

Validation Review

Procedure Split Ratio Study

Check the appropriate box: New Validation ☐ Modification ☒ Performance Check ☐

Sign and Date to indicate your acceptance of the procedure/instrument:

Examiner (if applicable) [Signature] Date 10/17/13

Unit Supervisor Melina B. Everett Date 10-29-13

Technical Leader (if different than Supervisor) Mel B. Everett Date 10-29-13

Crime Laboratory Regional Supervisor [Signature] Date 10-29-13

Quality Assurance Manager [Signature] Date 10-29-13

*All information must be submitted to the Quality Assurance Manager prior to the implementation of the procedure/instrument. If all technical staff training has not been completed, please include a proposed training plan and completion schedule.

Validation Study adjusting split ratios

Nashville Lab

October 2013

1. Introduction

The purpose of the validation study is to verify the performance of forensic chemistry drug identification with different split ratios. This study will verify the performance and accuracy of identifications by varying the split ratio. It will show that it has little to no impact on the identification.

2. Objective

Demonstrate the operation of the mass spectrometer detector and the accuracy of mass spectra obtained using different split ratios

3. Setup and Procedures

Forensic chemistry identification methods were used with the same parameters with the exception of the split ratio. Four drug standards were run using different split ratios. The spectra from these samples were compared to spectra obtained using the current method.

4. Results and Conclusion

The 4 drug standards were injected on the instrument using different split ratios. It was determined that these standards performed appropriately in comparison. The resulting spectra did not vary from the know spectra. All spectra were confirmed to be accurate when compared to the spectra generated using the current split ratio in the method.

5. Training and Competency

The instrument used for the study is configured the same as the other instruments used in the laboratory. Adjusting the split ratio will not significantly affect the methodology. The only effect this demonstrated was an increased response for residual samples.

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\METHAMPHETAMINE A.D

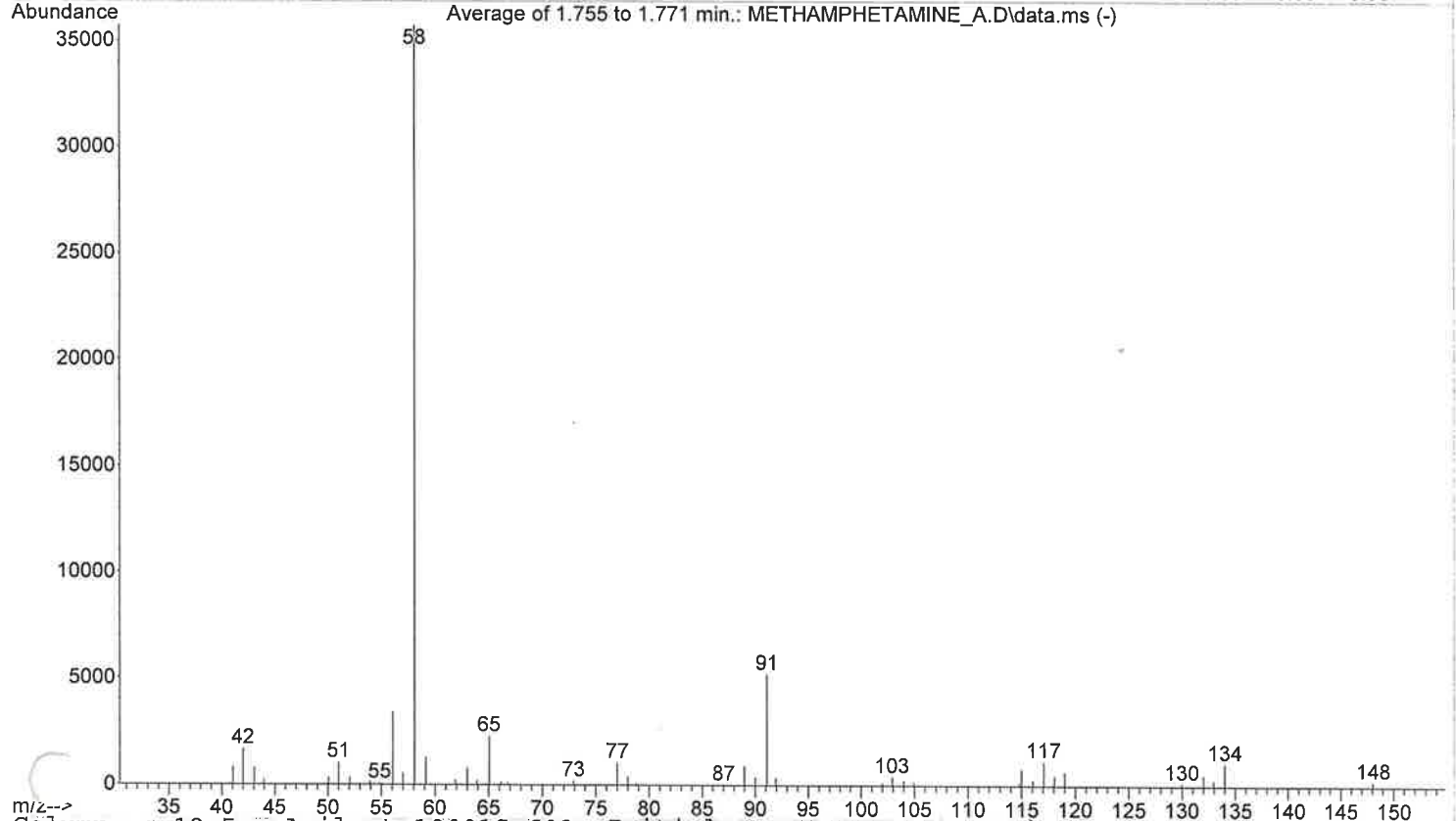
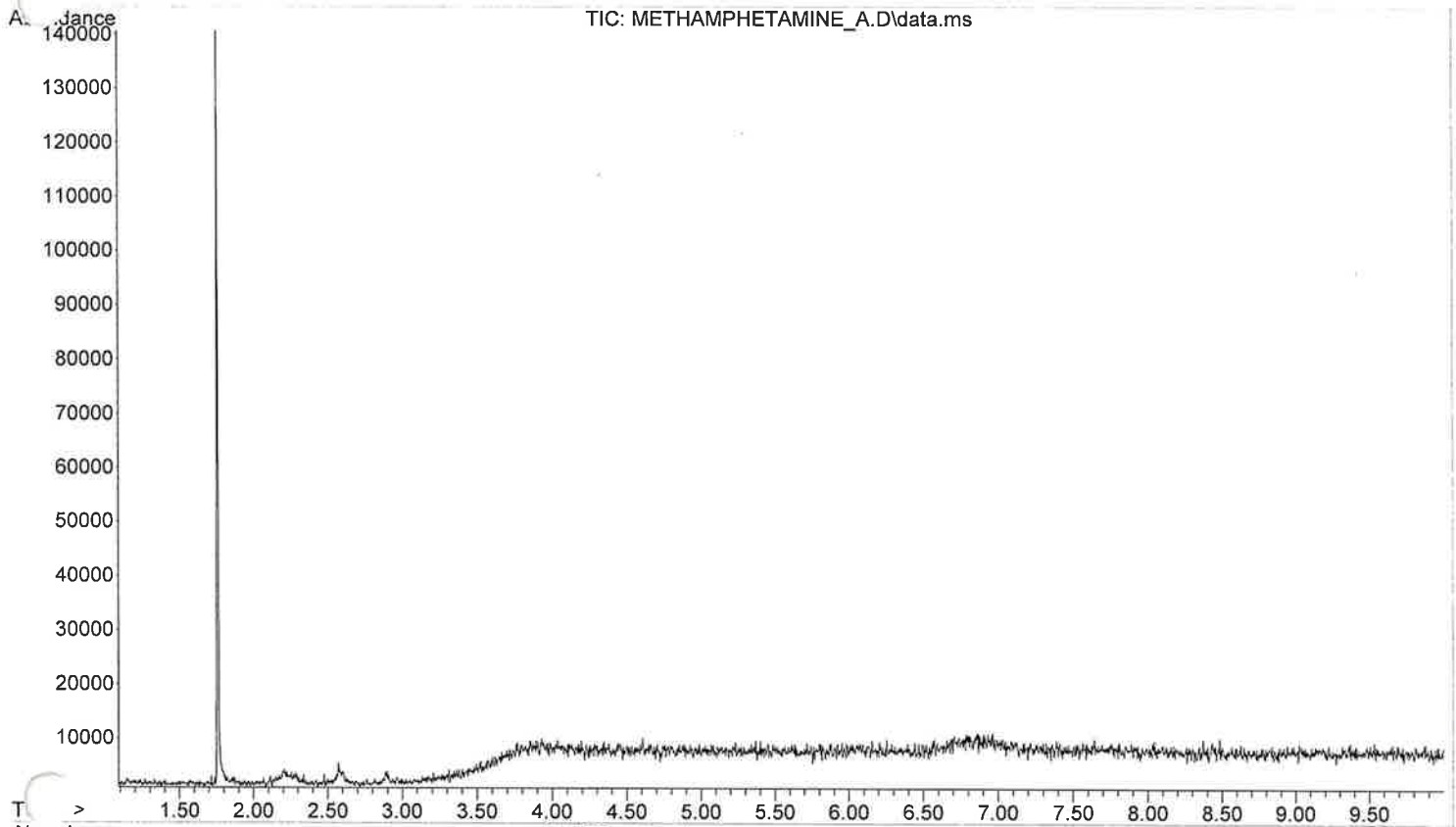
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 17 Oct 2013 15:51

Information: Methamphetamine 1:120 Misc. Information: Cerilliant lot#FE061710-02

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 120:1 Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00 280

2

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\METHAMPHETAMINE_B.D

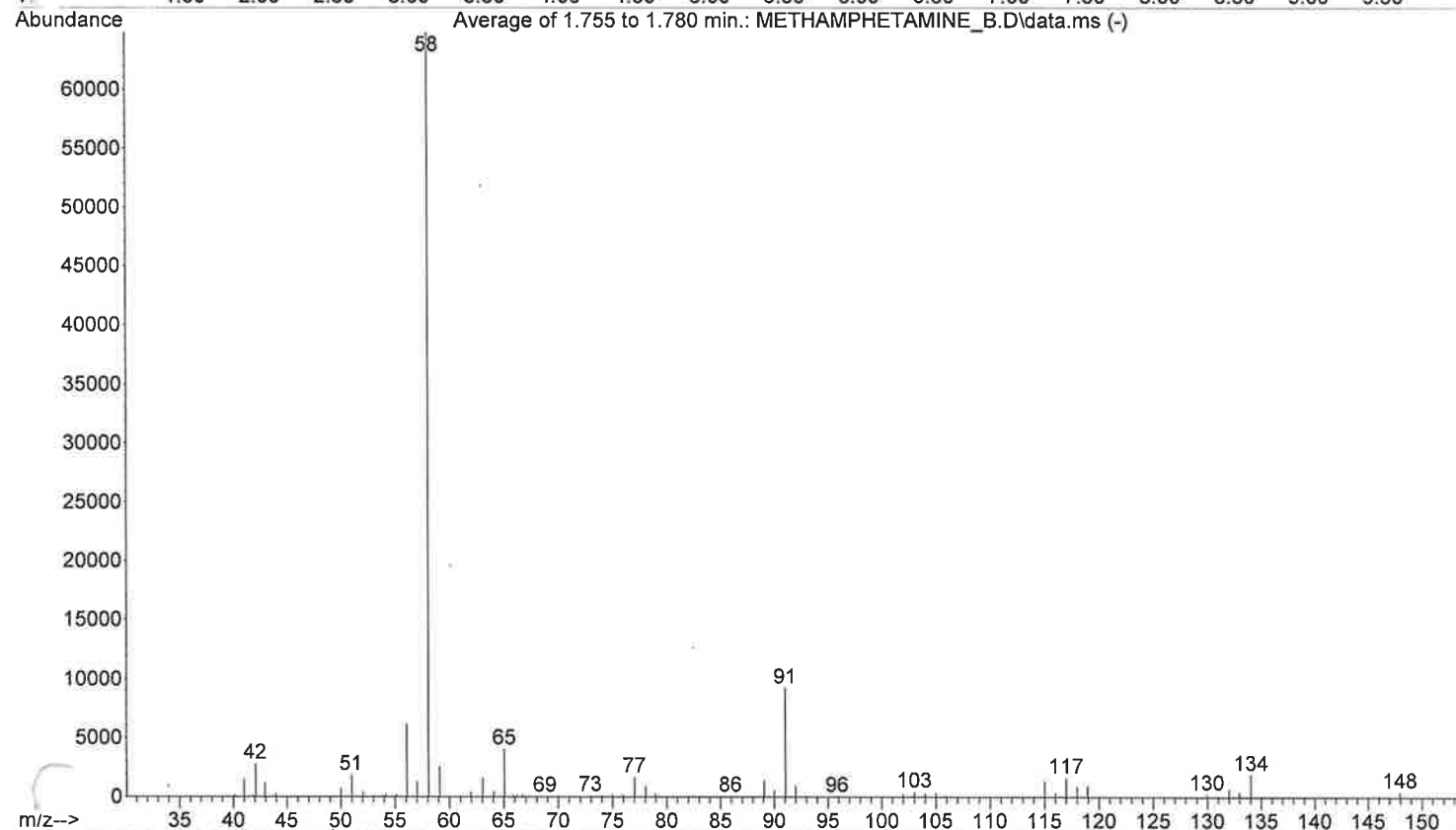
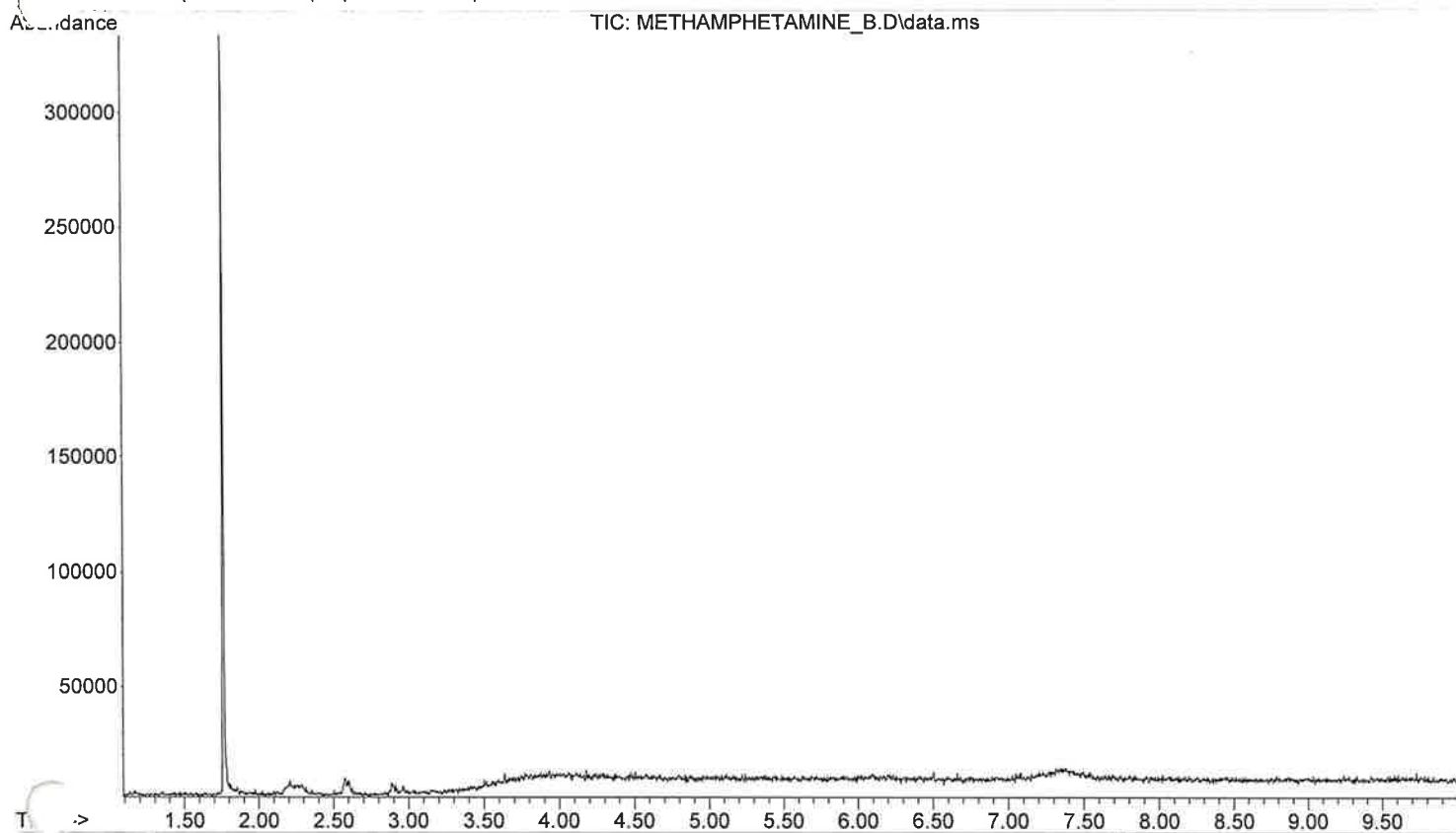
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 17 Oct 2013 15:39

Information: Methamphetamine 1:60 Misc. Information: Cerilliant lot#FE061710-02

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 60:1 Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280

