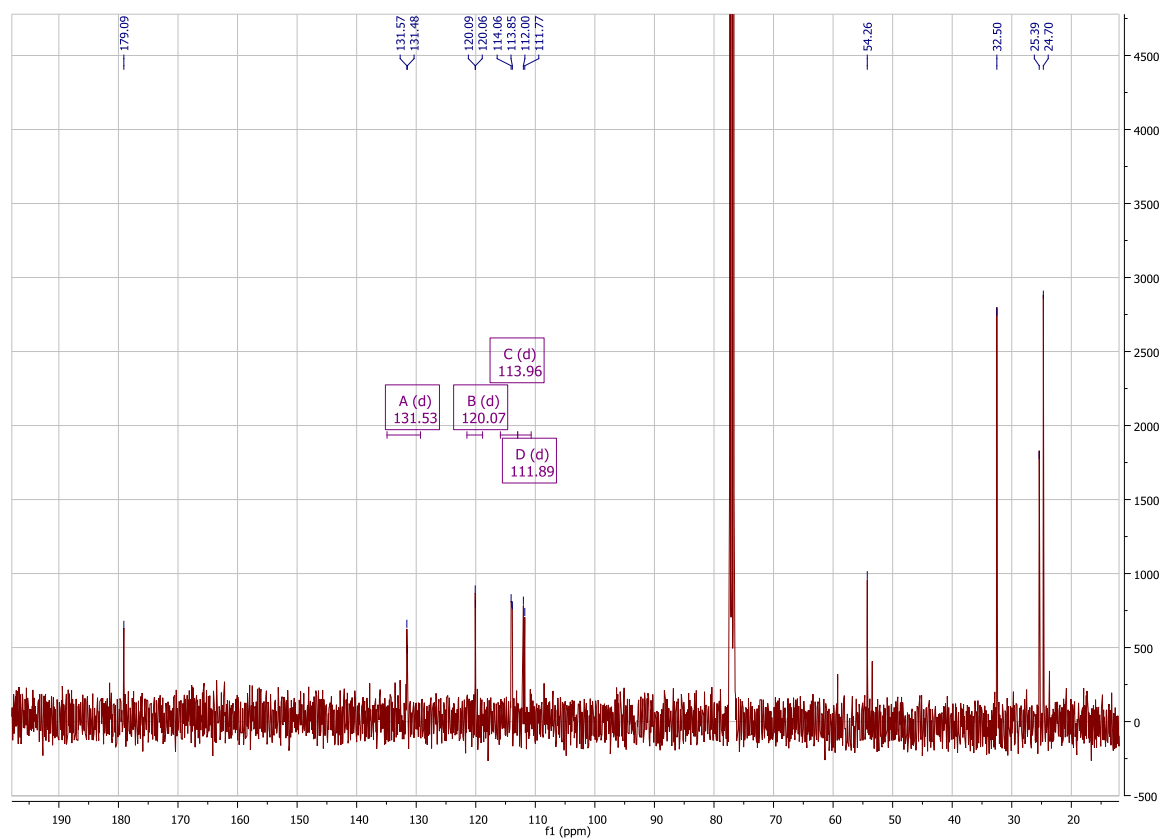
Infrared spectrum



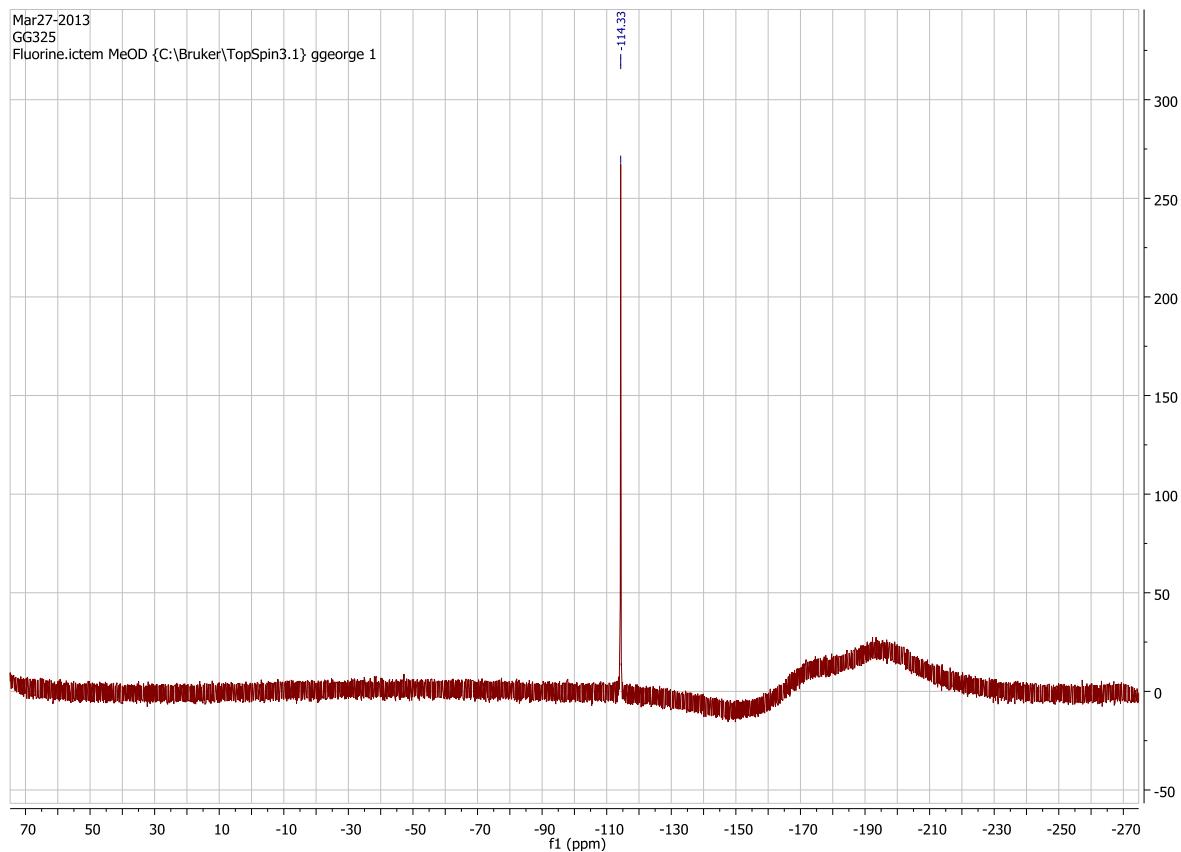
¹H NMR spectrum

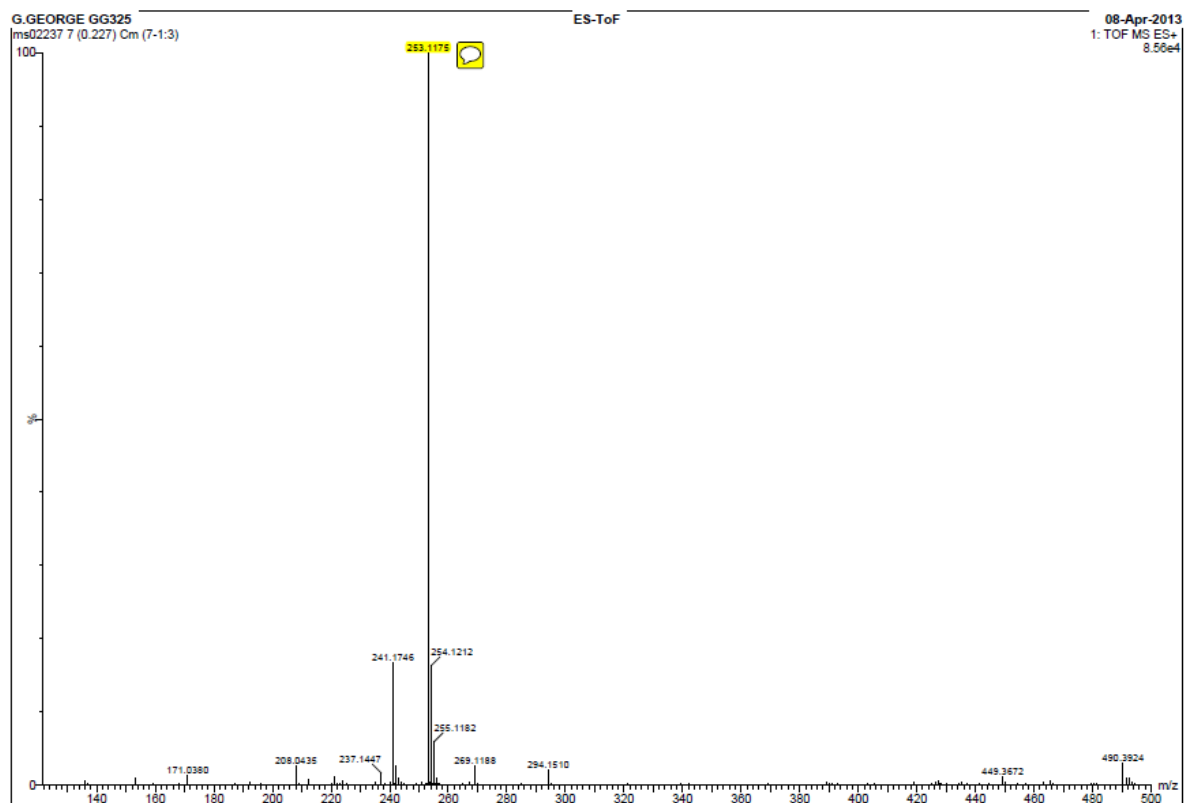


^{13}C NMR spectrum



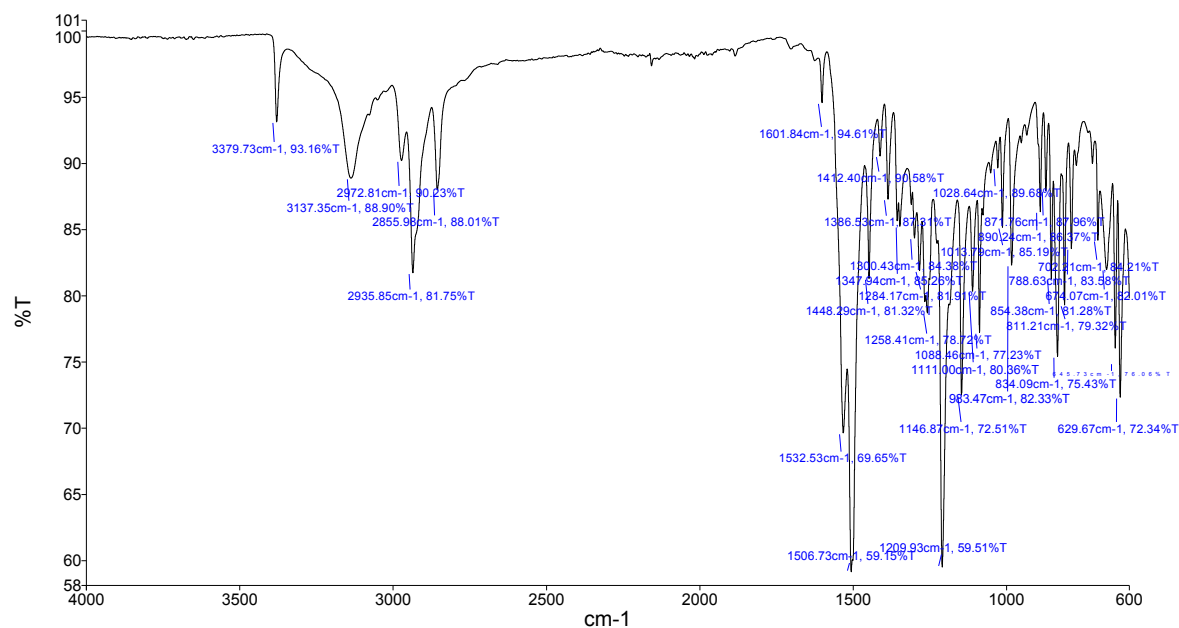
^{19}F NMR spectrum



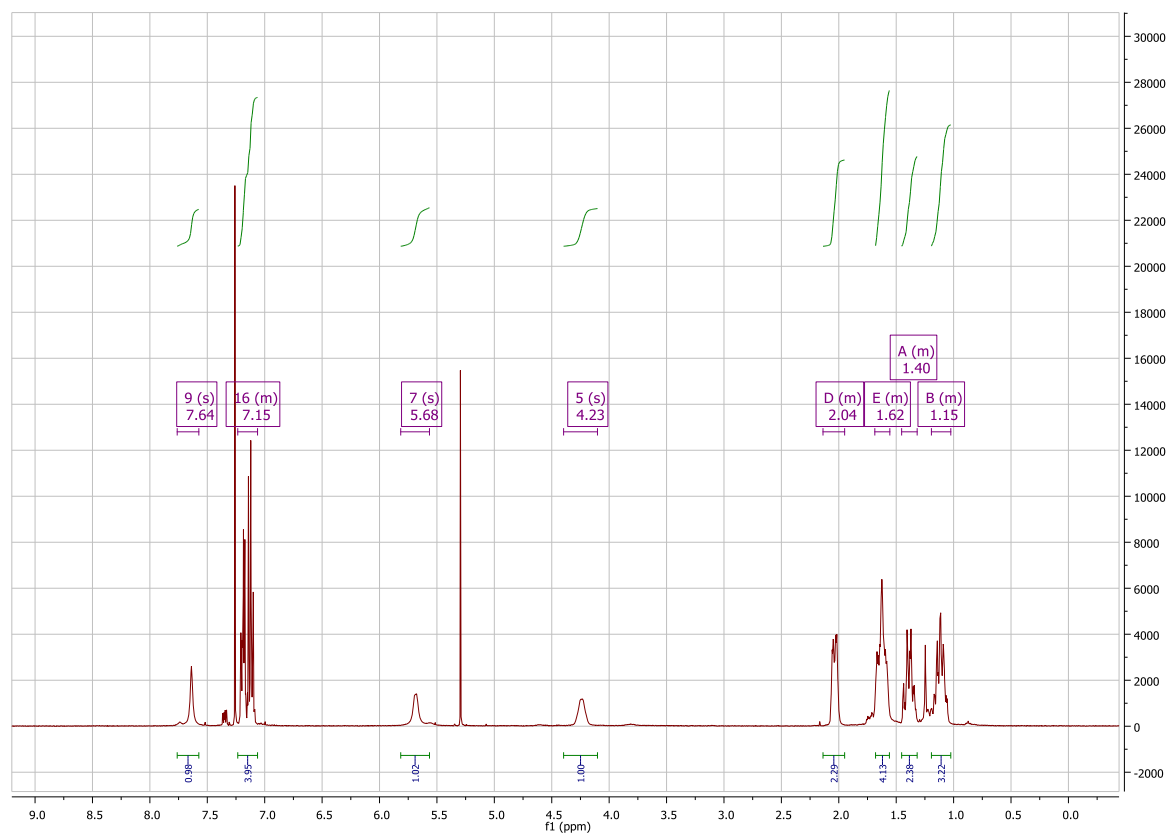


105. 3-(Cyclohexyl)-1-(4-fluorophenyl)thiourea

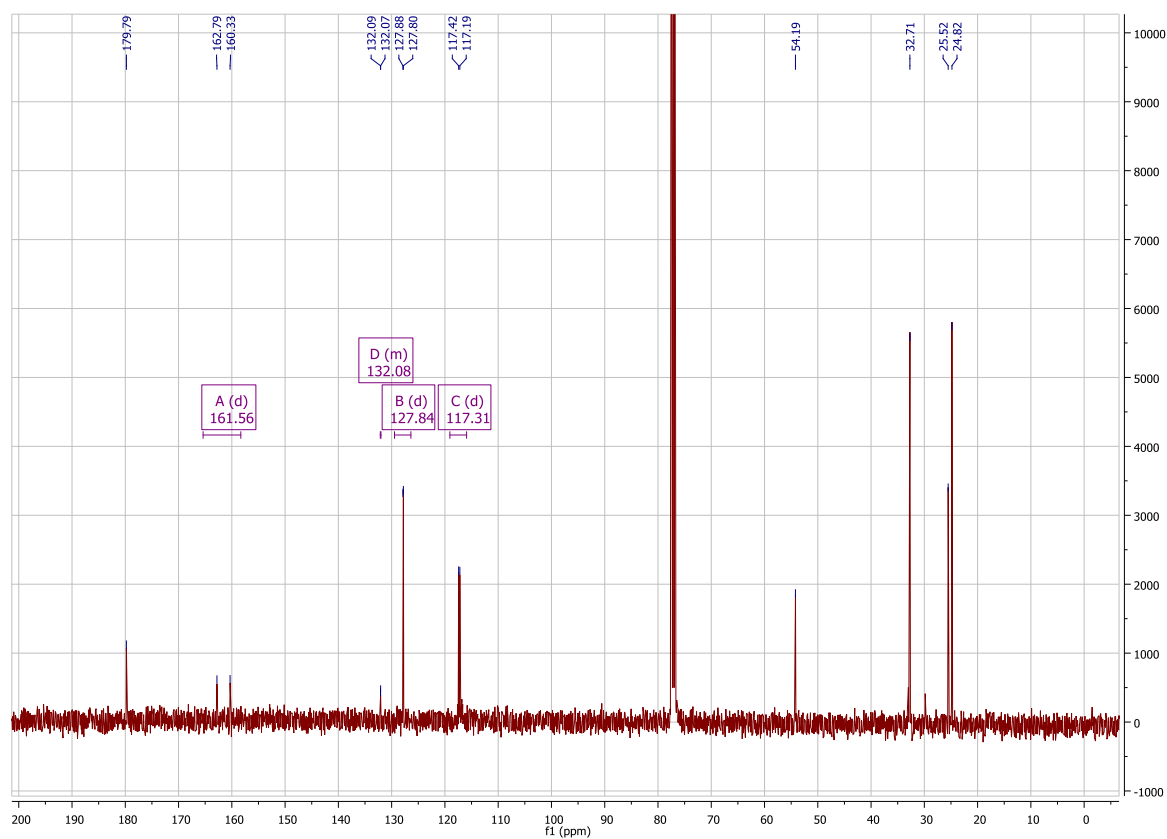
Infrared spectrum



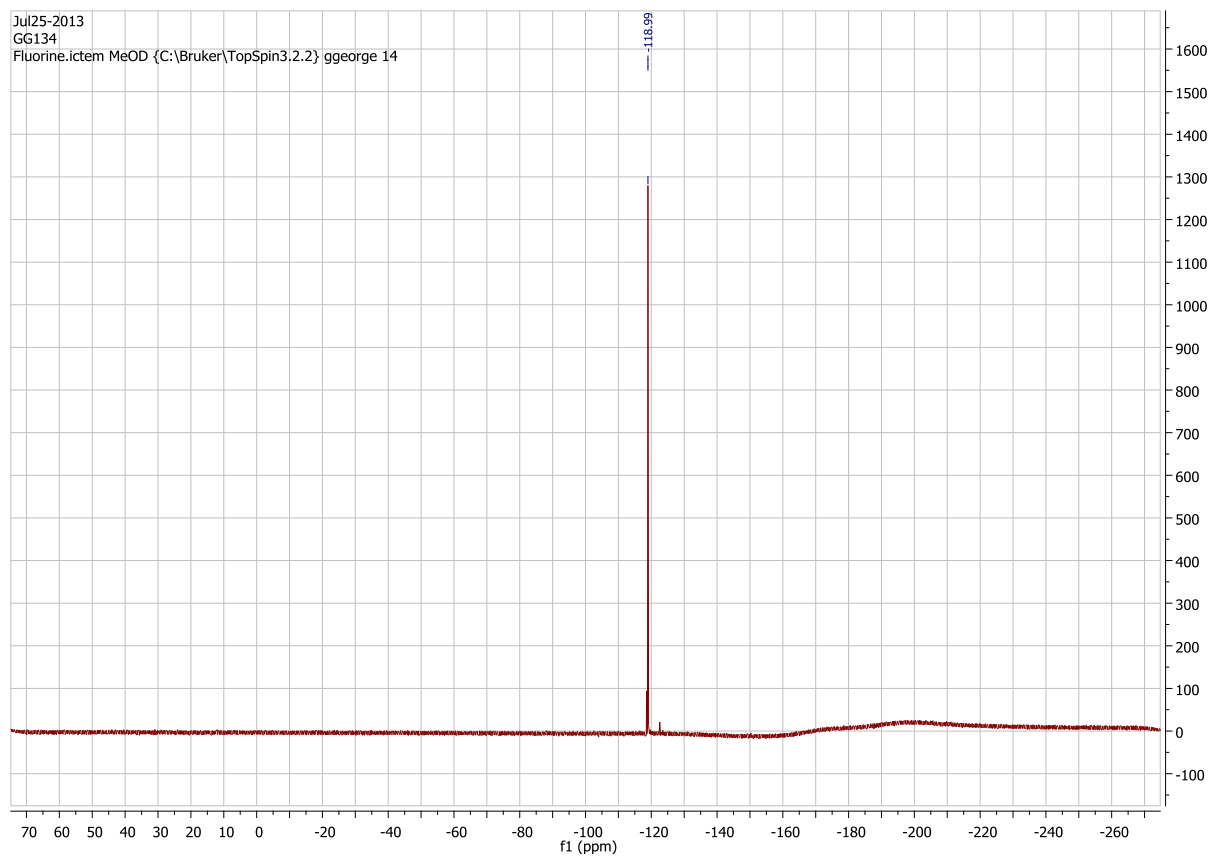
^1H NMR spectrum

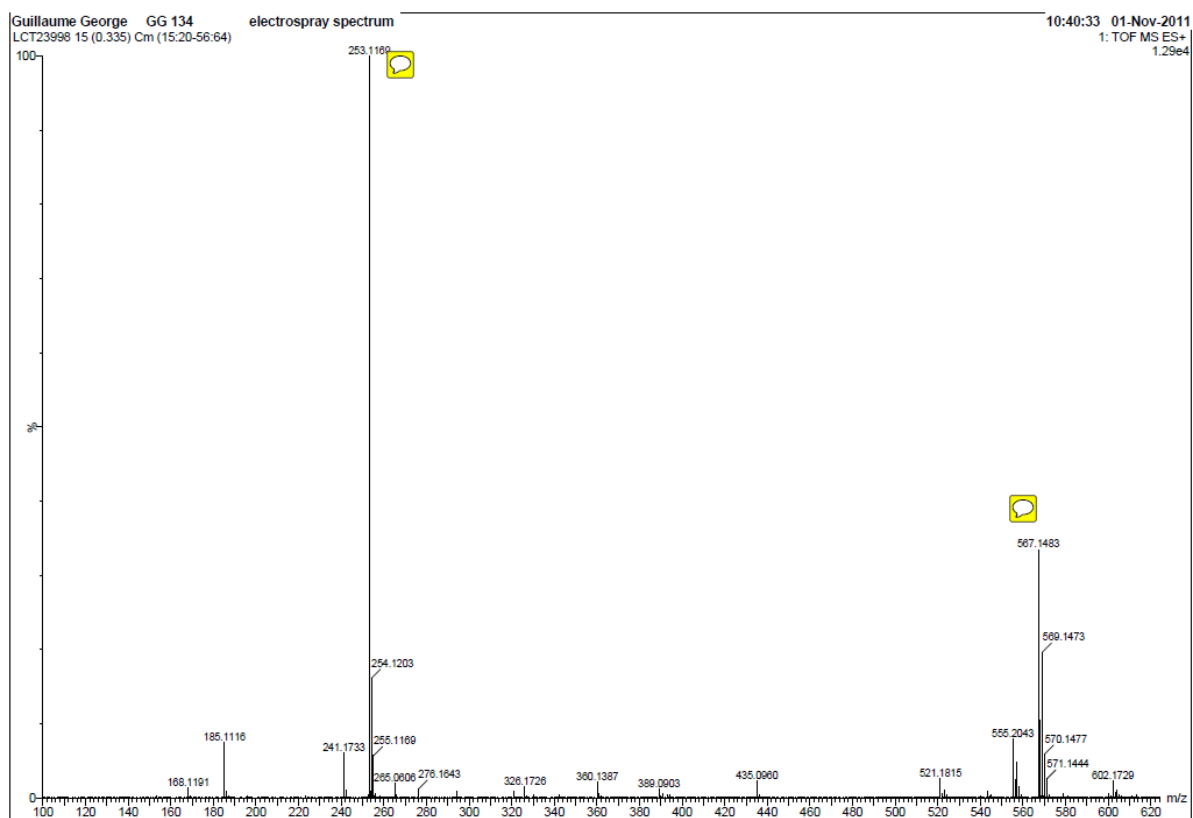


^{13}C NMR spectrum



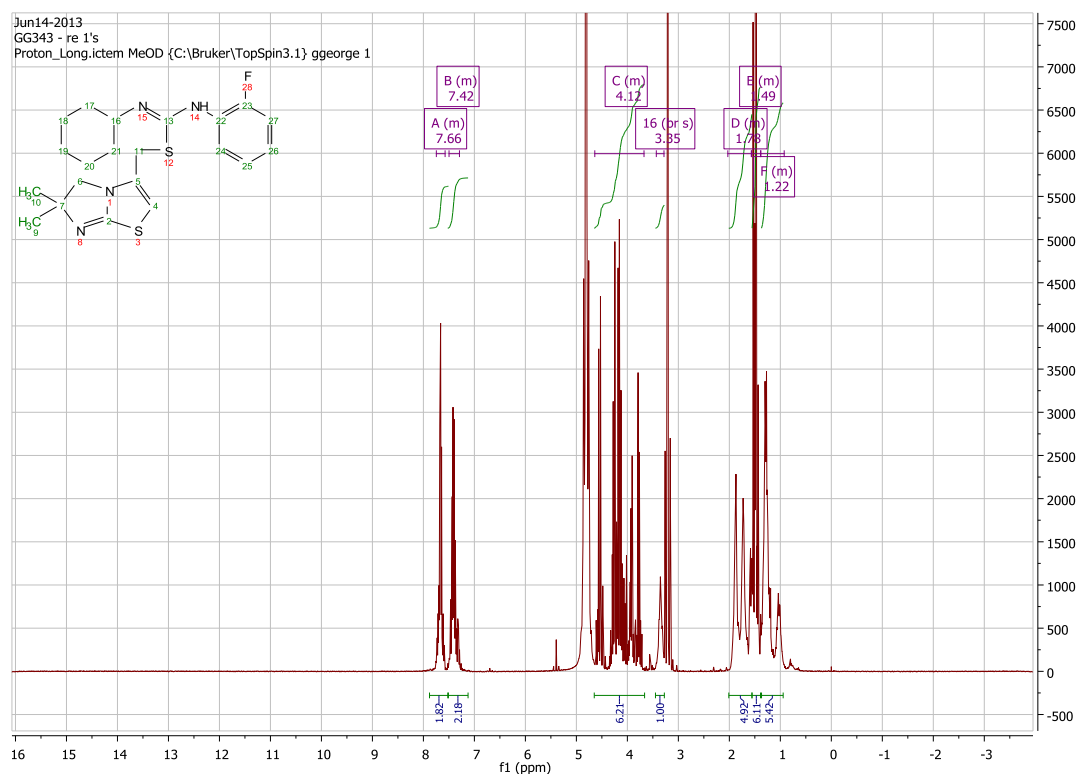
^{19}F NMR spectrum



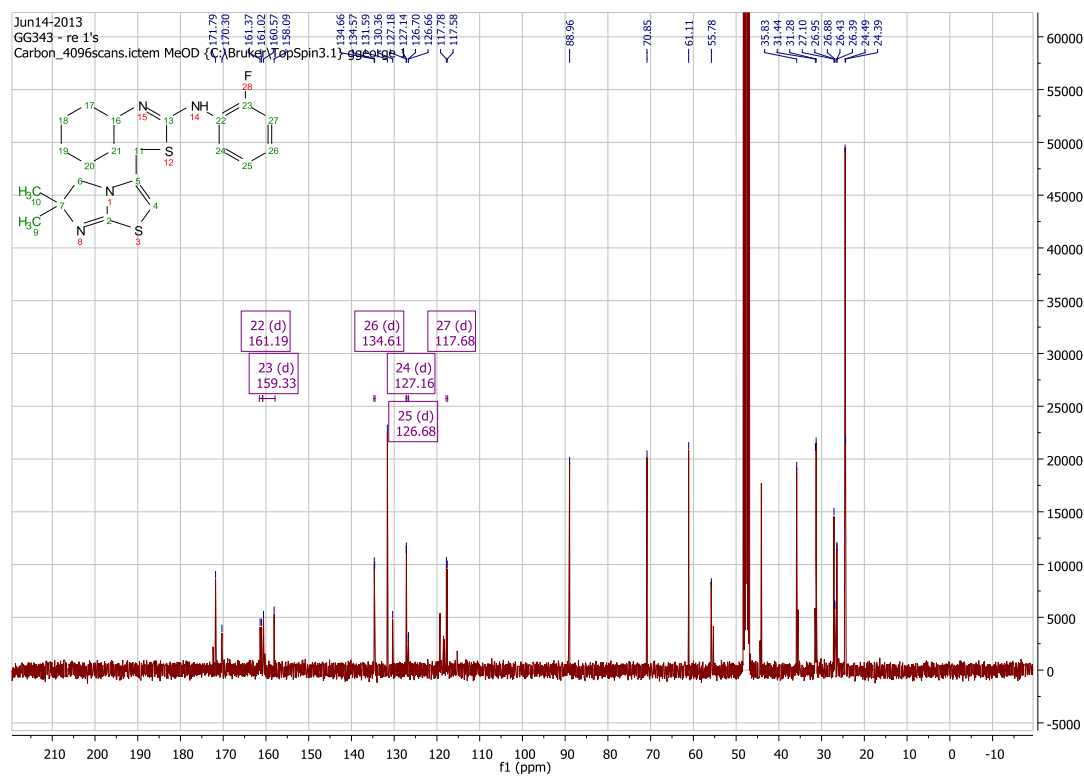


106. *N*³-Cyclohexyl-*N*-(2-fluorophenyl)[({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidamide .3TFA

¹H NMR spectrum



¹³C NMR spectrum



Jun14-2013
GG343 - re 1's
Fluorine.ictem MeOD {C:\Bruker\TopSpin3.1} ggeorge 1

The figure displays an NMR spectrum of compound 1 in MeOD. The main spectrum shows a sharp peak at -76.89 ppm and a multiplet at -122.10 ppm. An inset zooms in on the -122.10 ppm region, showing a complex multiplet with peaks labeled at -122.07, -122.08, -122.10, -122.11, and -122.13 ppm. The inset also shows a fit labeled 'A (dt) -122.10' and a fit labeled 'A (ddd) -122.10'.

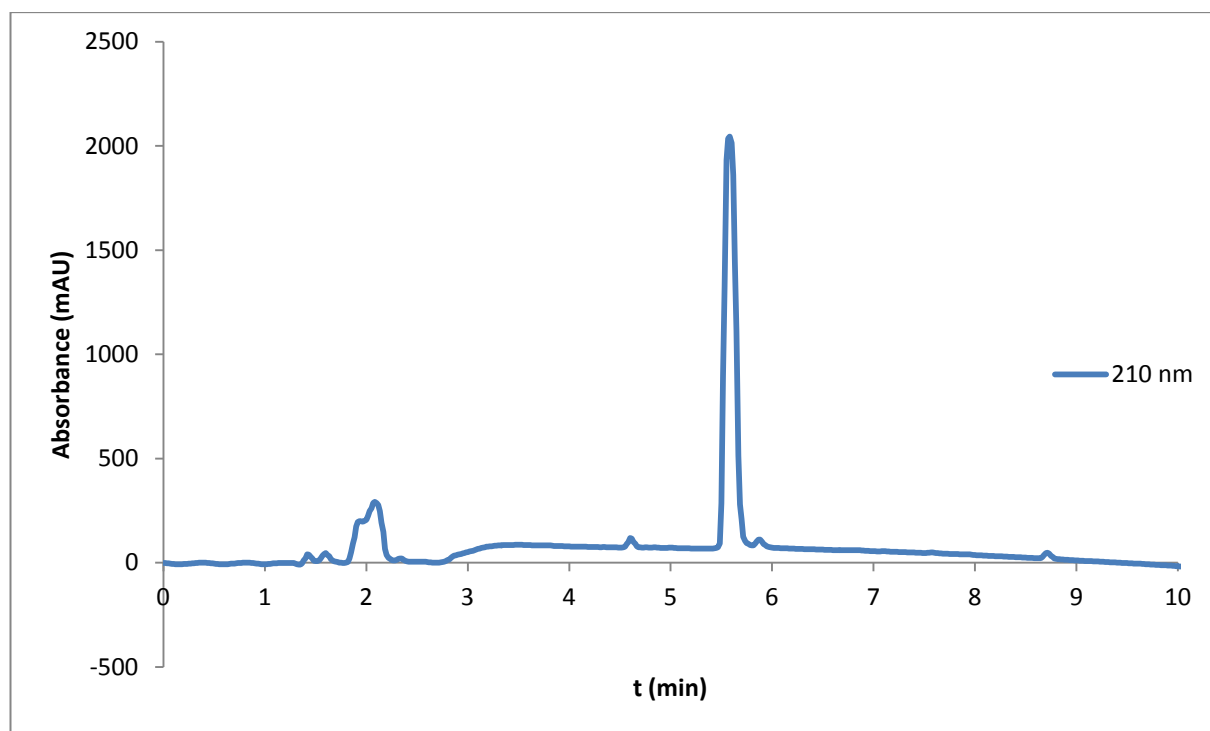
Guillaume George GG 147 electrospray spectrum
LCT24208 24 (0.533) Cm (24:30-73:81)

10:36:20 11-Nov-2011
1: TOF MS ES+
7.61e3

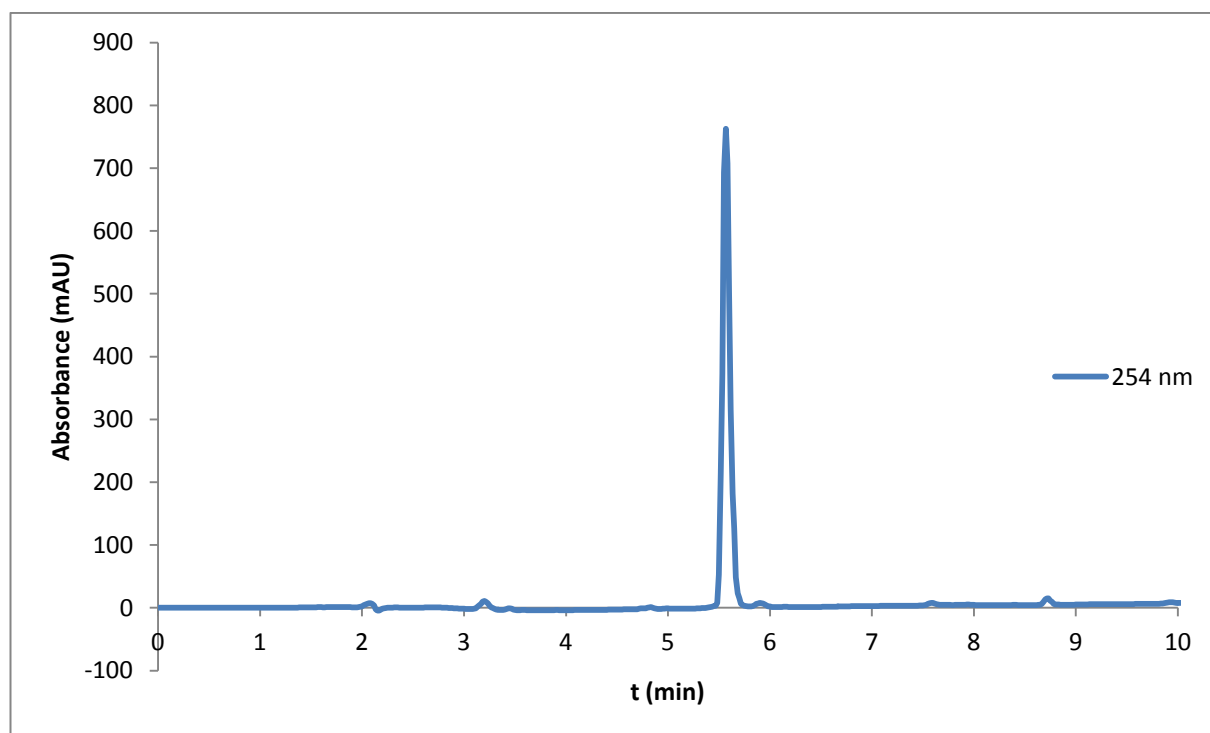
Mass spectrum plot showing relative intensity (0 to 100) versus m/z (100 to 460). The base peak is at m/z 419.1725. Other significant peaks are labeled with their m/z values.