Q

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\METHAMPHETAMINE_C.D

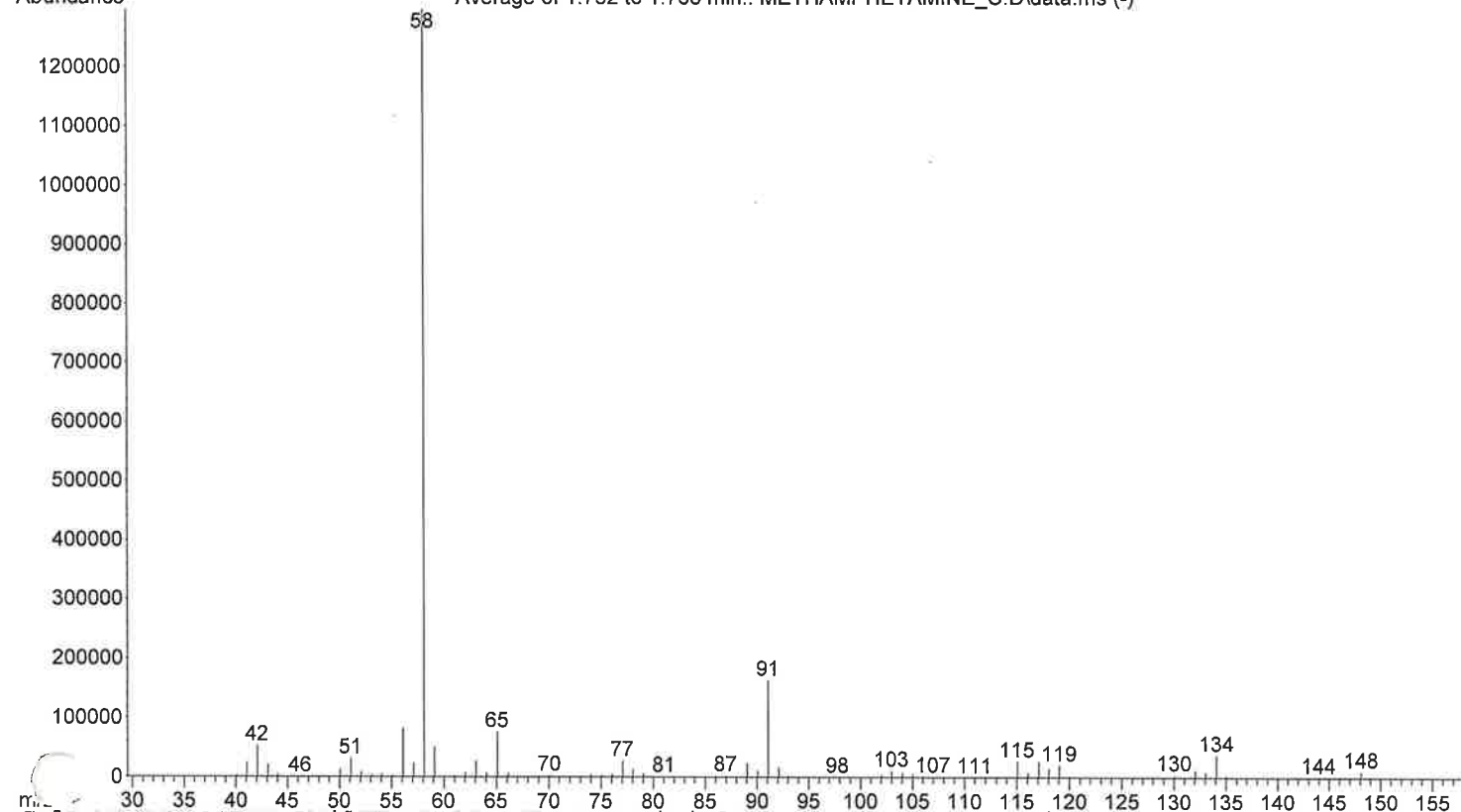
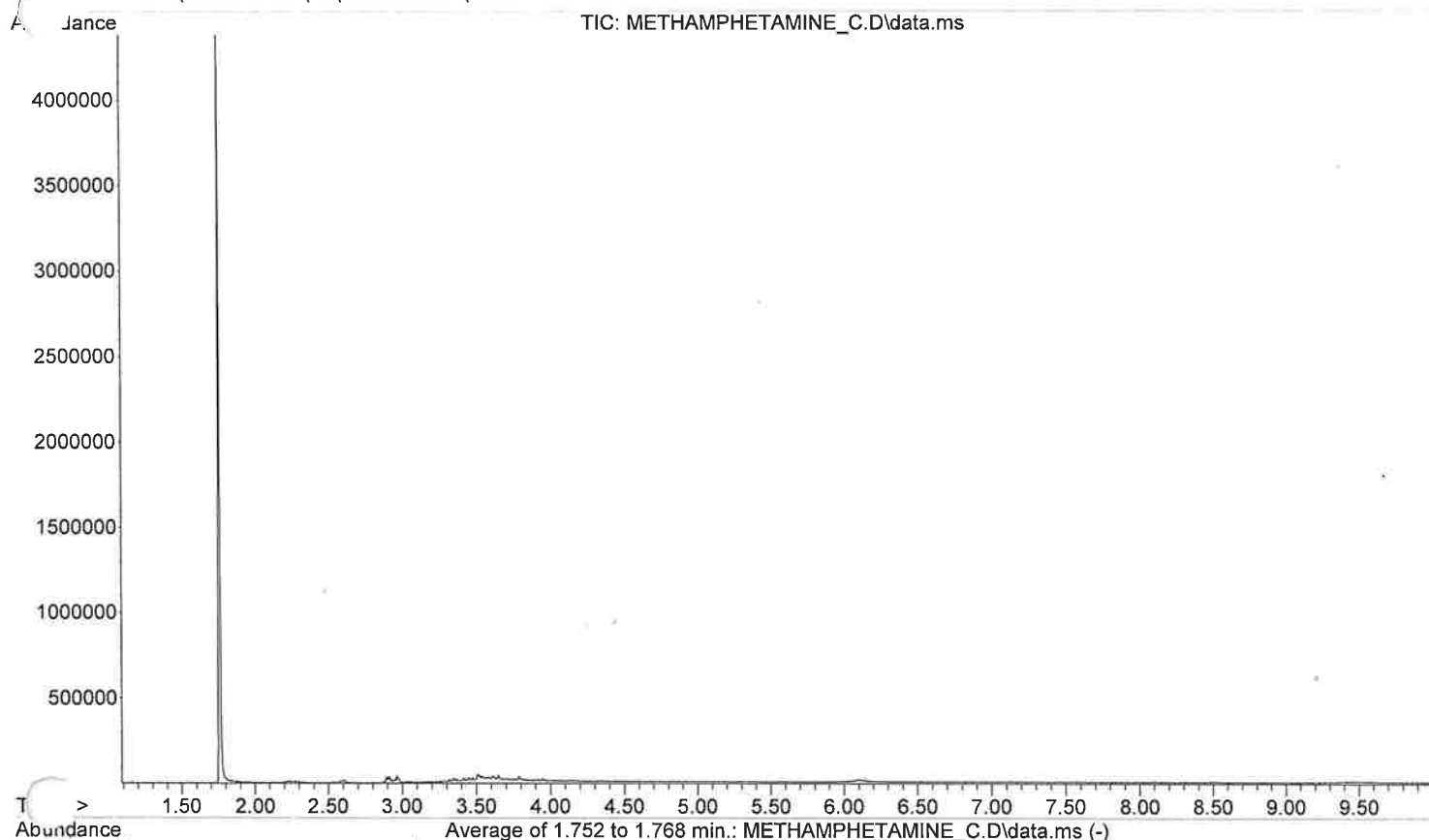
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 17 Oct 2013 15:27

Information: Methamphetamine 1:30 Misc. Information: Cerilliant lot#FE061710-02

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 30:1 Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280

[Handwritten signature]

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\METHAMPHETAMINE_D.D

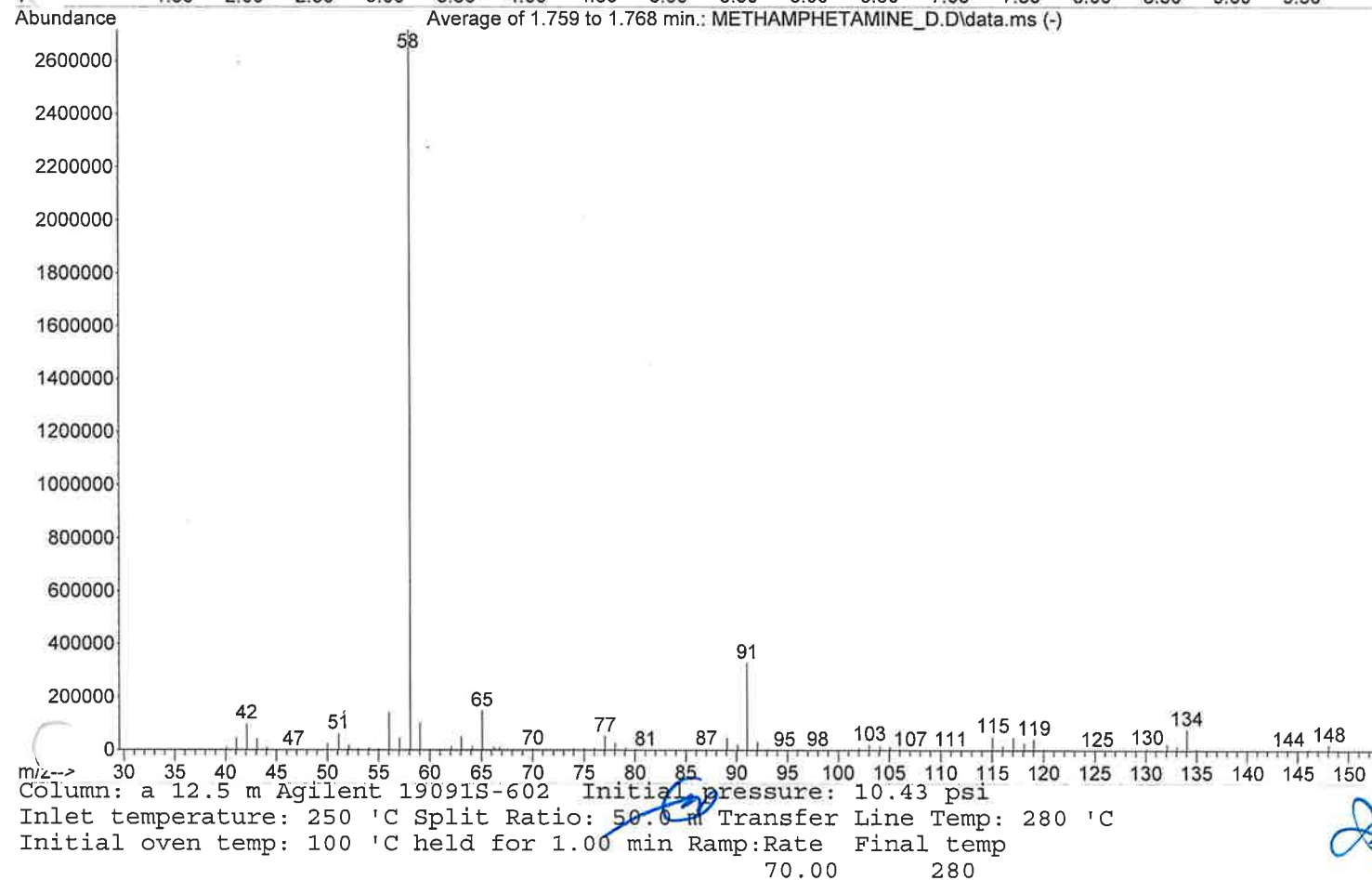
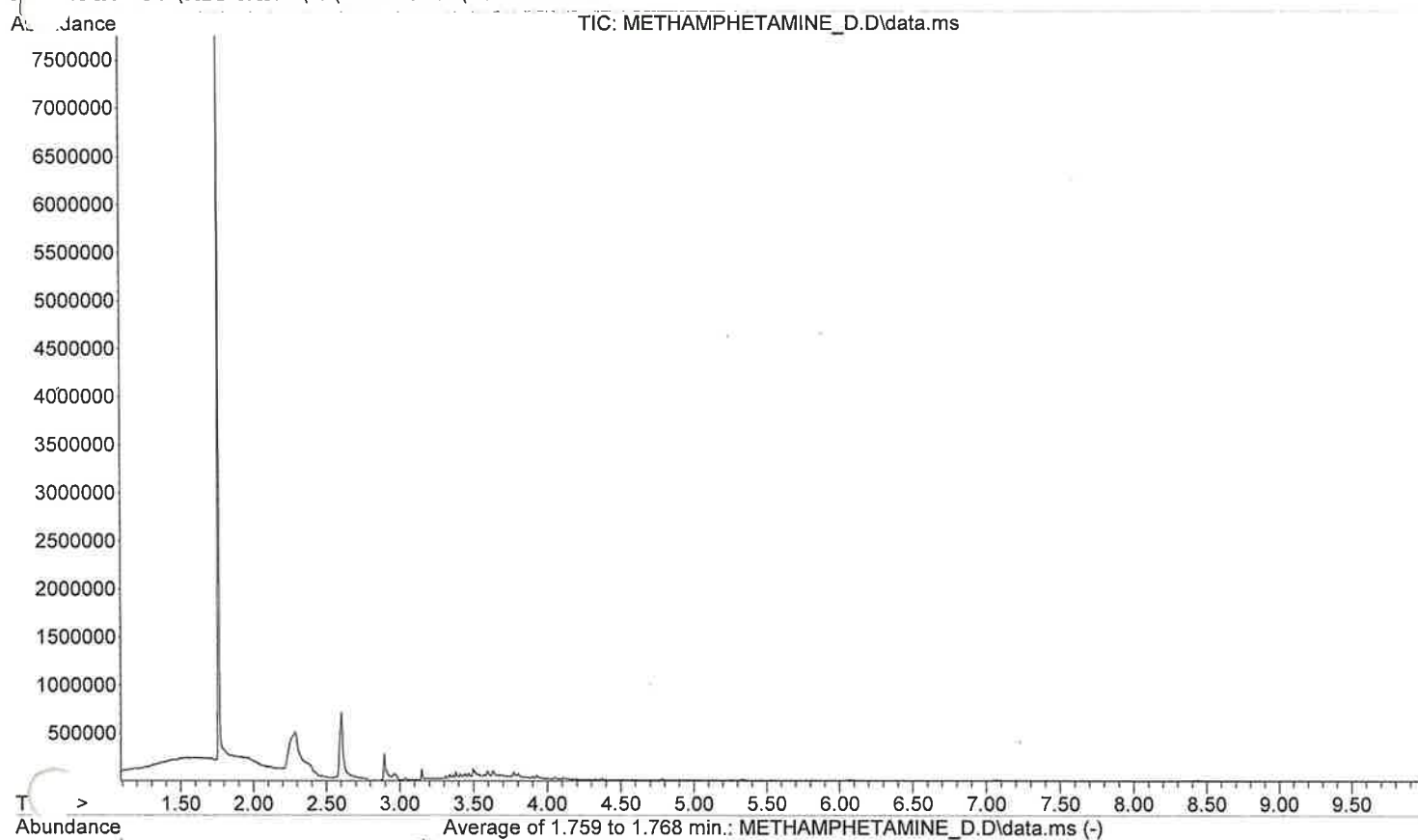
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 17 Oct 2013 16:03

Information: Methamphetamine Splitless Misc. Information: Cerilliant lot#FE061710-02

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\LSD_A.D

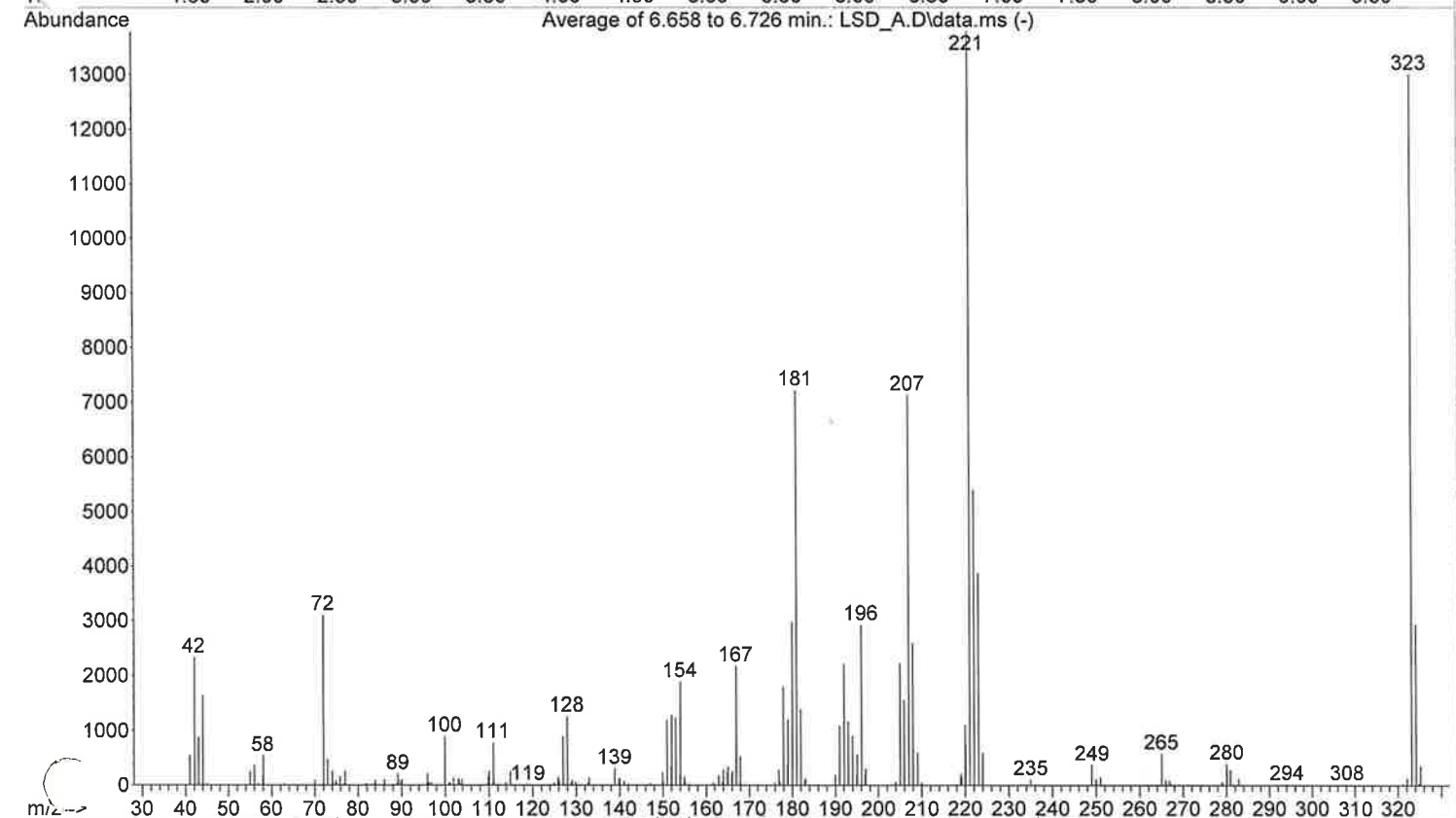
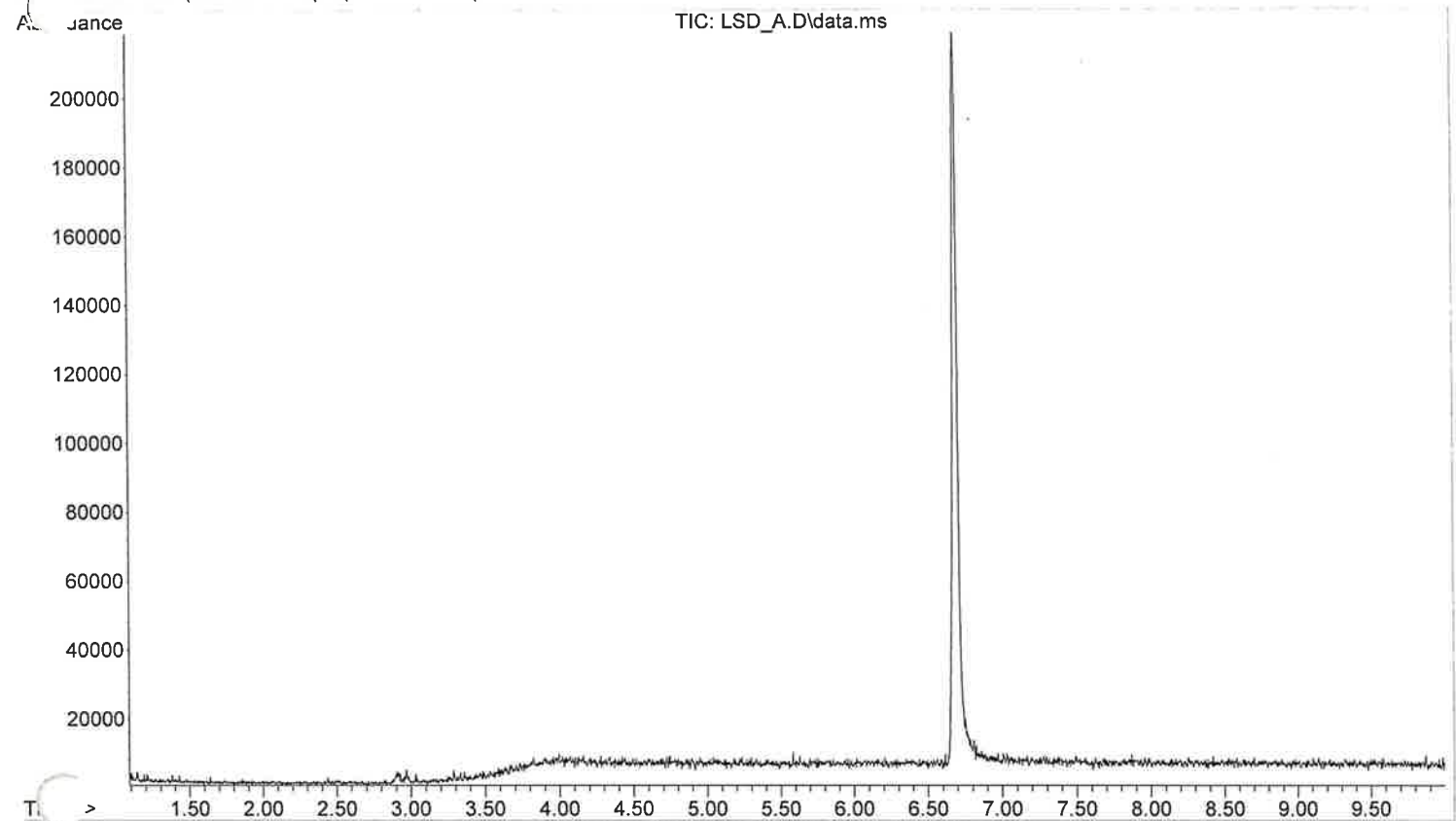
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 12:04