m/z	Relative Intensity (approx)
153.0724	5
168.0724	10
185.1097	5
201.0519	15
207.0362	10
230.6020	15
243.0679	10
265.1422	10
289.1180	10
297.1204	40
298.1229	15
299.1168	15
299.1620	10
325.1516	10
337.0948	35
338.0975	10
339.0936	10
369.1782	10
370.1818	10
407.2301	20
419.1725	100
420.1757	25
421.1709	15
422.1726	10
447.2062	5
459.1492	5

HPLC at 210 nm

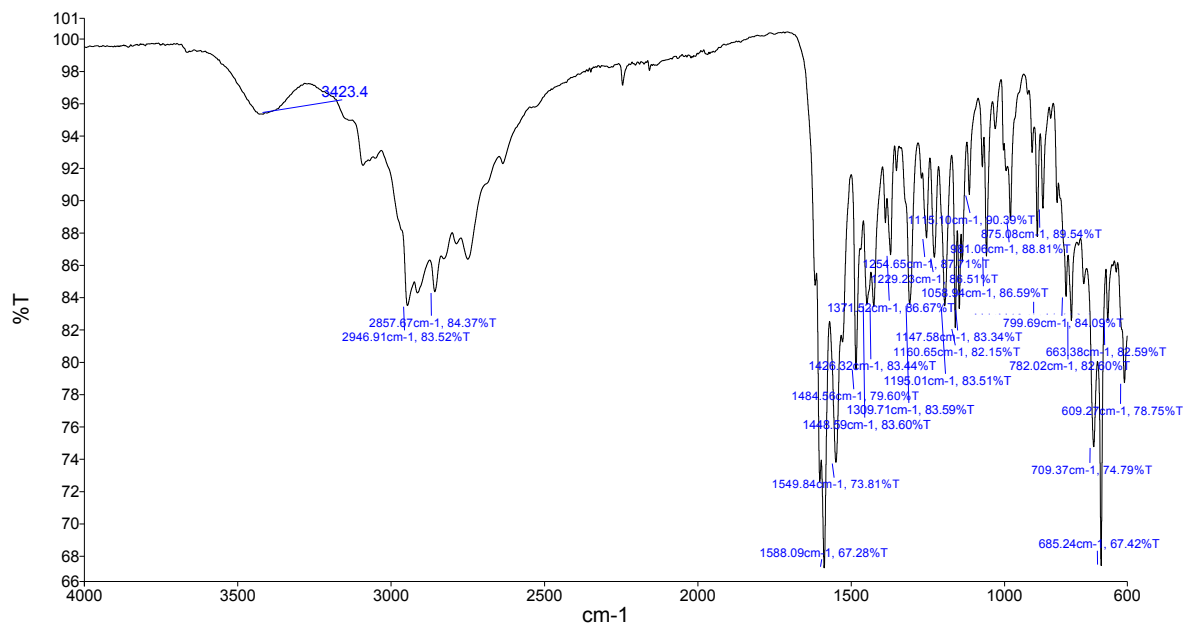
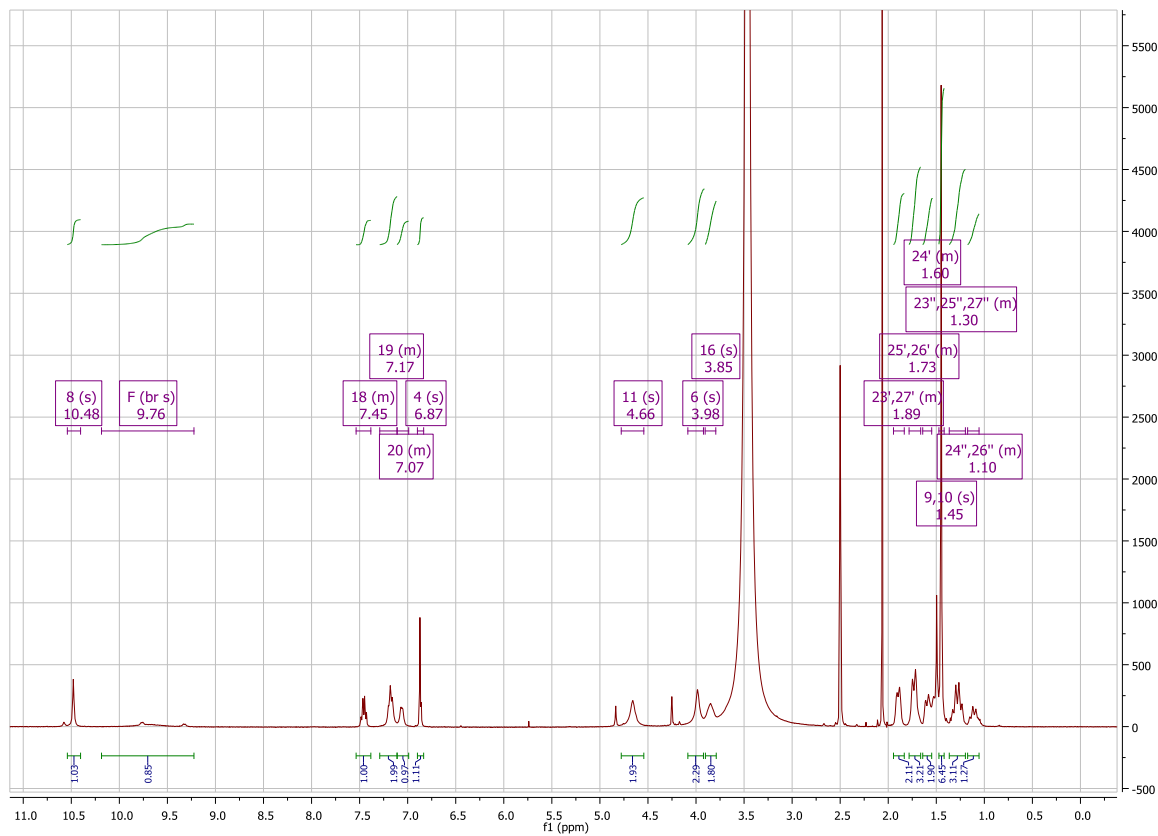


HPLC at 254 nm

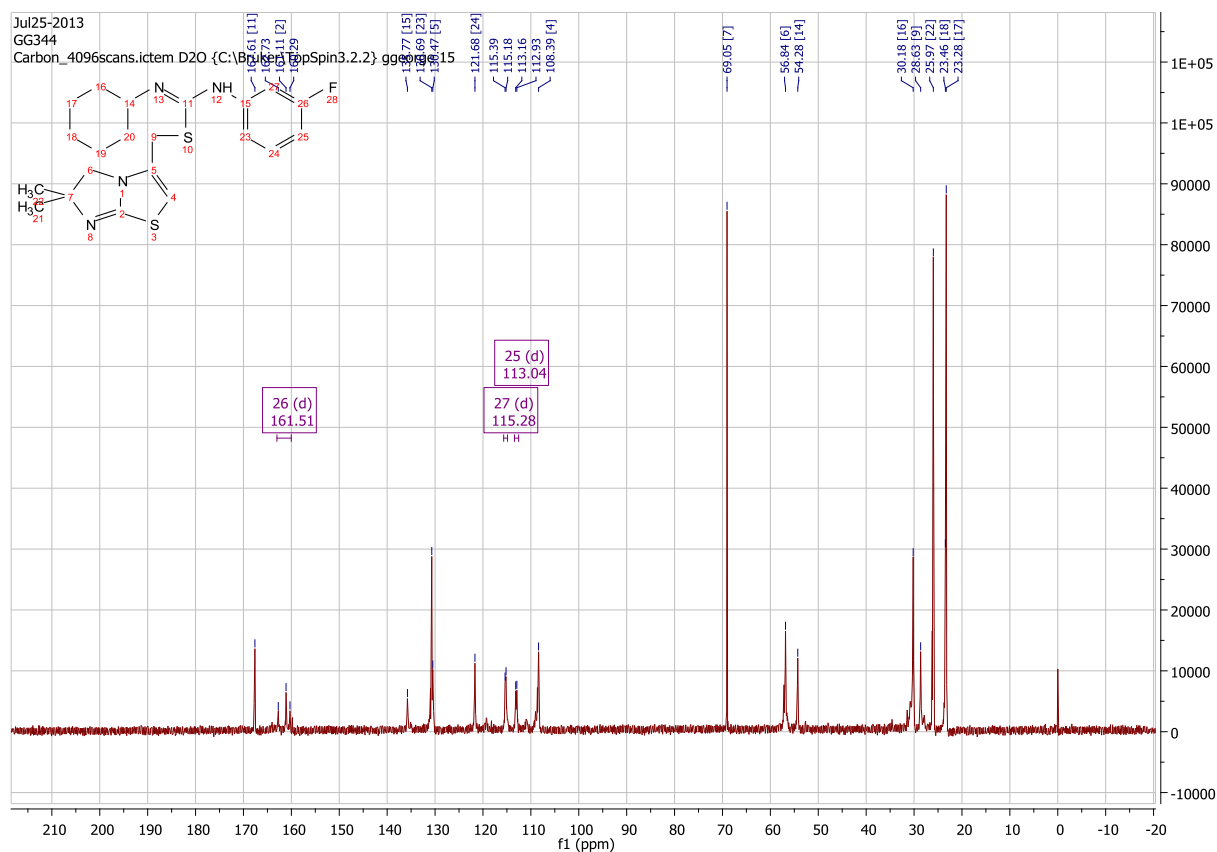


yl)methyl)sulfanyl]methanimidamide dihydrochloride salt

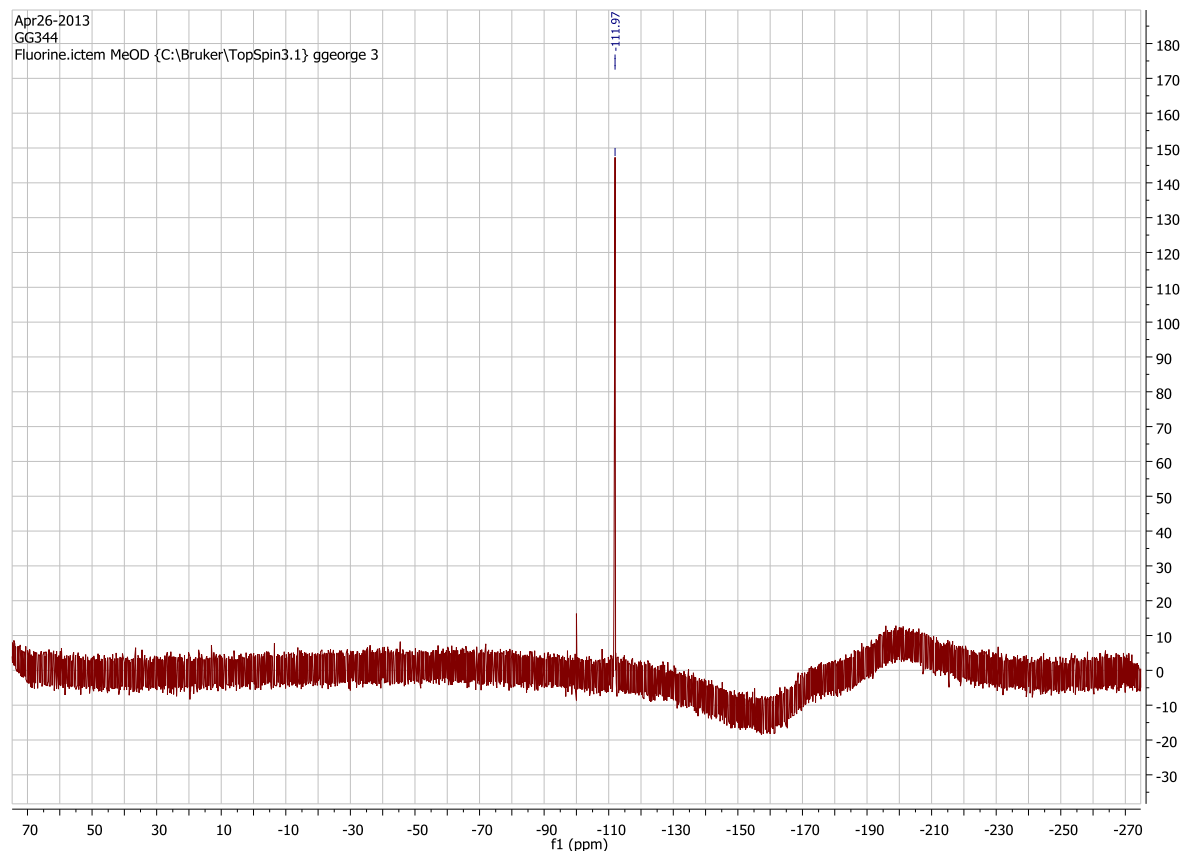
Infrared spectrum

 ^1H NMR spectrum

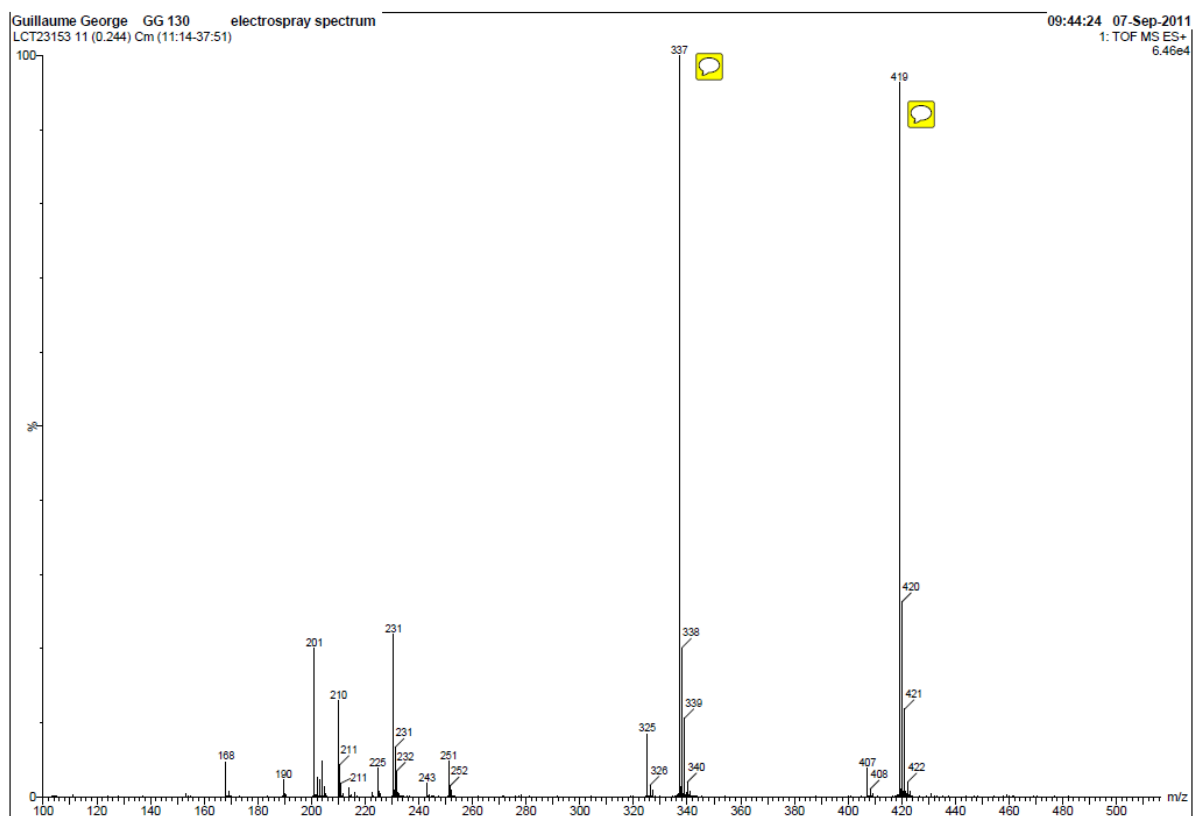
^{13}C NMR spectrum



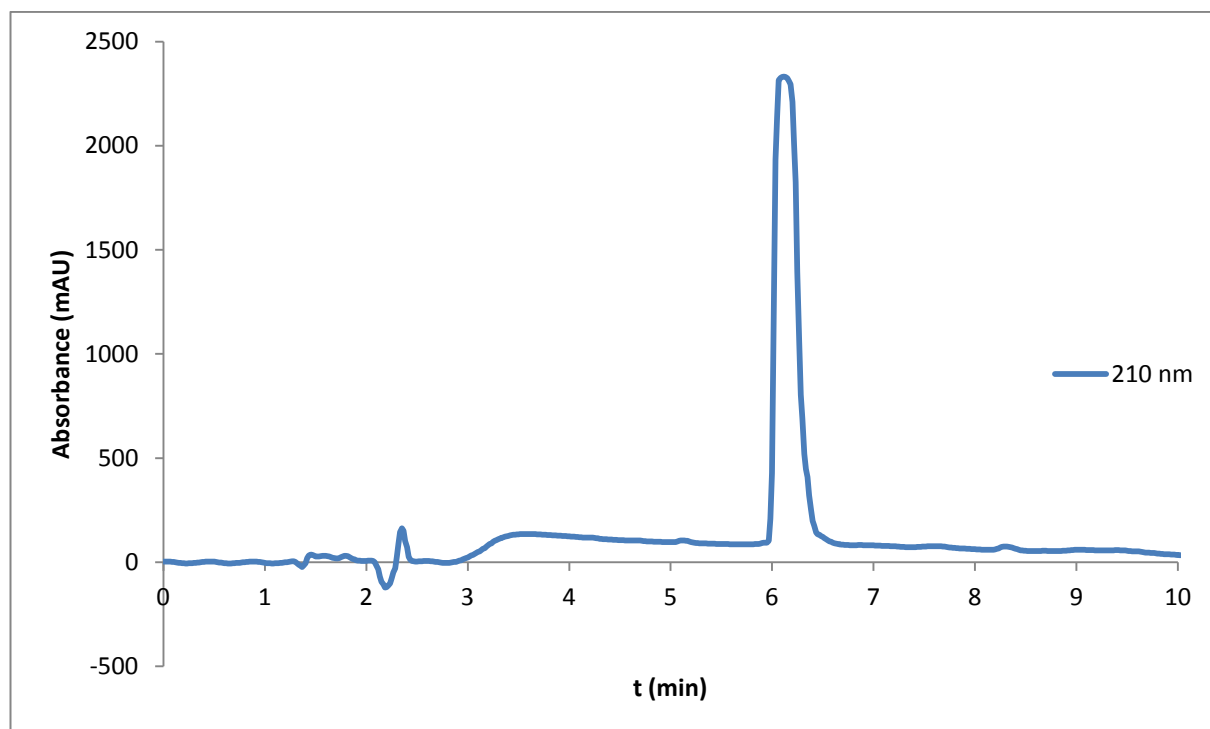
^{19}F NMR spectrum



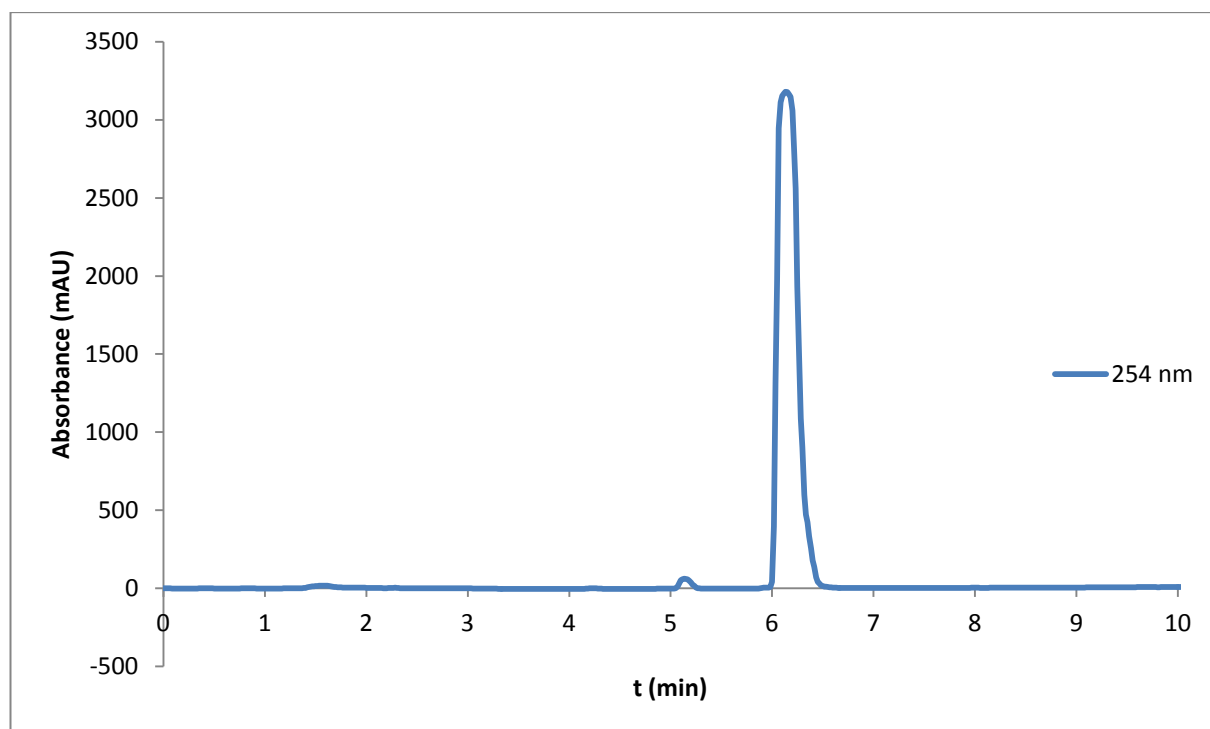
HRMS



HPLC trace at 210 nm

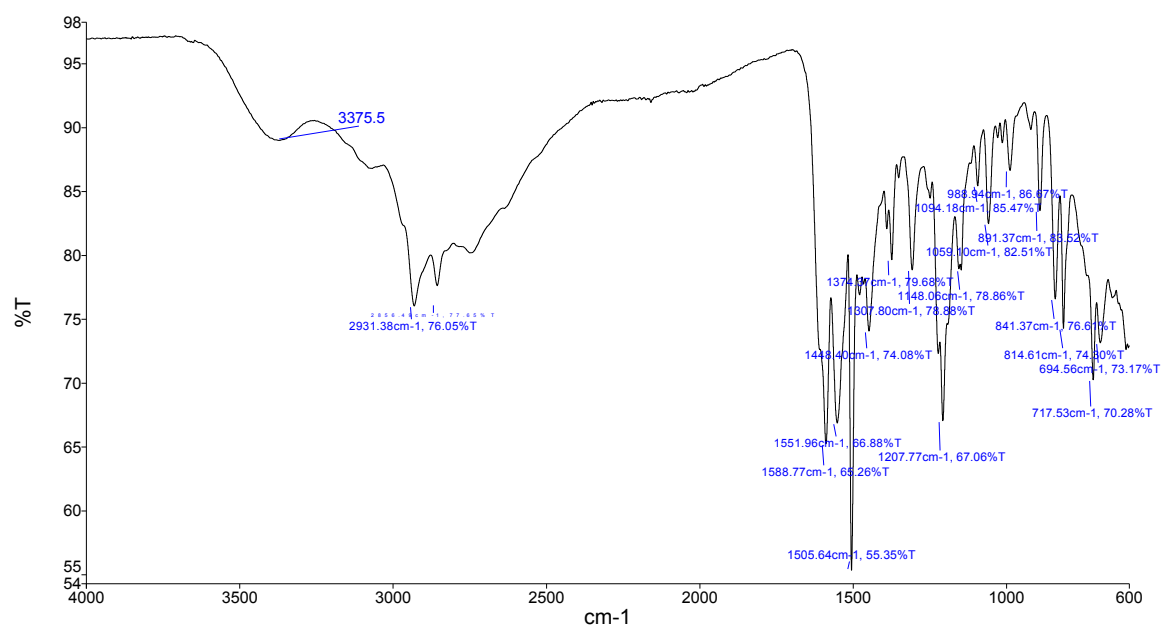


HPLC trace at 254 nm

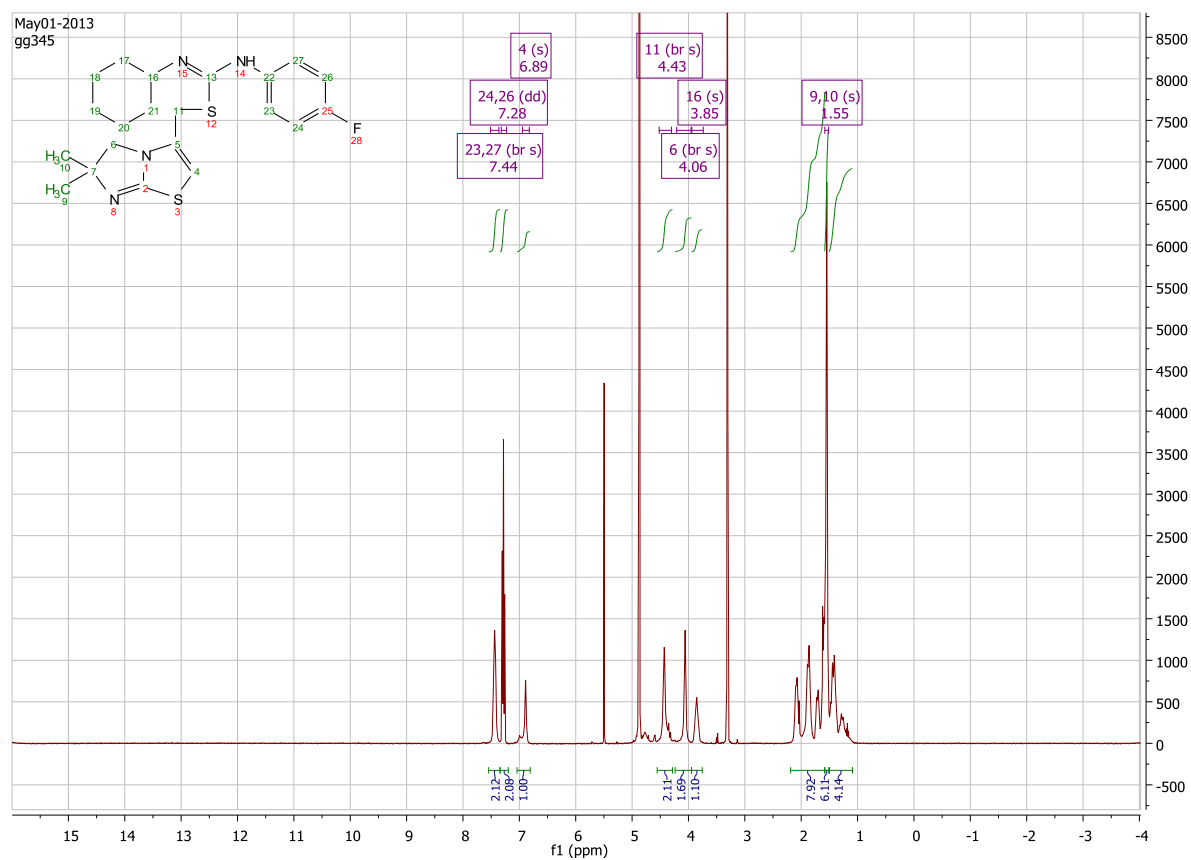


108. *N*'-Cyclohexyl-*N*-(4-fluorophenyl)[({6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidamide dihydrochloride salt