Information: LSD Split 1:120 Cerilliant lot#FE012111-04 Misc. Information: Acetonitrile Exp

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 120:1 Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280



Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\LSD_B.D

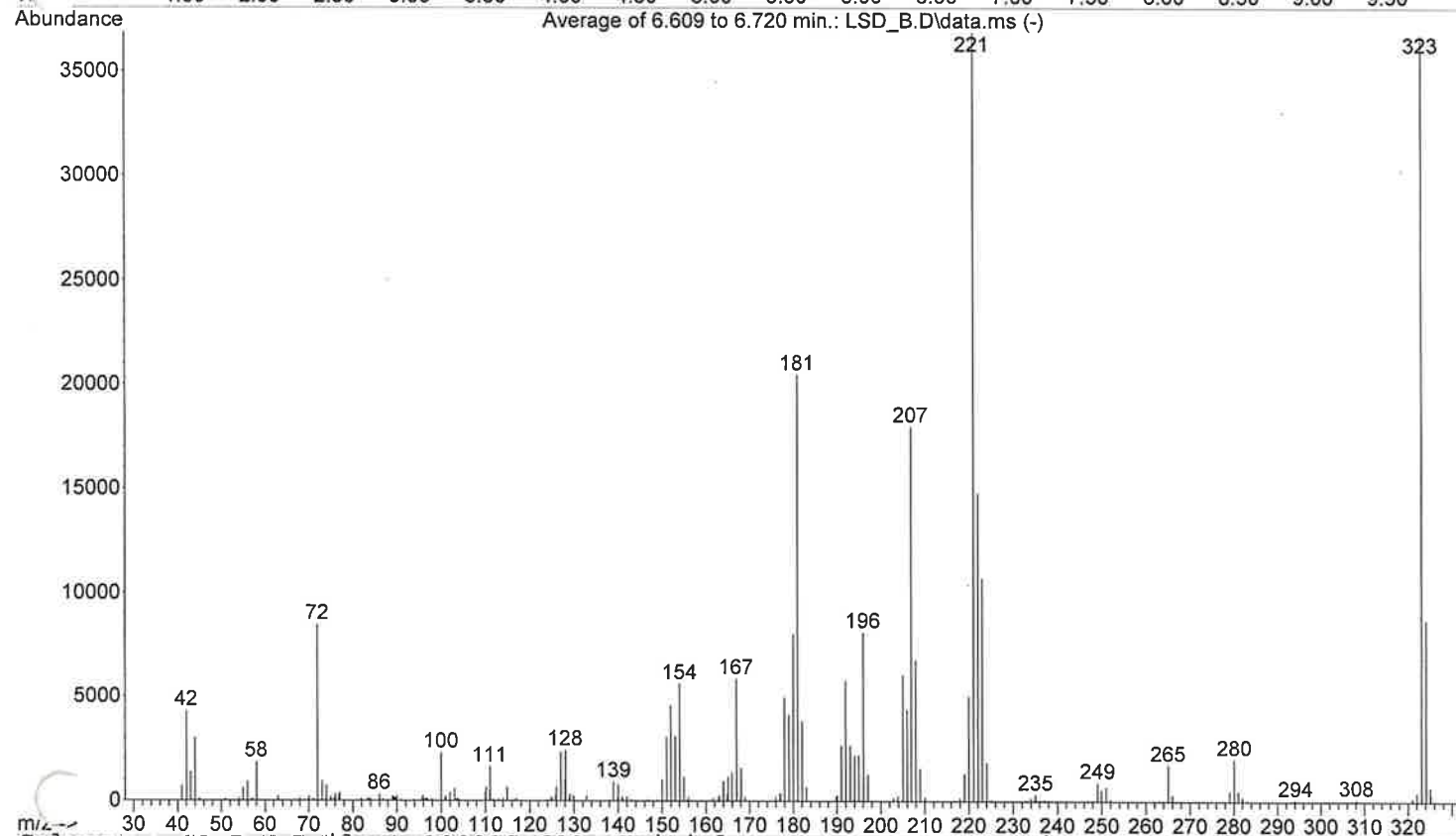
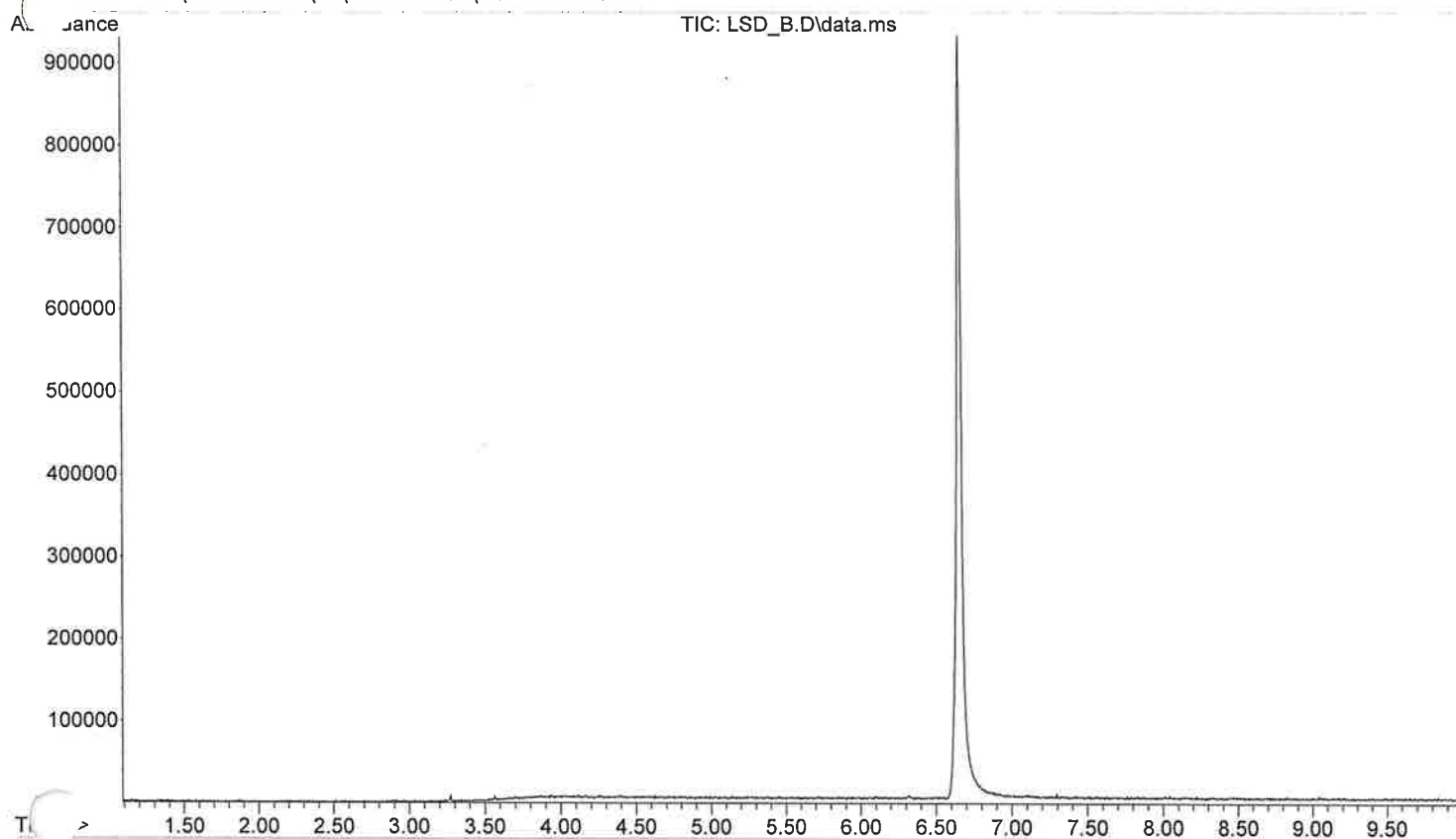
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 12:17

Information: LSD Split 1:60 Cerilliant lot#FE012111-04 Misc. Information: Acetonitrile Exp 2

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 °C Split Ratio: 60:1 Transfer Line Temp: 280 °C

Initial oven temp: 100 °C held for 1.00 min Ramp:Rate Final temp

70.00 280

3

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\LSD_C.D

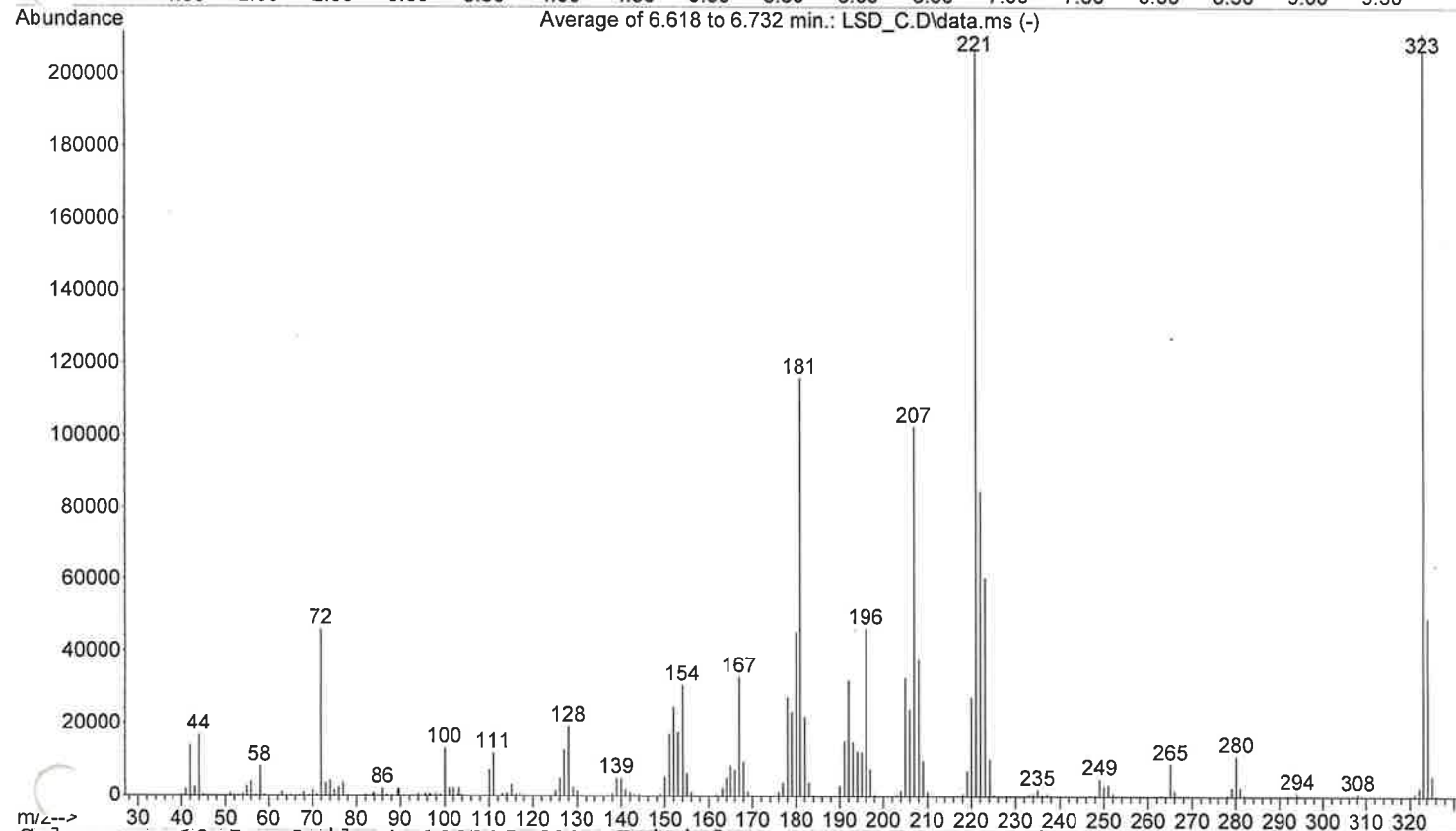
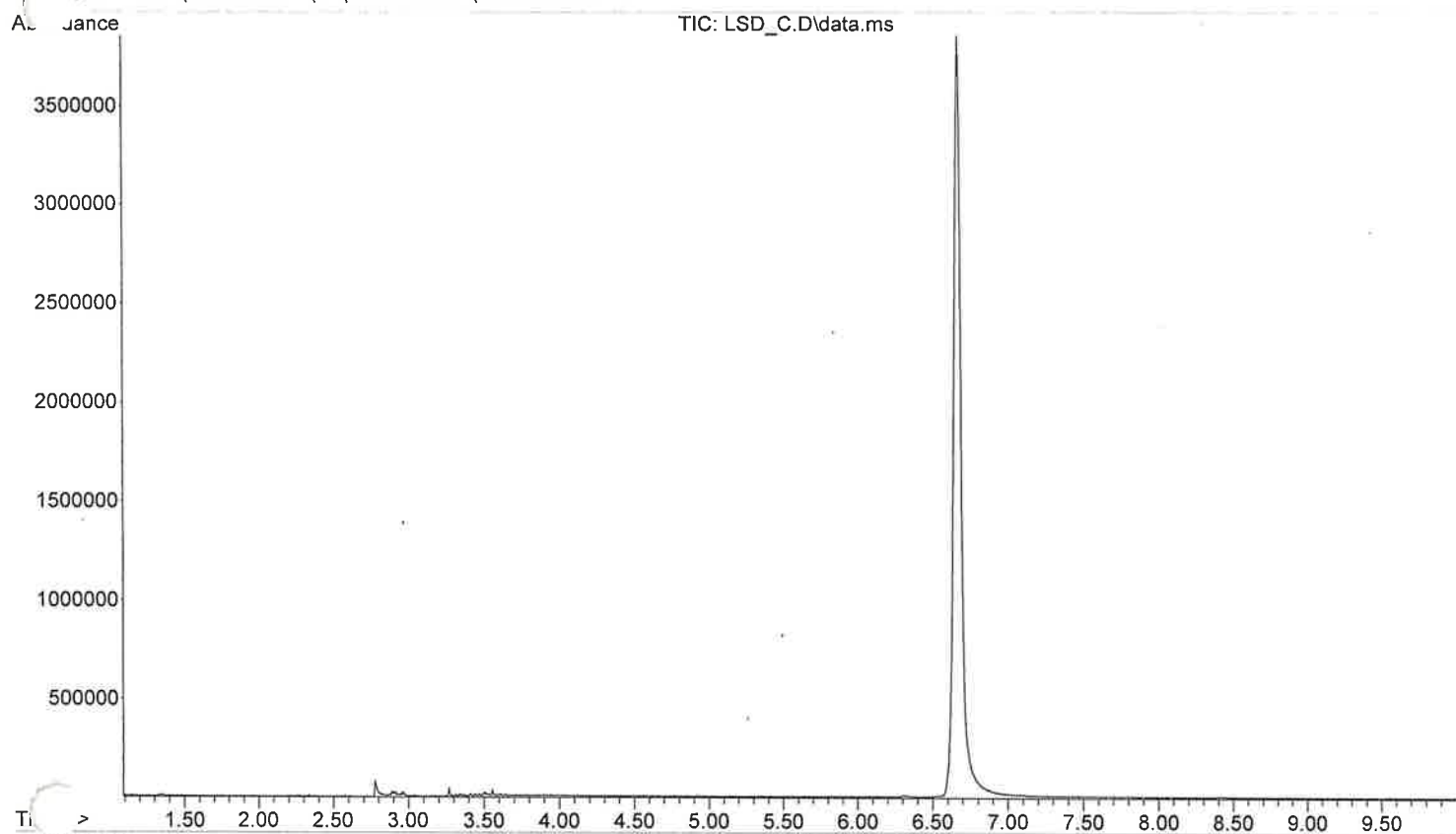
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 12:29

Information: LSD Split 1:30 Cerilliant lot#FE012111-04 Misc. Information: Acetonitrile Exp 2

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 °C Split Ratio: 30:1 Transfer Line Temp: 280 °C

Initial oven temp: 100 °C held for 1.00 min Ramp:Rate Final temp

70.00

280

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Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\LSD_D.D

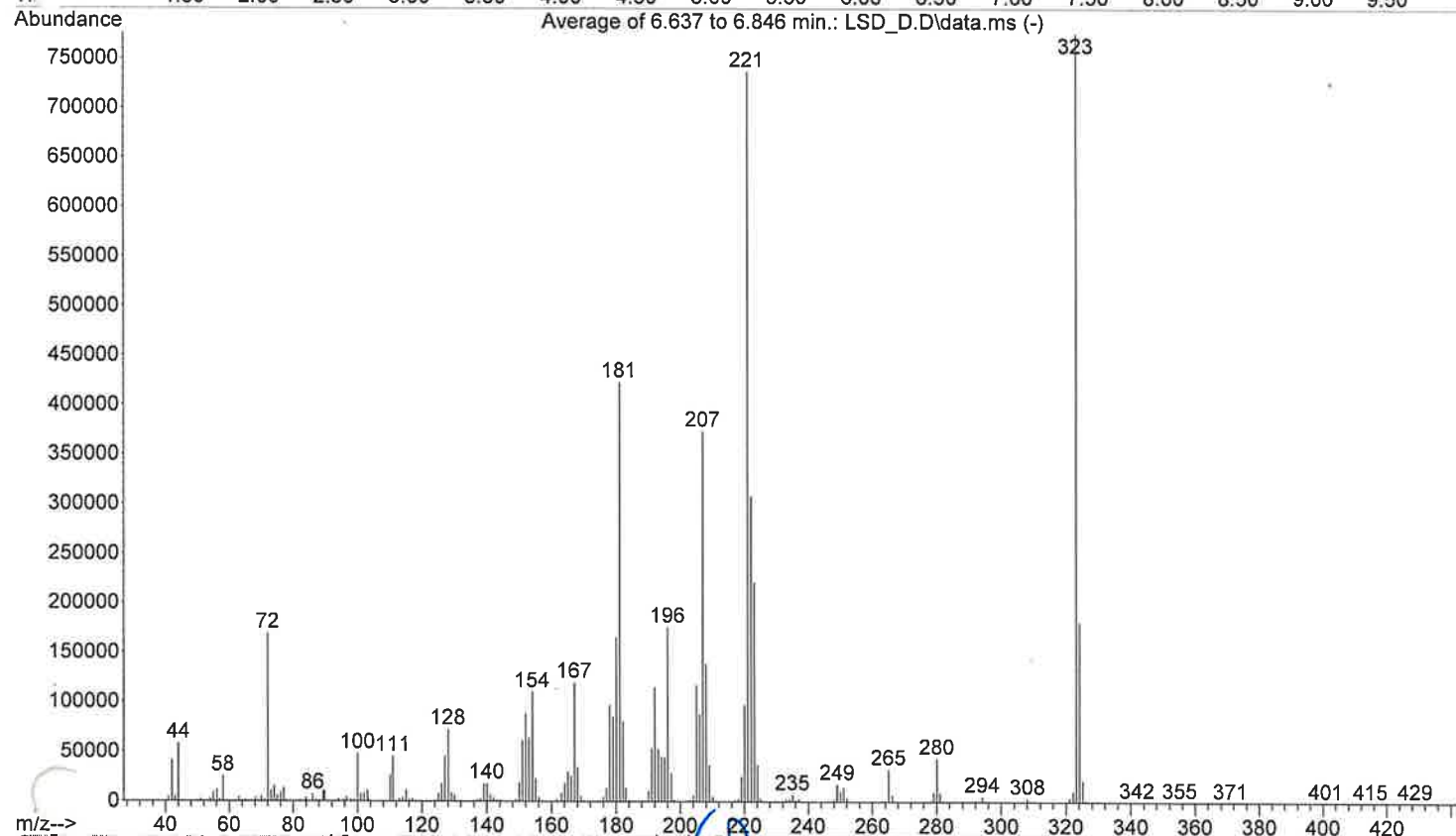
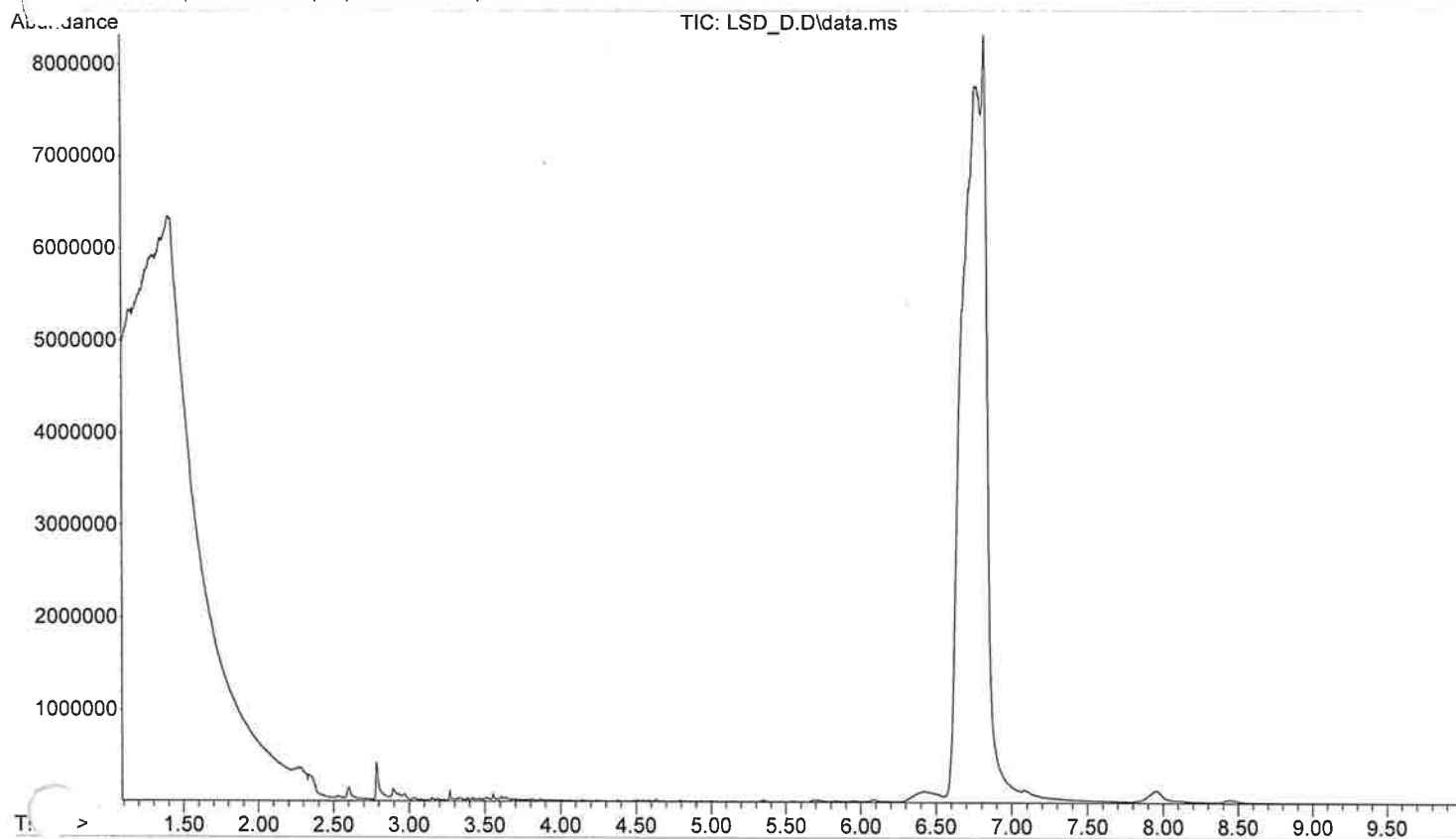
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 12:41

Information: LSD Splitless Cerilliant lot#FE012111-04 Misc. Information: Acetonitrile Exp 2/

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 50.0 m Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280

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Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\CLONAZEPAM_A.D

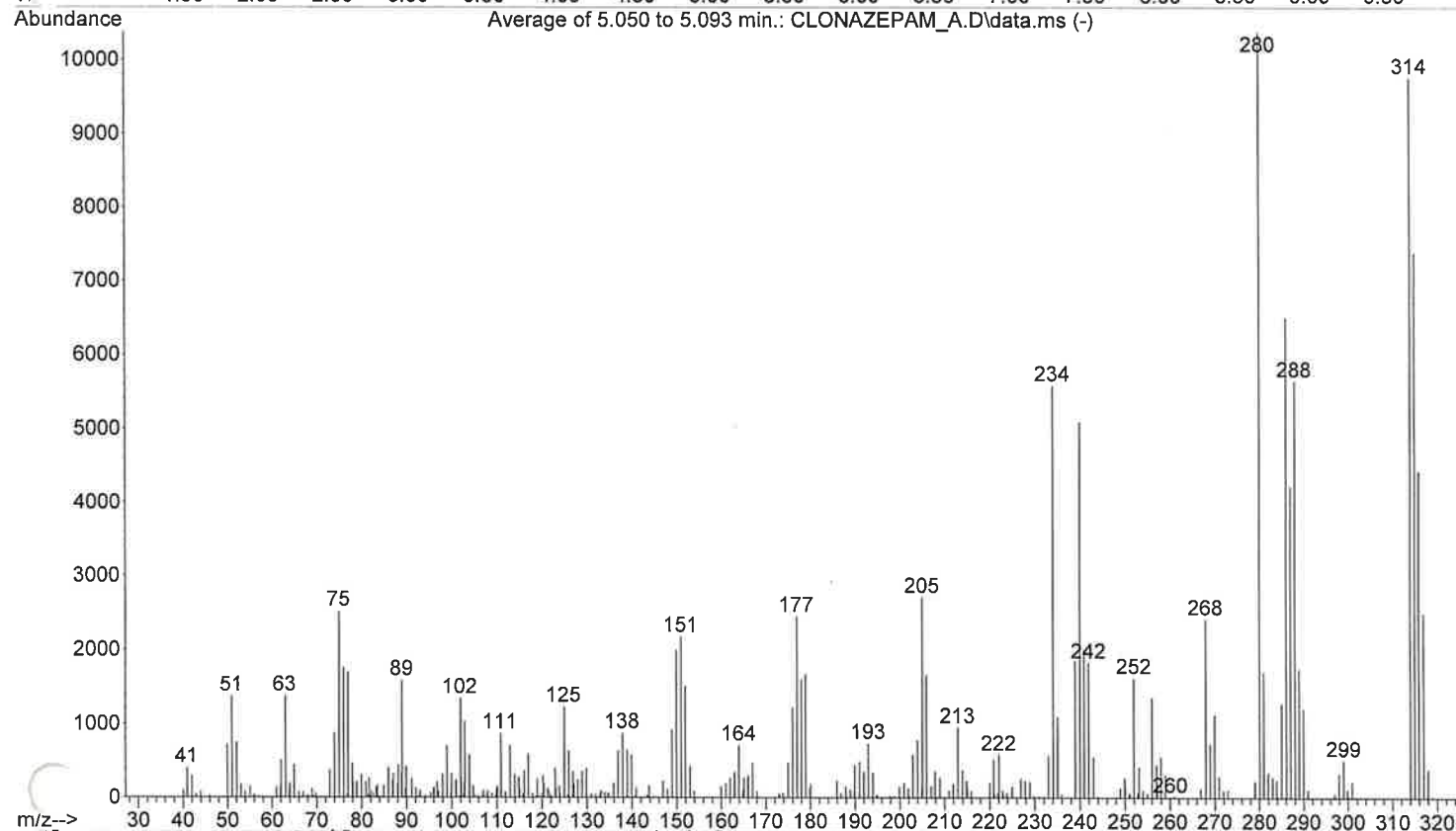
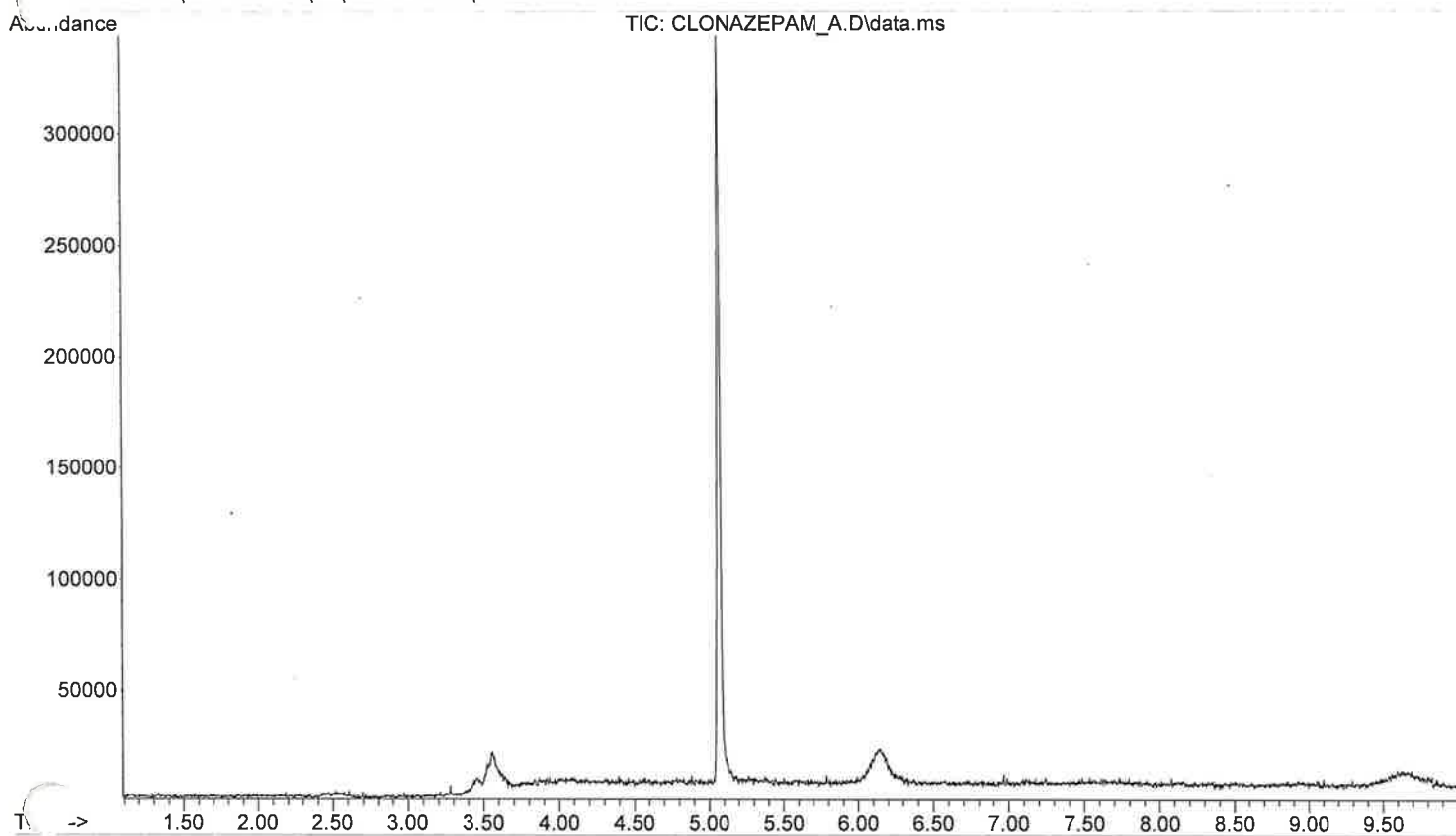
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 12:59

Information: Clonazepam 1:120 Misc. Information: Hoffman-Roche Lot#012077

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 120:1 Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280



Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\CLONAZEPAM B.D

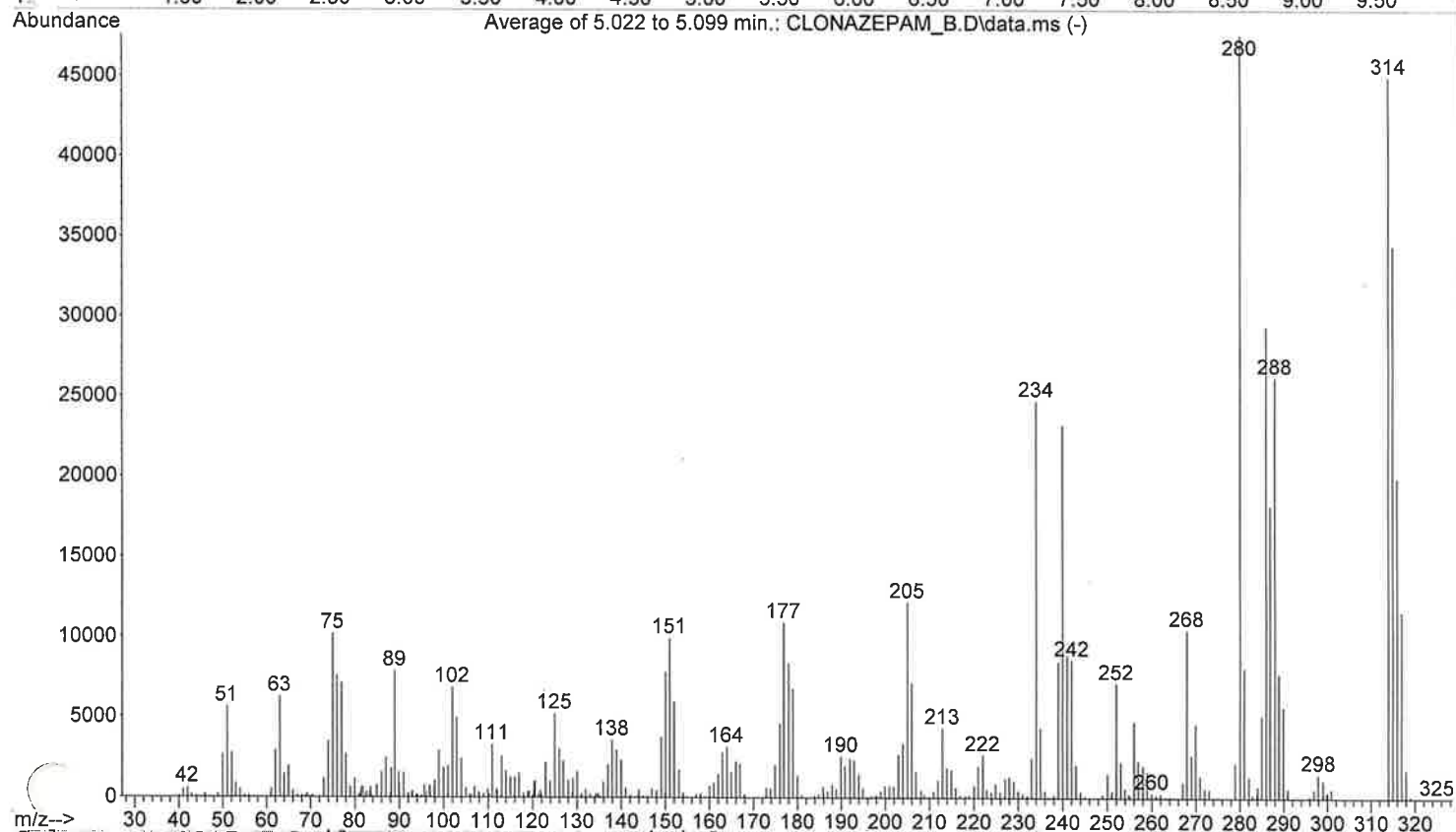
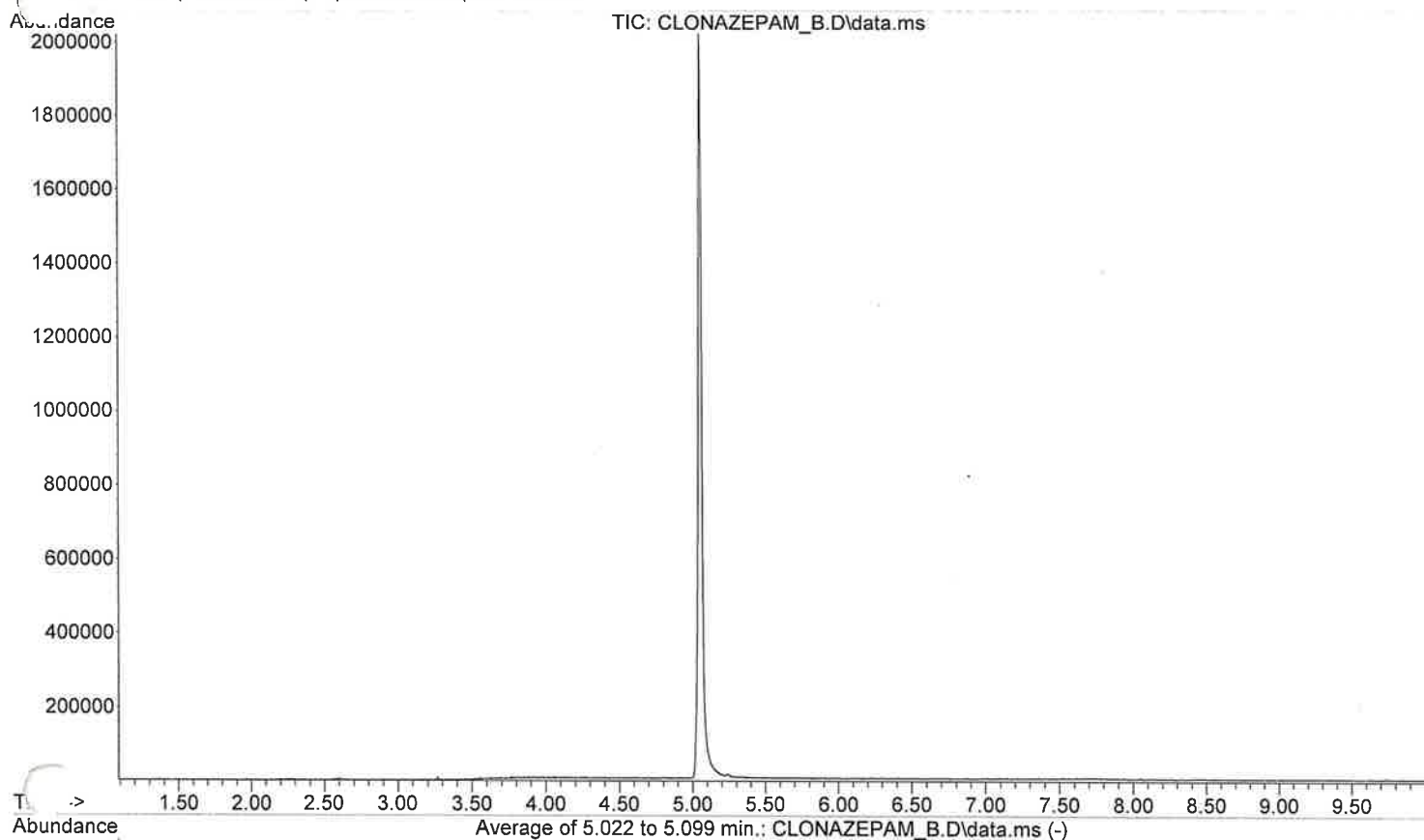
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 13:11