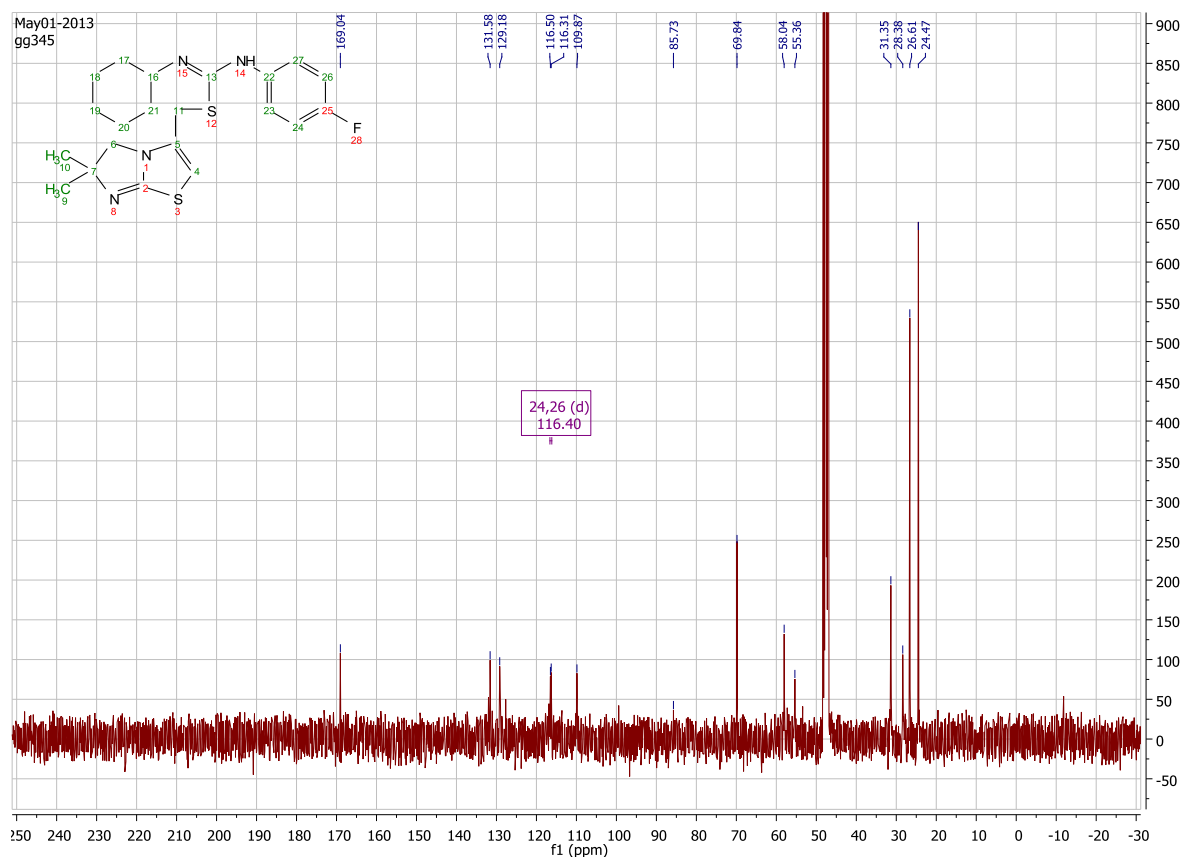
Infrared spectrum



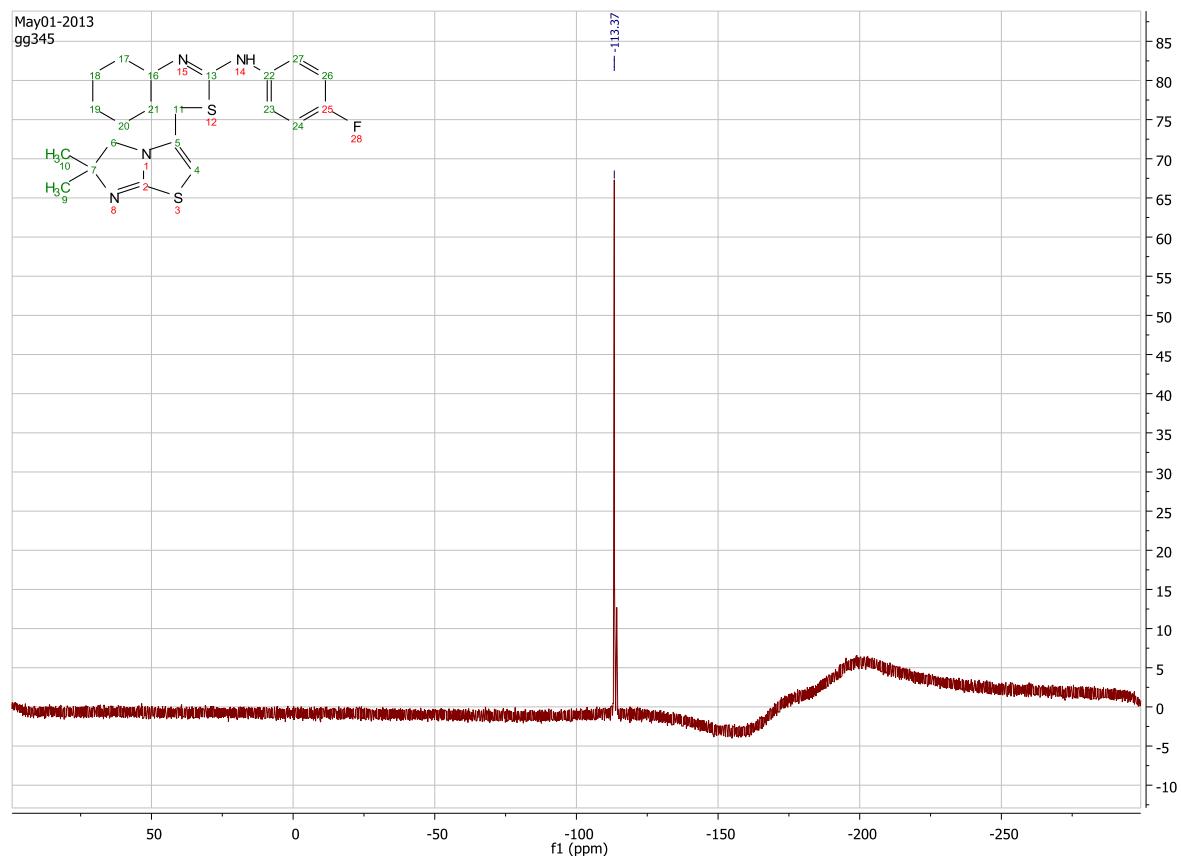
¹H NMR spectrum



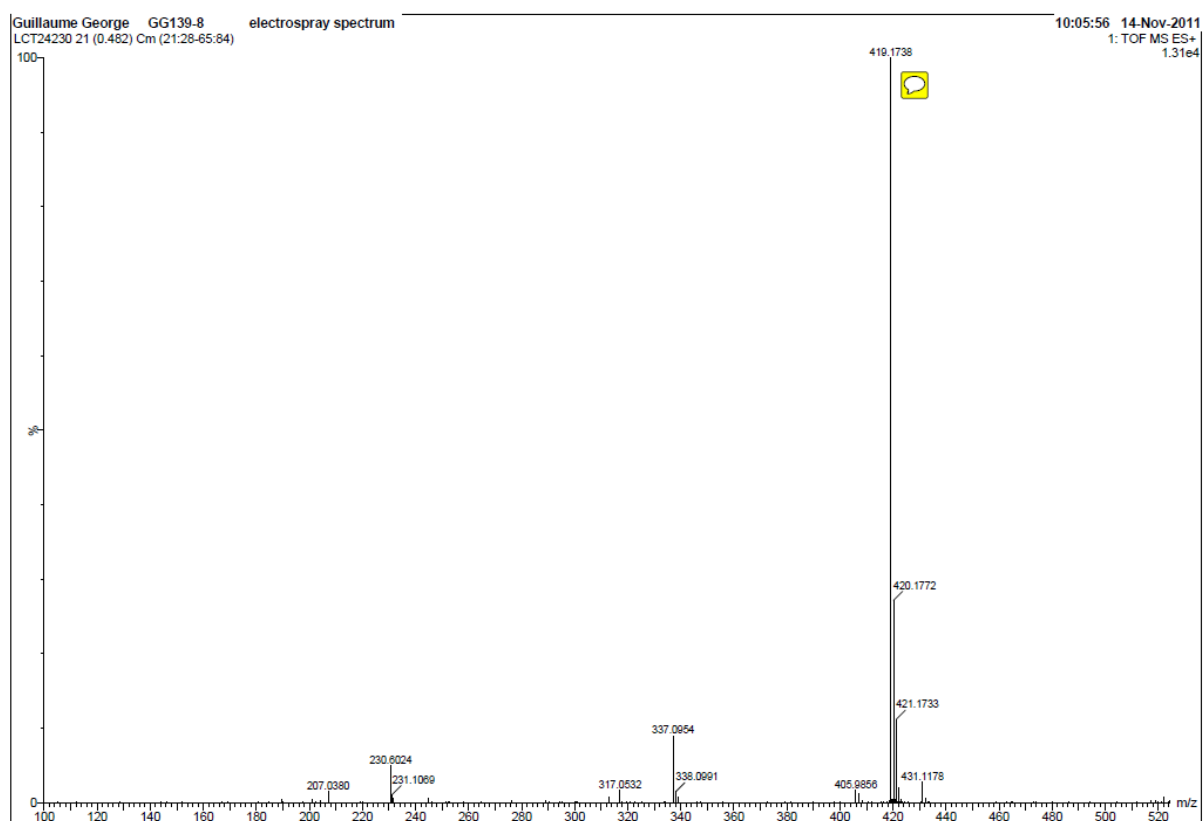
^{13}C NMR spectrum



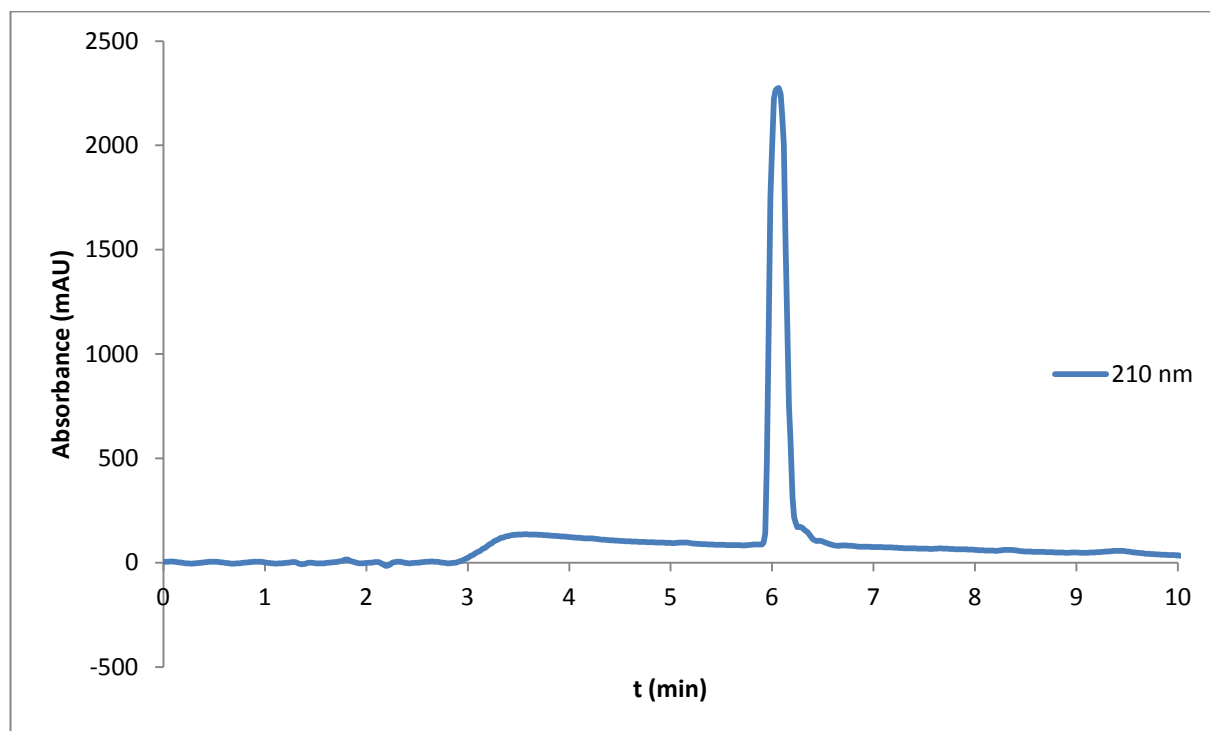
^{19}F NMR spectrum



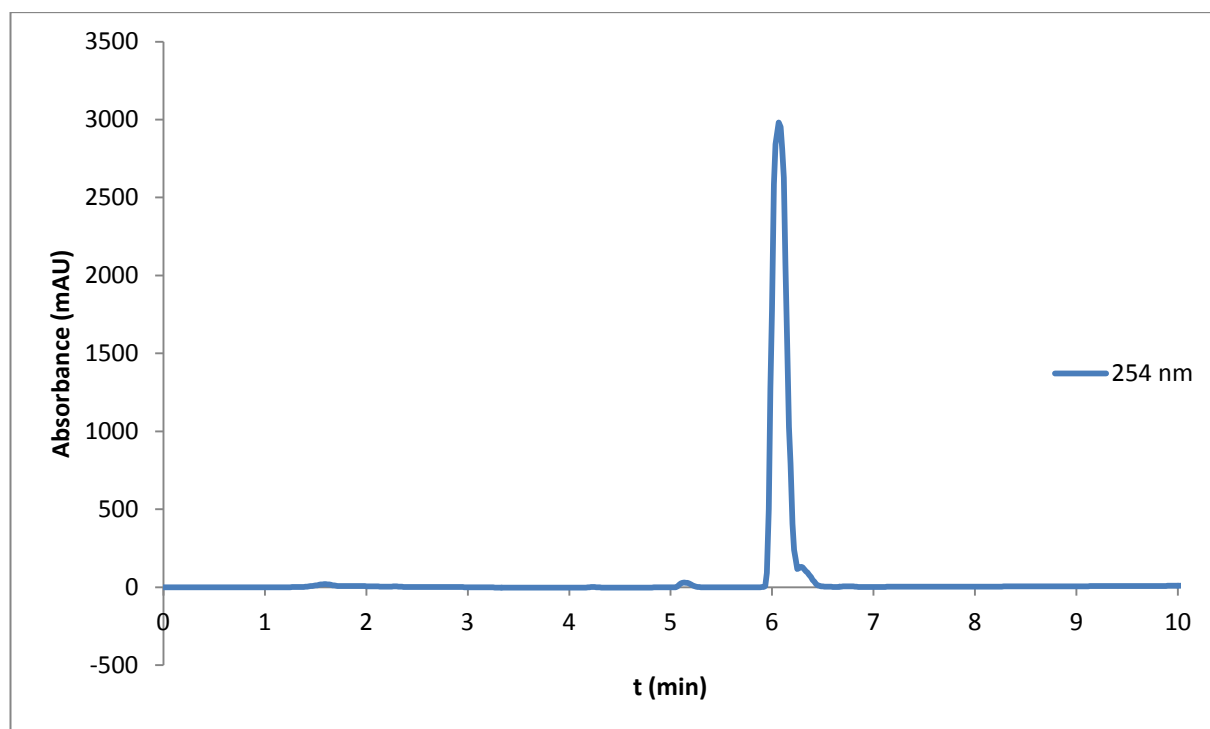
HRMS



HPLC trace at 210 nm

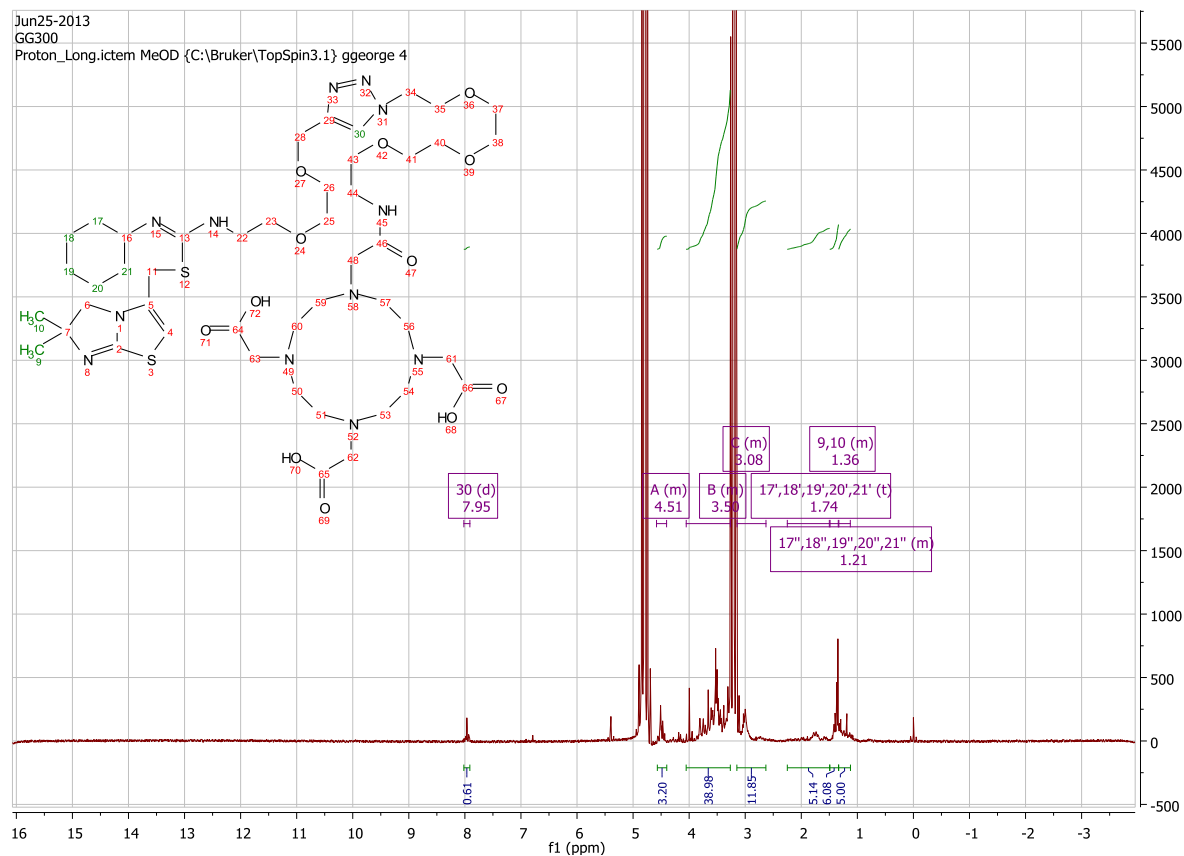


HPLC trace at 254 nm

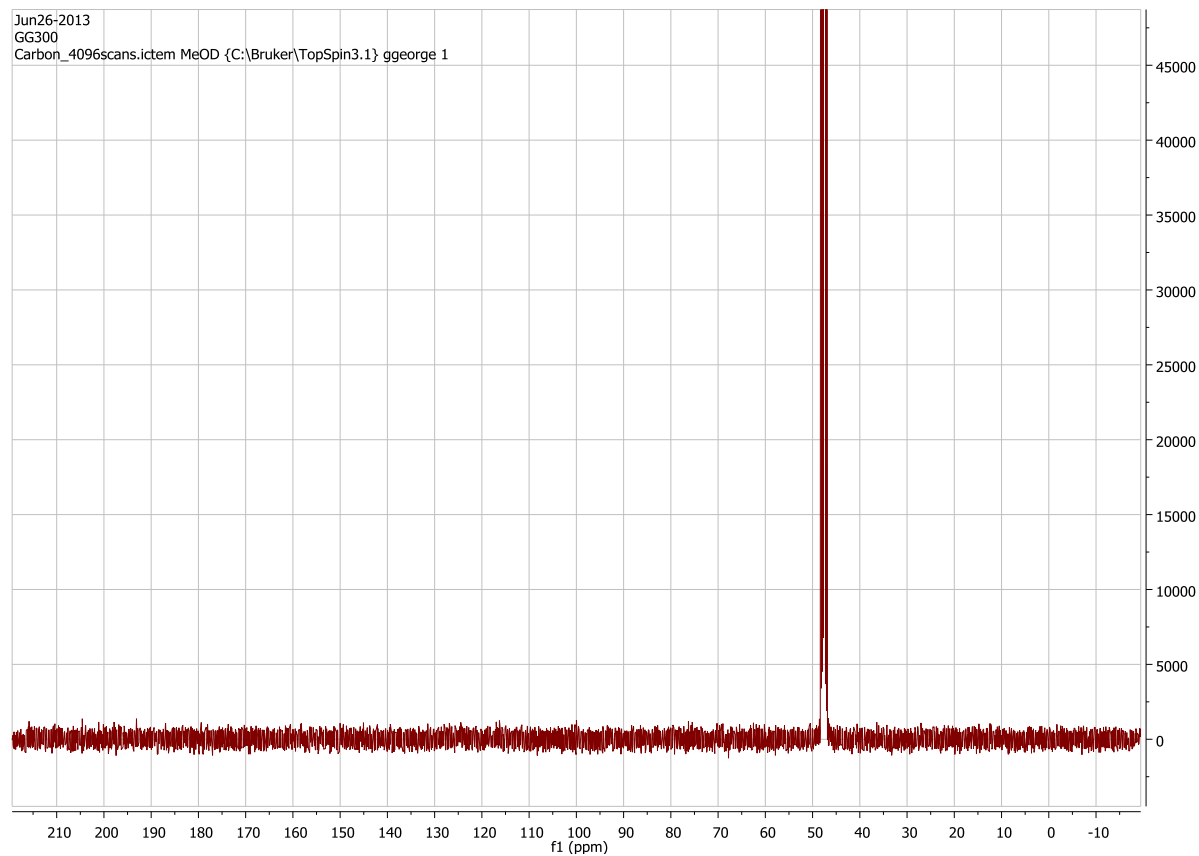


110. 2-[4,10-bis(carboxymethyl)-7-[(2-[2-(2-{2-[4-(N-cyclohexyl-1-{2-[2-((6,6-dimethyl-5*H*,6*H*-imidazo[2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl)methanimidoyl]amino)ethoxy]ethoxy)methyl)-1*H*-1,2,3-triazol-1-yl]ethoxy]ethoxy)ethoxy]ethyl]carbamoyl)methyl]-1,4,7,10-tetraazacyclododecan-1-yl]acetic acid .7TFA

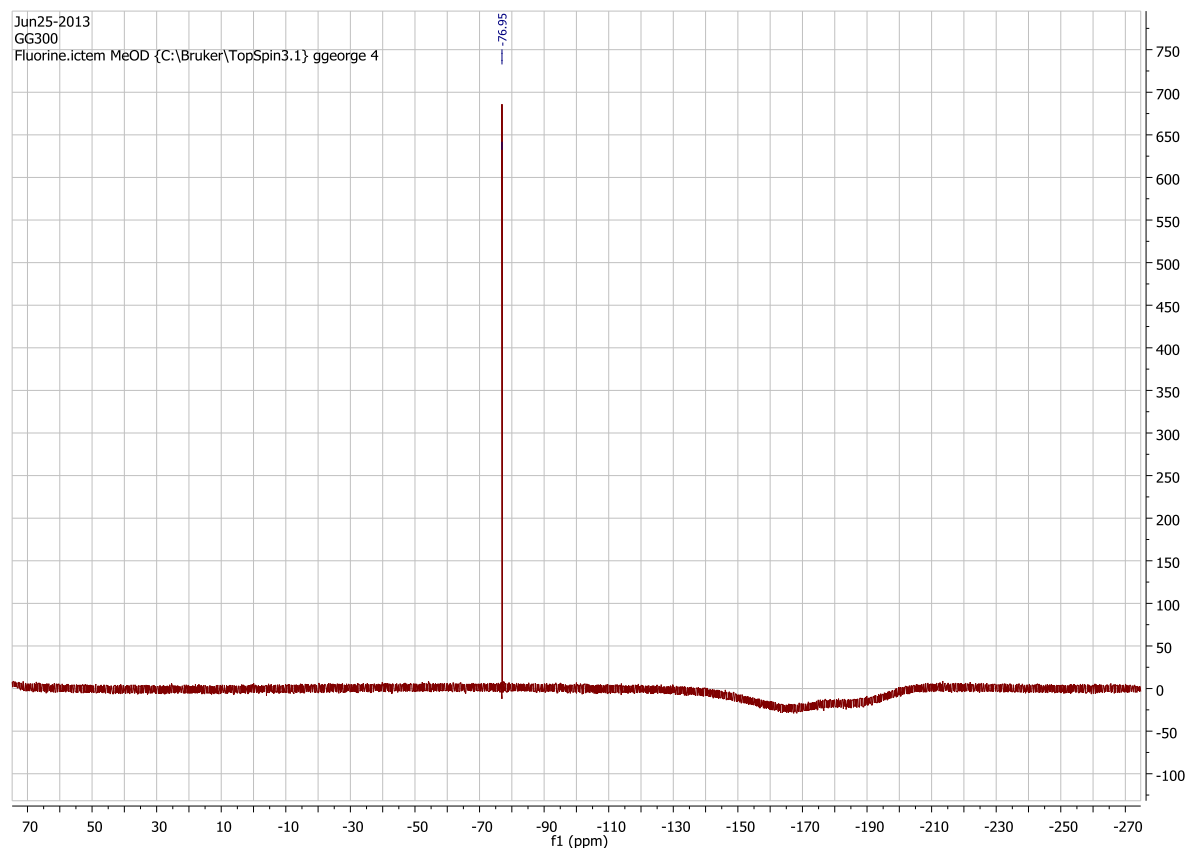
¹H NMR spectrum



^{13}C NMR spectrum

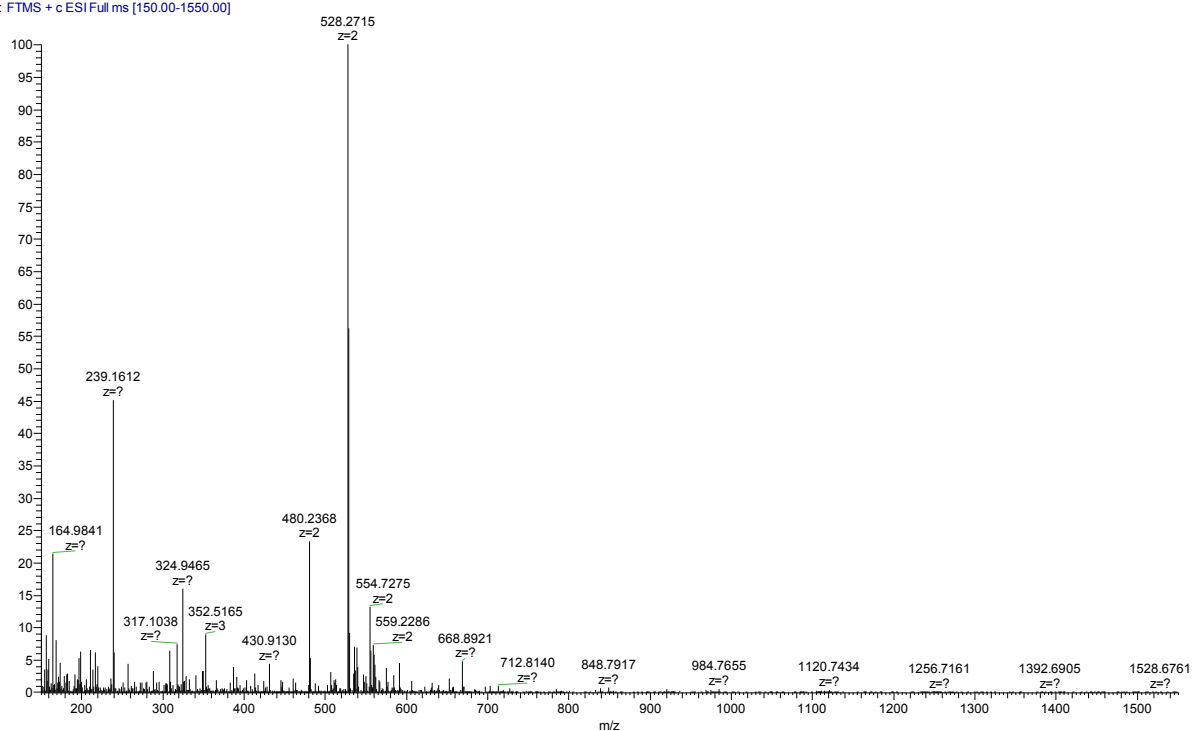


^{19}F NMR spectrum

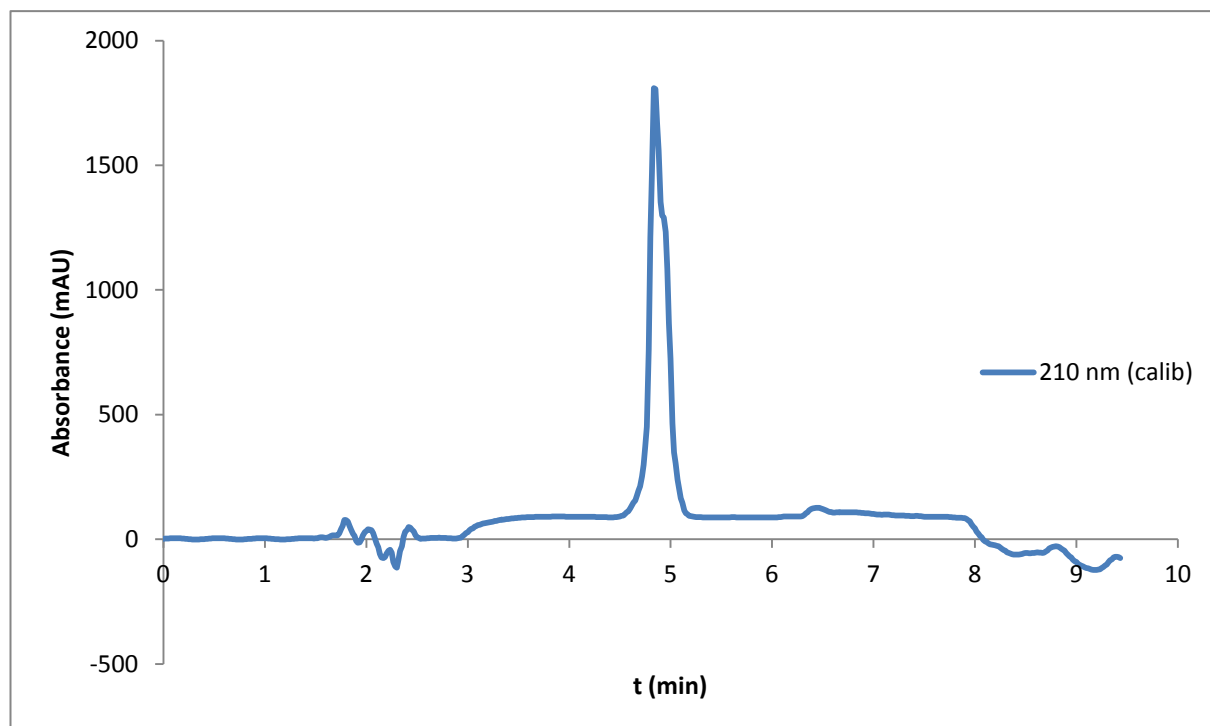


HRMS

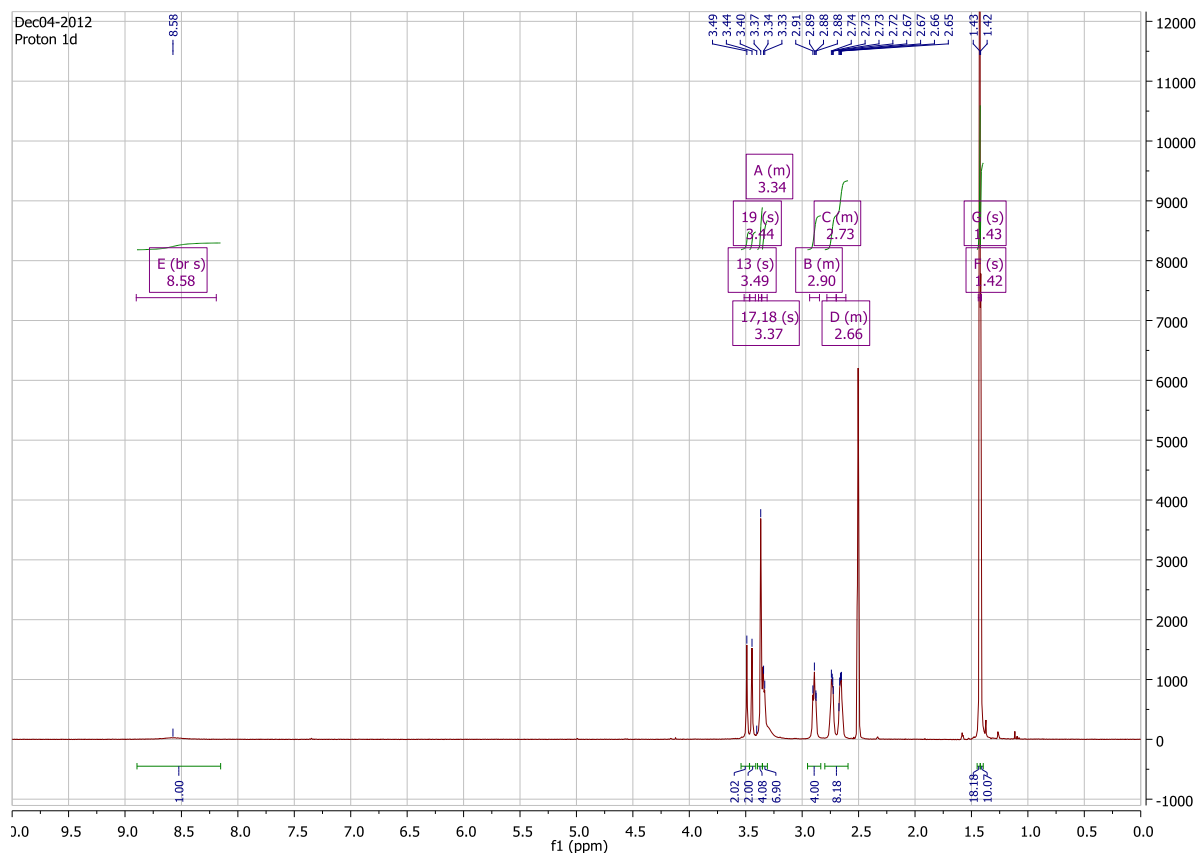
MS03871_GGEORGE_GG300_130625092800 #158-247 RT: 0.20-0.32 AV: 90 NL: 2.21E8
T: FTMS + c ESI Full ms [150.00-1550.00]



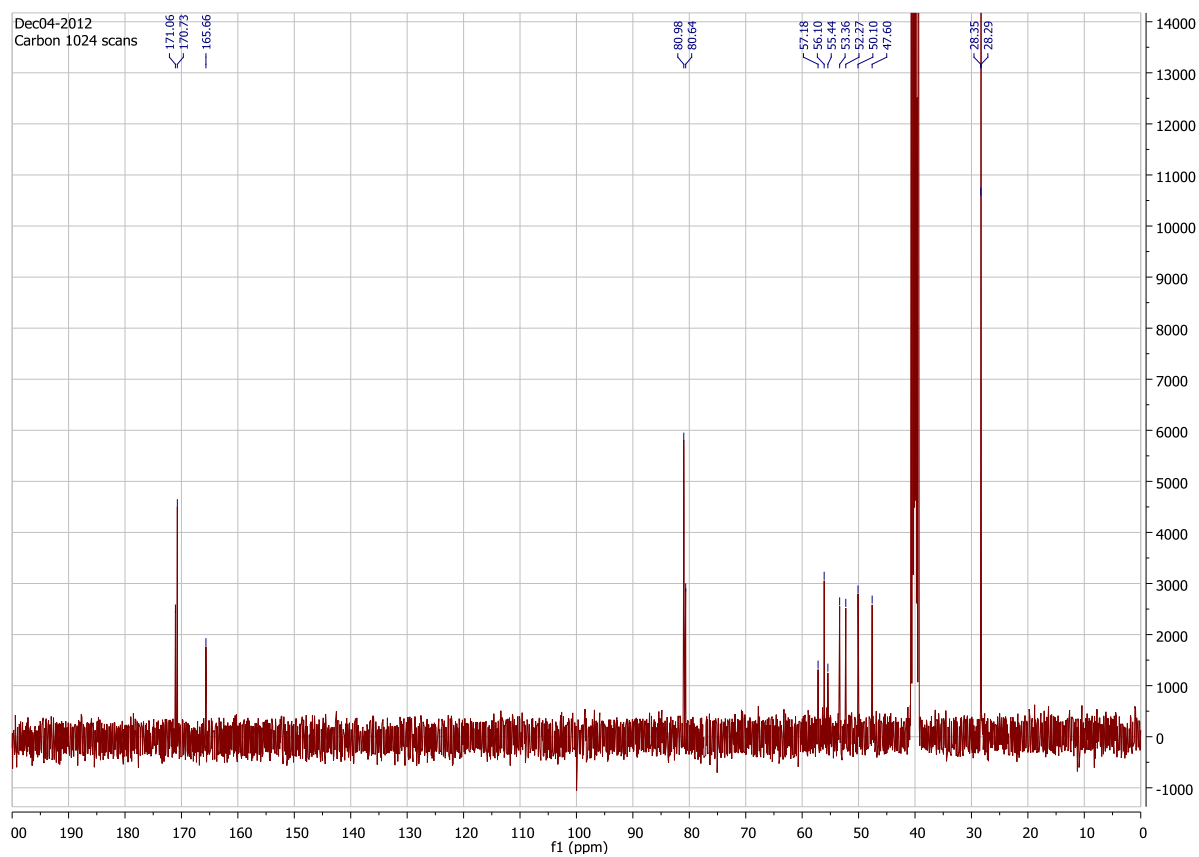
HPLC trace at 210 nm



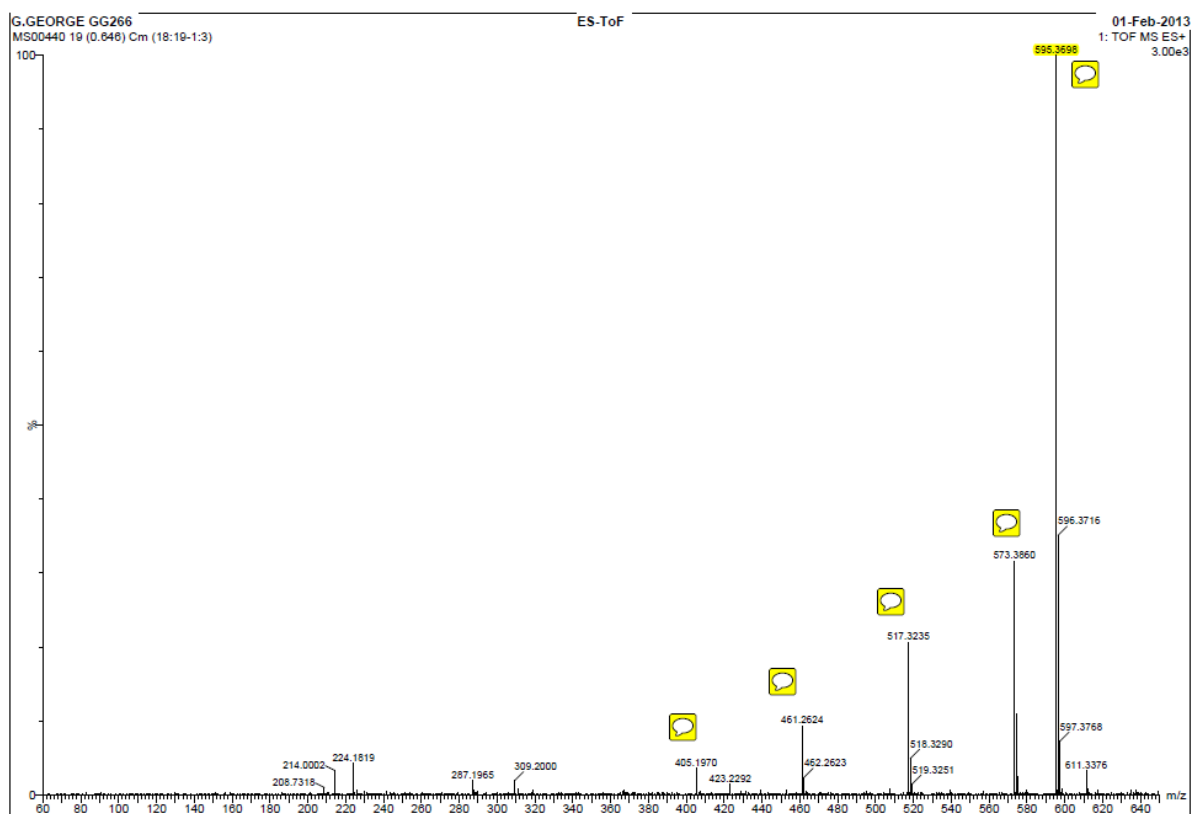
Infrared spectrum



^{13}C NMR spectrum

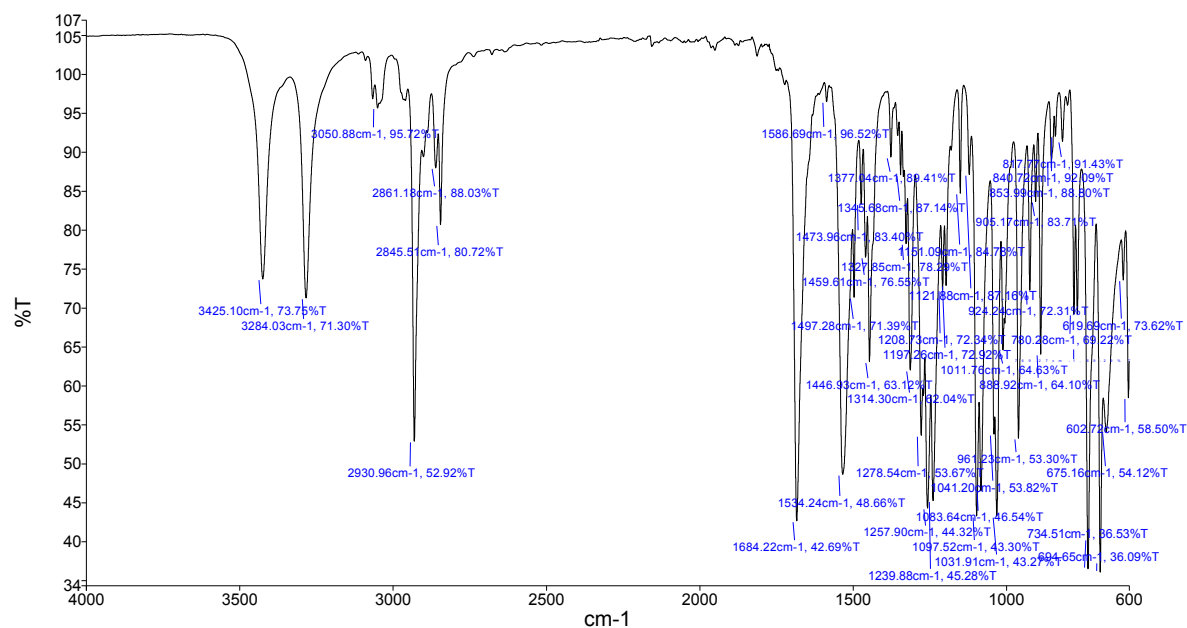


HRMS

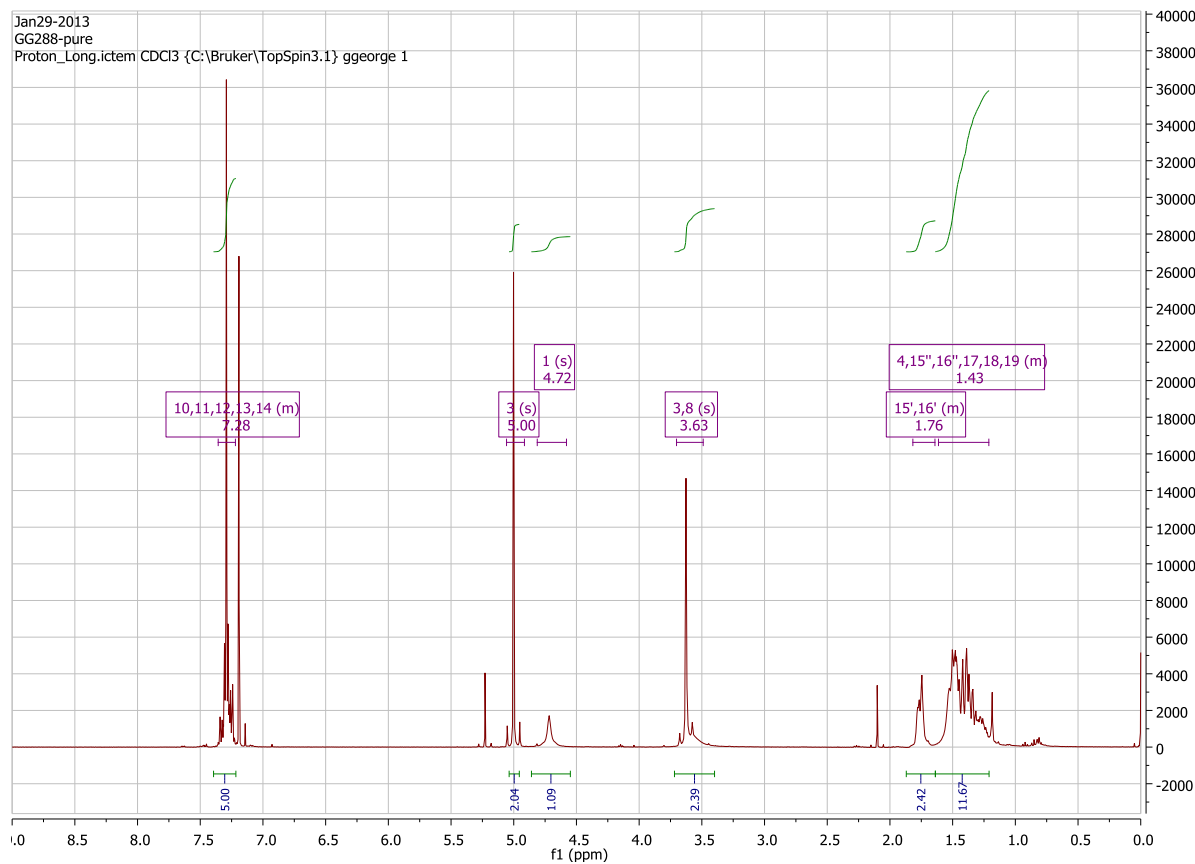


115. Benzyl N-[1-(hydroxymethyl)cyclohexyl]carbamate

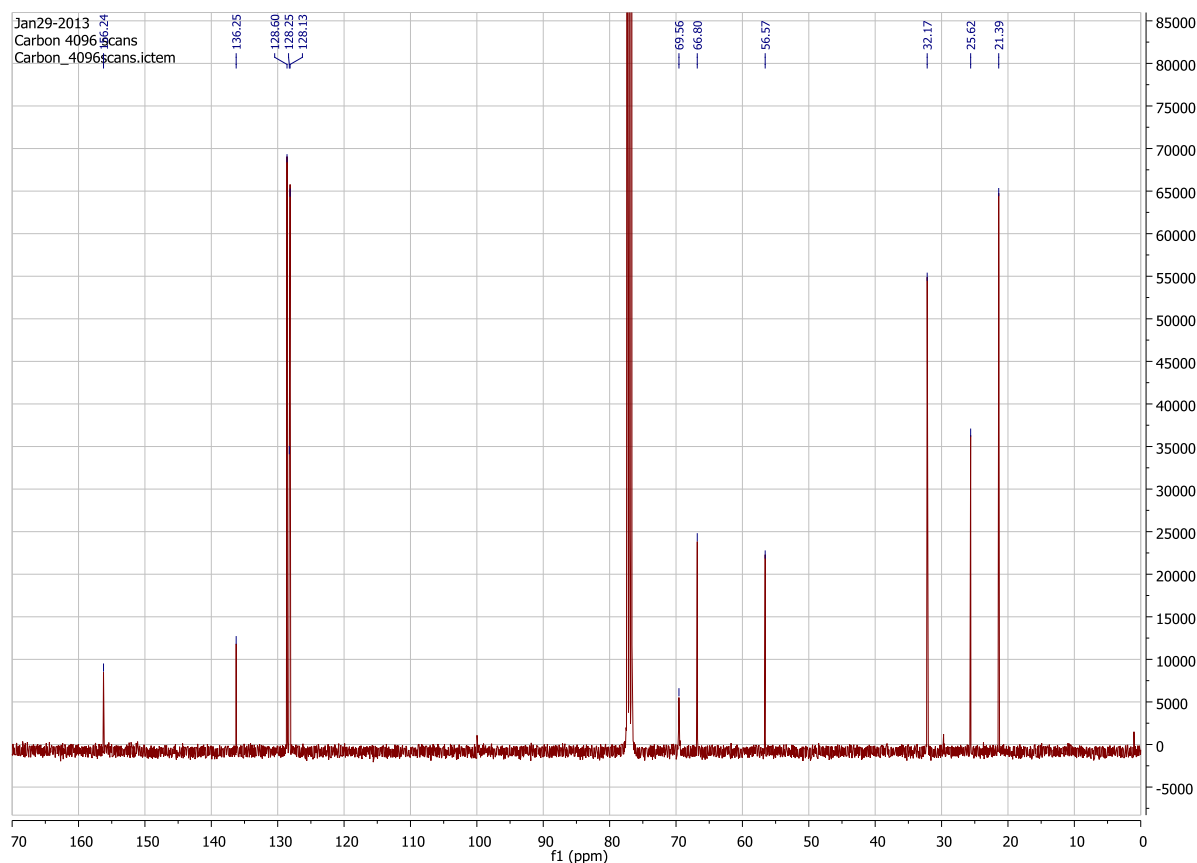
Infrared spectrum



^1H NMR spectrum



¹³C NMR spectrum



HRMS

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 8.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

749 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 15-15 H: 5-300 N: 0-10 O: 0-10 Na: 0-1 S: 0-2 I: 0-1

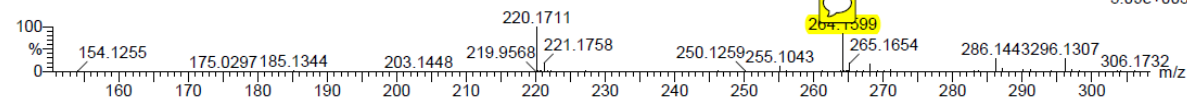
G.GEORGE GG257

LCT28624 6 (0.199) Cm (6:8-20:29)

09:24:01 20-Aug-2012

1: TOF MS ES+

5.09e+003



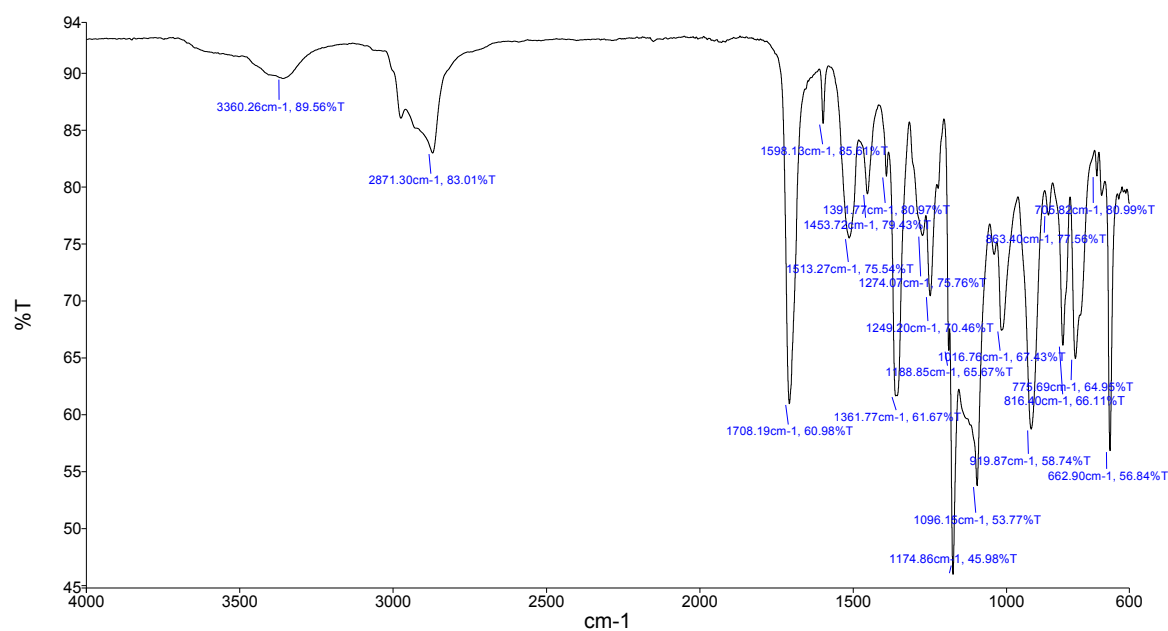
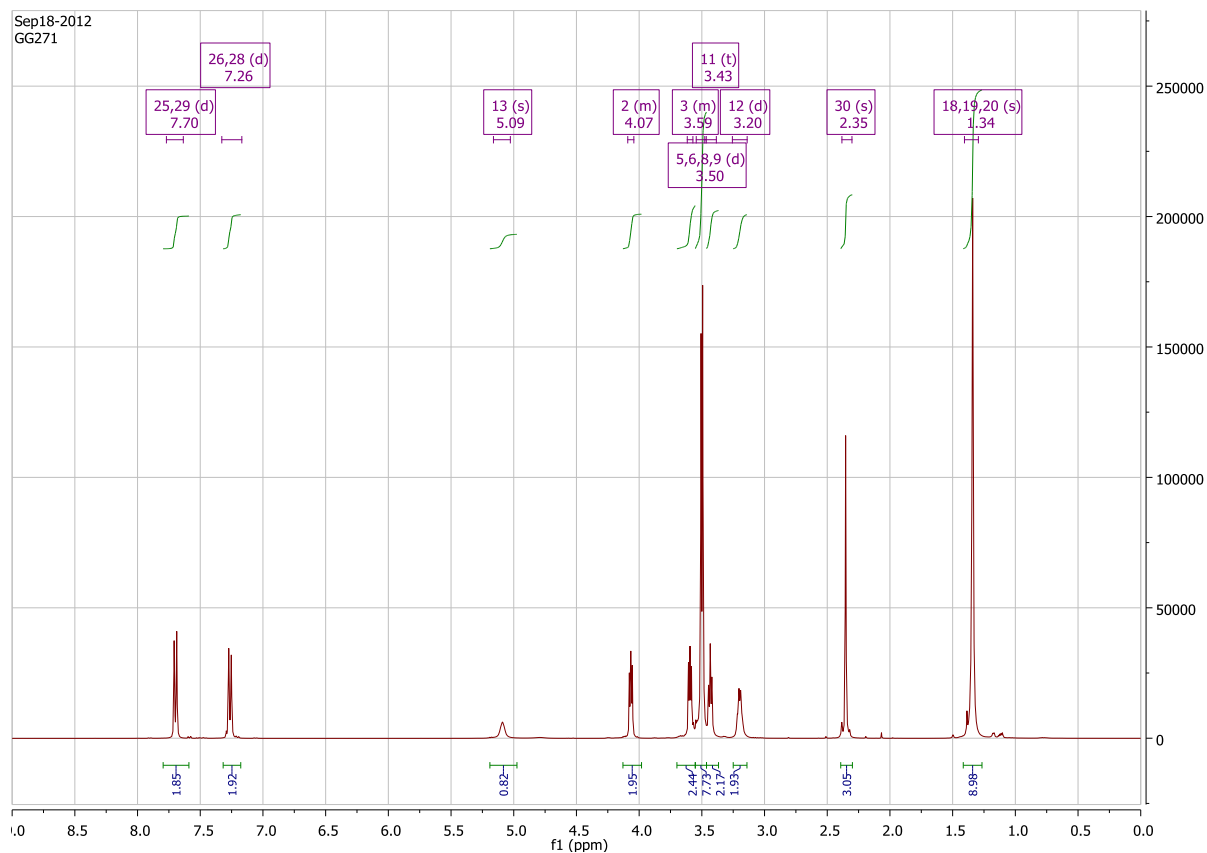
Minimum: -1.5
Maximum: 5.0 8.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
264.1599	264.1600	-0.1	-0.4	5.5	89.6	0.0	C15 H22 N O3

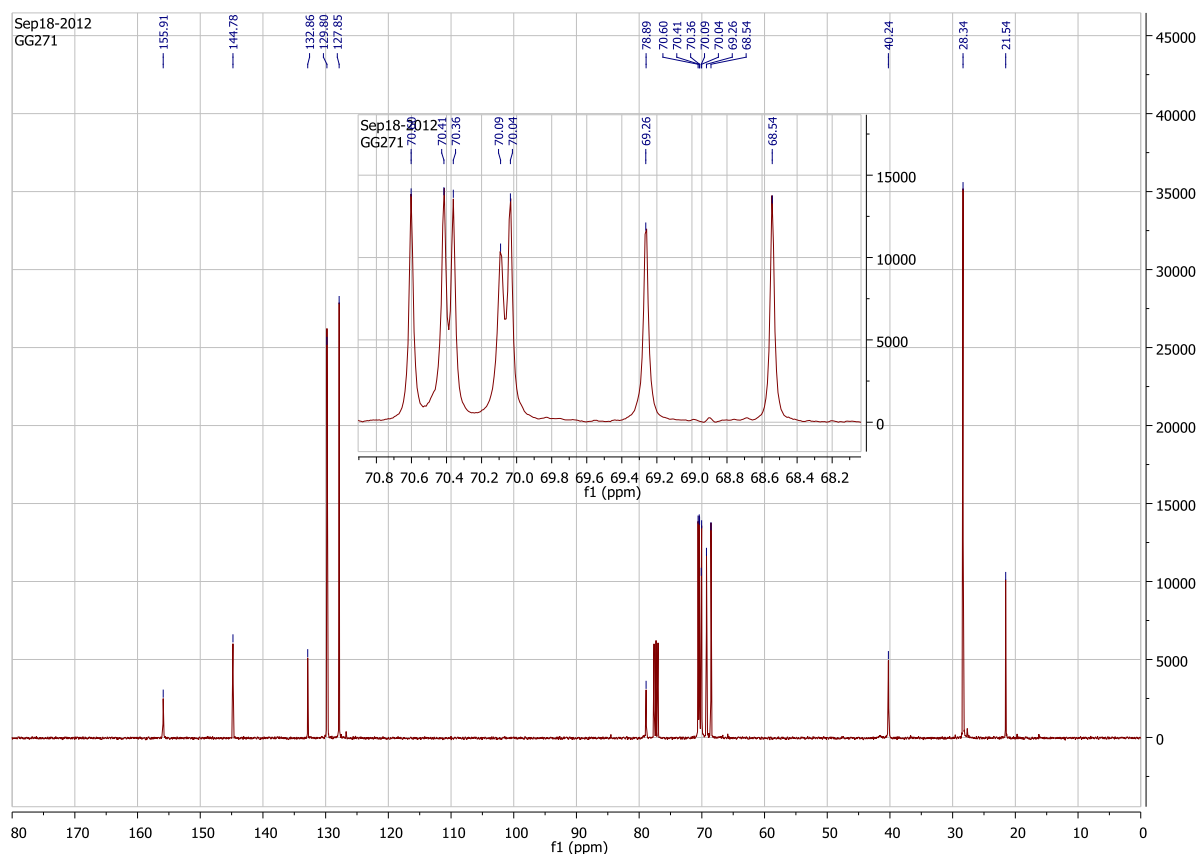
116.

tert-Butyl*N*-{2-[2-(2-{2-[(4-

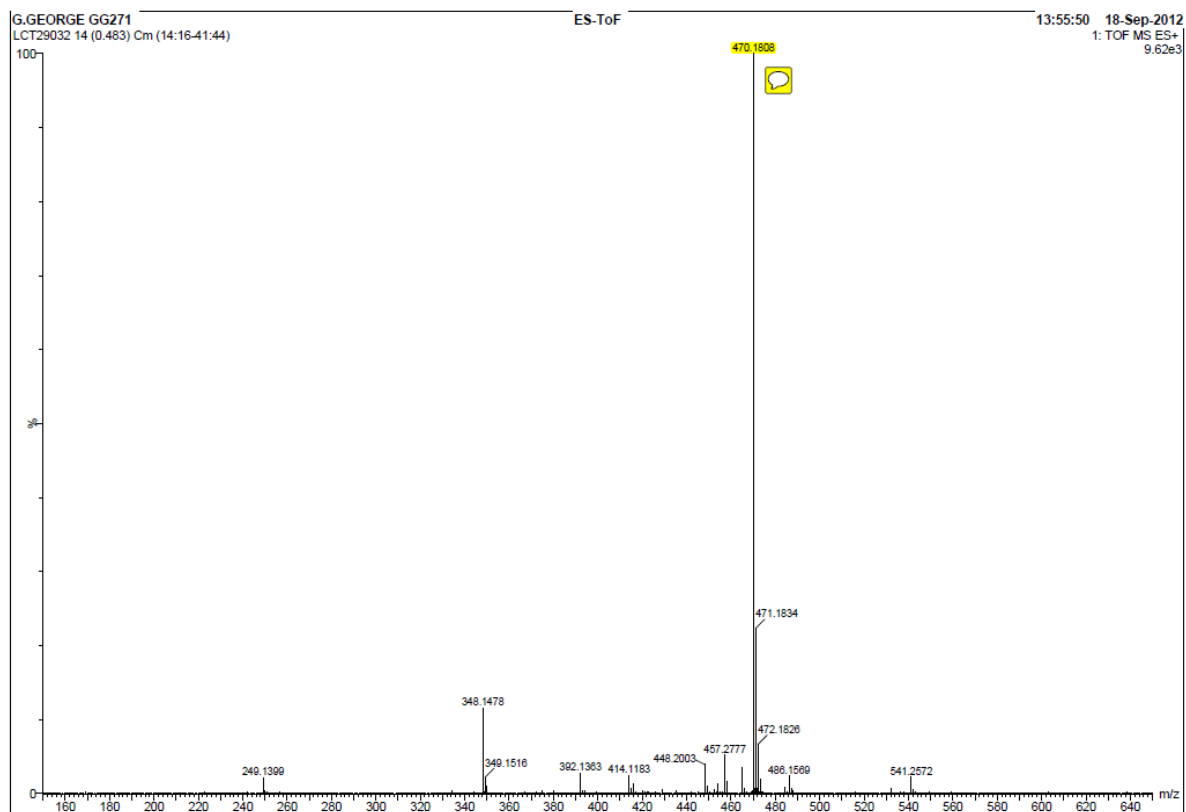
methylbenzenesulfonyl)oxy]ethoxy}ethoxy)ethoxy]ethyl}carbamate

Infrared spectrum¹H NMR spectrum

^{13}C NMR spectrum

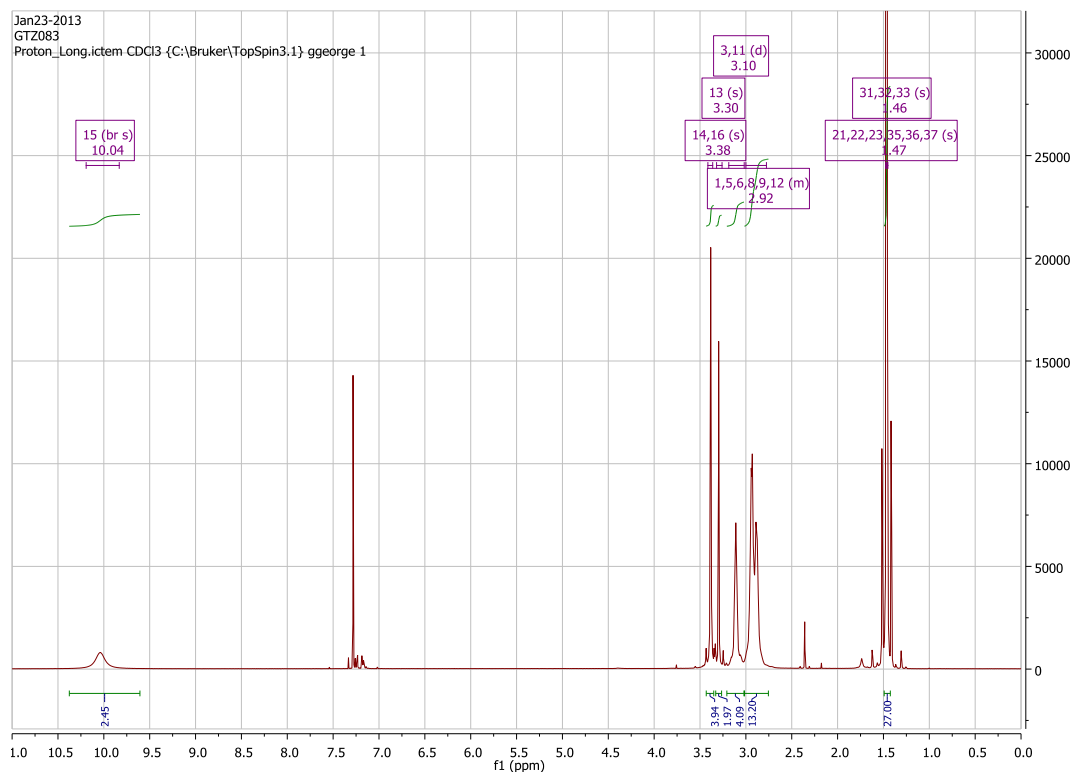


HRMS

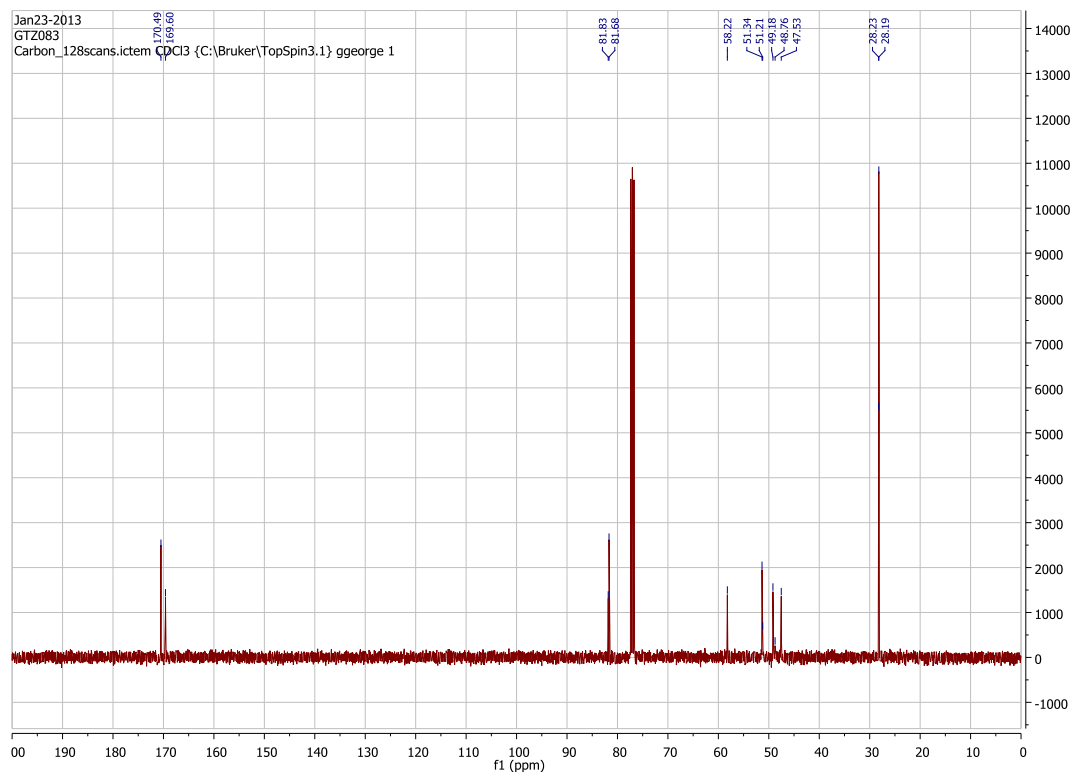


121. *tert*-Butyl 2-{4,7-bis[2-(*tert*-butoxy)-2-oxoethyl]-1,4,7,10-tetraazacyclododecan-1-yl}acetate hydrobromide

¹H NMR spectrum



¹³C NMR spectrum



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

116 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 26-26 H: 5-300 N: 0-10 O: 0-10

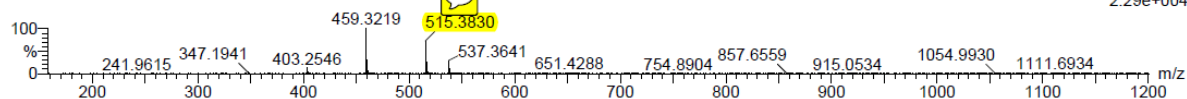
G.GEORGE GG259

LCT28684 12 (0.398) Cm (12:14-43:53)

14:26:28 21-Aug-2012

1: TOF MS ES+

2.29e+004

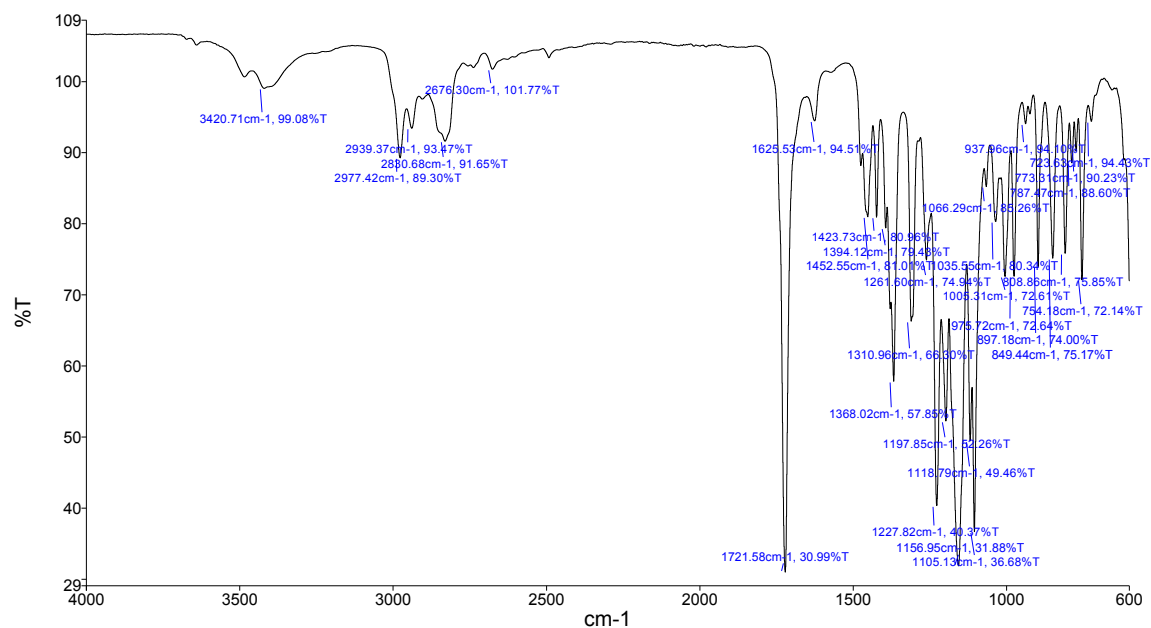


Minimum: -1.5
Maximum: 5.0 10.0 50.0

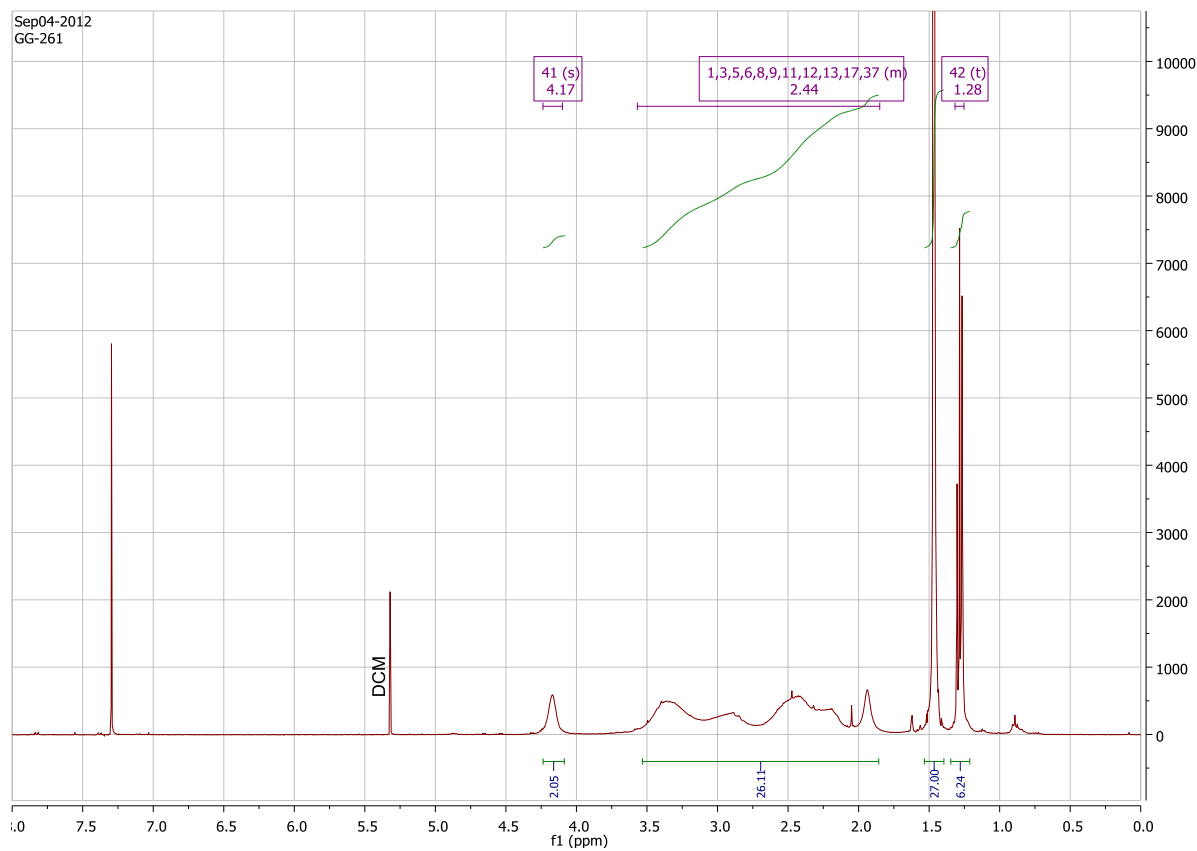
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
515.3830	515.3809	2.1	4.1	3.5	193.1	0.0	C26 H51 N4 O6

123. Ethyl 2-{4,7,10-tris[2-(*tert*-butoxy)-2-oxoethyl]-1,4,7,10-tetraazacyclododecan-1-yl}acetate

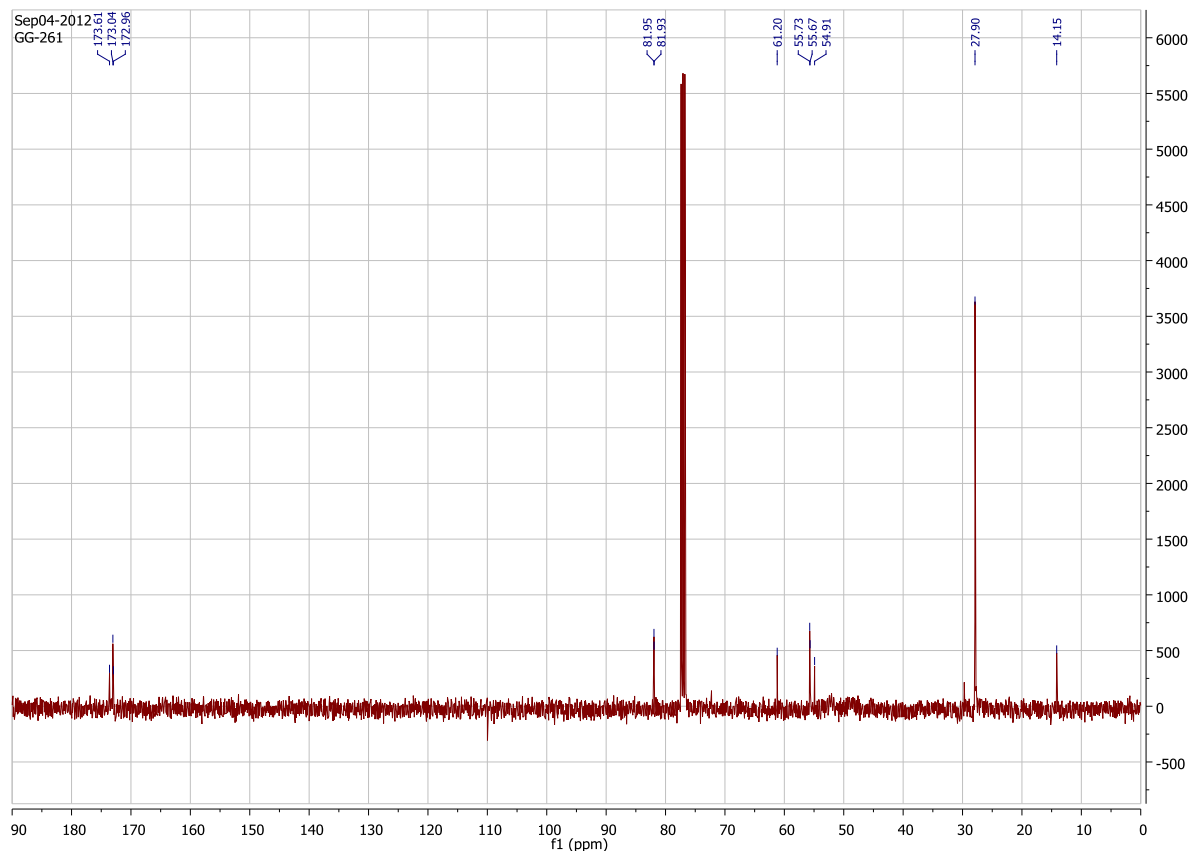
Infrared spectrum



¹H NMR spectrum



¹³C NMR spectrum



HRMS

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

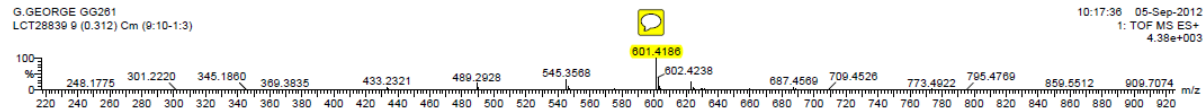
464 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 30-30 H: 5-300 N: 0-10 O: 0-10 Na: 0-1 I: 0-1

G.GEORGE GG261
LCT28839 9 (0.312) Cm (9:10-1:3)

10:17:36 05-Sep-2012
1: TOF MS ES+
4.38e+003



Minimum:

Maximum:

5.0

10.0

-1.5

50.0

Mass

Calc. Mass

mDa

PPM

DBE

1-FIT

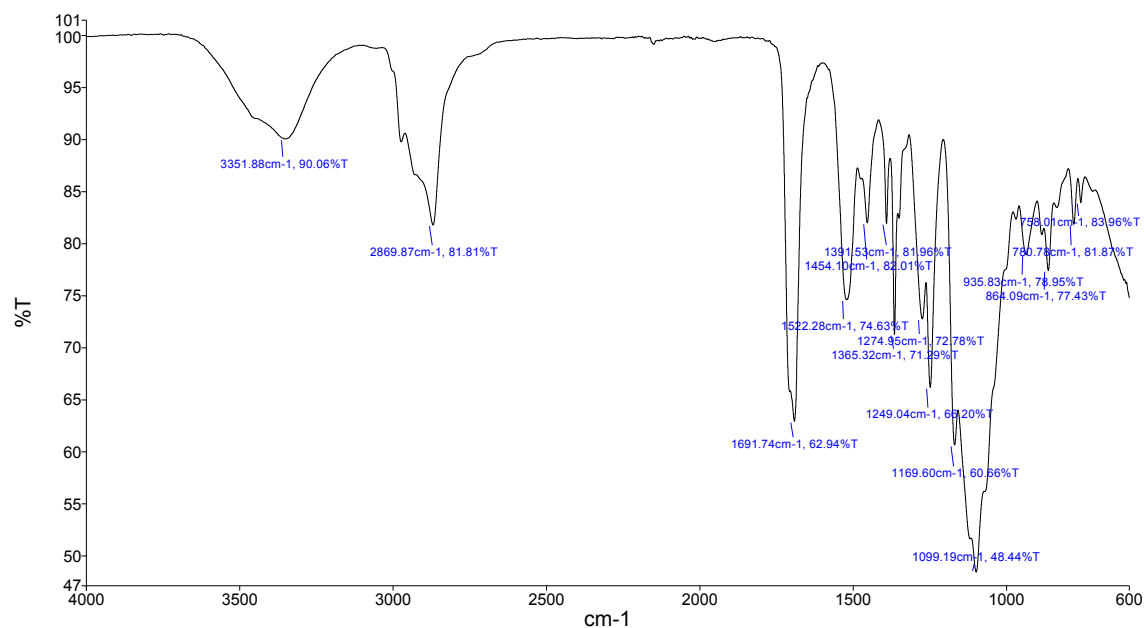
1-FIT (Norm)