Information: Clonazepam 1:60 Misc. Information: Hoffman-Roche Lot#012077

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 °C Split Ratio: 60:1 Transfer Line Temp: 280 °C

Initial oven temp: 100 °C held for 1.00 min Ramp:Rate Final temp

70.00

280

2

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\CLONAZEPAM C.D

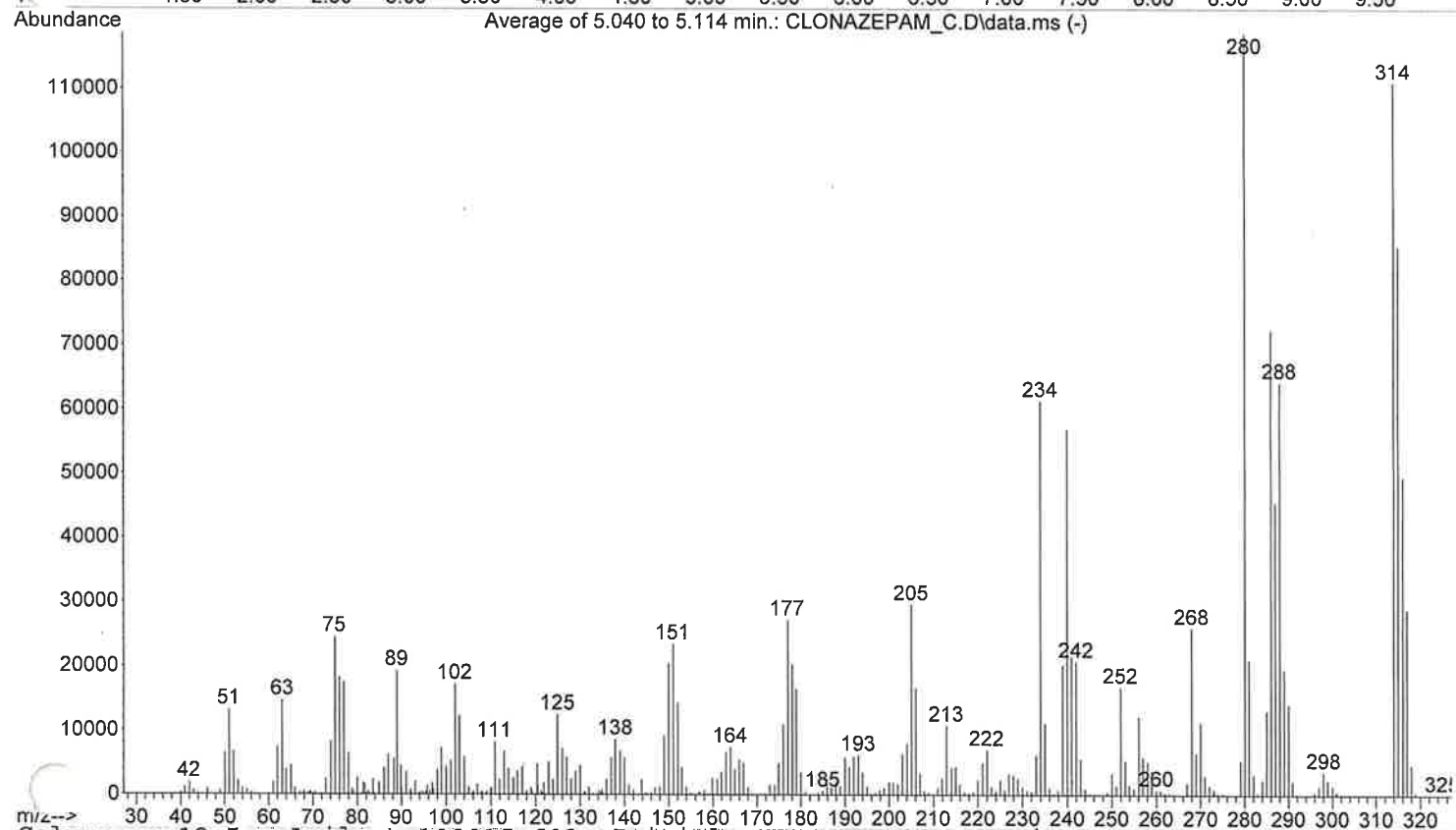
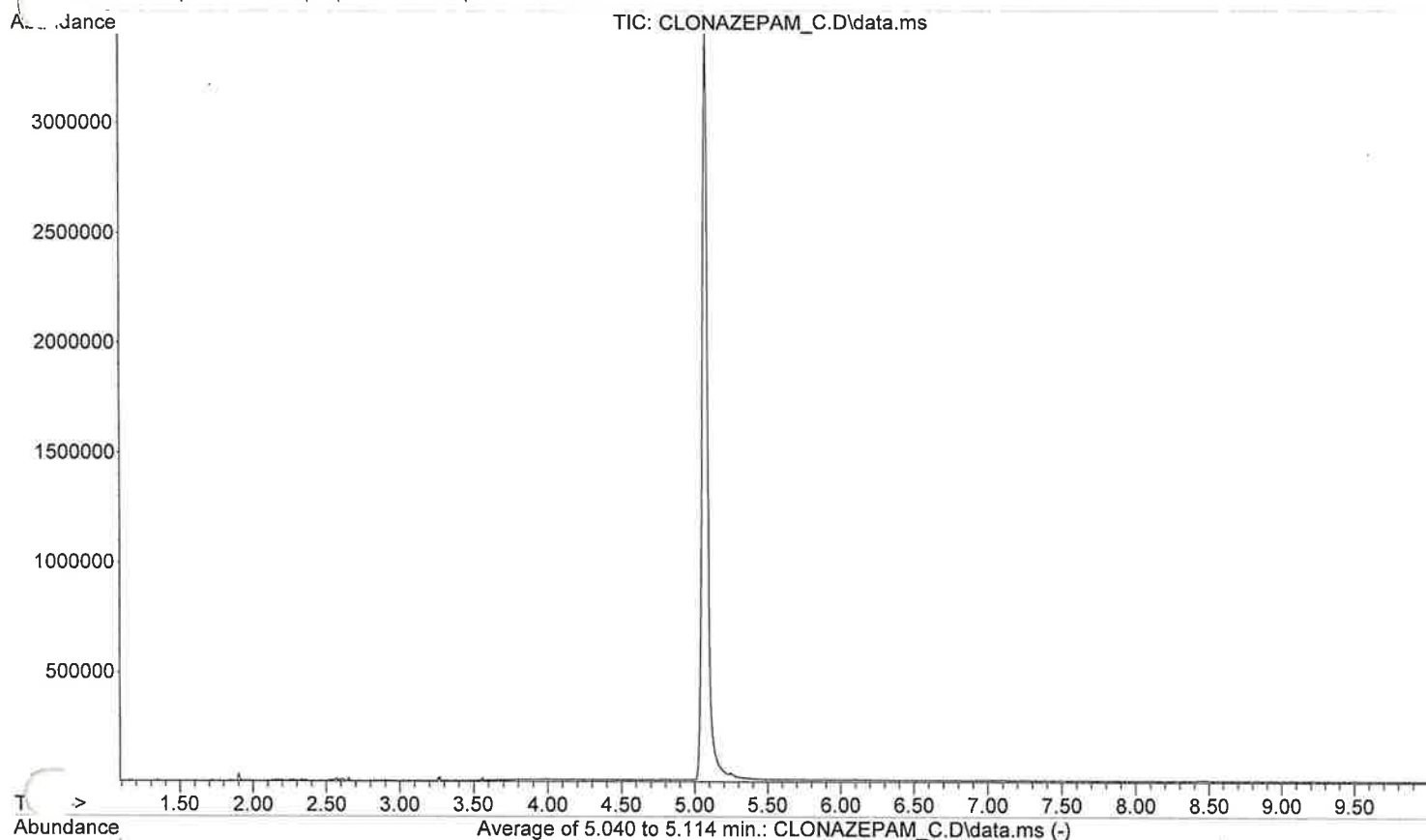
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 13:32

Information: Clonazepam 1:30 Misc. Information: Hoffman-Roche Lot#012077

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 30:1 Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280

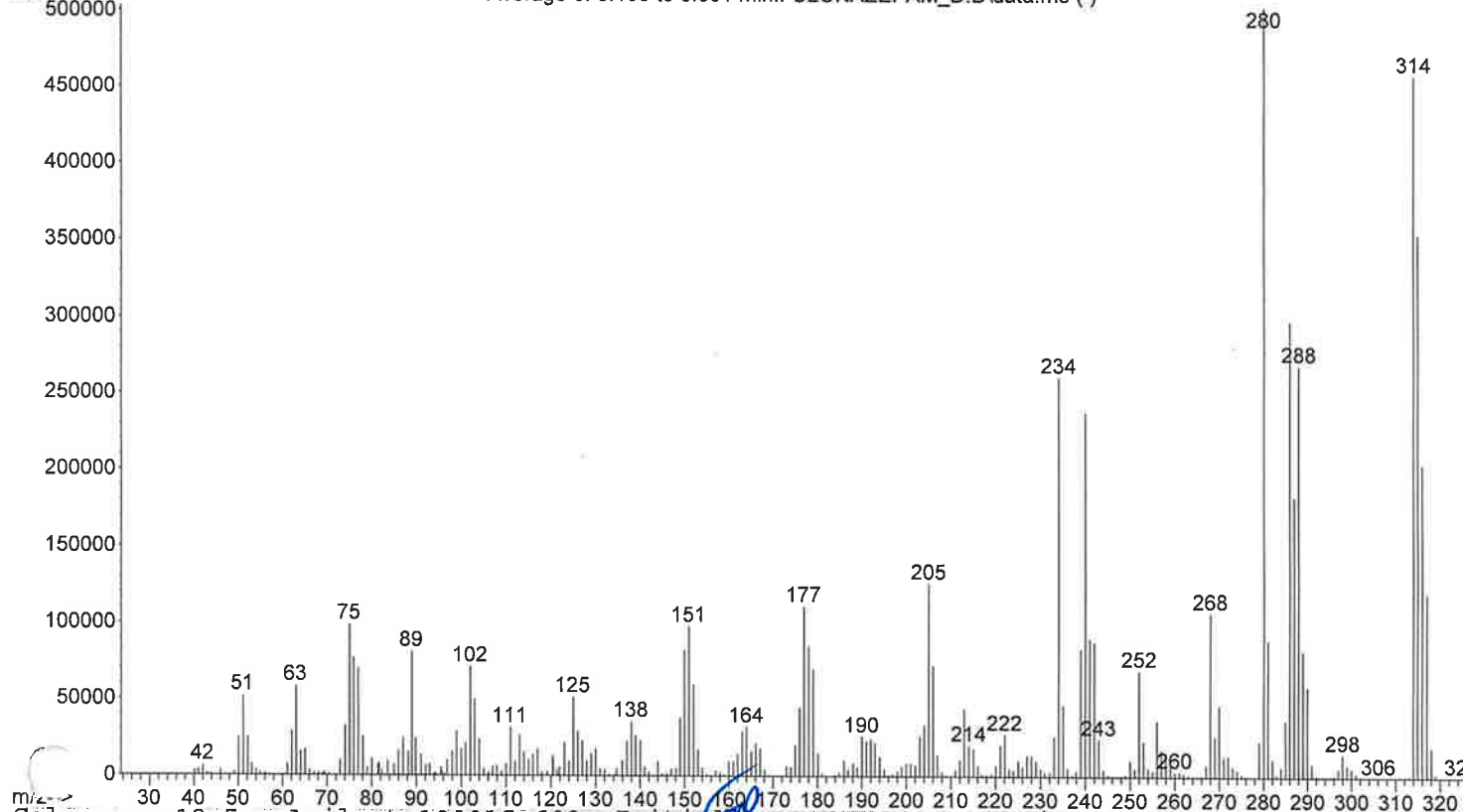
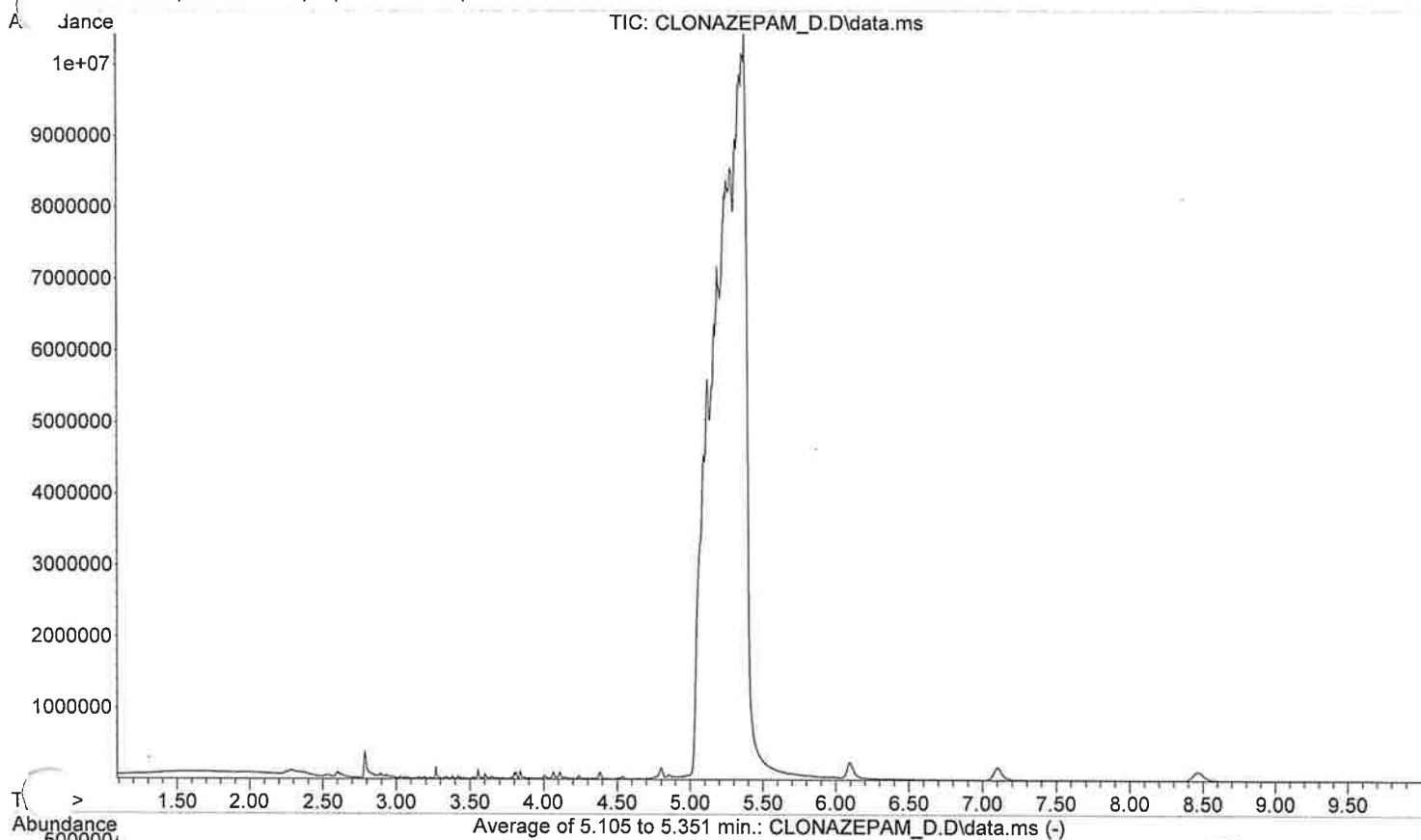
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Tennessee Bureau of Investigation: Nashville Crime Laboratory
Name of file: D:\JAS\CLONAZEPAM D.D
Instrument: Donna 2 (model: 5975 Serial number: US10423616)
Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 13:45

Information: Clonazepam Splitless Misc. Information: Hoffman-Roche Lot#012077

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi
Inlet temperature: 250 °C Split Ratio: 50.0 m Transfer Line Temp: 280 °C
Initial oven temp: 100 °C held for 1.00 min Ramp:Rate Final temp
70.00 280

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\OXYCODONE A.D

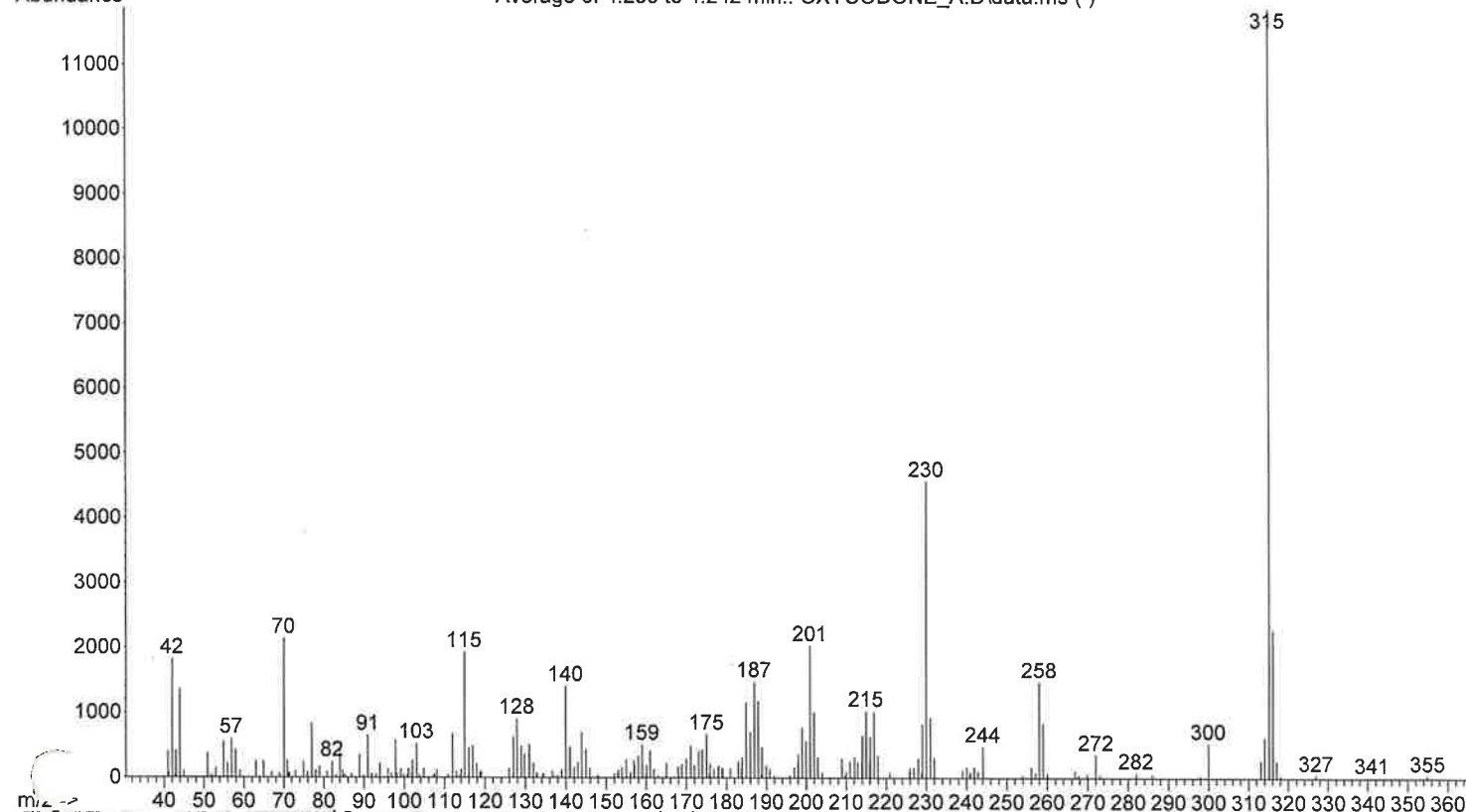
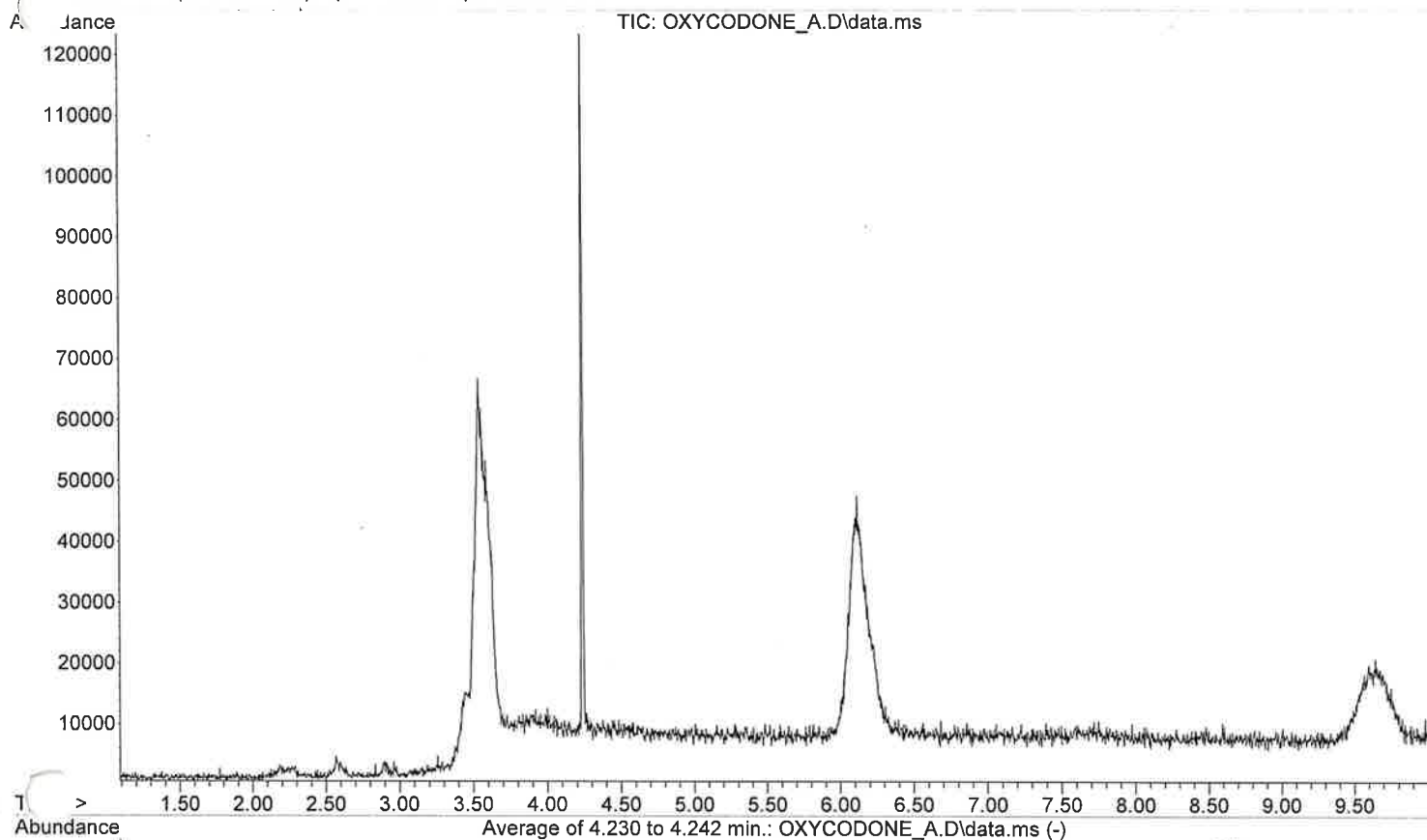
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 13:57

Information: Clonazepam 1:120 Misc. Information: Cerilliant lot#FE092910-02

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 °C Split Ratio: 120:1 Transfer Line Temp: 280 °C

Initial oven temp: 100 °C held for 1.00 min Ramp:Rate Final temp

70.00

280

[Handwritten signature]

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\OXYCODONE_B.D

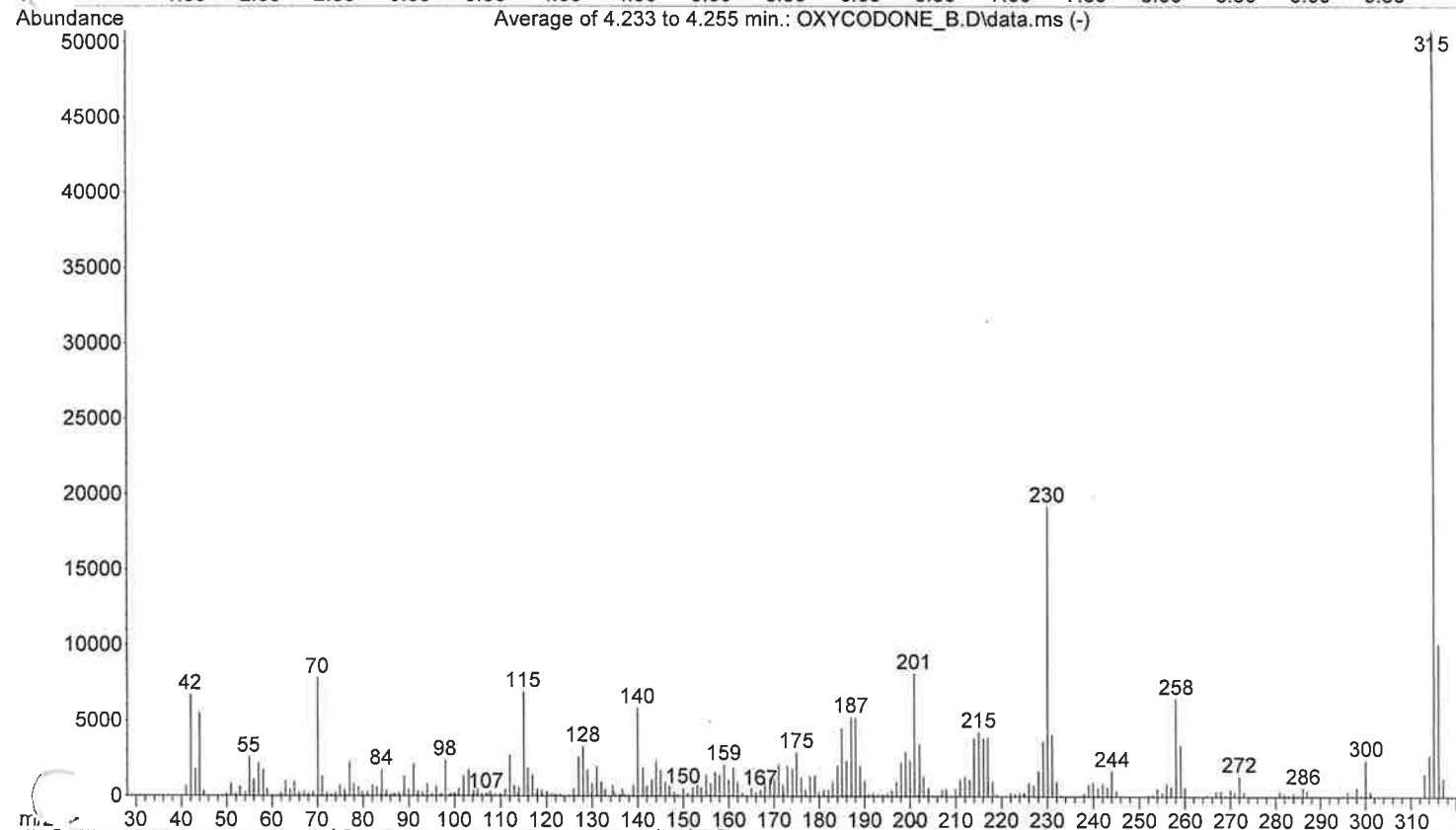
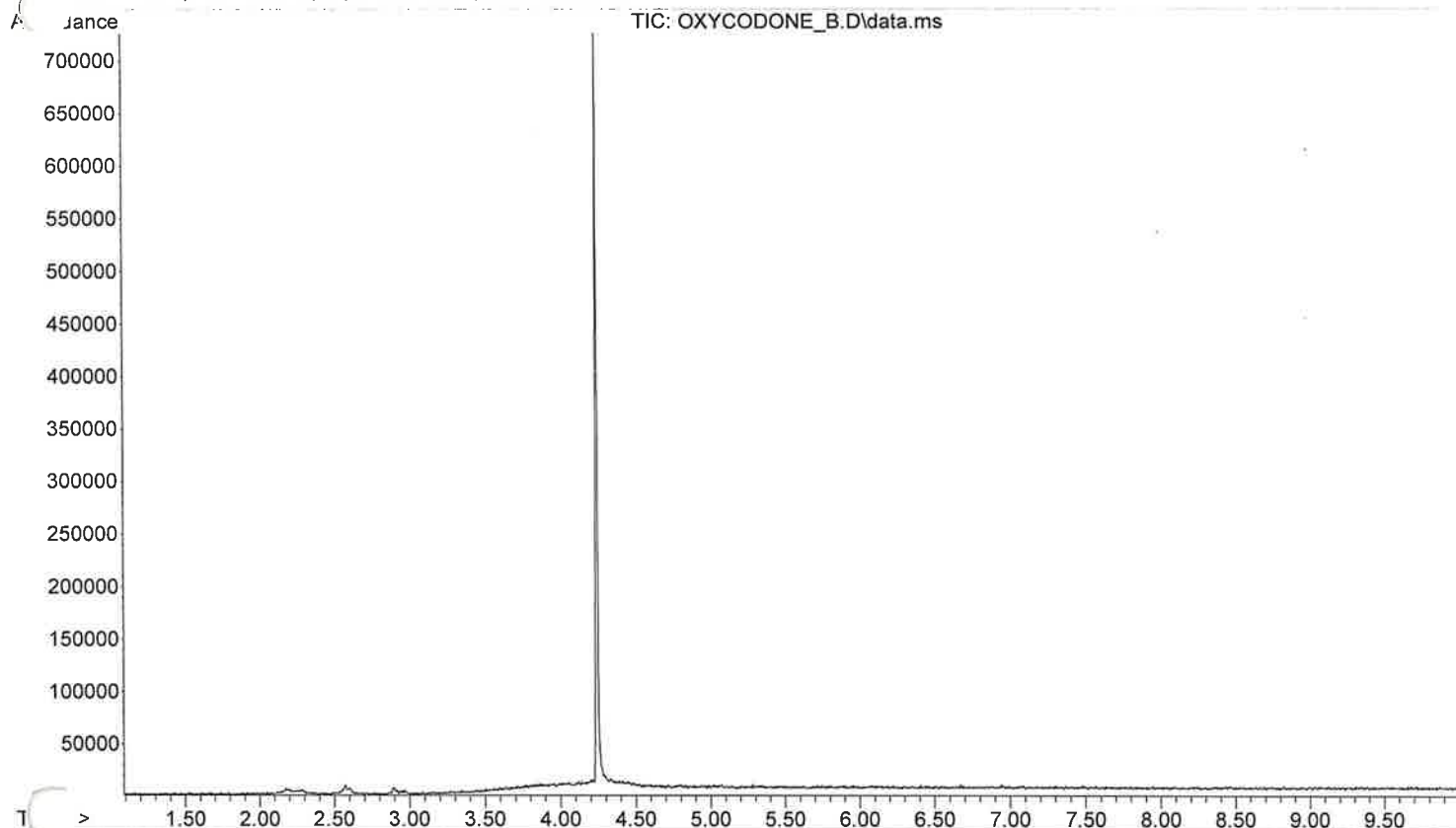
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 14:18

Information: Clonazepam 1:60 Misc. Information: Cerilliant lot#FE092910-02

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 60:1 Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280

3

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\OXYCODONE_C.D

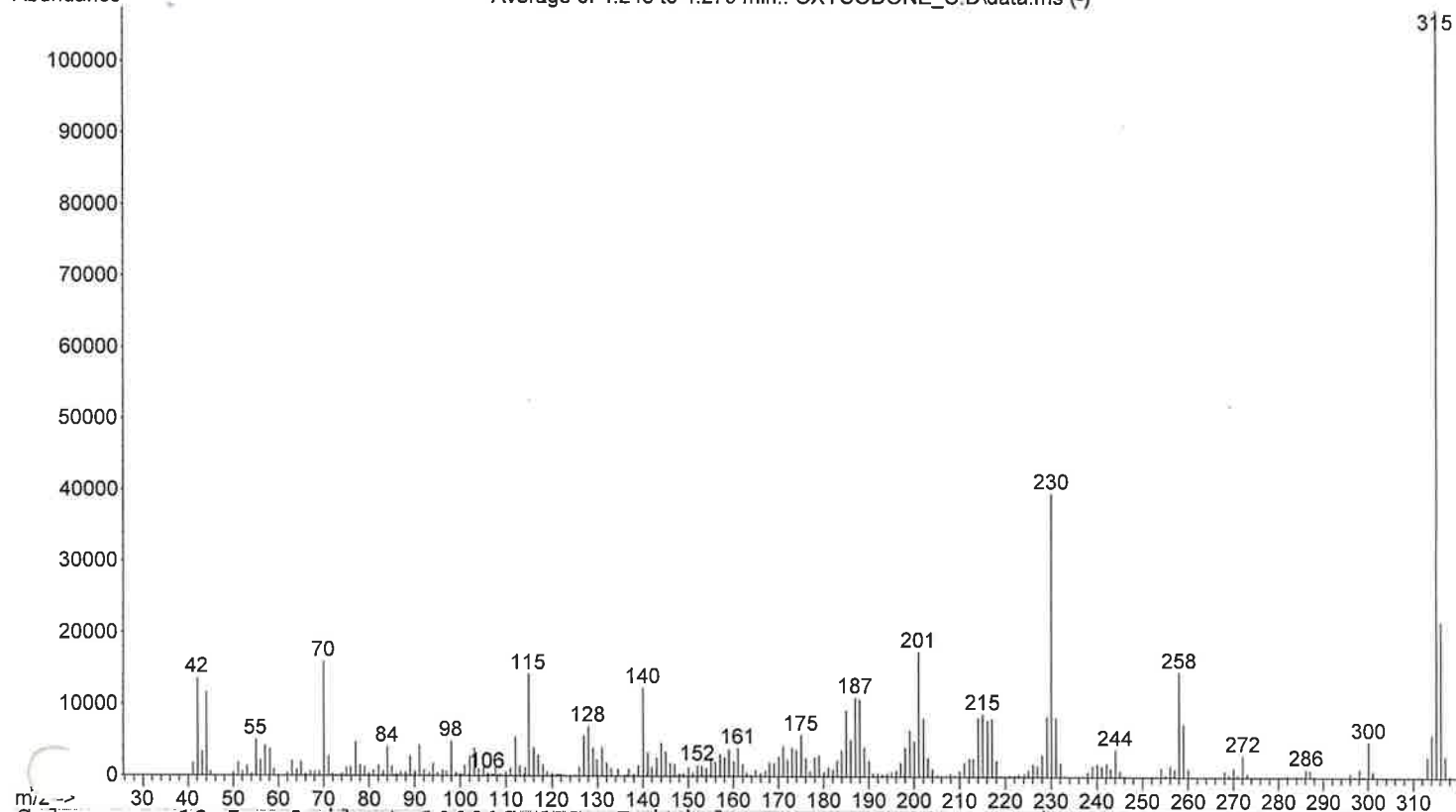
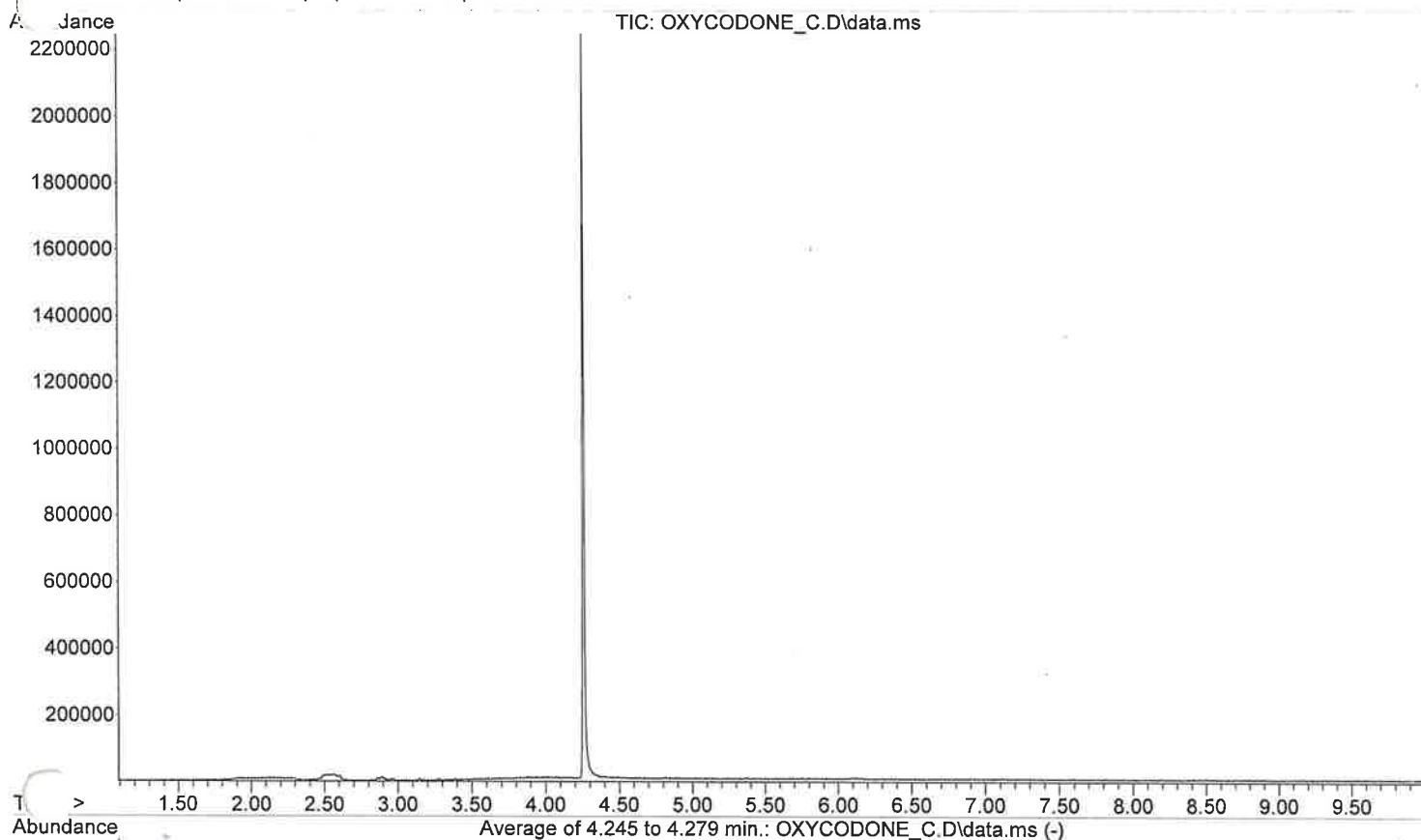
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 14:33