Formula

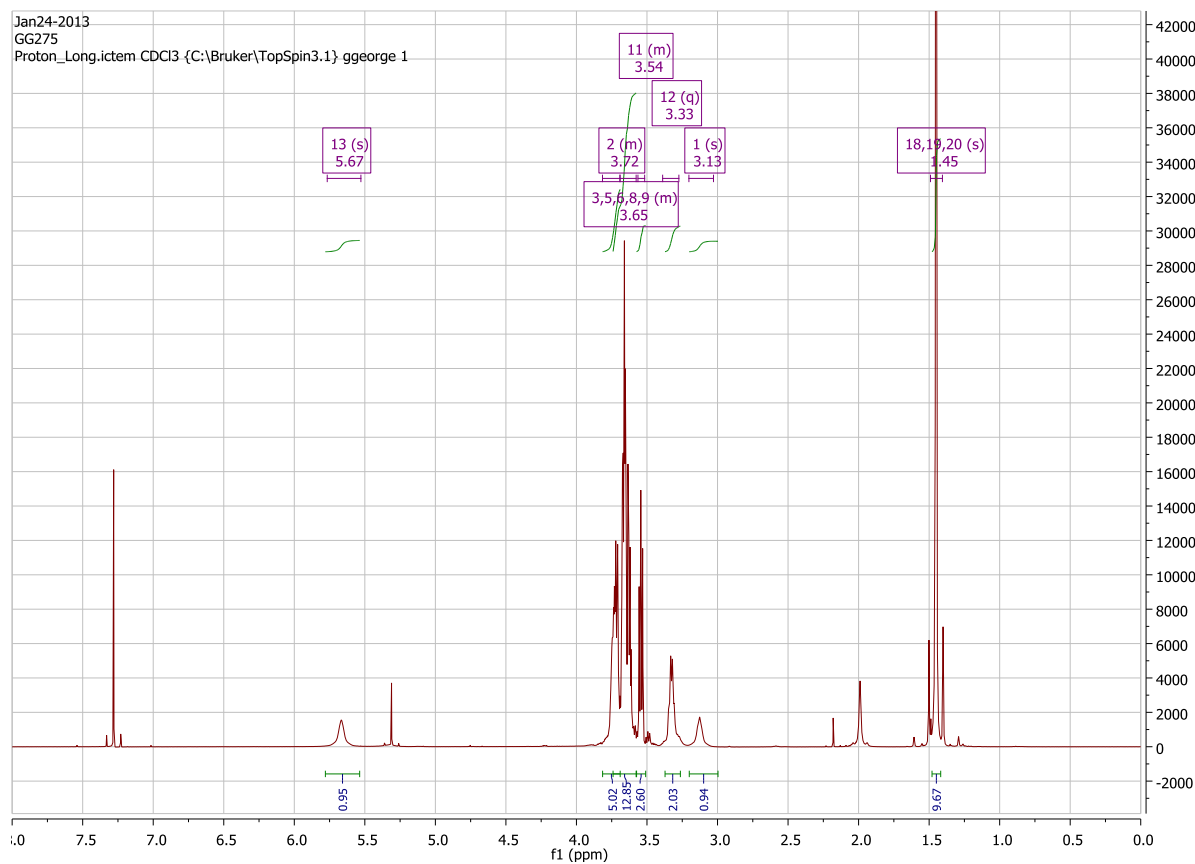
601.4186 601.4176 1.0 1.7 4.5 125.3 0.0 C30 H57 N4 O8

125. *tert*-Butyl *N*-(2-{2-[2-(2-hydroxyethoxy)ethoxy]ethoxy}ethyl)carbamate

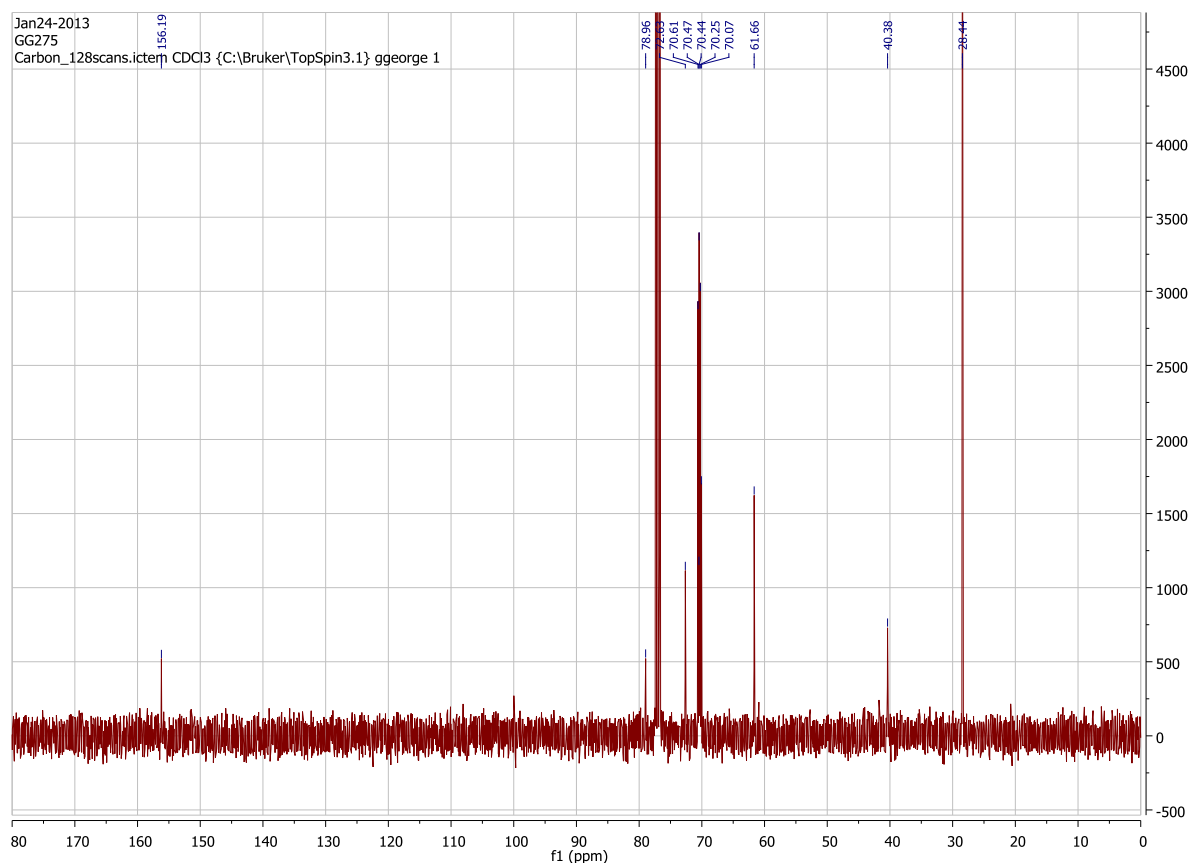
Infrared spectrum



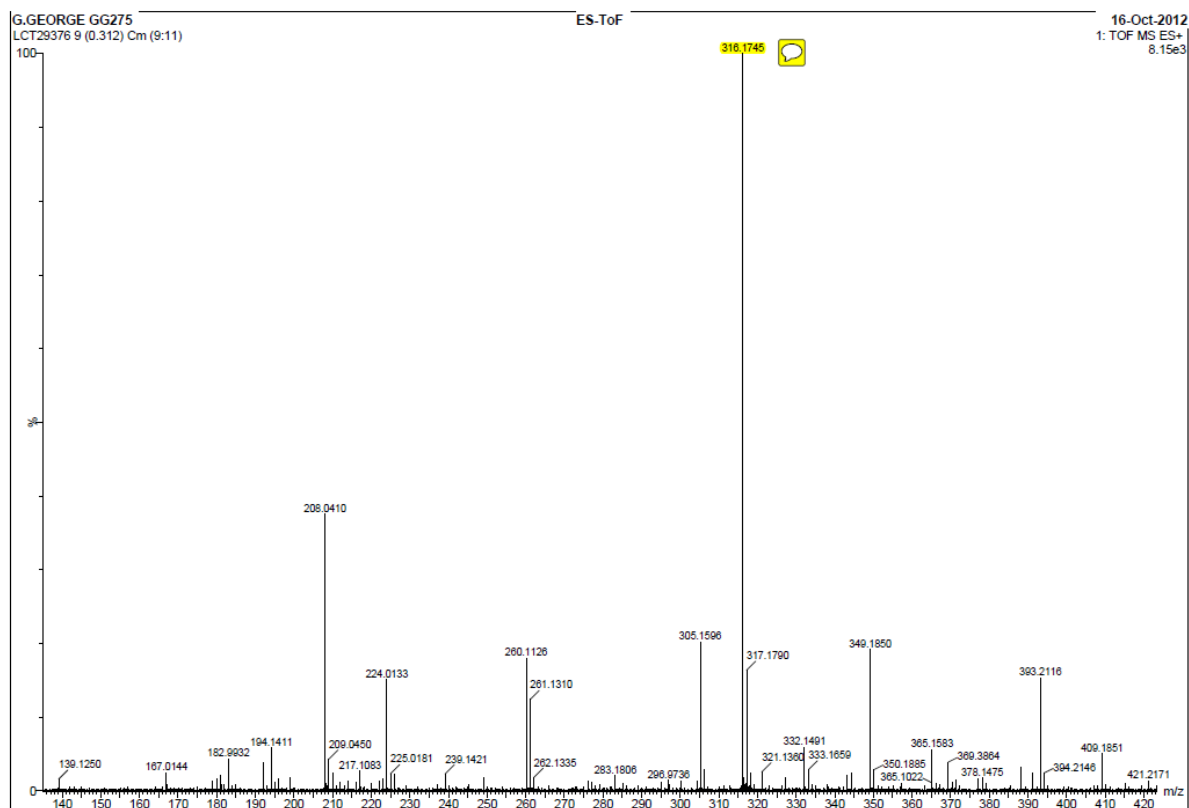
¹H NMR spectrum



^{13}C NMR spectrum

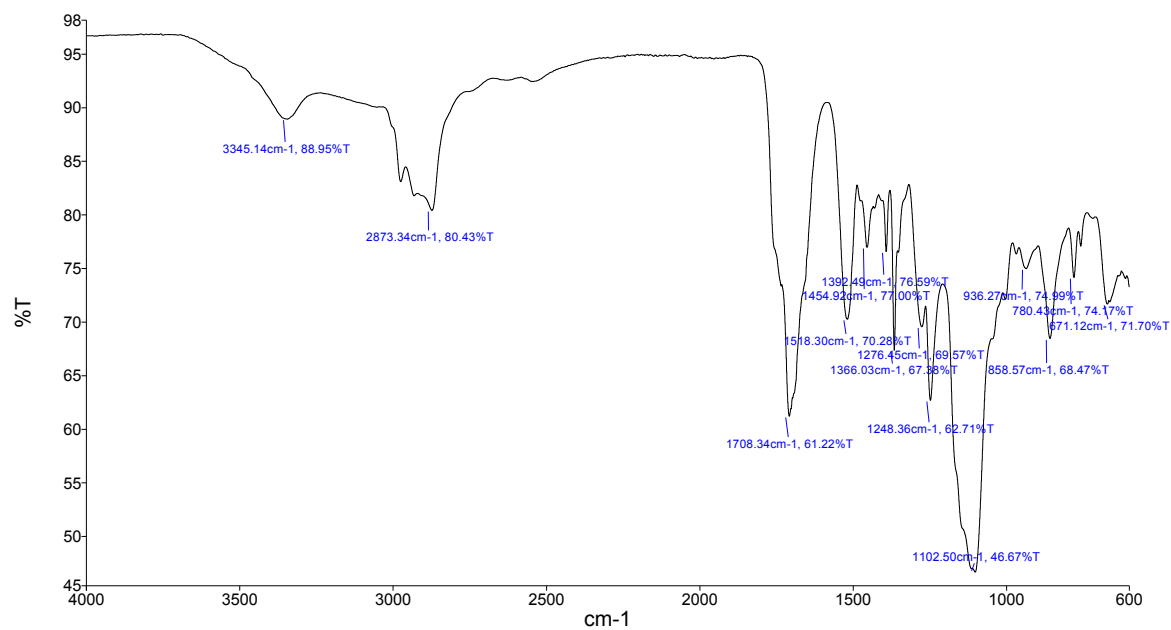


HRMS

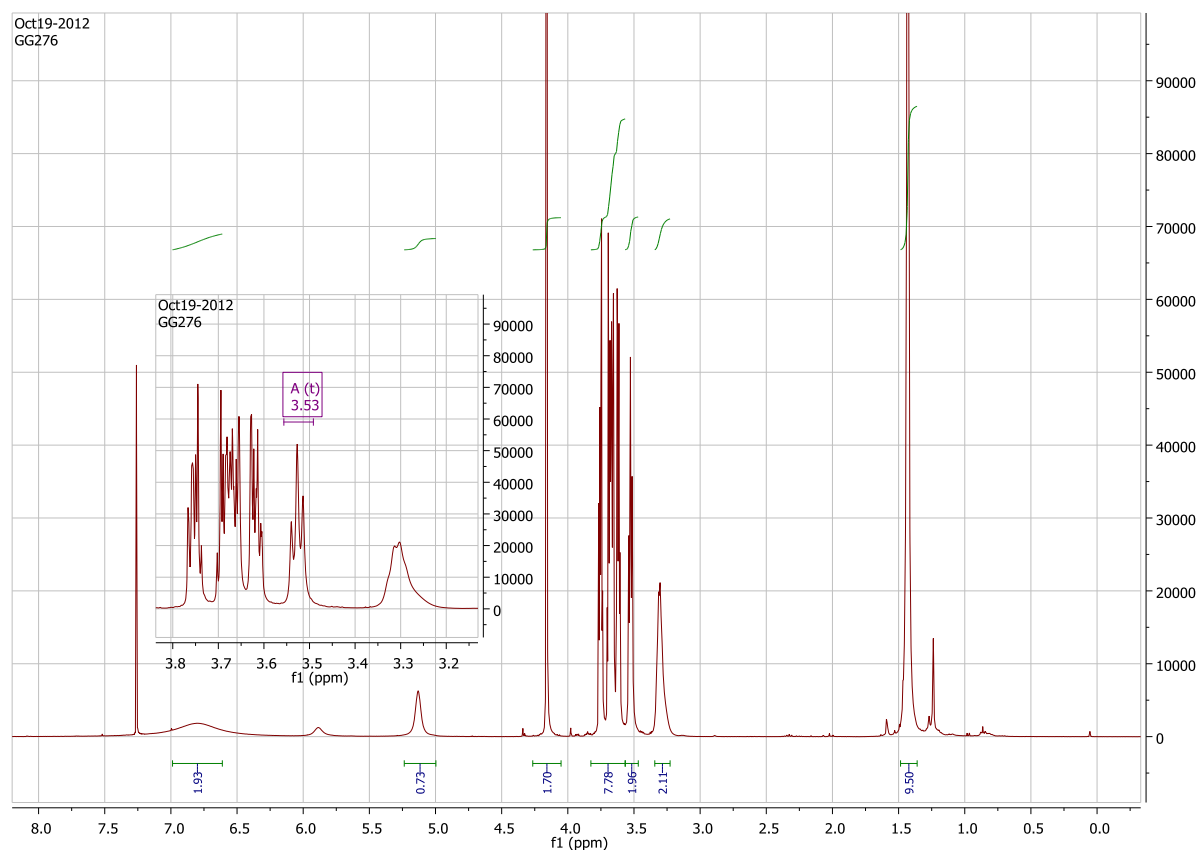


126. 2-{2-[2-(2-[(Tert-butoxy)carbonyl]amino)ethoxy]ethoxy}ethoxy}acetic acid

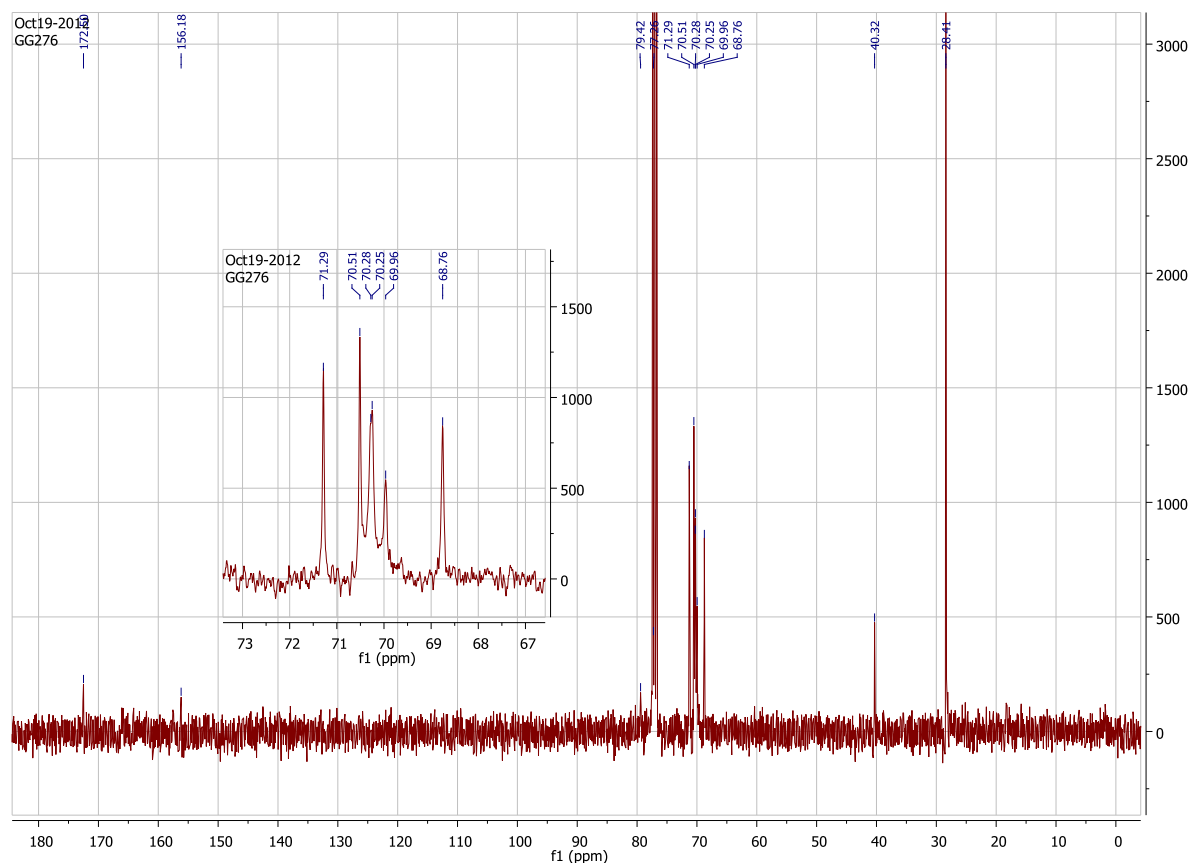
Infrared spectrum



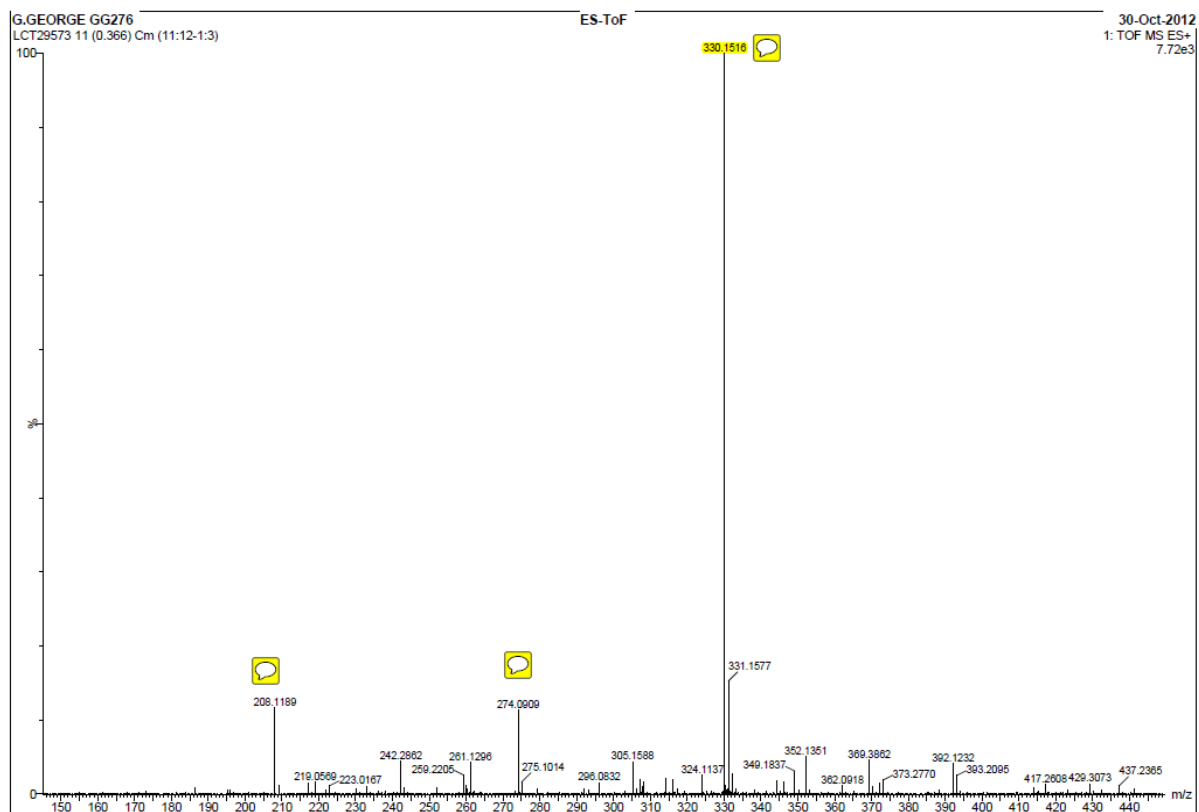
¹H NMR spectrum



^{13}C NMR spectrum



HRMS



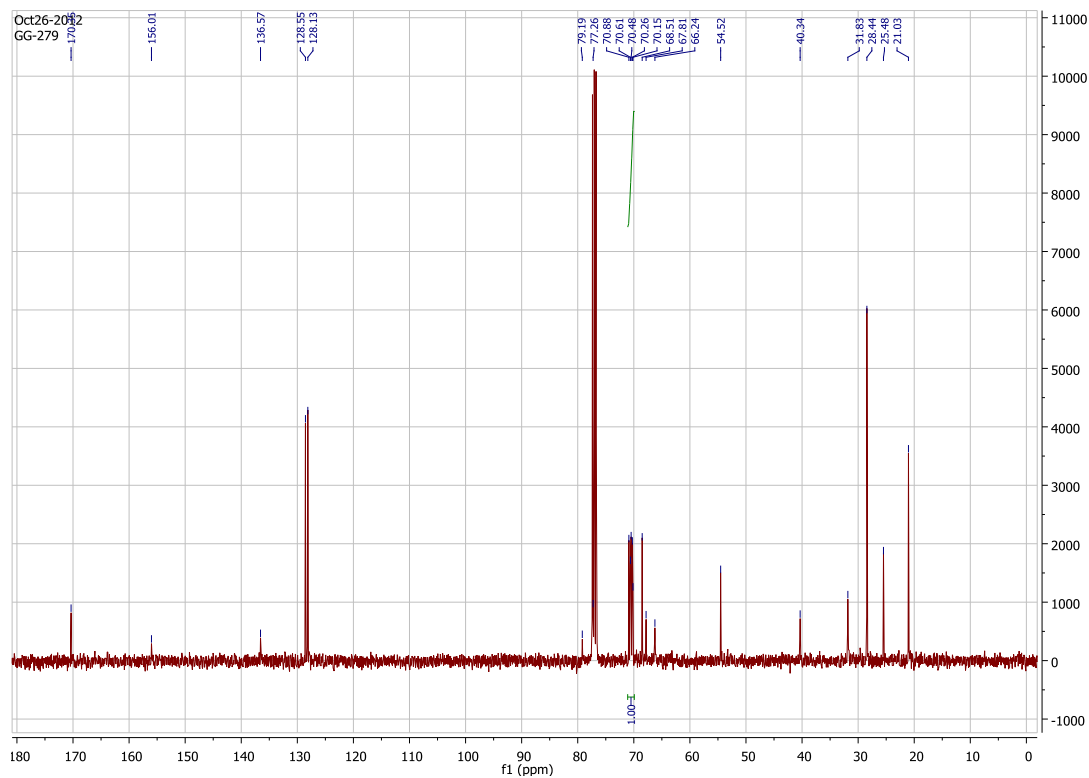
127. (1-{[(Benzyloxy)carbonyl]amino}cyclohexyl)methyl
butoxy)carbonyl]amino}ethoxy}ethoxy}acetate

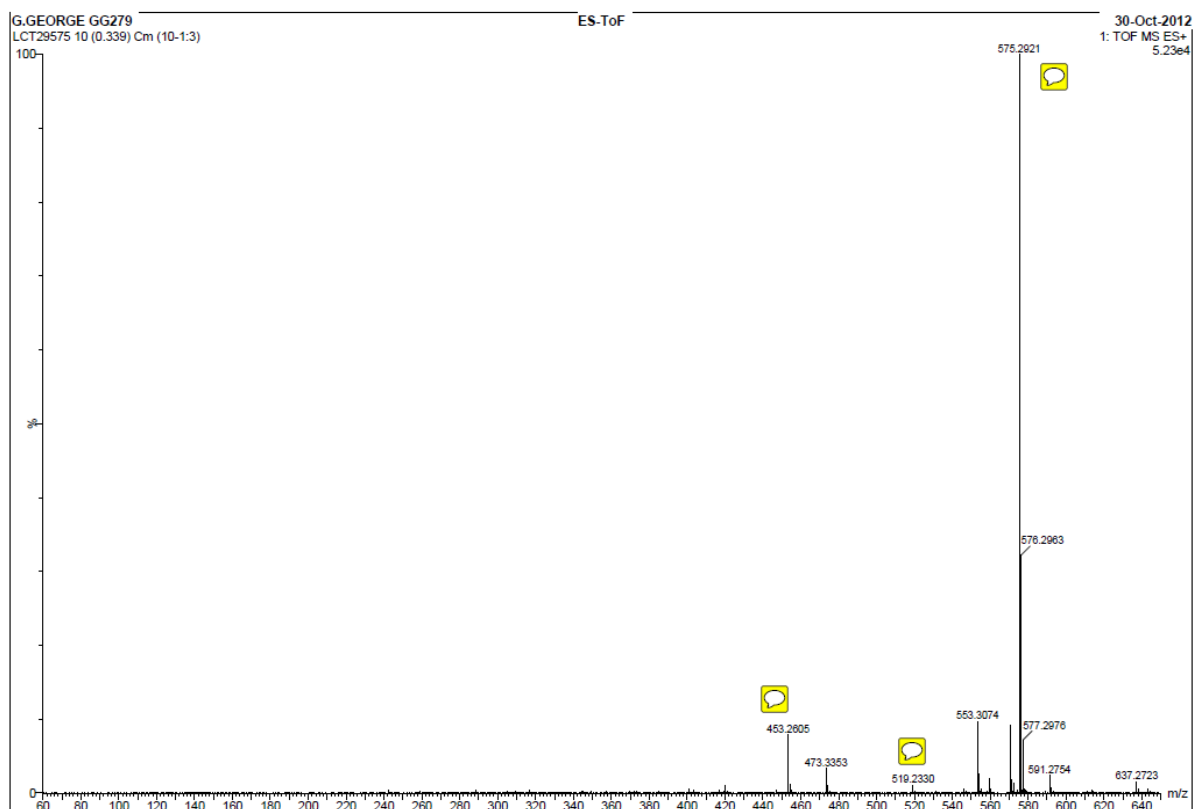
2-{2-[2-(2-{[(tert-

^1H NMR spectrum



^{13}C NMR spectrum

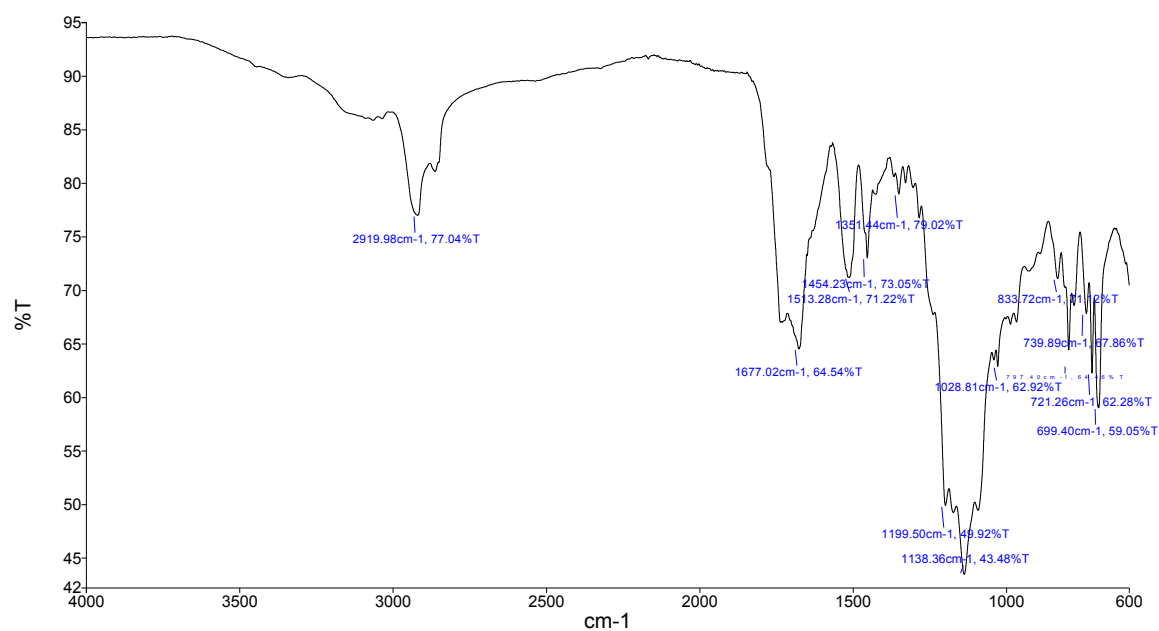




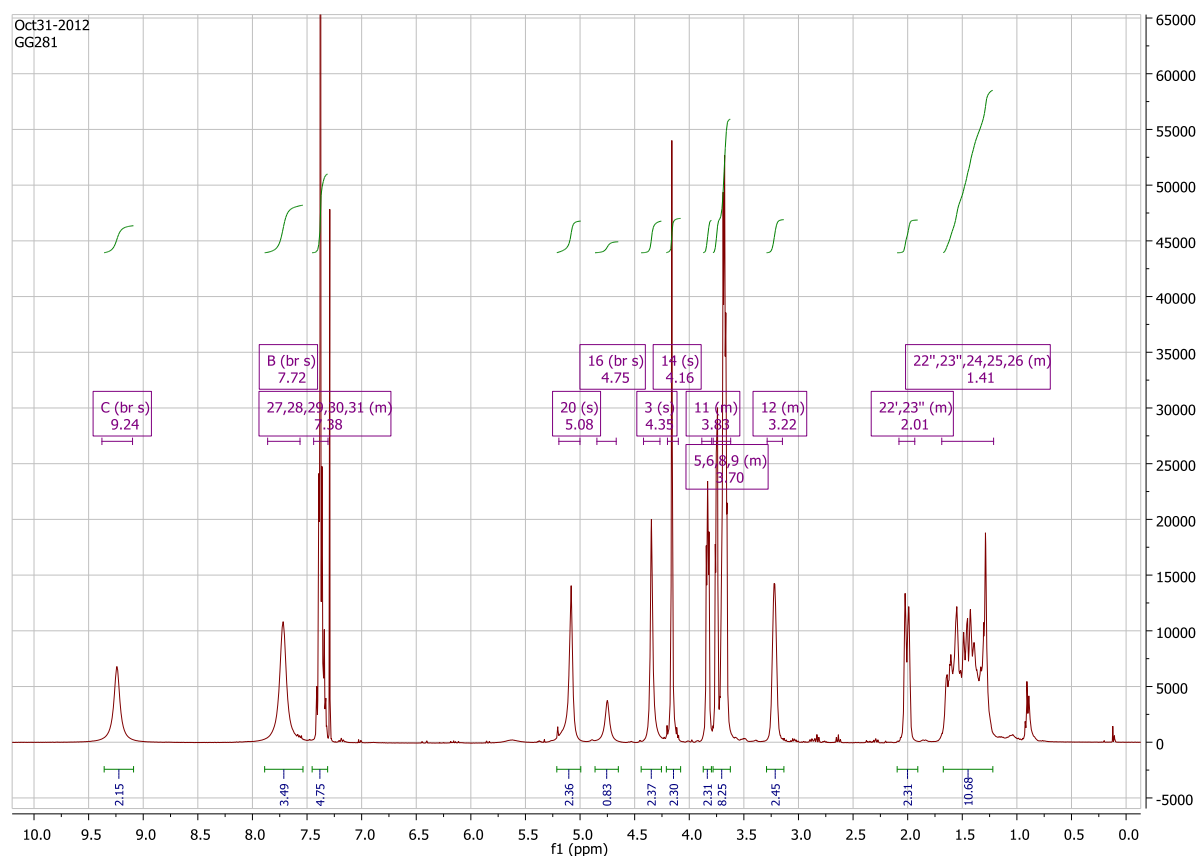
128. (1-{[(Benzyloxy)carbonyl]amino}cyclohexyl)methyl
aminoethoxy)ethoxy]ethoxy}acetate

2-{2-[2-(2-

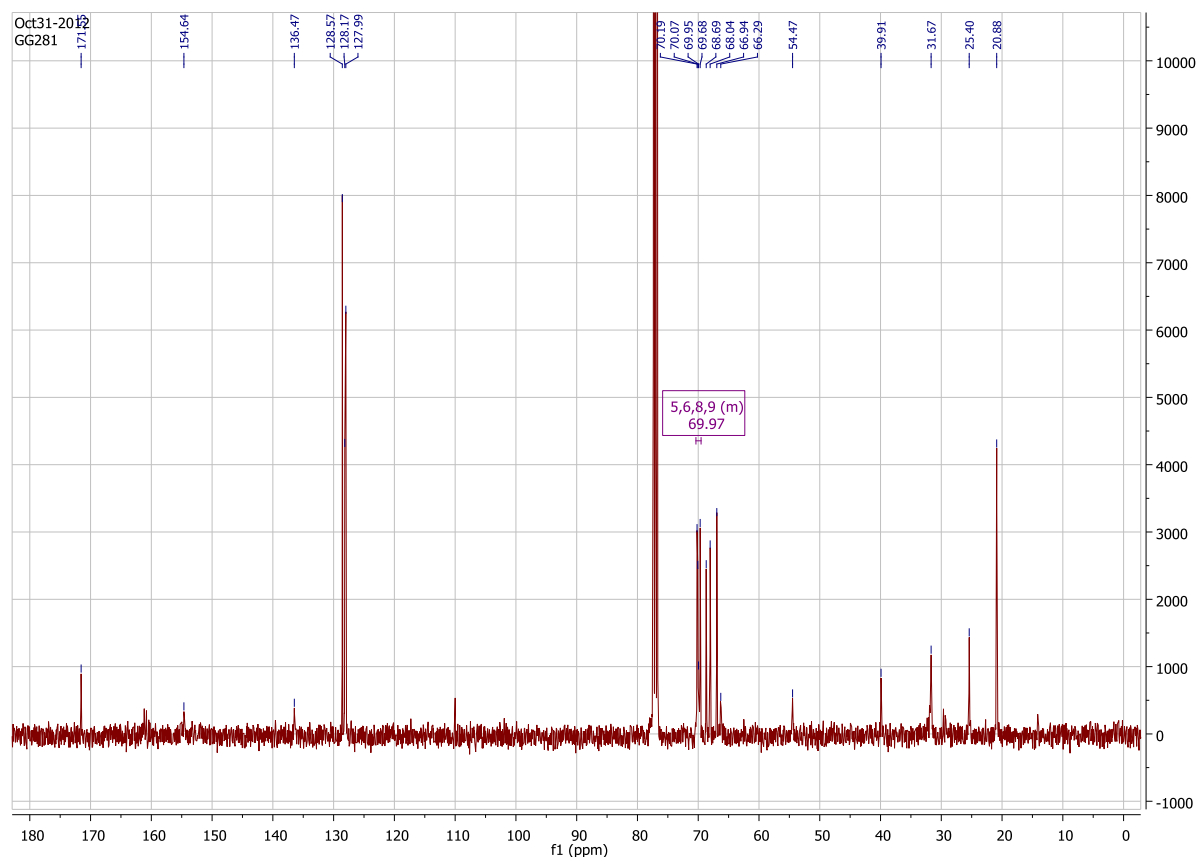
Infrared spectrum



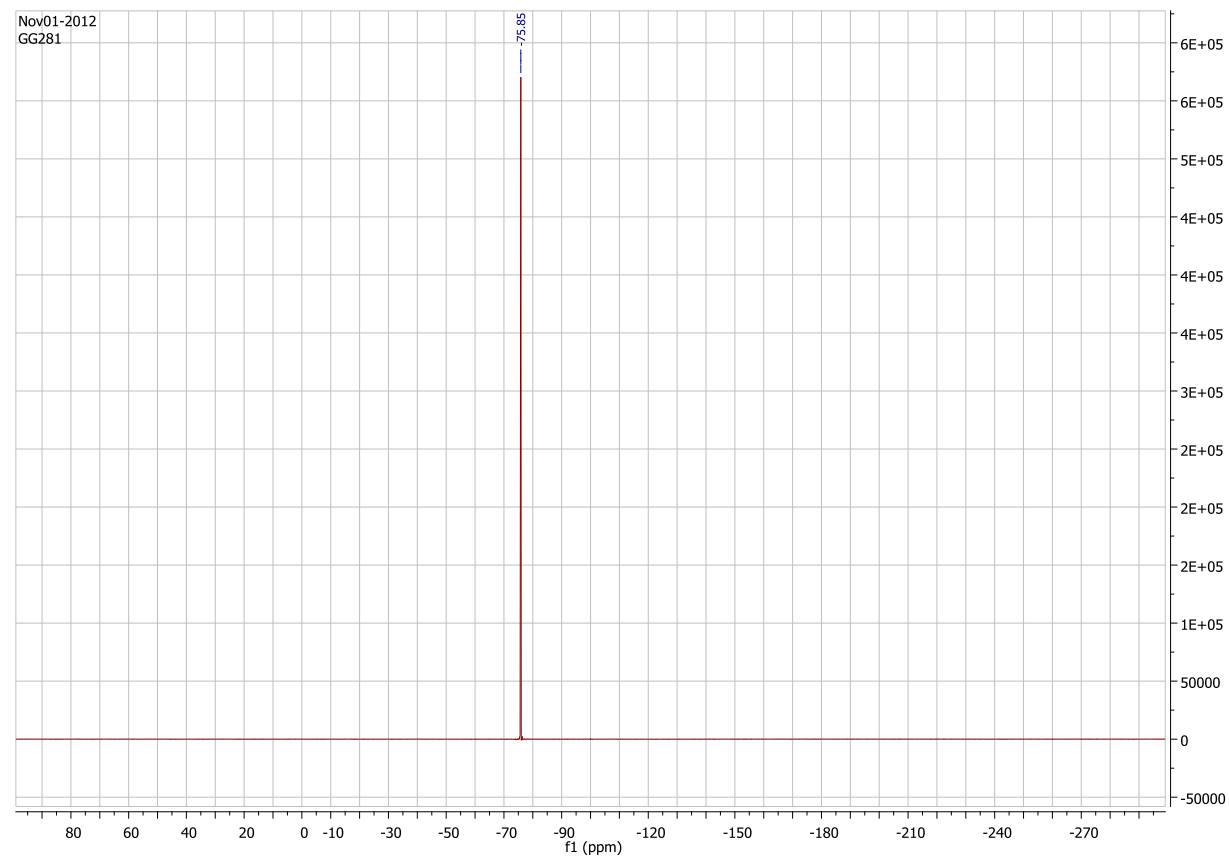
¹H NMR spectrum

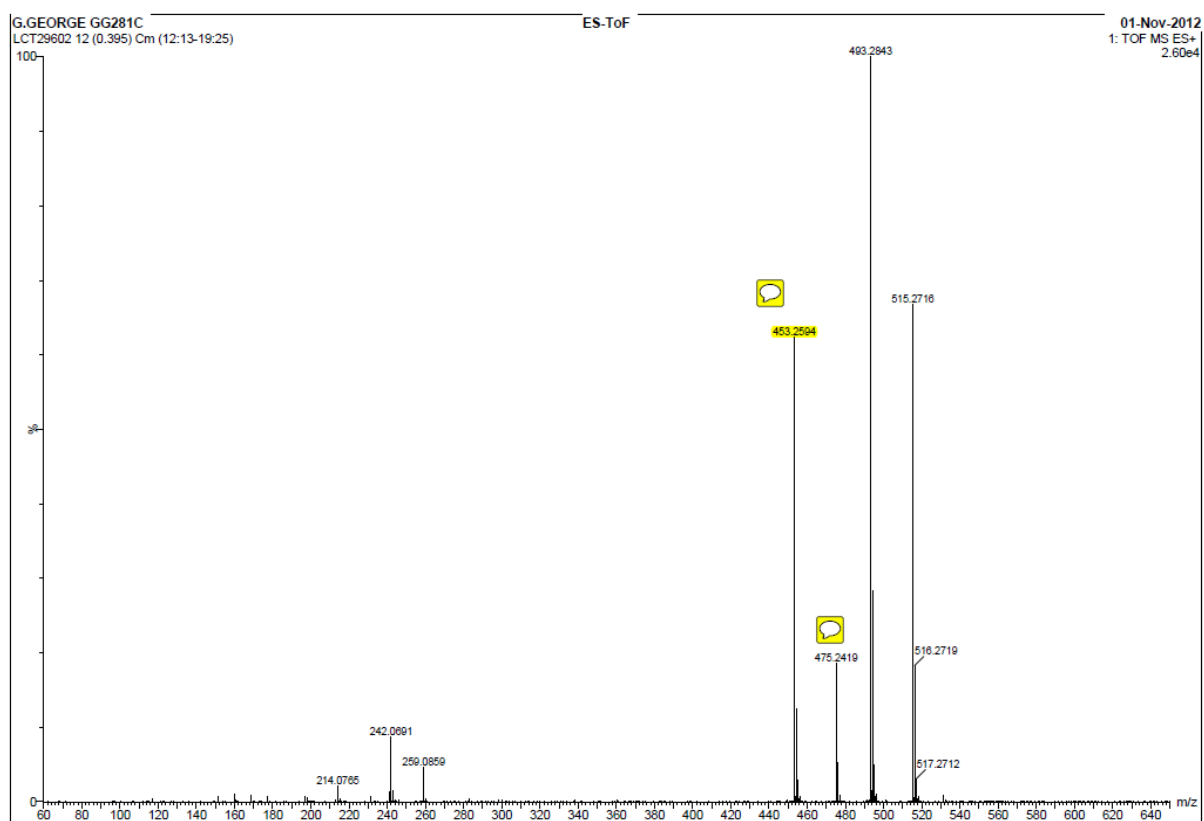


^{13}C NMR spectrum



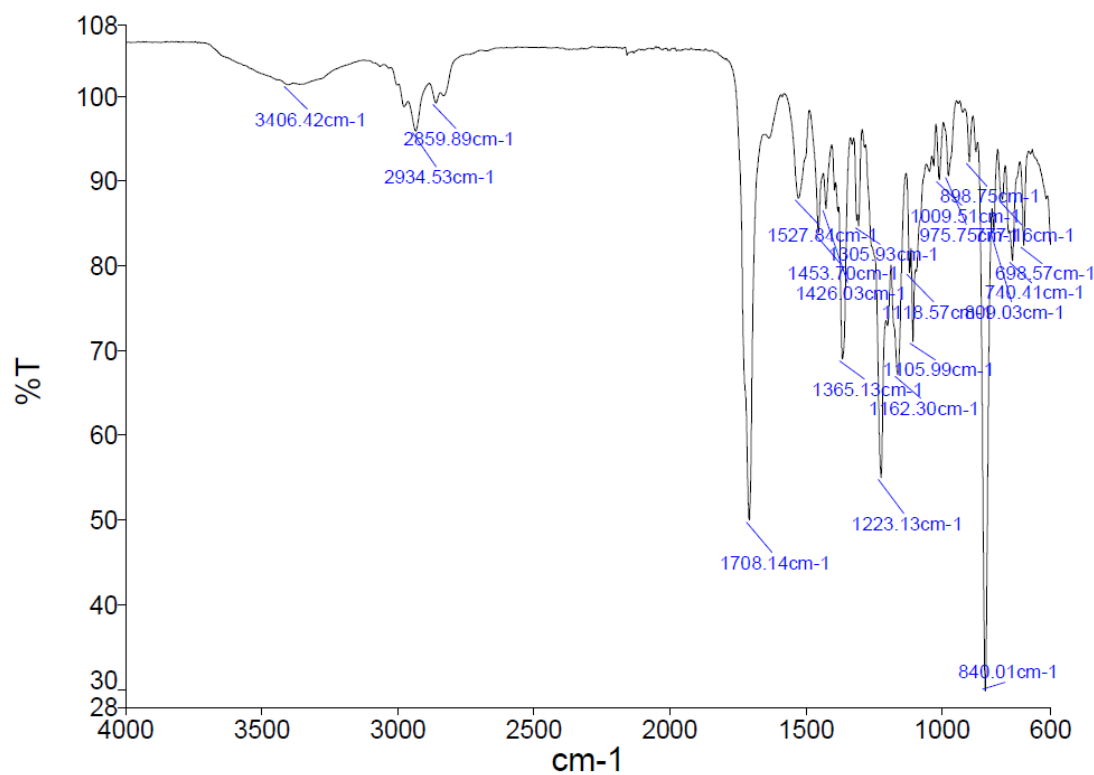
^{19}F NMR spectrum



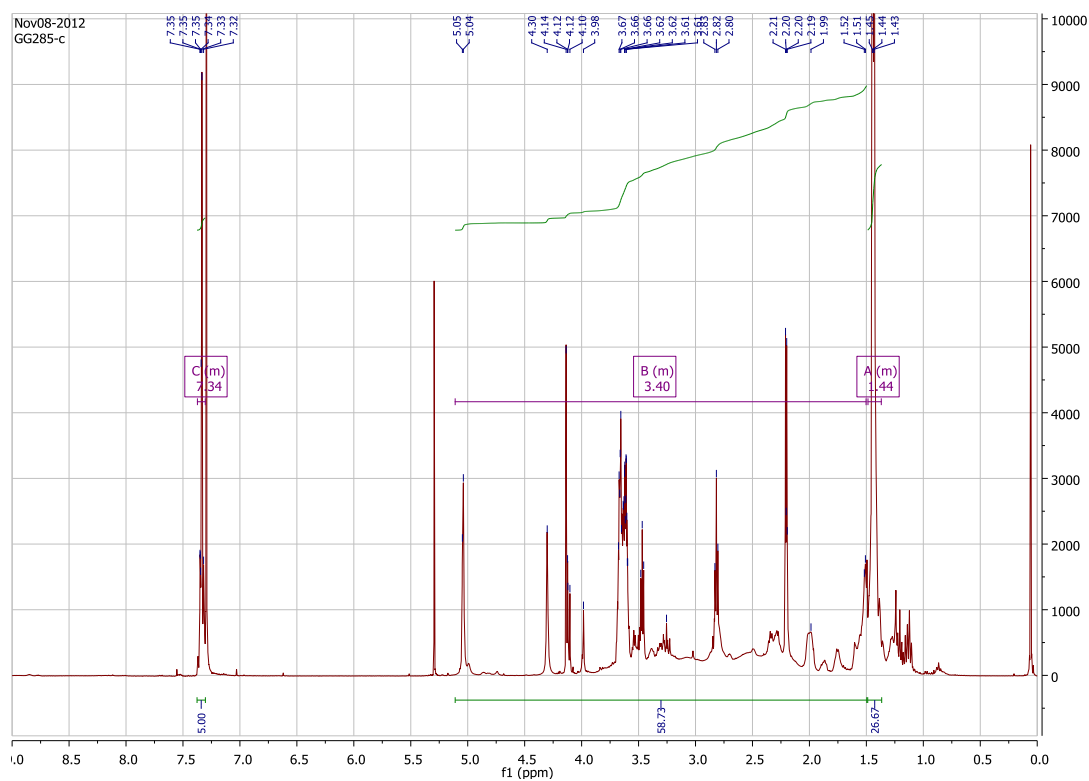


129. (1-{[(Benzyloxy)carbonyl]amino}cyclohexyl)methyl 2-(2-{2-[2-(2-{4,7,10-tris[2-(*tert*-butoxy)-2-oxoethyl]-1,4,7,10-tetraazacyclododecane-1-yl}acetamido)ethoxy]ethoxy}ethoxy)acetate

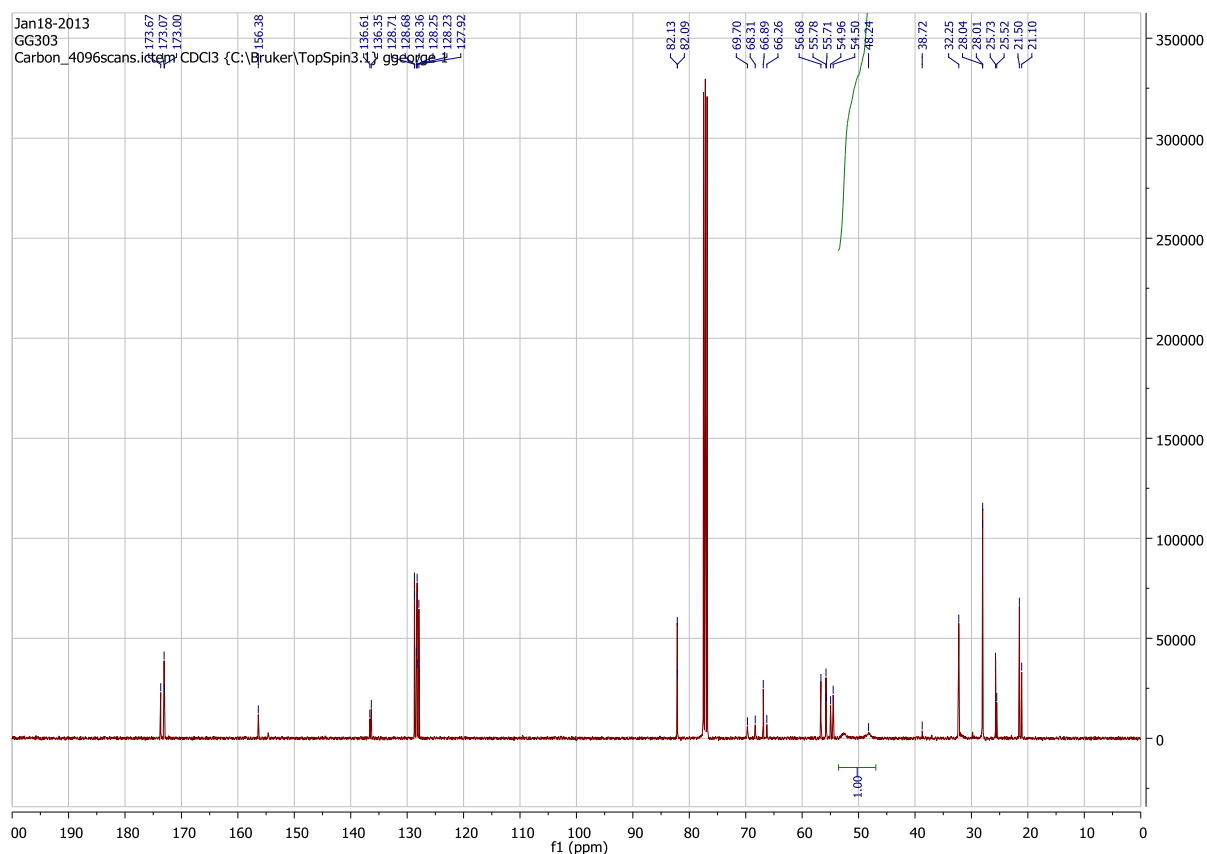
Infrared spectrum



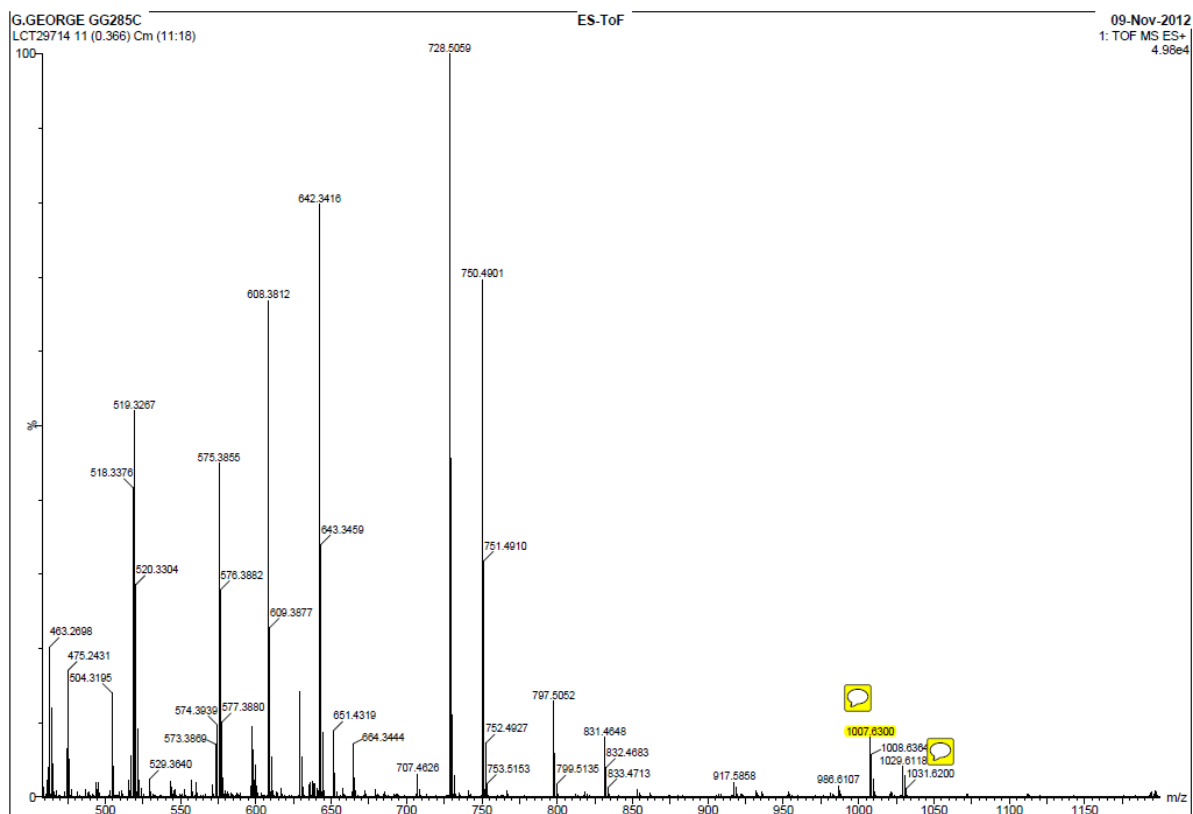
^1H NMR spectrum



^{13}C NMR spectrum

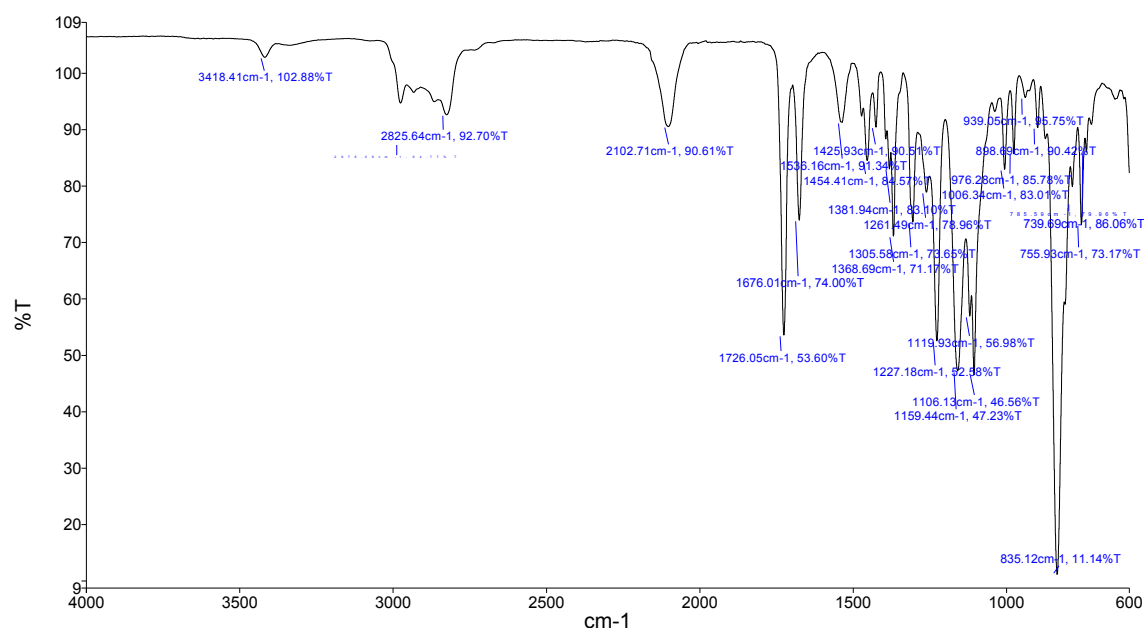


HRMS

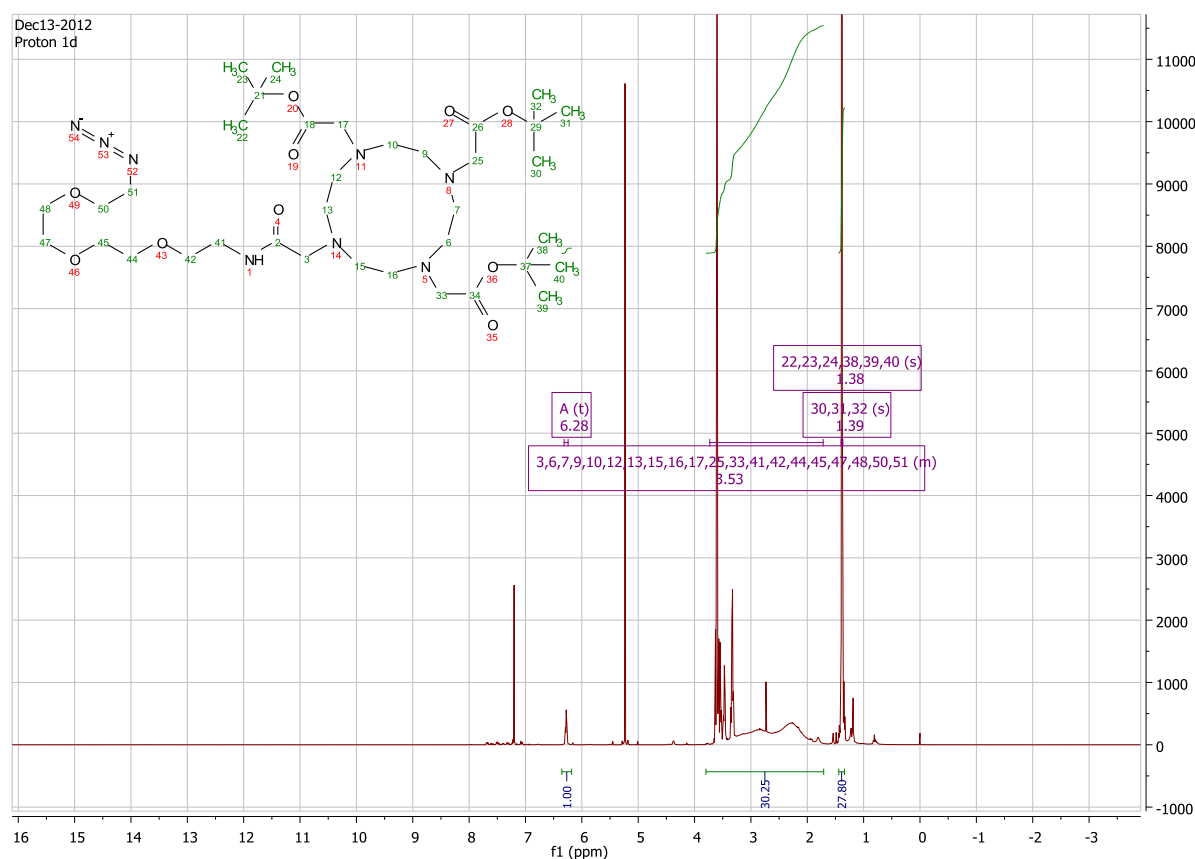


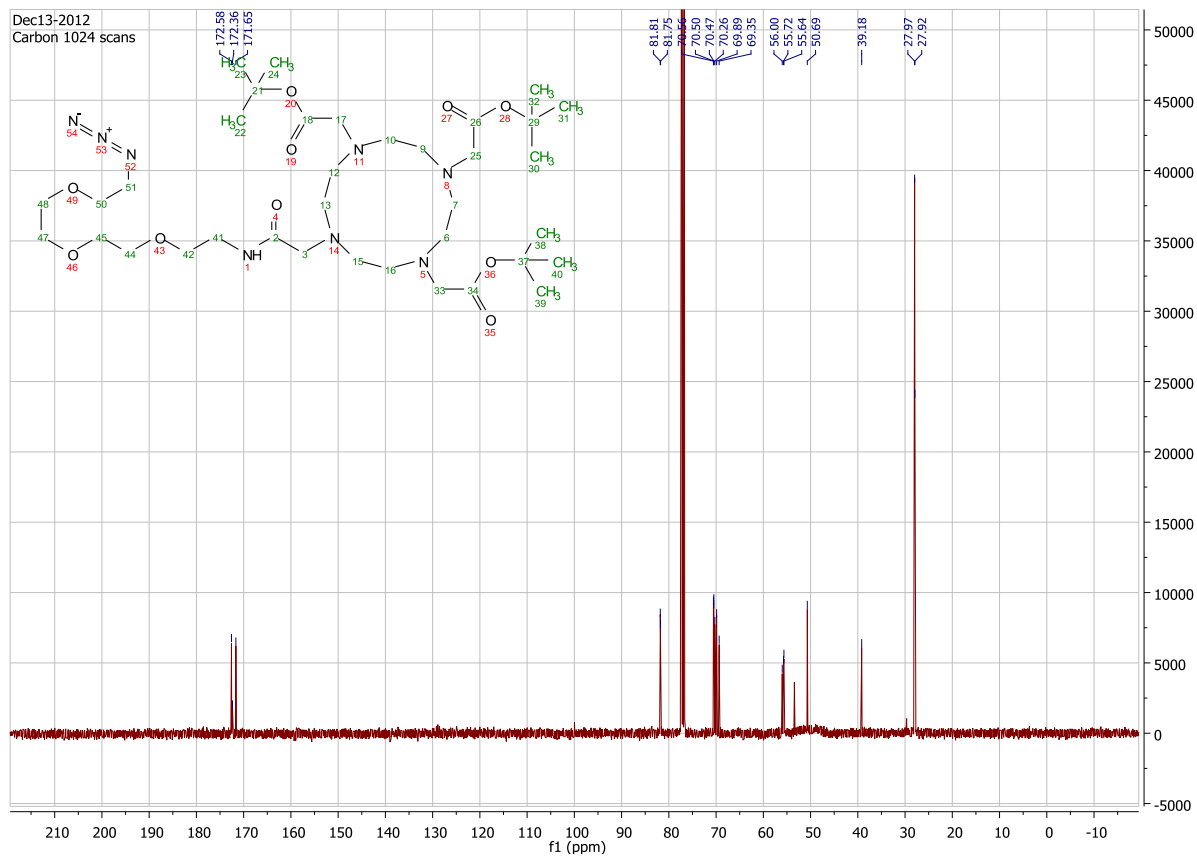
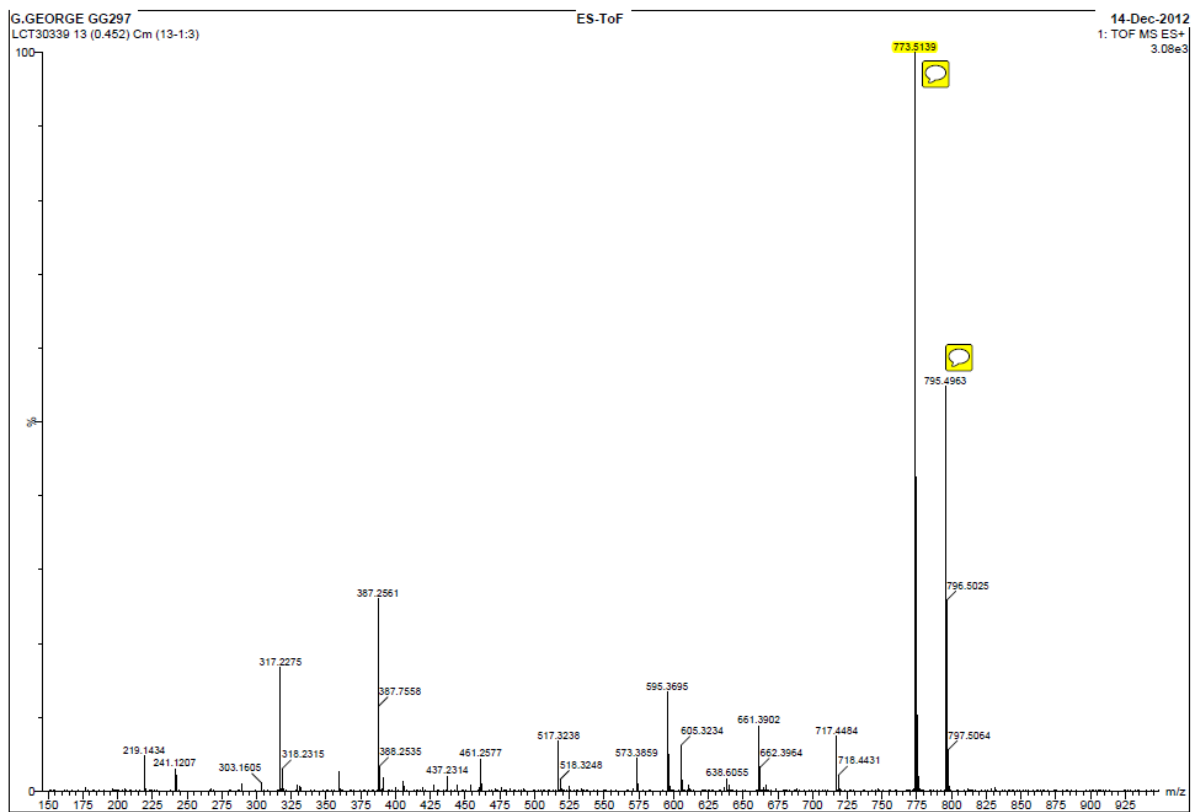
131. *tert*-Butyl 2-(7-[(2-{2-[2-(2-azidoethoxy)ethoxy]ethoxy}ethyl)carbamoyl]methyl}-4,10-bis[2-(*tert*-butoxy)-2-oxoethyl]-1,4,7,10-tetraazacyclododecan-1-yl)acetate

Infrared spectrum



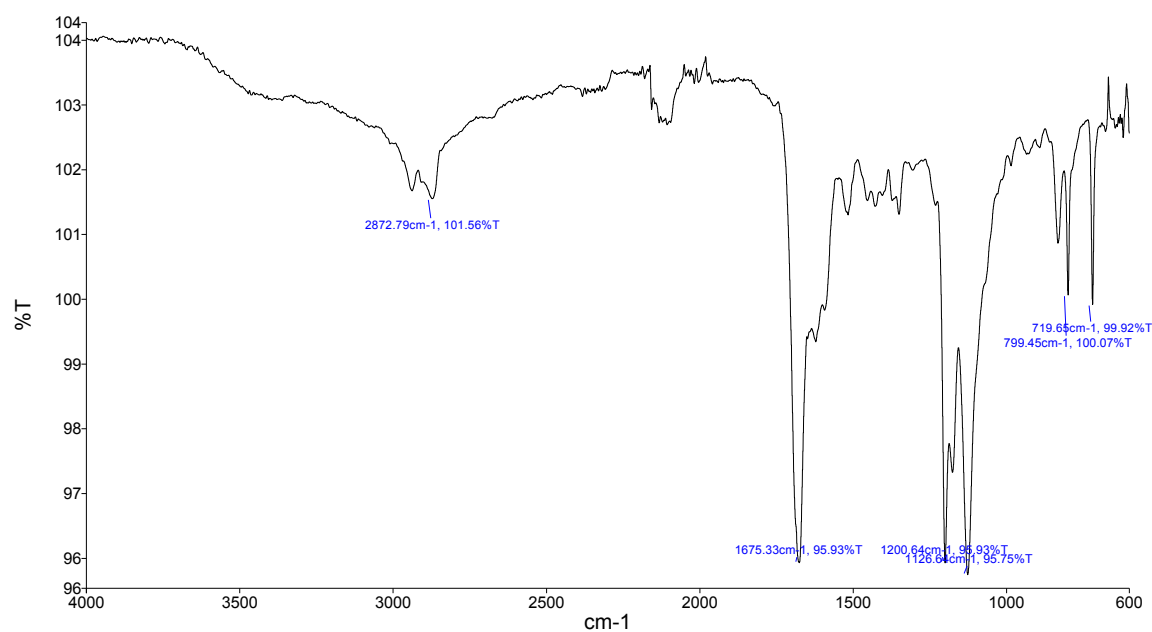
¹H NMR spectrum



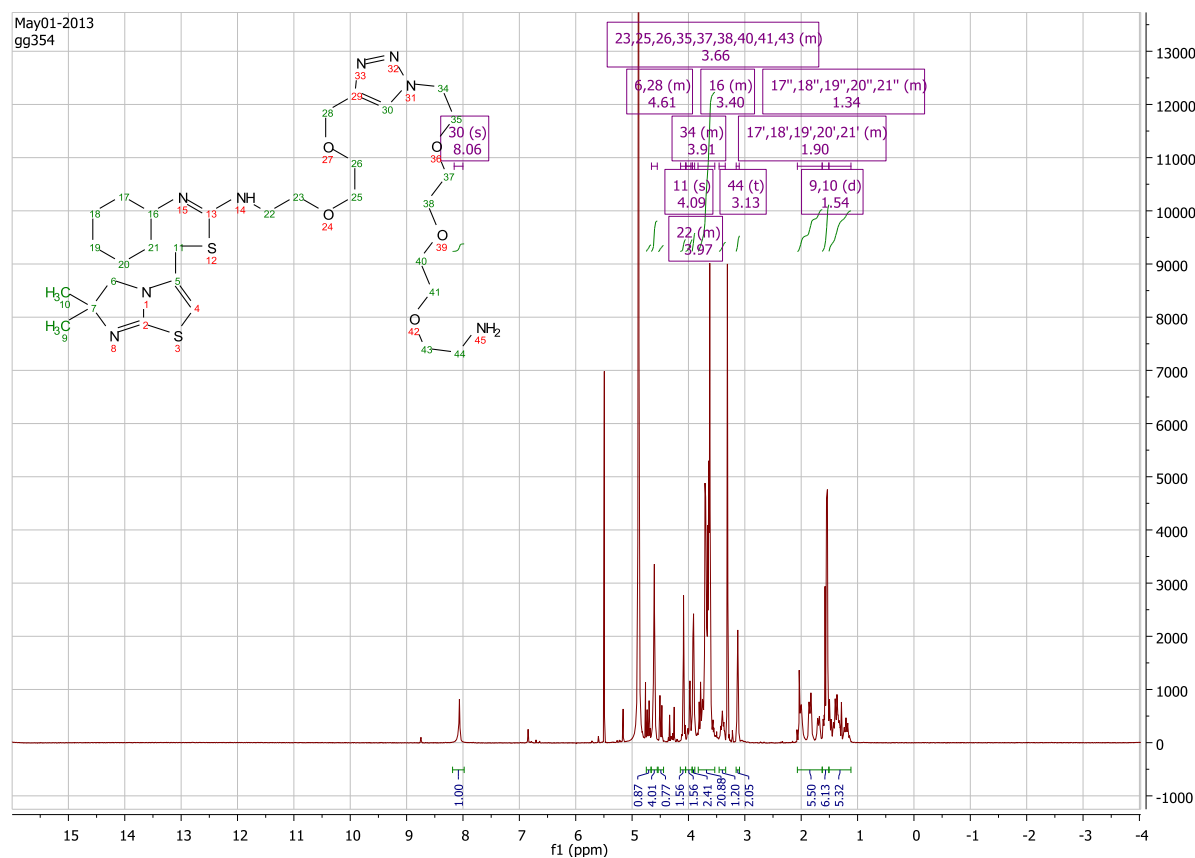
^{13}C NMR spectrum*HRMS*

132. 2-(2-([1-(2-{2-[2-(2-aminoethoxy)ethoxy]ethoxy}ethyl)-1H-1,2,3-triazol-4-yl]methoxy}ethoxy)-N-[(cyclohexylimino)[(6,6-dimethyl-5H,6H-imidazo[2,1-b][1,3]thiazol-3-yl)methyl]sulfanyl)methyl]ethan-1-amine .4TFA