Information: Clonazepam 1:30 Misc. Information: Cerilliant lot#FE092910-02

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 30:1 Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280

[Handwritten signature]

Tennessee Bureau of Investigation: Nashville Crime Laboratory

Name of file: D:\JAS\OXYCODONE D.D

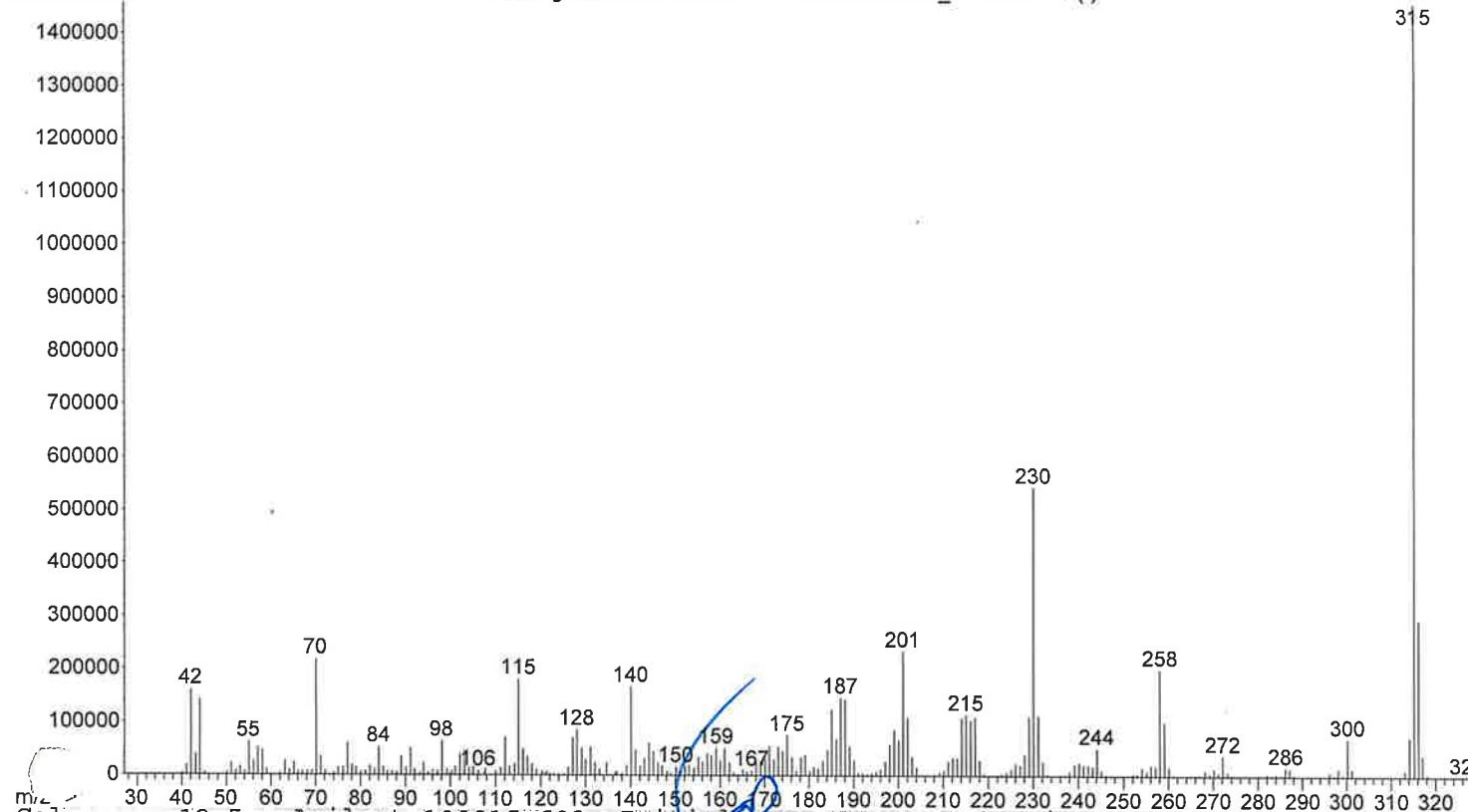
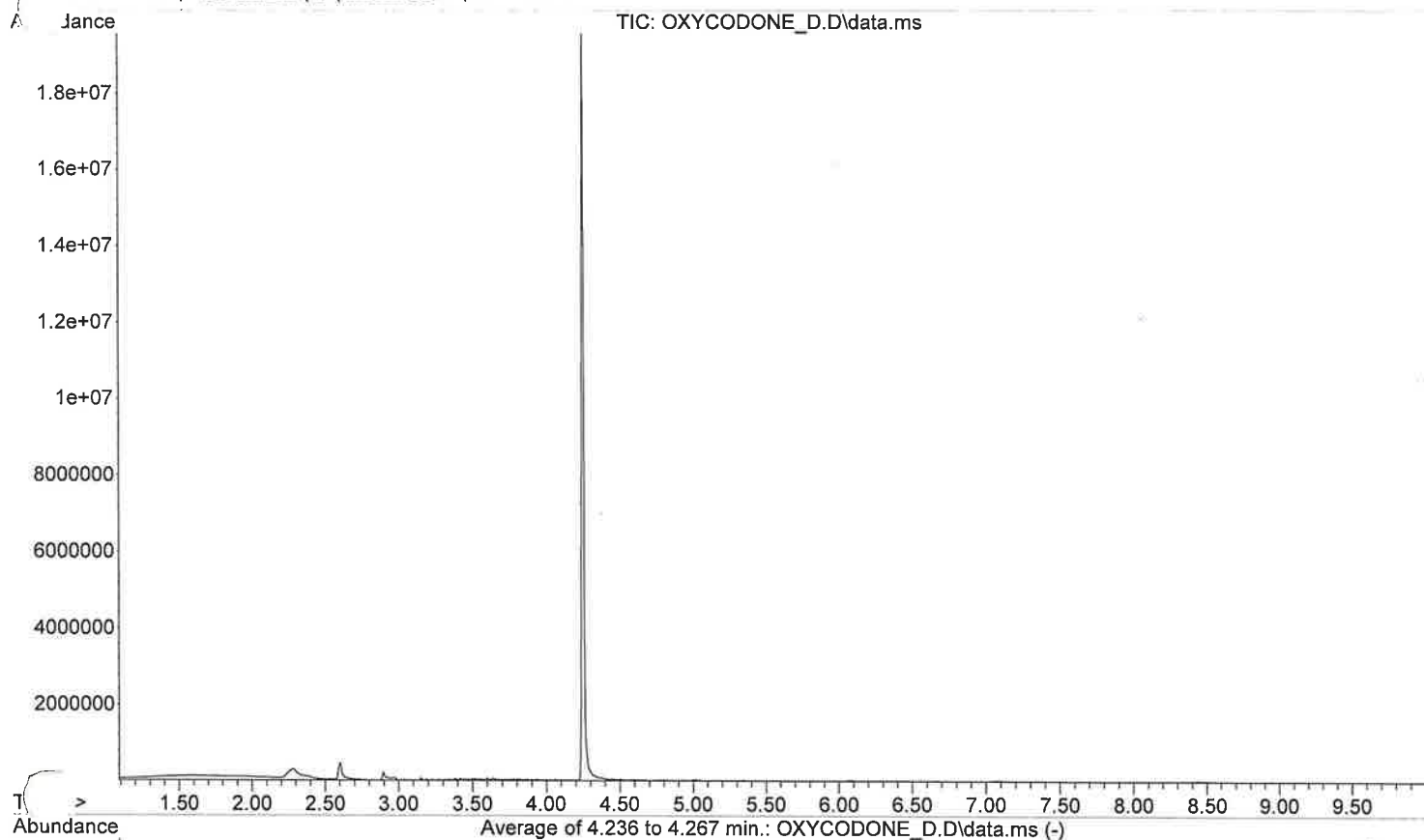
Instrument: Donna 2 (model: 5975 Serial number: US10423616)

Tuned on 16 Oct 2013 10:34 am Scanned from 40.00 to 550.00 amu

Acquired by JAS on 16 Oct 2013 14:46

Information: ~~Clonazepam~~ Splitless Misc. Information: Cerilliant lot#FE092910-02

Method: C:\MSDCHEM\1\METHODS\MSDRUGS.M



Column: a 12.5 m Agilent 19091S-602 Initial pressure: 10.43 psi

Inlet temperature: 250 'C Split Ratio: 50.0 m Transfer Line Temp: 280 'C

Initial oven temp: 100 'C held for 1.00 min Ramp:Rate Final temp

70.00

280

Validation Review

Procedure modification to msDRUGS method to allow for
more sample to get on column for low concentration compounds

Check the appropriate box: New Validation ☐ Modification ☒ Performance Check ☐

Sign and Date to indicate your acceptance of the procedure/instrument:

Examiner (if applicable) John Swartz Date 4/26/16

Unit Supervisor Glenn B. Everett Date 4-26-16

Technical Leader (if different than Supervisor) Glenn B. Everett Date 4-26-16

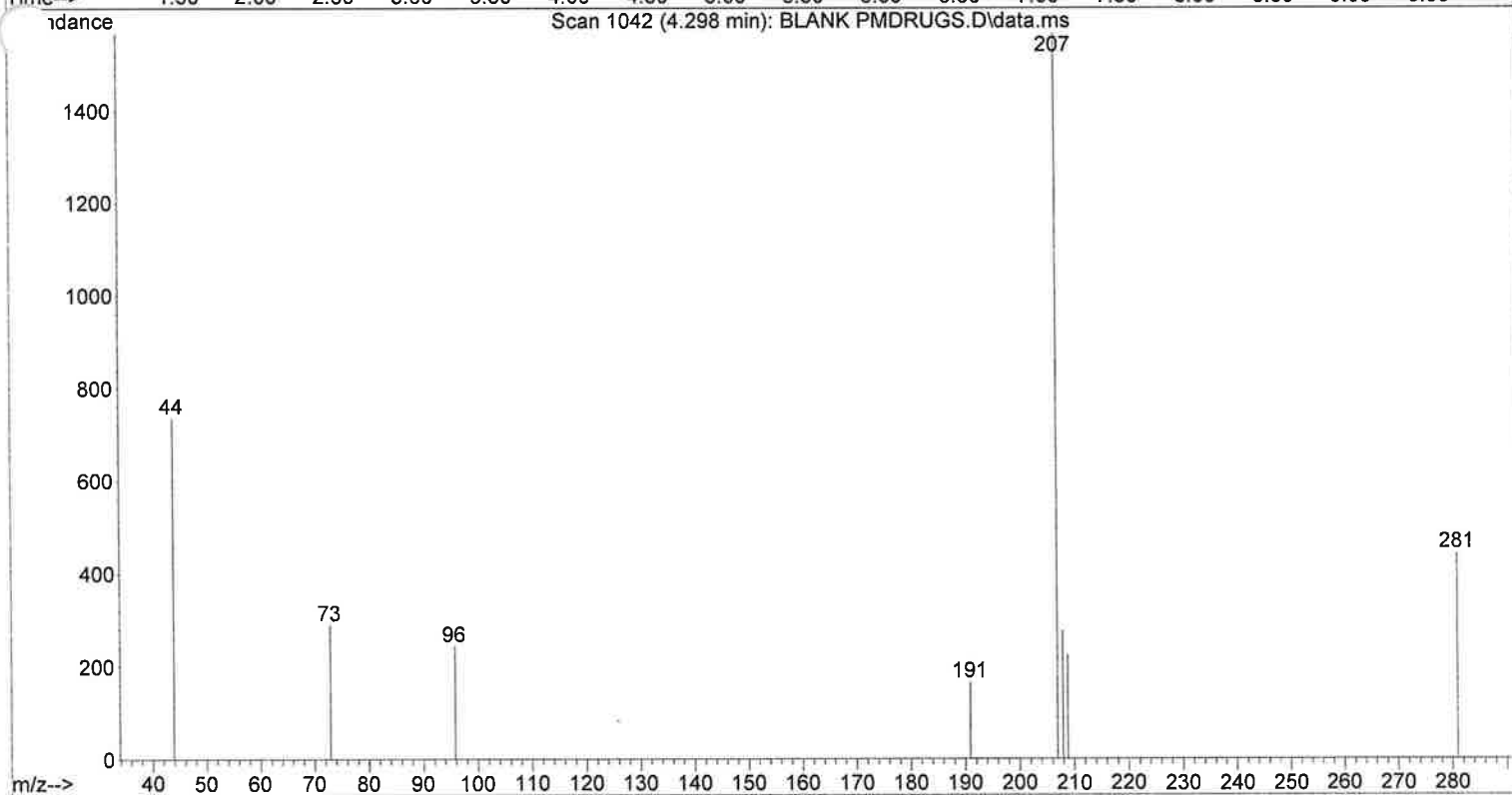
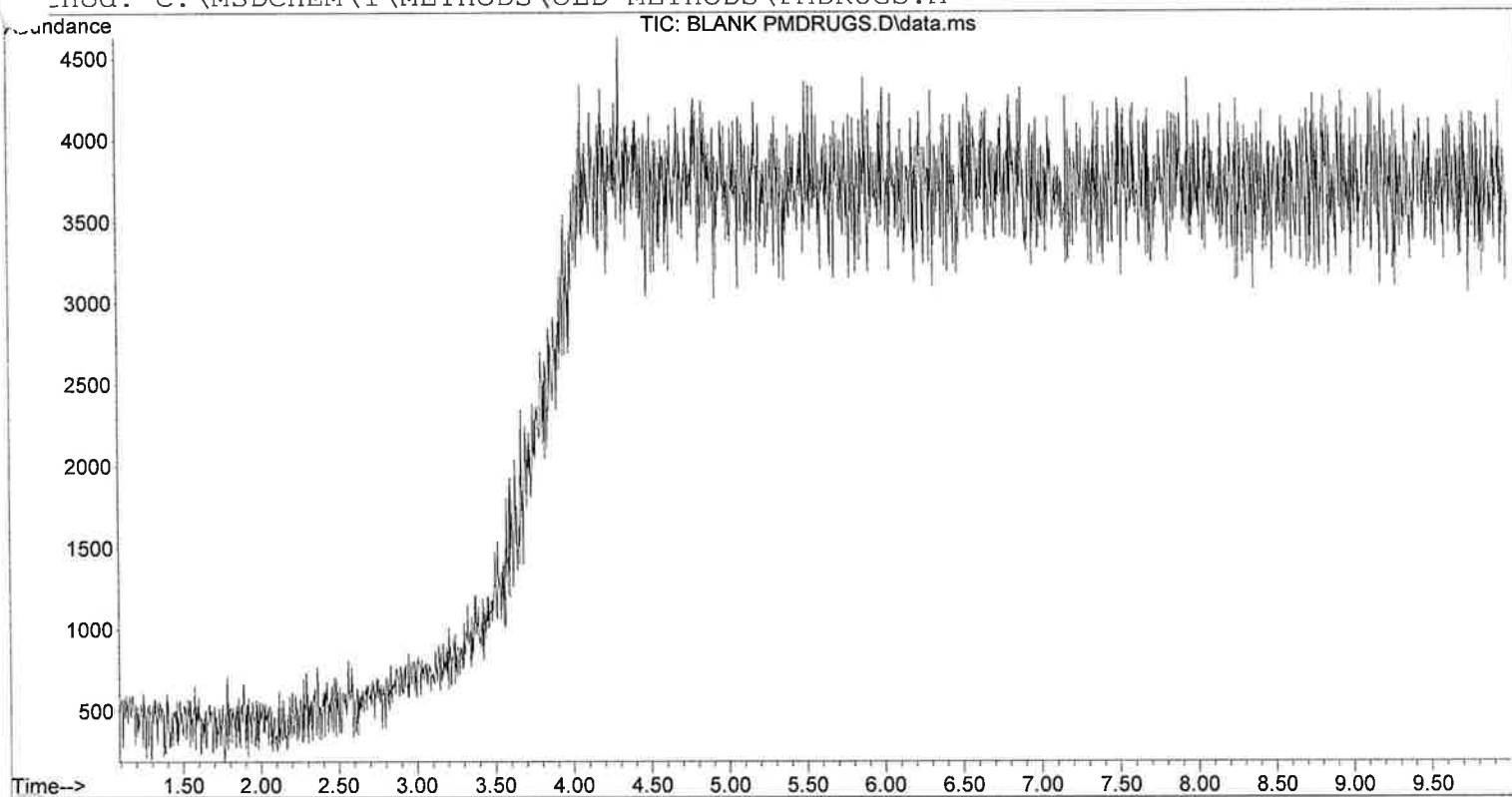
Crime Laboratory Regional Supervisor Jennifer Hall Date 4.26.16

Quality Assurance Manager Chad D. Johnson Date 04/26/16

*All information must be submitted to the Quality Assurance Manager prior to the implementation of the procedure/instrument. If all technical staff training has not been completed, please include a proposed training plan and completion schedule.

A PMDRUGS method was created. The PMDRUGS method is a modification of the previously validated MSDRUGS method. A validation study of the effects of split ratio was previously performed as well. The PMDRUGS method is a method that allows for more of the sample to get on the column when the analyst is dealing with low concentration compounds. A side by side study was performed with the MSDRUGS method and the PMDRUGS method by injecting the same sample volume (1 ul) of a known check standard mixture containing cocaine, pethidine, and hydrocodone. All three compounds were identified in both methods with the only variance being a greater abundance of all three on the PMDRUGS method.

Tennessee Bureau of Investigation: Nashville Crime Laboratory
Name of file: D:\Data\2016\JLS\HAND INJECTION\BLANK PMDRUGS.D
Instrument: Eggroll (model: 5975 Serial number: US61633037)
Tuned on 25 Apr 2016 8:53 am
Acquired by JOHN SCOTT, JR. on 26 Apr 2016 8:52
Information: BLANK Misc. Information: MEOH
Method: C:\MSDCHEM\1\METHODS\OLD METHODS\PMDRUGS.M



Injection Temp = 250.0000 C Transfer Line = 280.0000 C

Split Ratio = 75.0000 Column Flow = 1.0006 mL/min

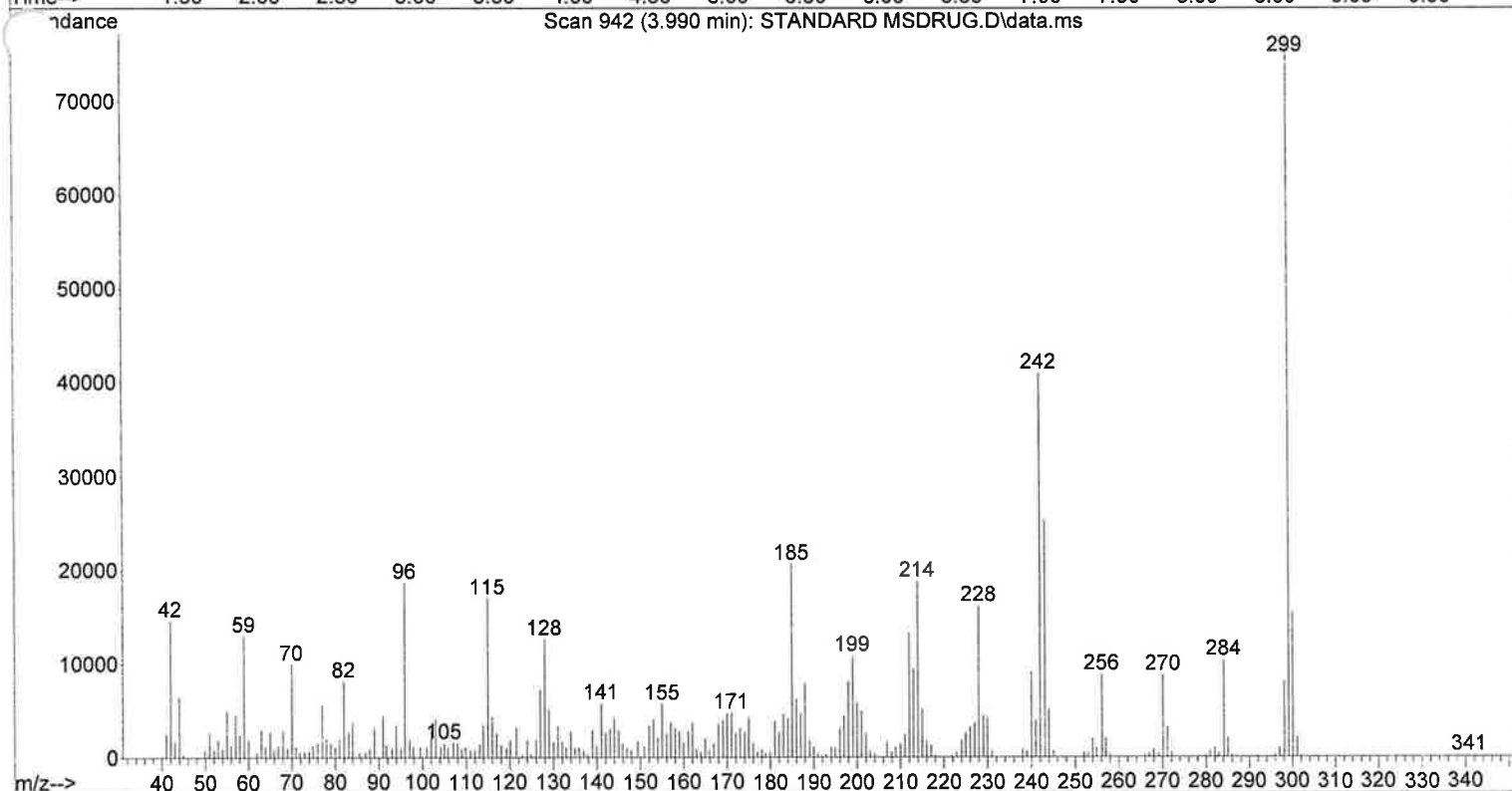
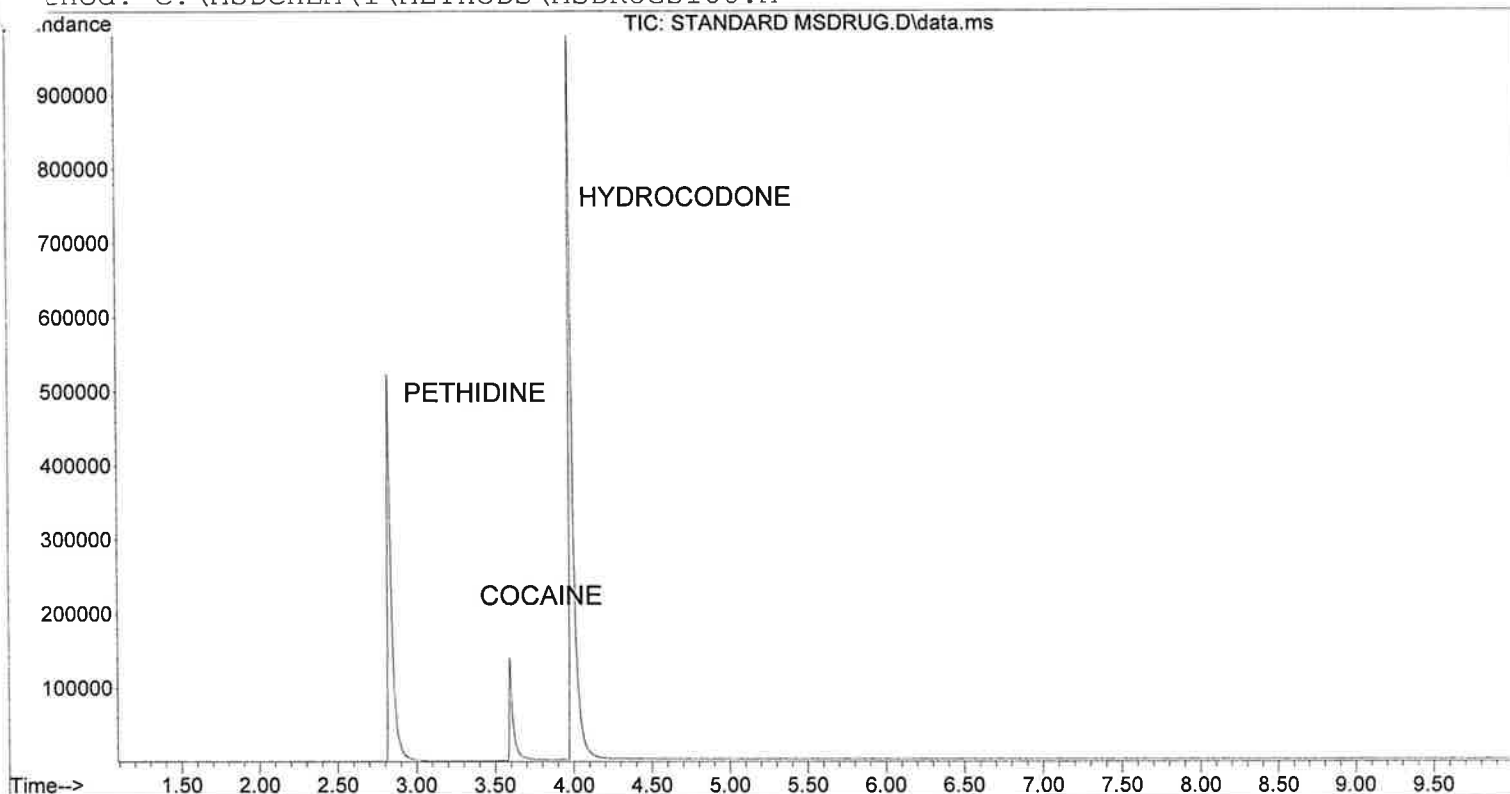
The column is a 12.5 meter 19091S-602

Scanned from 40.00 amu to 550.00 amu

Initial temp 100 C for 1 min, Ramped at 70 C/min to 280 C

AP

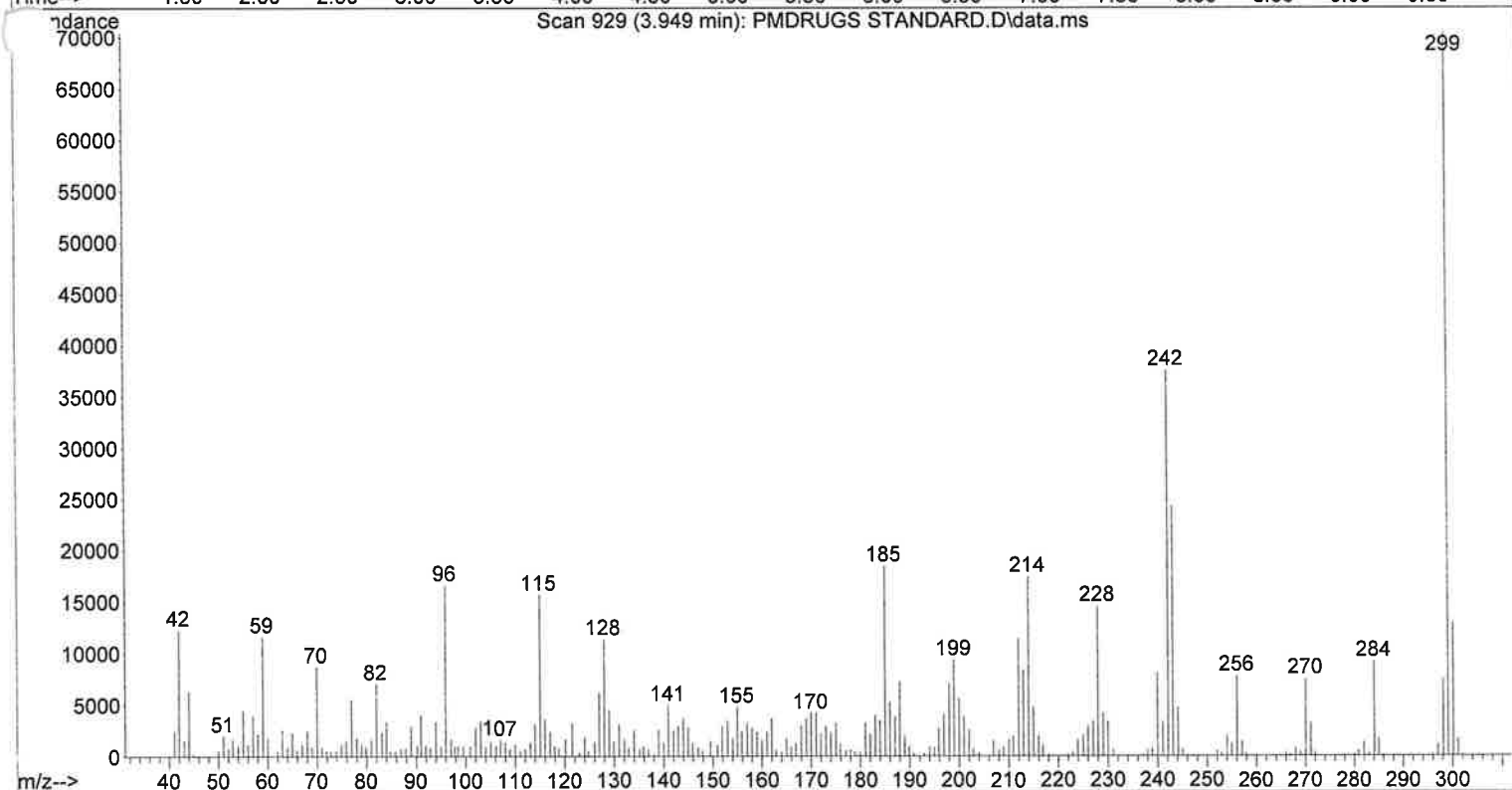
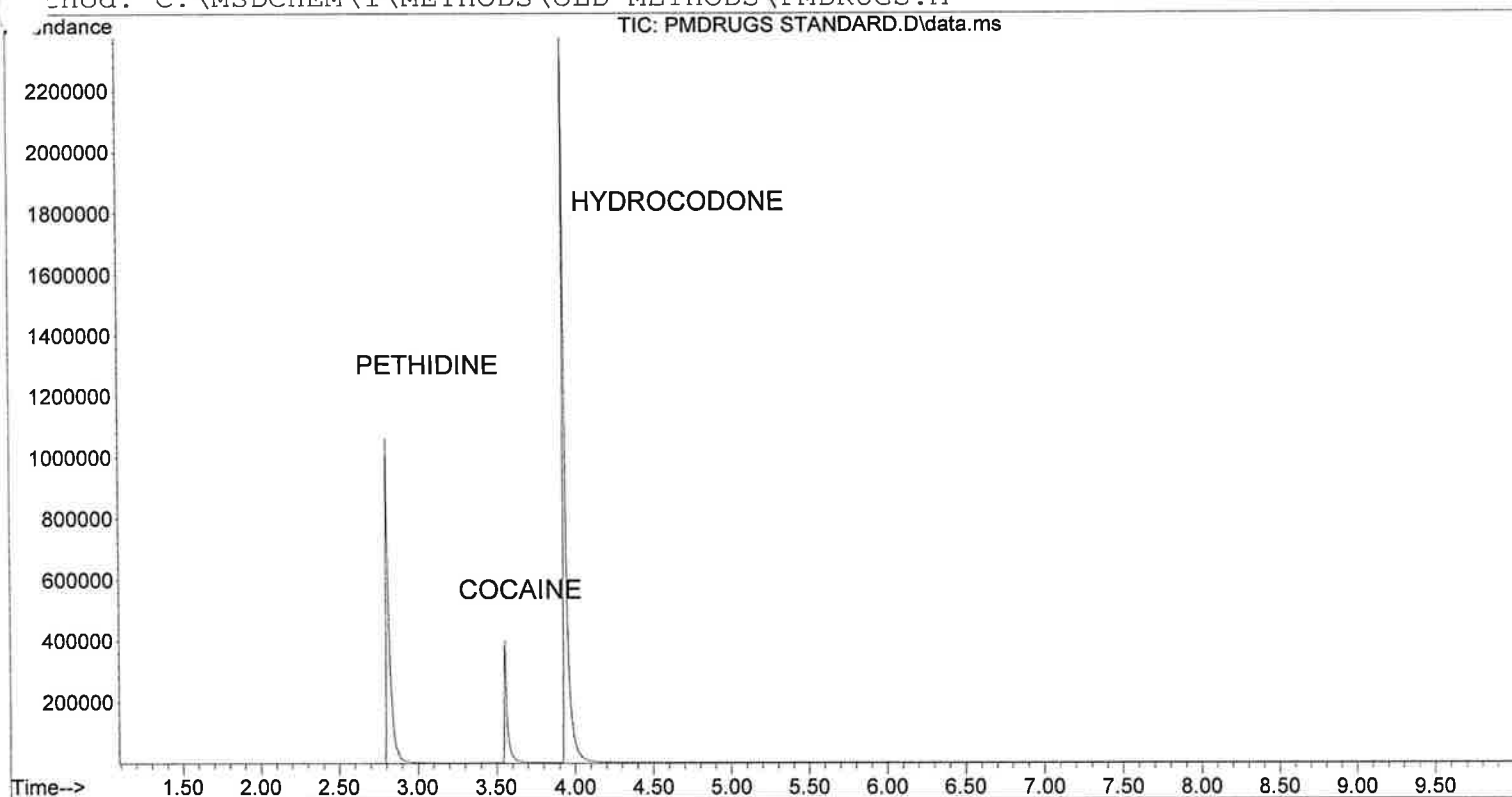
Tennessee Bureau of Investigation: Nashville Crime Laboratory
Name of file: D:\Data\2016\JLS\HAND INJECTION\STANDARD MSDRUG.D
Instrument: Eggroll (model: 5975 Serial number: US61633037)
Tuned on 25 Apr 2016 8:53 am
Acquired by JOHN SCOTT, JR. on 26 Apr 2016 8:38
Information: PETHIDINE, COCAINE, HYDROCODONE STANDARDS Misc. Information: MEOH
Method: C:\MSDCHEM\1\METHODS\MSDRUGS100.M



Injection Temp = 250.0000 C Transfer Line = 280.0000 C
Split Ratio = 75.0000 Column Flow = 1.0006 mL/min
The column is a 12.5 meter 19091S-602
Scanned from 40.00 amu to 550.00 amu
Initial temp 100 C for 1 min, Ramped at 70 C/min to 280 C

4

Tennessee Bureau of Investigation: Nashville Crime Laboratory
Name of file: D:\Data\2016\JLS\HAND INJECTION\PMDRUGS STANDARD.D
Instrument: Eggroll (model: 5975 Serial number: US61633037)
Tuned on 25 Apr 2016 8:53 am
Acquired by JOHN SCOTT, JR. on 26 Apr 2016 9:36
Information: PETHIDINE, COCAINE, HYDROCODONE STANDARDS Misc. Information: MEOH
Method: C:\MSDCHEM\1\METHODS\OLD METHODS\PMDRUGS.M



Injection Temp = 250.0000 C Transfer Line = 280.0000 C
Split Ratio = 75.0000 Column Flow = 1.0024 mL/min
The column is a 12.5 meter 19091S-602
Scanned from 40.00 amu to 550.00 amu
Initial temp 100 C for 1 min, Ramped at 70 C/min to 280 C

A