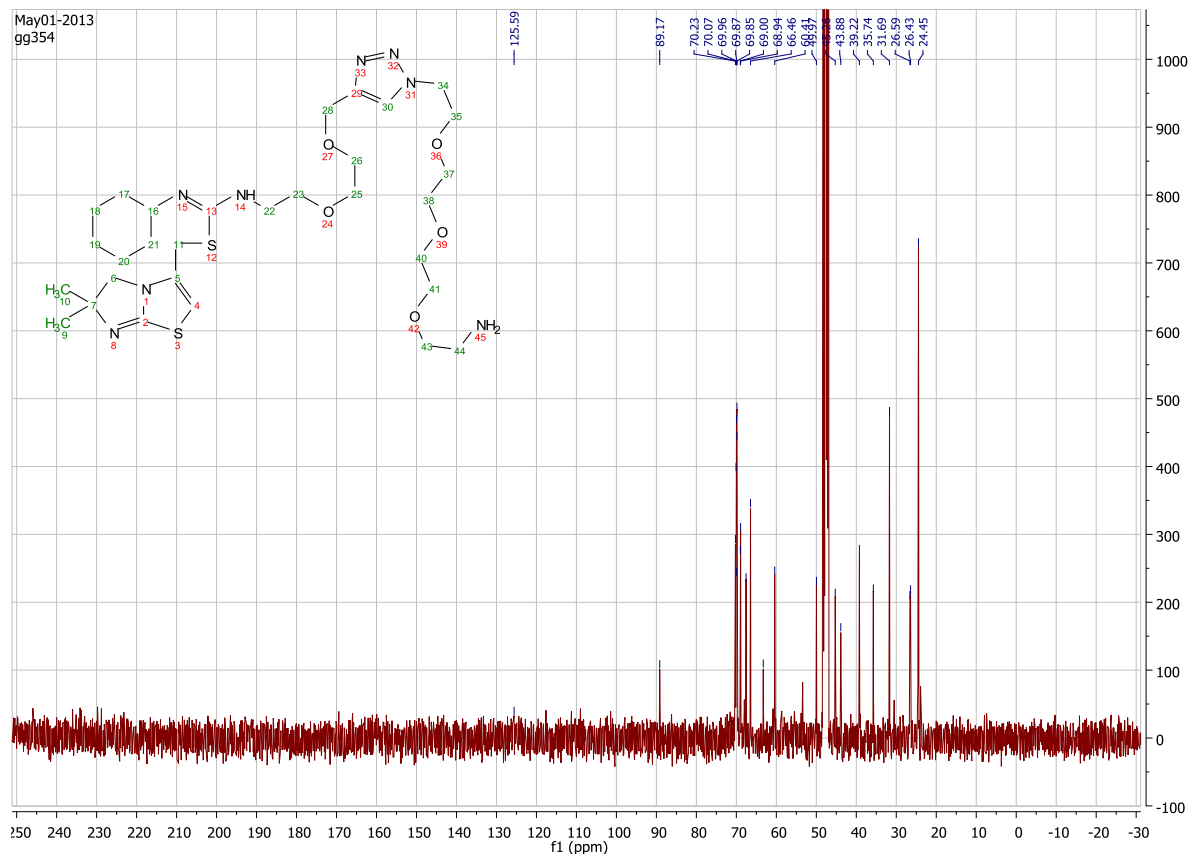
Infrared spectrum



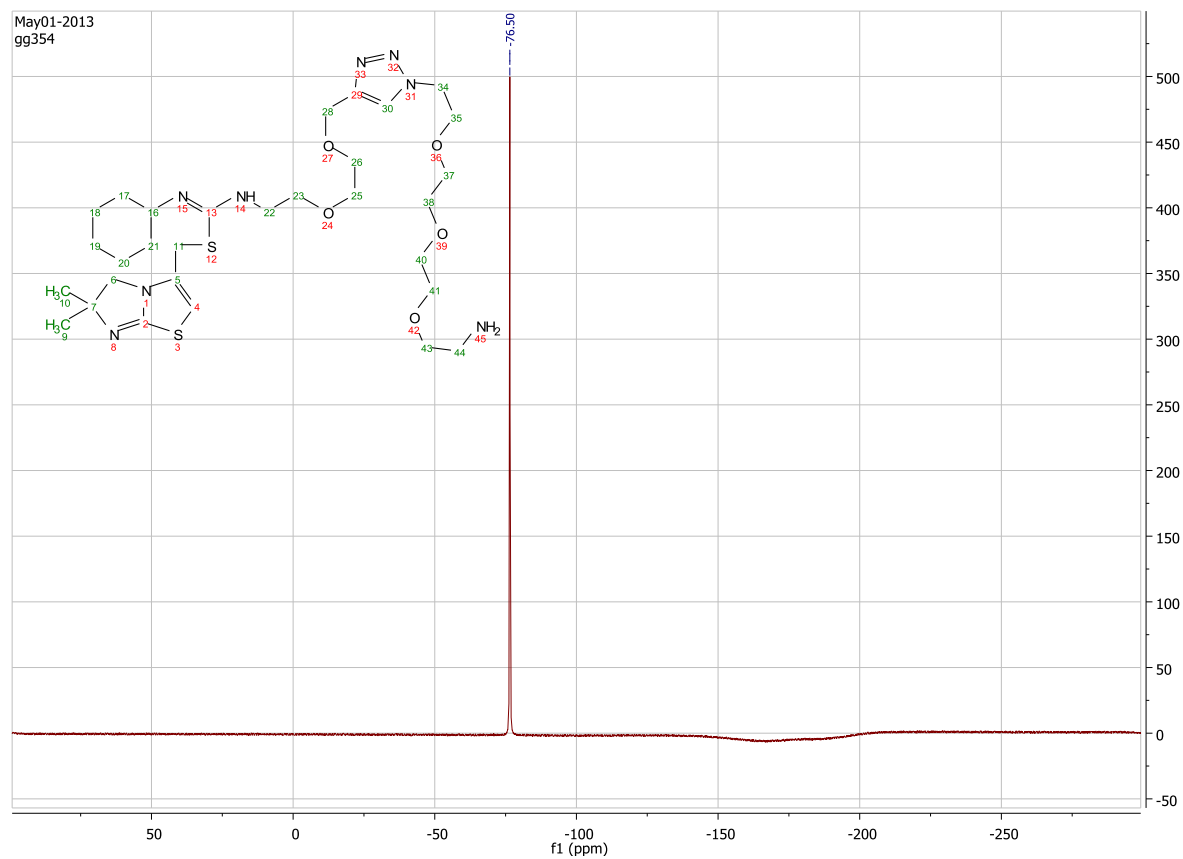
¹H NMR spectrum

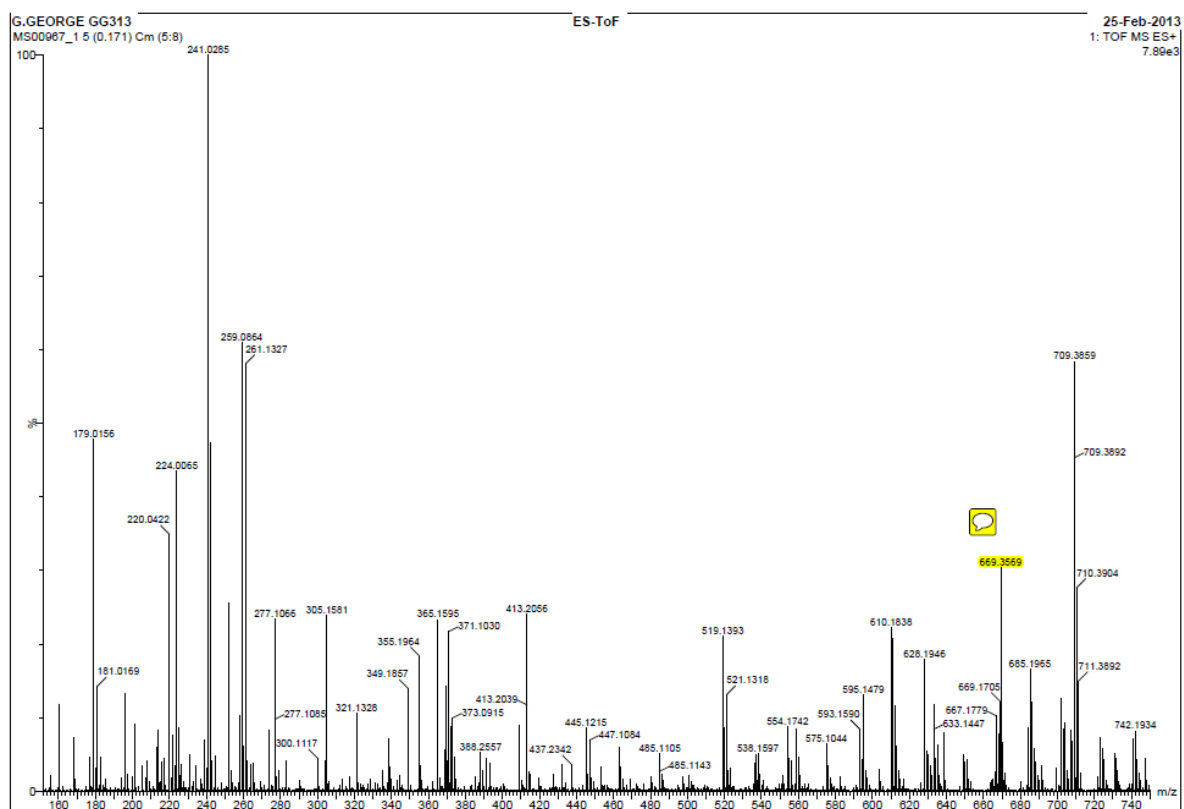


^{13}C NMR spectrum



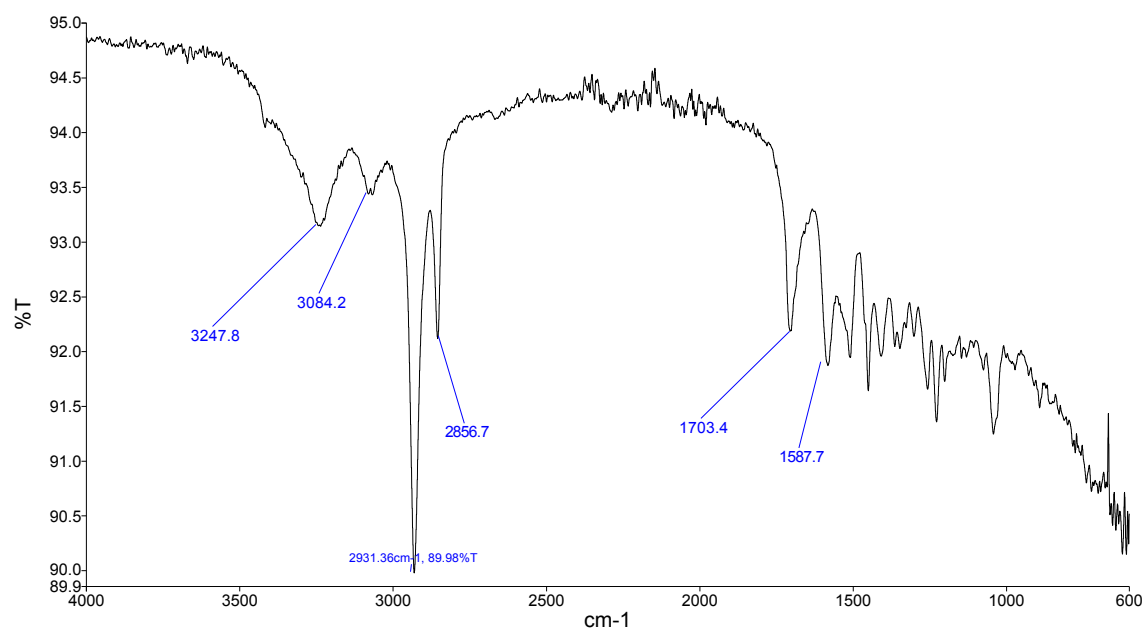
^{19}F NMR spectrum



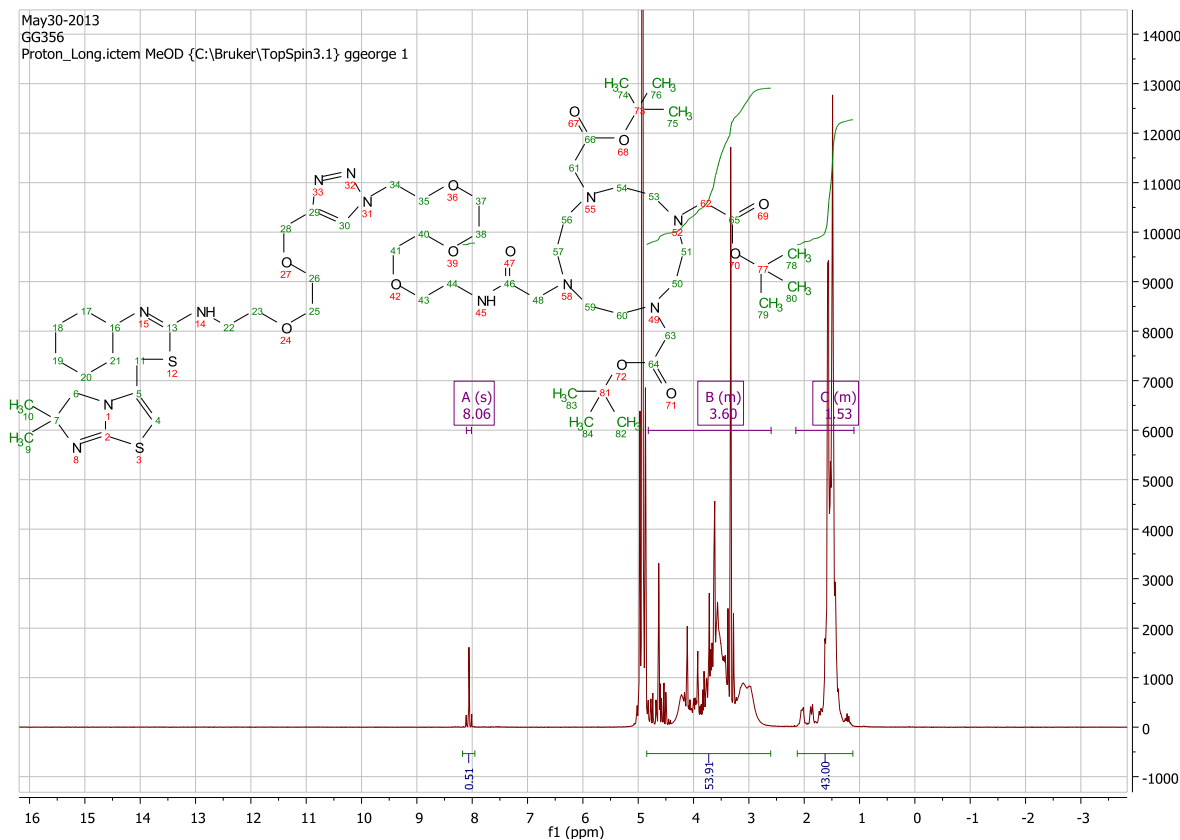


133. *tert*-Butyl 2-{4,10-bis[2-(*tert*-butoxy)-2-oxoethyl]-7-[(2-[2-(2-{2-[4-(*N*-cyclohexyl-1-{2-[2-((6,6-dimethyl-5*H*,6*H*-imidazo{2,1-*b*][1,3]thiazol-3-yl)methyl)sulfanyl]methanimidoyl}amino)ethoxy]ethoxy)methyl)-1*H*-1,2,3-triazol-1-yl]ethoxy}ethoxy)ethoxy]ethyl}carbonyl)methyl]-1,4,7,10-tetraazacyclododecan-1-yl}acetate .7TFA

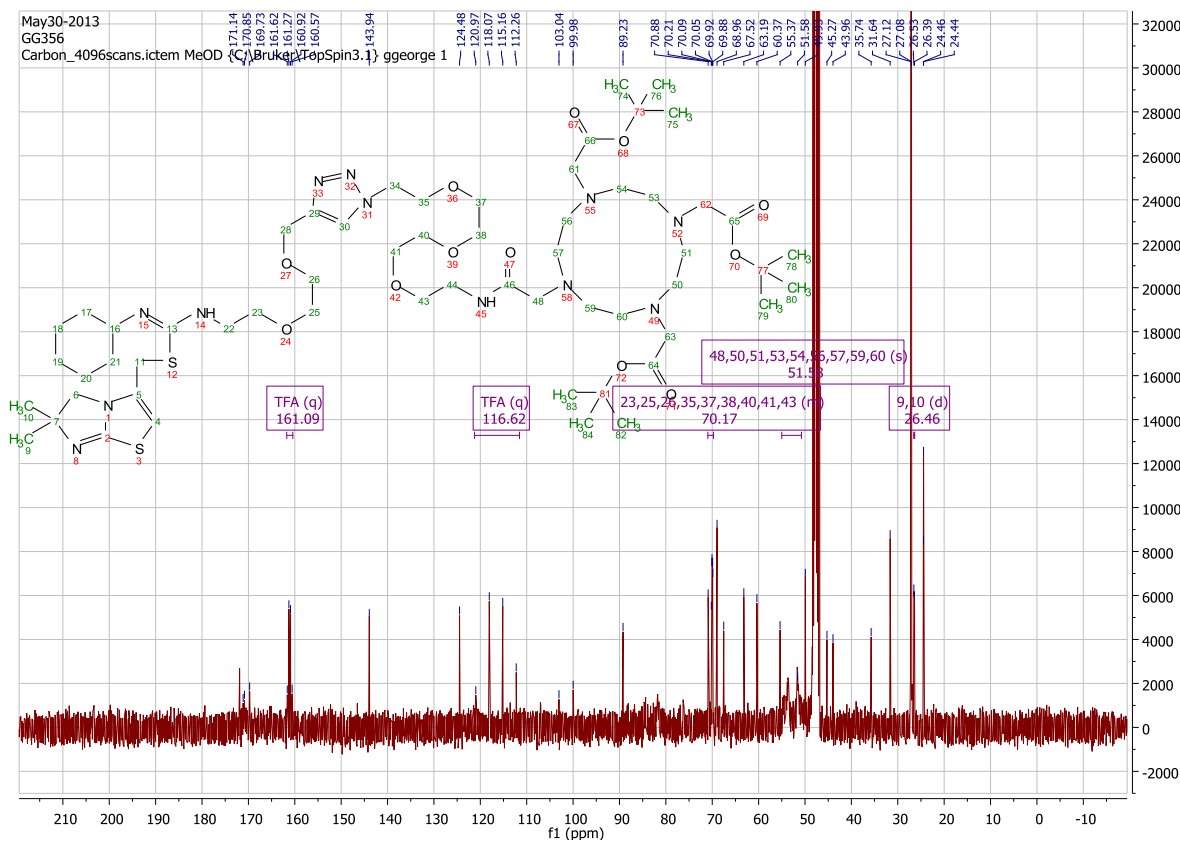
Infrared spectrum



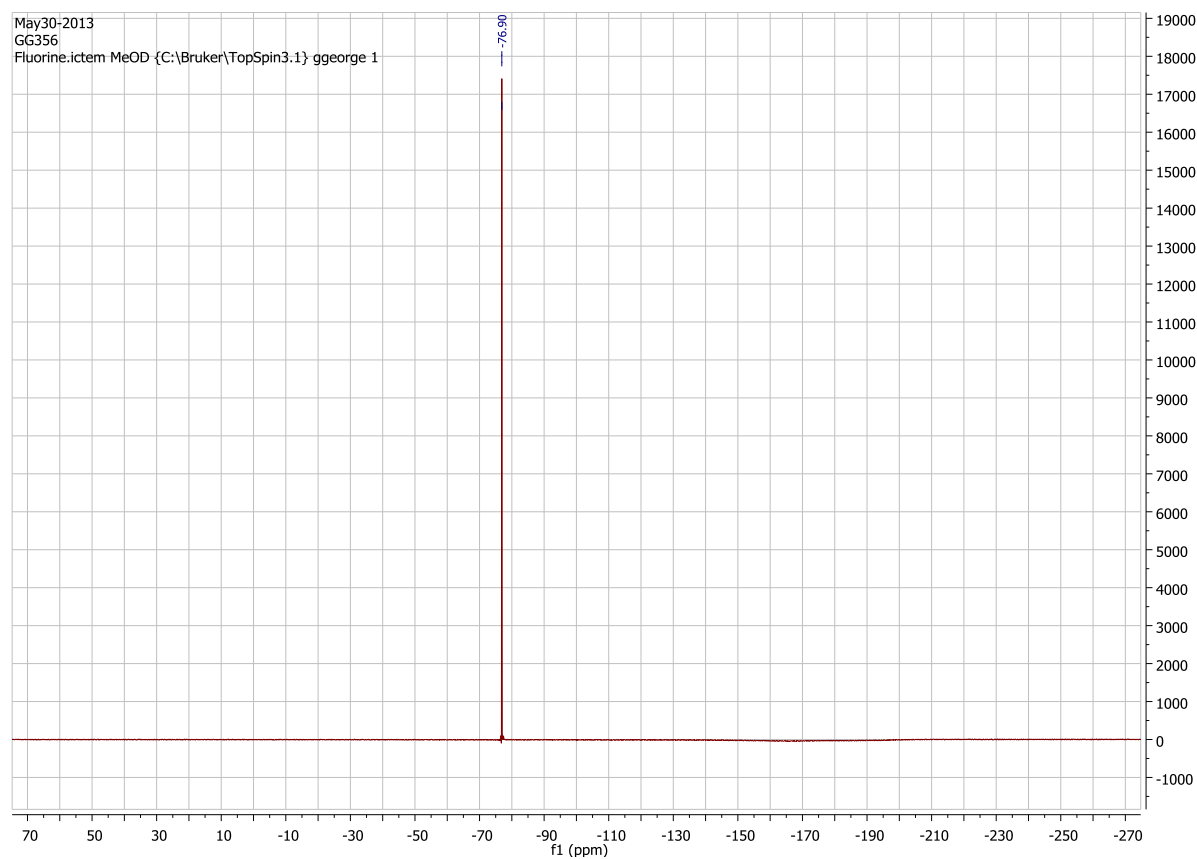
^1H NMR spectrum



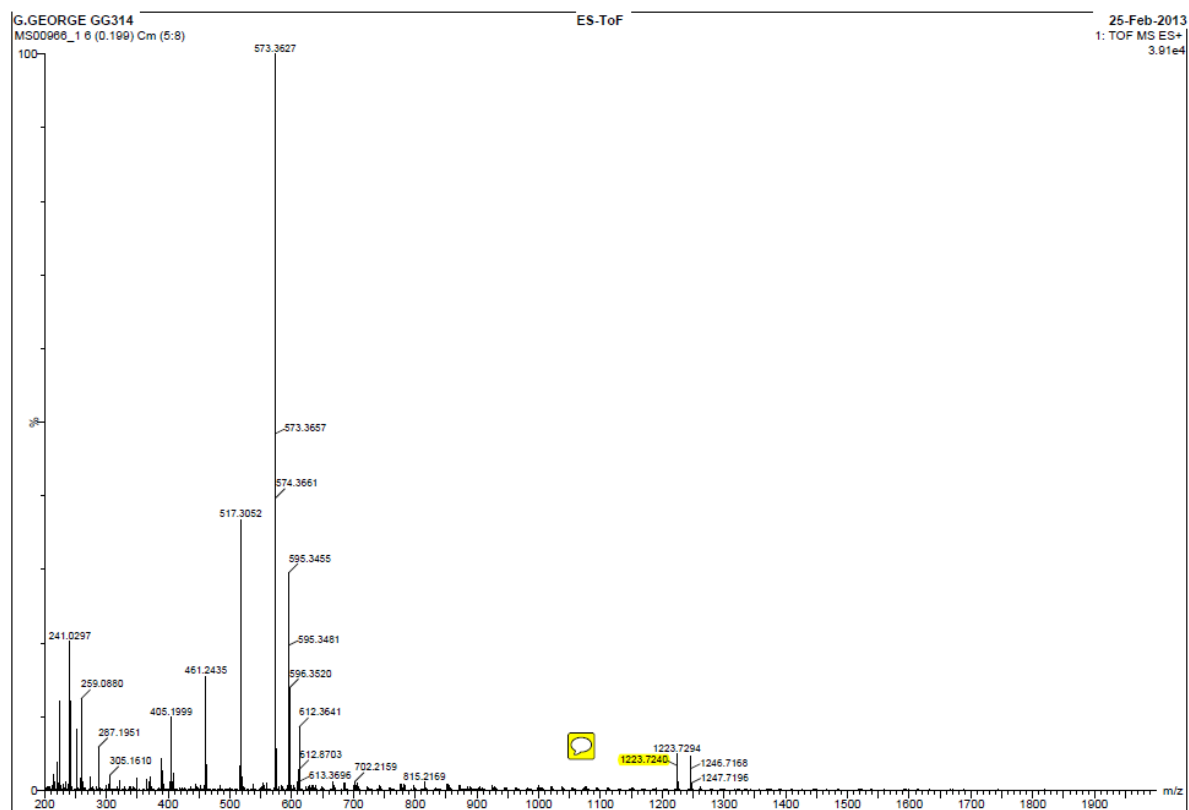
^{13}C NMR spectrum



^{19}F NMR spectrum

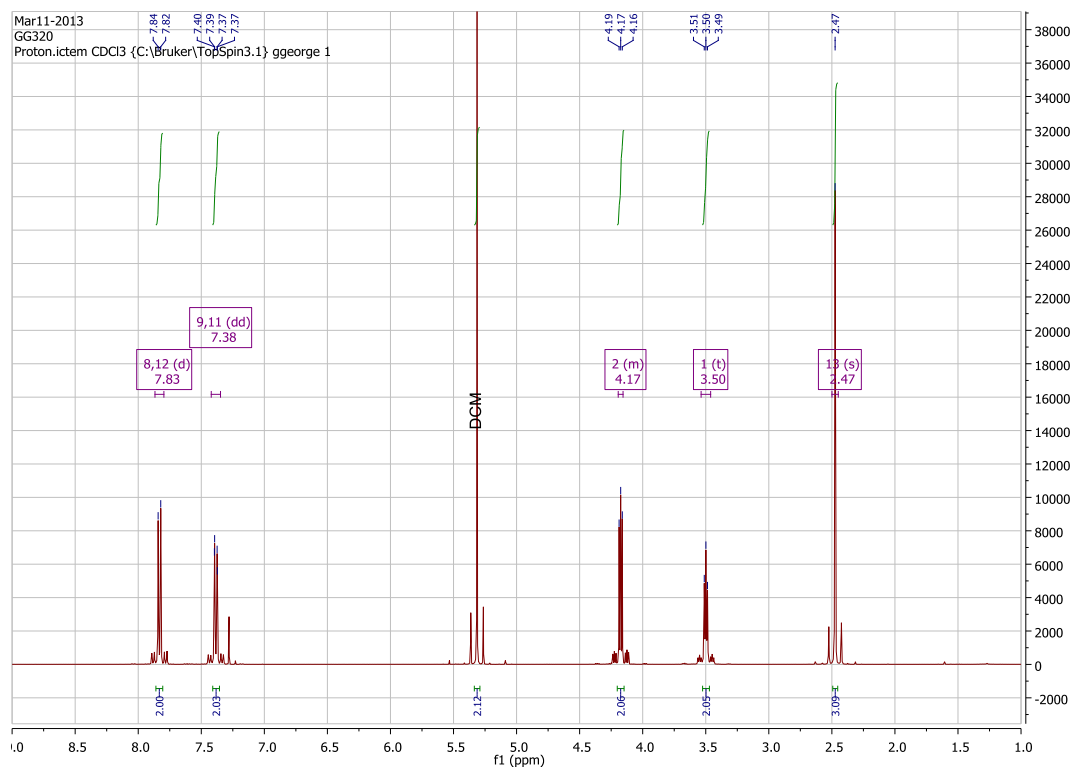


HRMS

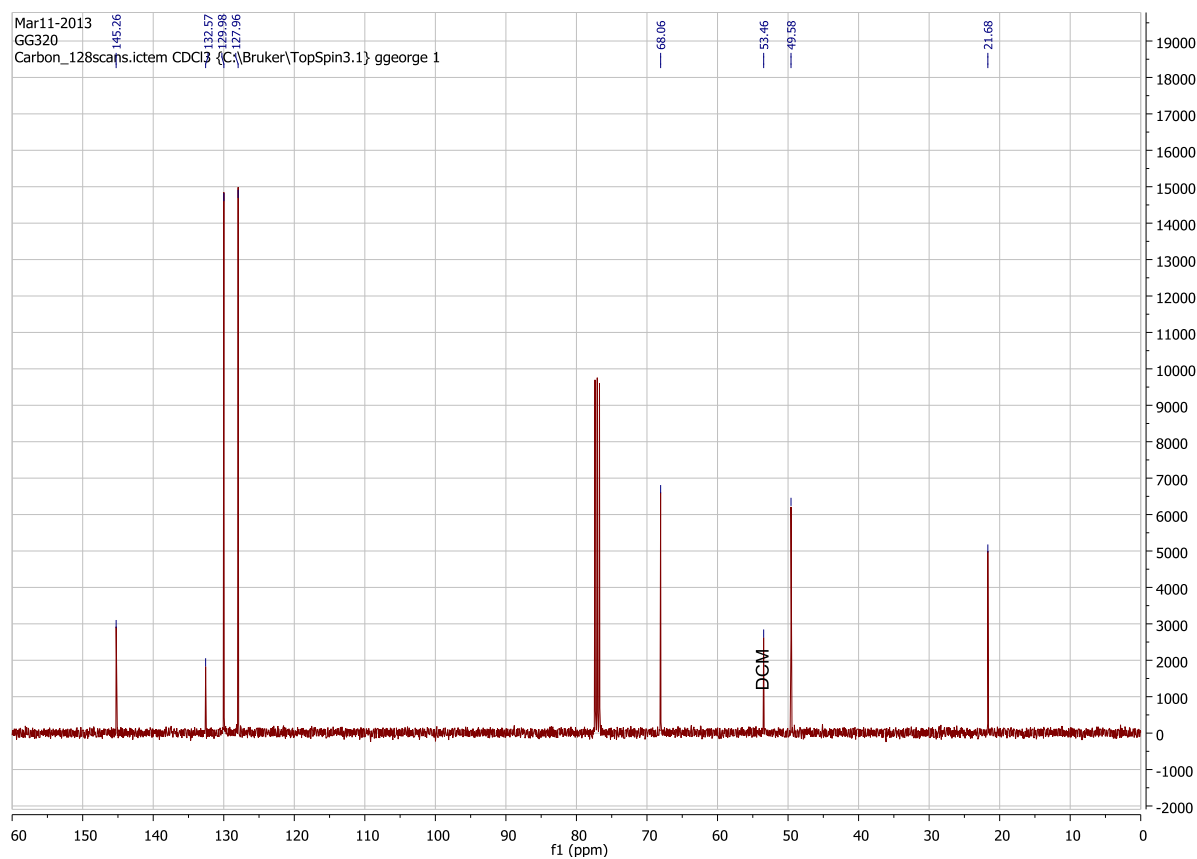


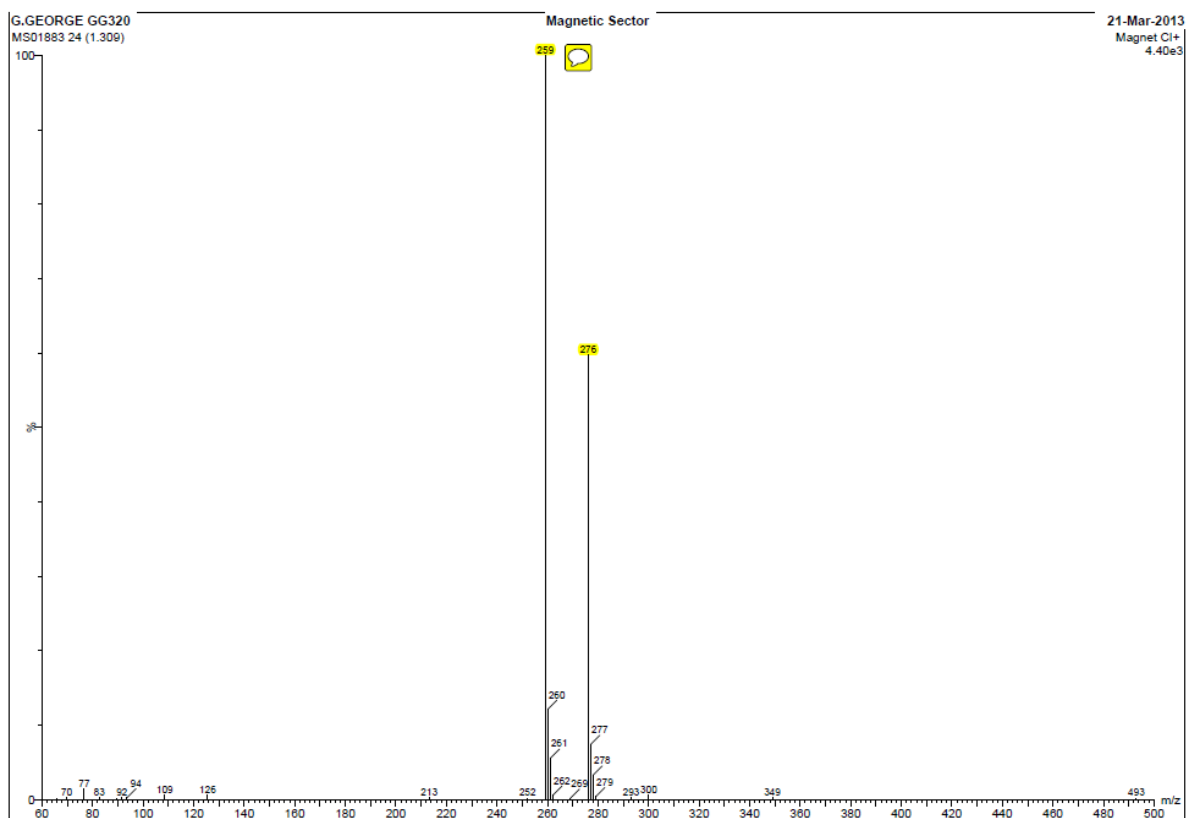
134. 1-[(2-Azidoethoxy)sulfonyl]4-methylbenzene

^1H NMR spectrum



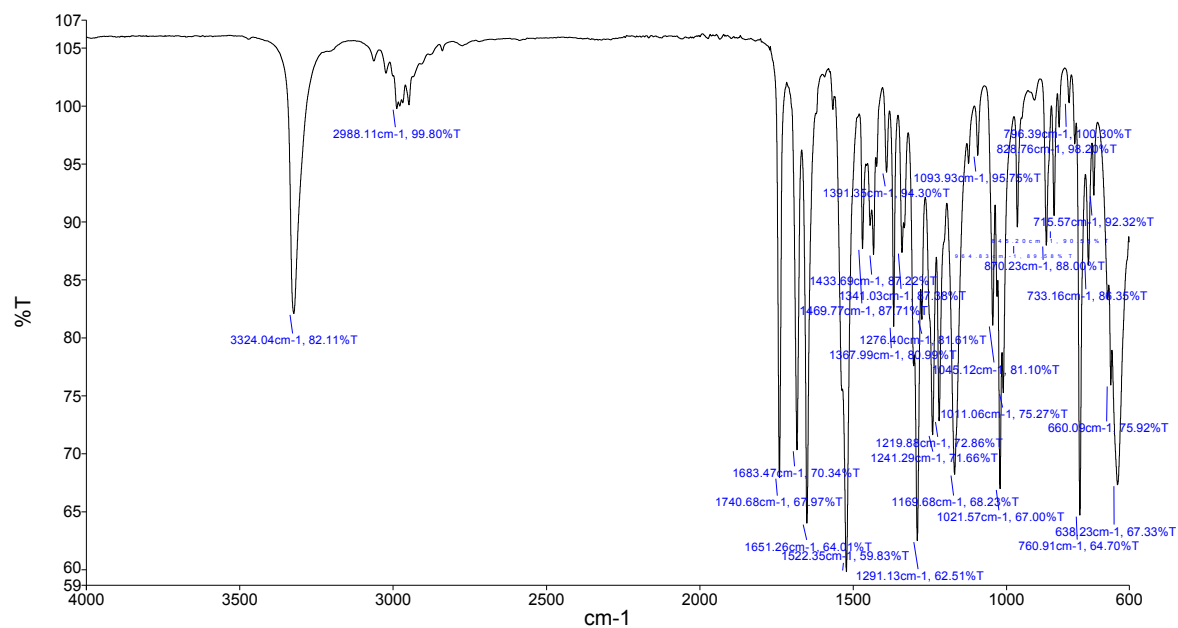
^{13}C NMR spectrum



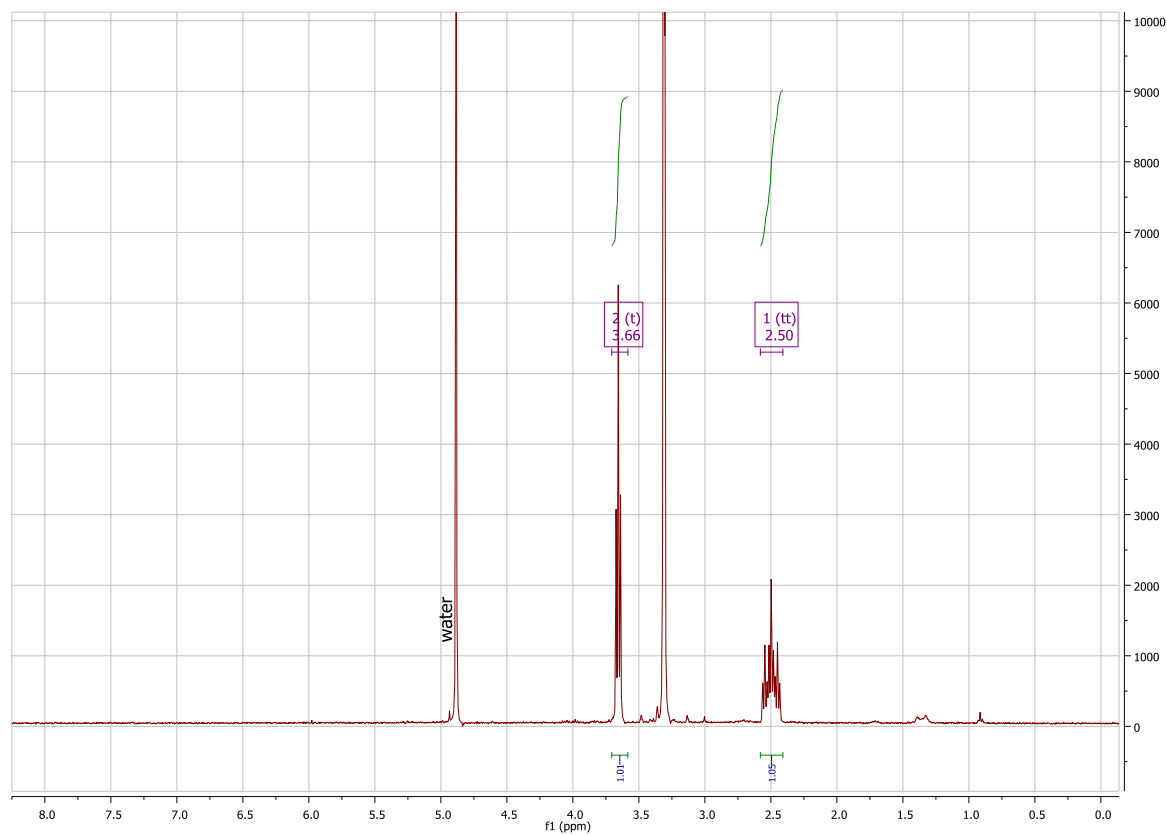


135. 10-Azido-1,1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-heptafluorodecane

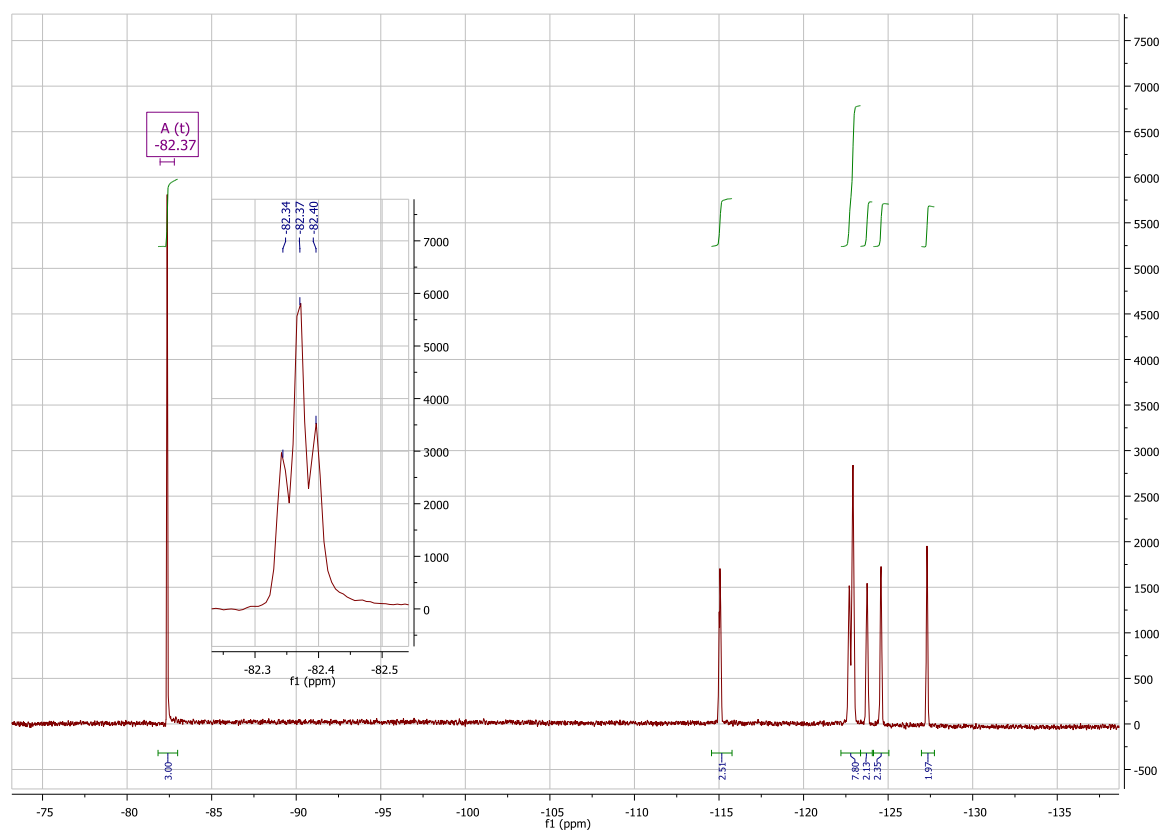
Infrared spectrum



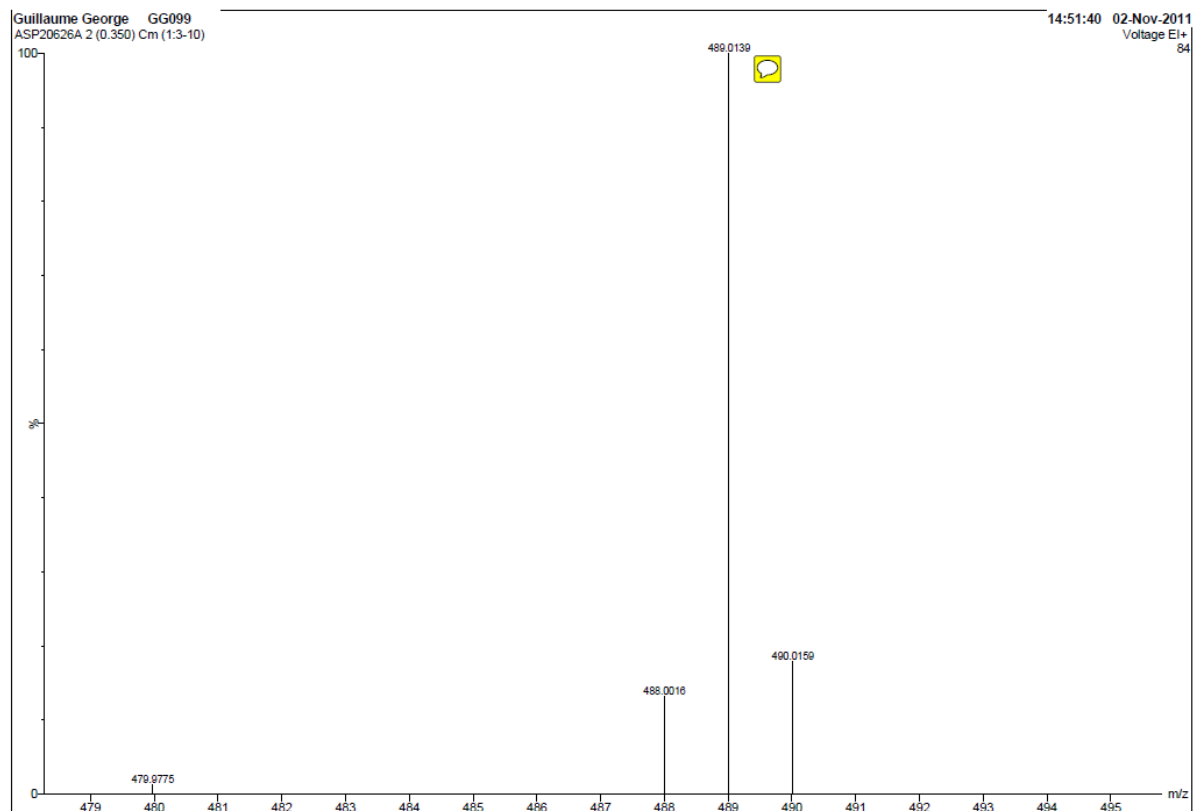
^1H NMR spectrum



^{19}F NMR spectrum

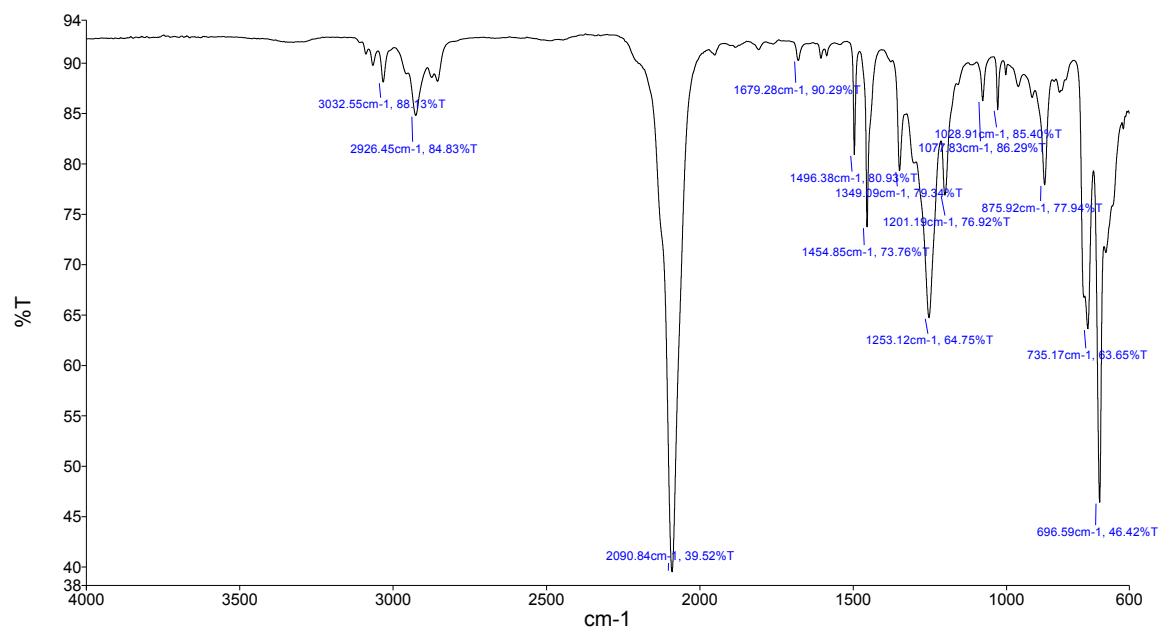


HRMS

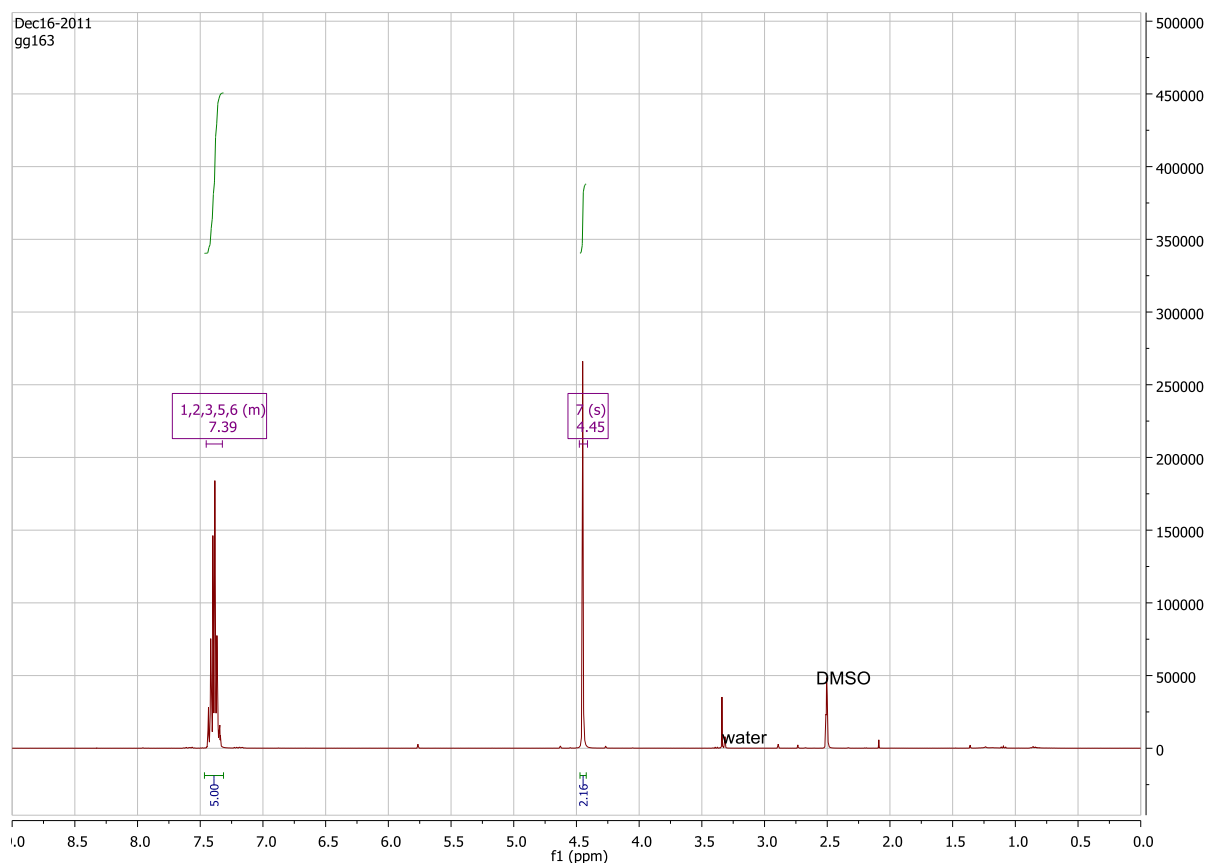


136. (Azidomethyl)benzene

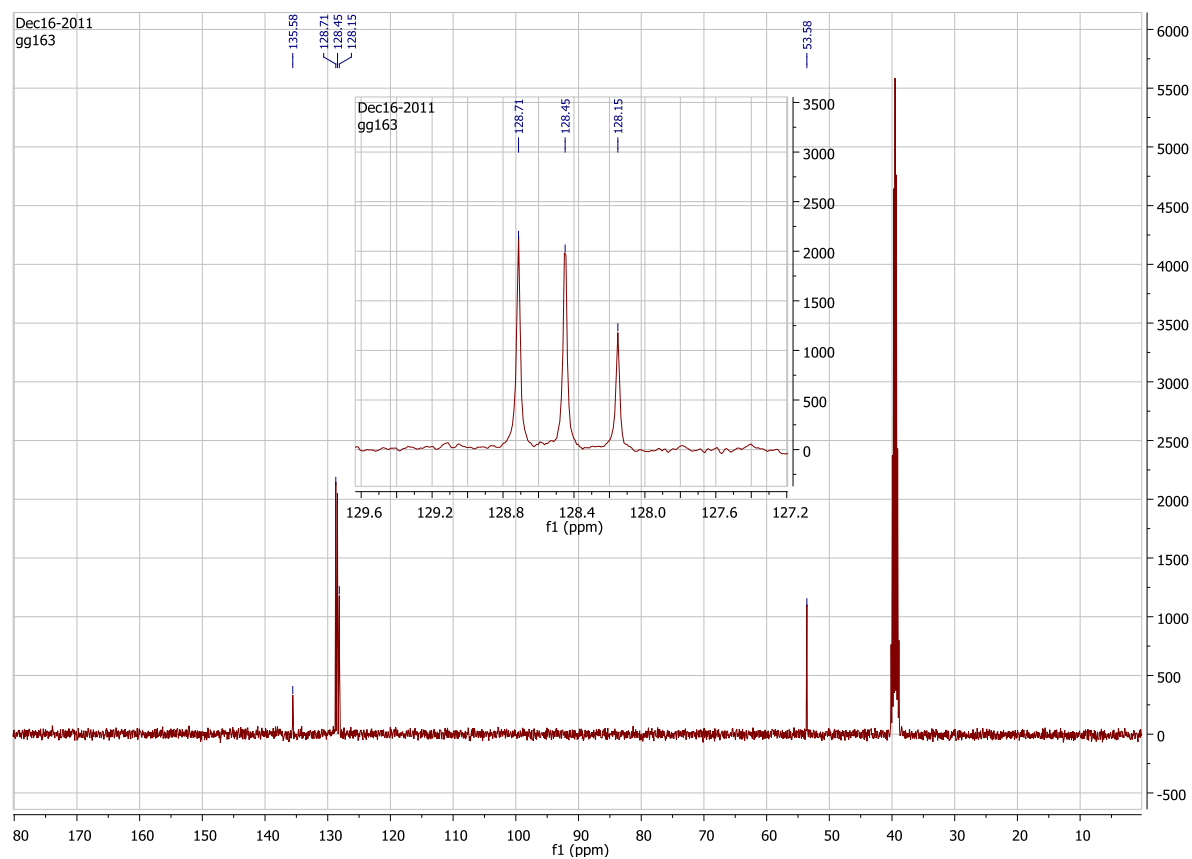
Infrared spectrum



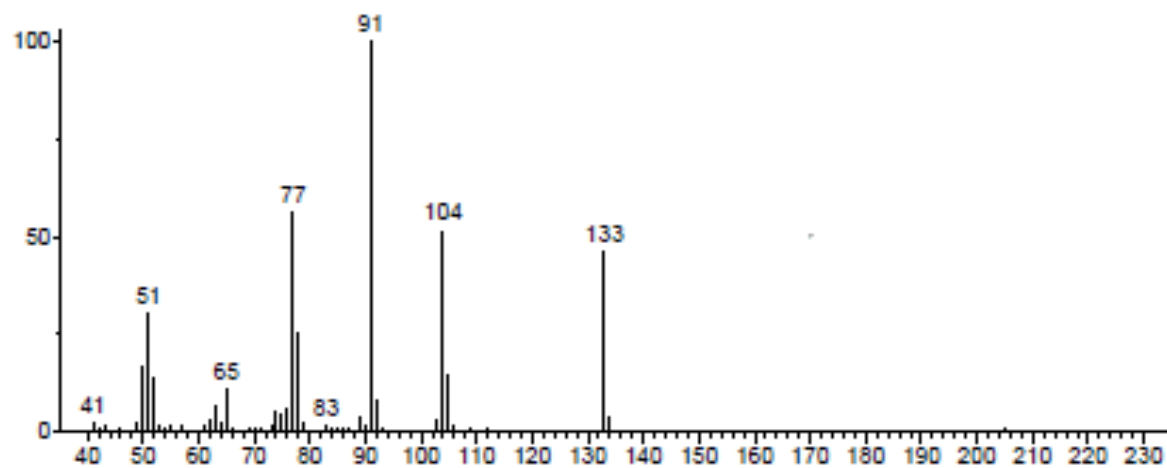
¹H NMR spectrum



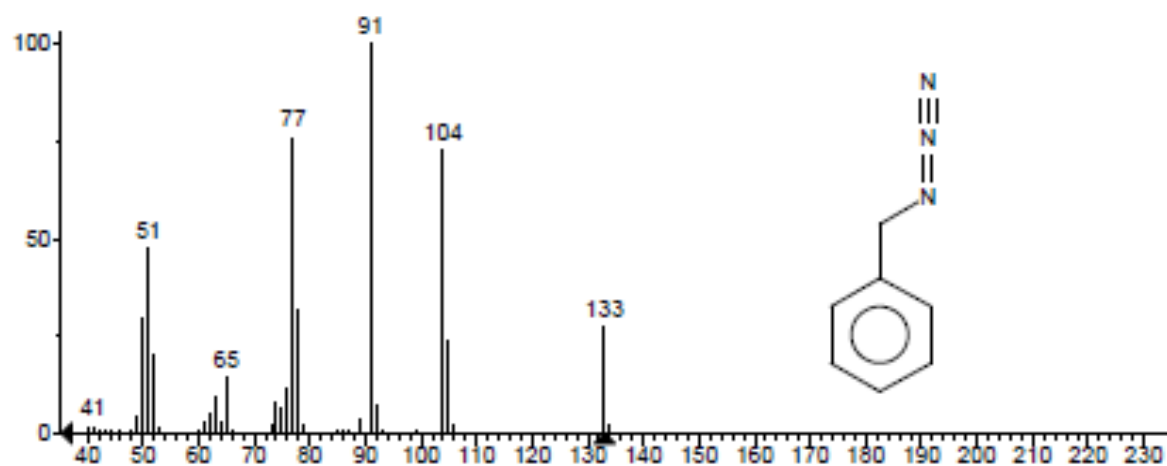
^{13}C NMR spectrum

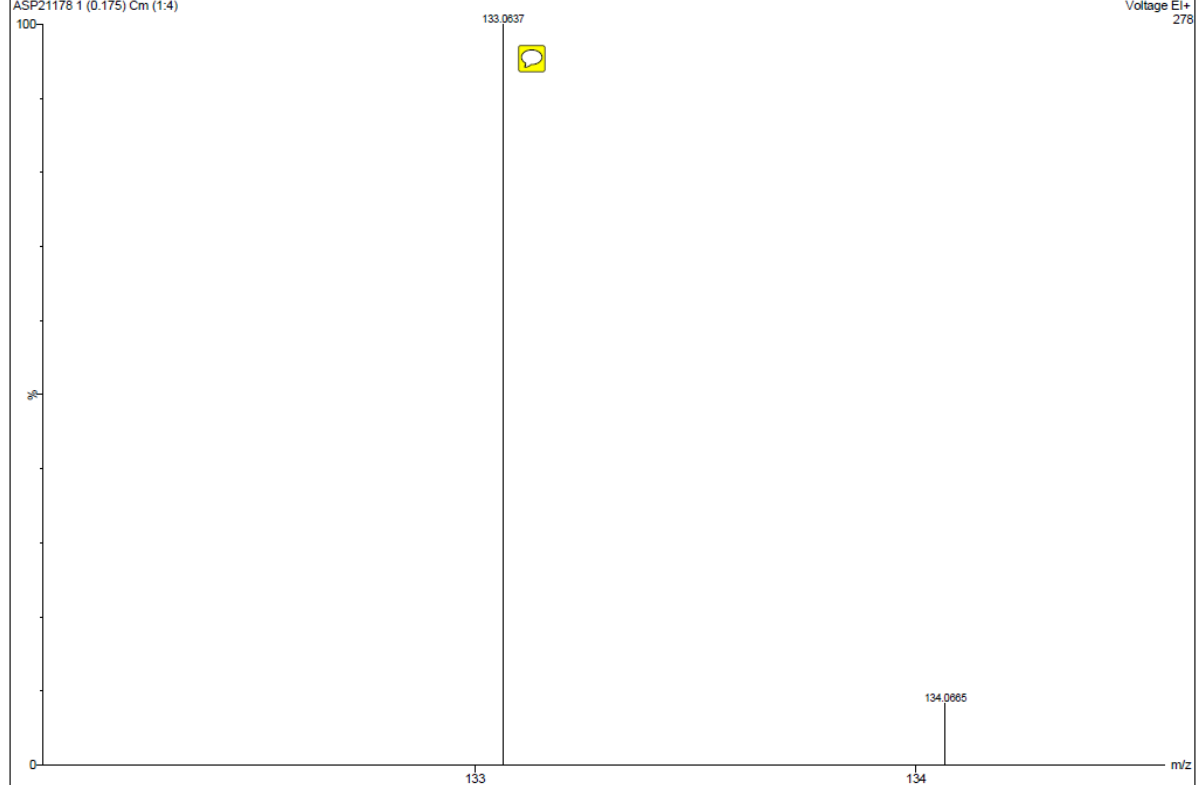


Unknown: ASP21170 3 (0.364)
Compound In Library Factor = 162



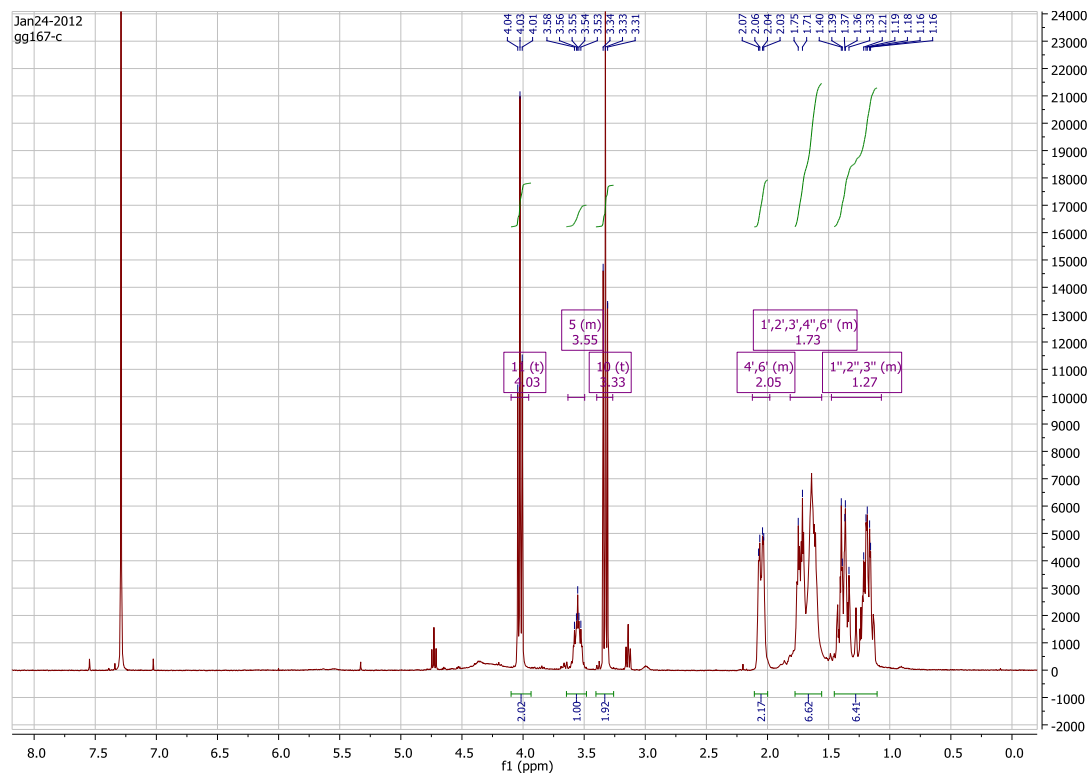
Hit 1 : Benzene, (azidomethyl)-
C7H7N3; MF: 888; RMF: 909; Prob 73.7%; CAS: 622-79-7; Lib: mainlib; ID: 44778.



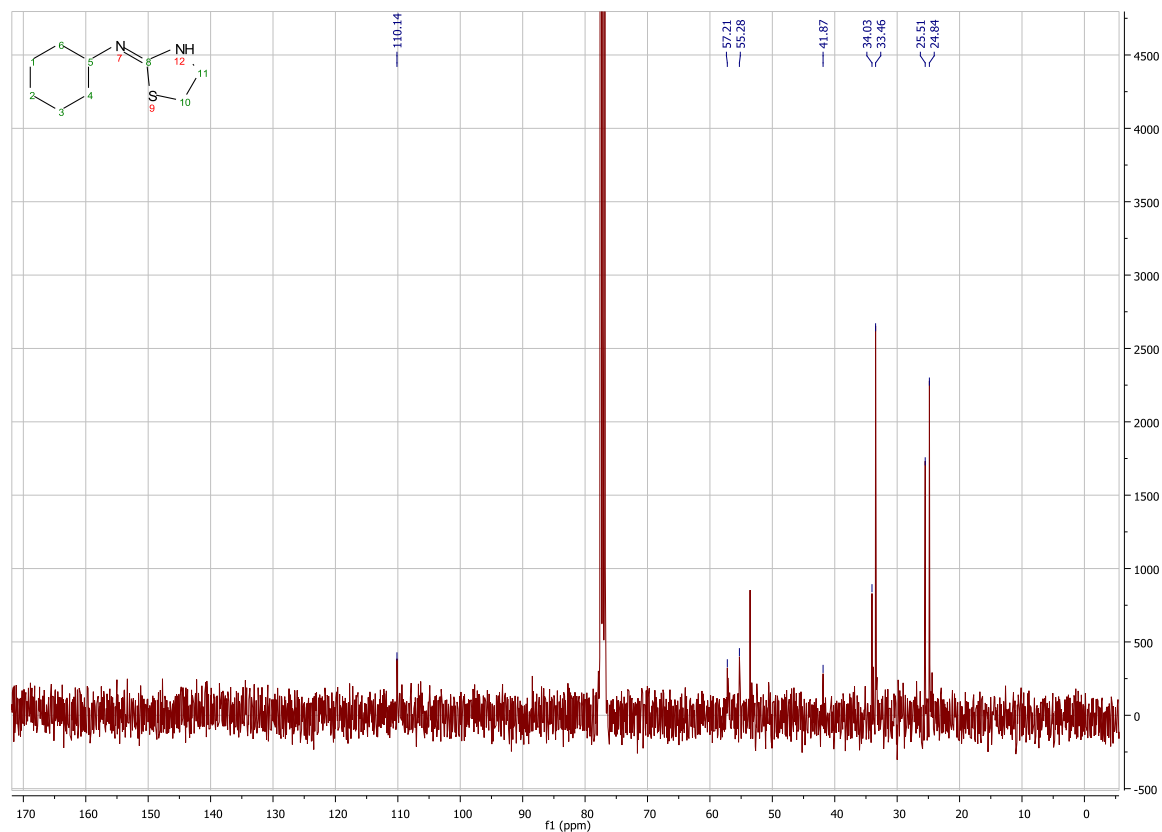


xxx. *N*-Cyclohexyl-1,3-thiazolidin-2-imine

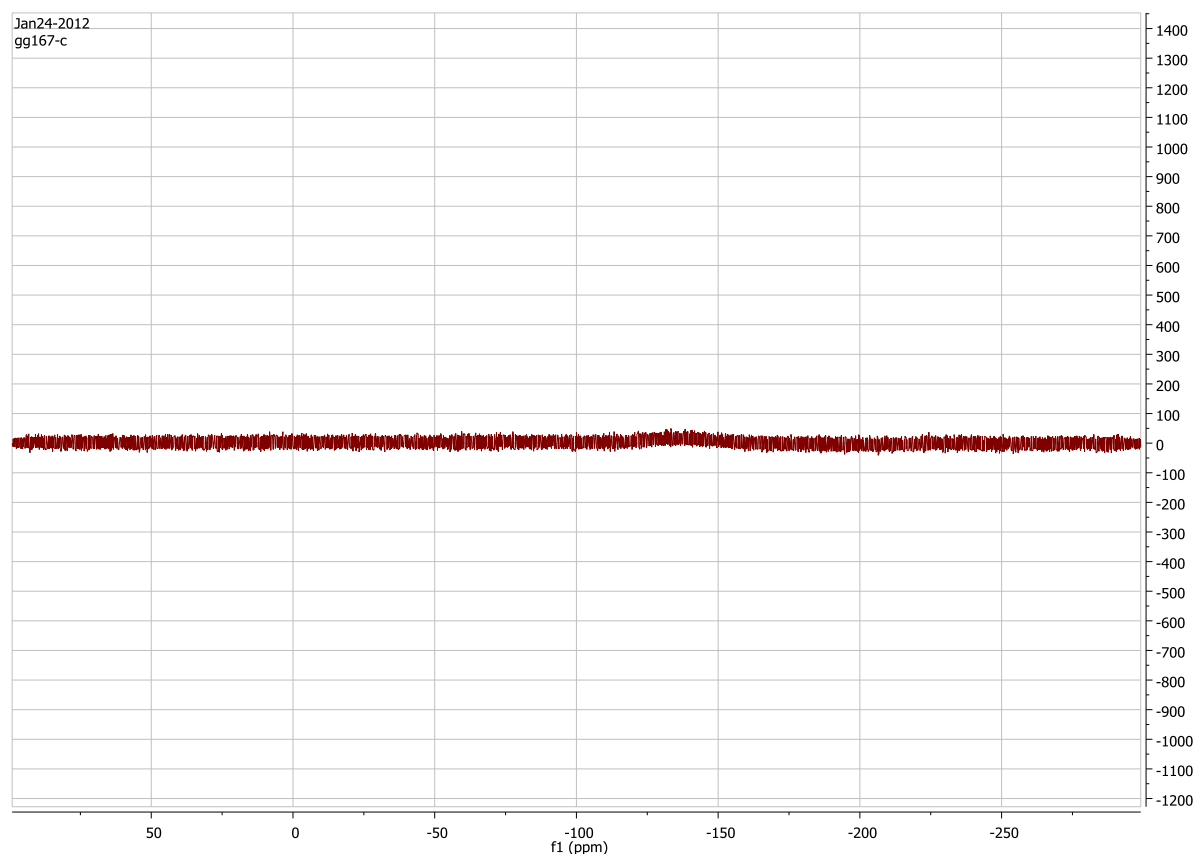
^1H NMR spectrum



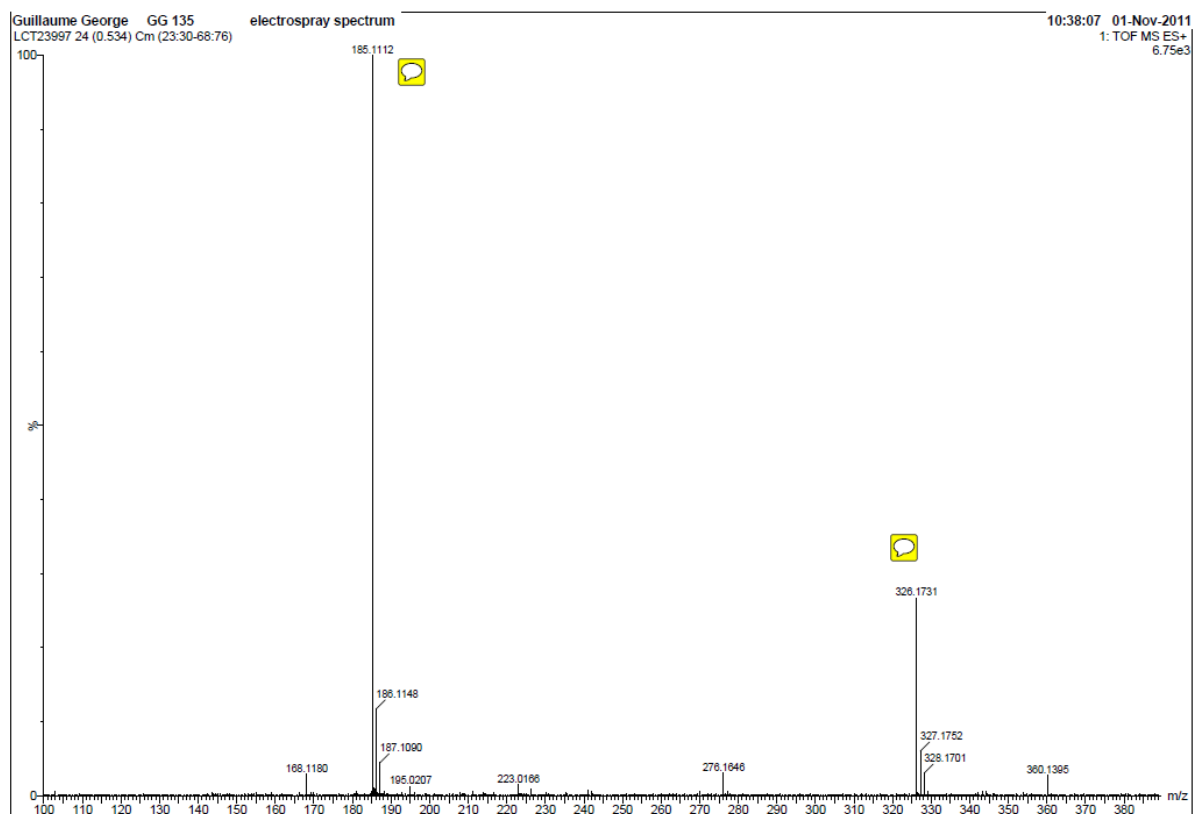
^{13}C NMR spectrum



^{19}F NMR spectrum



HRMS



xxx.

Cyclo(–Gly–D-Tyr–{[1-(3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,13,13,14,14,15,15,16,16,17,17,18,18,19,19-pentatriacontafluorononadecyl)-1*H*-1,2,3-triazol-4-yl]Ala}–Arg–Nal–)

HRMS

