

Validation Review

Procedure Allyl Nitrite GC/MS Method Validation

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

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

Examiner (if applicable) Ella Carpenter Date 5/16/17

Unit Supervisor Blinn B. Everett Date 5/16/17

Technical Leader (if different than Supervisor) Blinn B. Everett Date 5/16/17

Crime Laboratory Regional Supervisor Jennifer Hall Date 5.19.17

Quality Assurance Manager CUDG Date 05/18/17

*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.

Forensic Chemistry Method Validation
Alkyl Nitrite GC/MS Method - MS DRUGS 60 NIT

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Forensic Chemistry Method Validation

Alkyl Nitrite GC/MS Method - MS DRUGS 60NIT

1. Purpose:

Alkyl nitrites are controlled substances that are not regularly encountered in TBI FCU casework. Due to their low molecular weights, these compounds will often elute well before the solvent delay used in other methods has completed.

The electronic ionization of these compounds results in ion fragments with smaller m/z ratios that are not detected by the FCU's routine fragment scan range (40 – 500 m/z.)

This method has been developed to satisfy the distinct parameters required to successfully identify these compounds.

2. Required Instrumental Parameters

The basic method parameters are listed below.

<i>Starting temperature</i>	60°C
<i>Solvent delay</i>	Disabled
<i>Ramp rate</i>	70°C/min
<i>Ending temperature</i>	280°C
<i>Total run time</i>	10 minutes
<i>Fragment Scan Range</i>	5 – 500 m/z

Appendix A outlines the method in its entirety.

3. Procedure

Three working alkyl nitrite standards will be analyzed using this method. Due to volatility of the standards, headspace injections will be conducted using a gas-tight syringe. No further extraction is necessary. Standards can be heated within the injection vials to ensure adequate vapor concentration.

An air blank will be used as the procedural blank for this method. Air column "washes" will be performed between each standard run. The syringe will also be rinsed with methanol and thoroughly dried after each injection to eliminate cross contamination.

The standards will be manually injected into the instrument since the FCU does not have the equipment necessary for headspace autosampling.

Repeatability analyses will be performed using independent duplicate samples to ensure reproducible spectra. All analysis will be performed on the GC/MS identified as Espresso in the FCU. Further information on this instrument is on file within the FCU.

Forensic Chemistry Method Validation

Alkyl Nitrite GC/MS Method - MSDRUGS60NIT

4. Working Standards

Standard	Source	Lot Number
Butyl nitrite	Sigma	08311CI
Isobutyl nitrite	Sigma	DI08910AI
tert-Butyl nitrite	Sigma	TU00708TS

5. Results and Conclusions

Each standard demonstrated the correct fragmentation pattern when compared to previously verified standards. The standards also demonstrated satisfactory spectra for repeat analyses.

METHOD CONTROL PARAMETERS

Method Information For: C:\MSDCHEM\1\METHODS\MSDRUGS60NIT.M
Method Sections To Run:

- ☐ Save Copy of Method With Data
- ☐ Instrument Control Pre-Run Cmd/Macro =
- ☐ Data Analysis Pre-Run Cmd/Macro =
- ☒ Data Acquisition
- ☒ Data Analysis
- ☐ Instrument Control Post-Run Cmd/Macro =
- ☐ Data Analysis Post-Run Cmd/Macro =

Method Comments:

Manual injection 60-280 for 10 minutes

END OF METHOD CONTROL PARAMETERS

C:\MSDCHEM\1\METHODS\MSDRUGS60NIT.M

Control Information

Sample Inlet : GC
Injection Source : Manual
Injection Location: Front
Mass Spectrometer : Enabled

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6890 GC METHOD

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OVEN

Initial temp: 60 'C (On) Maximum temp: 325 'C
Initial time: 0.00 min Equilibration time: 0.50 min
Ramps:
Rate Final temp Final time
1 70.00 280 6.86
2 0.0 (Off)
Post temp: 0 'C
Post time: 0.00 min
Run time: 10.00 min

FRONT INLET (SPLIT/SPLITLESS)

Mode: Split
Initial temp: 250 'C (On)
Pressure: 13.39 psi (On)
Split ratio: 200:1
Split flow: 293.4 mL/min
Total flow: 298.4 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 2.00 min
Gas type: Helium

BACK INLET (SPLIT/SPLITLESS)

Mode: Split
Initial temp: 250 'C (On)
Pressure: 10.38 psi (On)
Split ratio: 50:1
Split flow: 103.6 mL/min
Total flow: 108.6 mL/min
Gas saver: Off
Gas type: Helium

COLUMN 1

Capillary Column
Model Number: J&W 19091S-602
HP1-MS
Max temperature: 350 'C
Nominal length: 12.5 m
Nominal diameter: 200.00 um
Nominal film thickness: 0.33 um
Mode: constant flow
Initial flow: 1.5 mL/min
Nominal init pressure: 13.39 psi
Average velocity: 69 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

COLUMN 2

Capillary Column
Model Number: Agilent 19091Z-213
HP-1 Methyl Siloxane
Max temperature: 325 'C
Nominal length: 30.0 m
Nominal diameter: 320.00 um
Nominal film thickness: 1.00 um
Mode: constant pressure
Pressure: 10.38 psi
Nominal initial flow: 2.1 mL/min
Average velocity: 35 cm/sec
Inlet: Back Inlet
Outlet: Back Detector
Outlet pressure: ambient

FRONT DETECTOR (NO DET)

BACK DETECTOR (FID)

Temperature: 250 'C (On)
Hydrogen flow: 40.0 mL/min (Off)
Air flow: 450.0 mL/min (Off)
Mode: Constant makeup flow
Makeup flow: 45.0 mL/min (On)
Makeup Gas Type: Helium
Flame: Off
Electrometer: On
Lit offset: 2.0

SIGNAL 1
Data rate: 50 Hz
Type: back detector
Save Data: On
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

SIGNAL 2
Data rate: 50 Hz
Type: back detector
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
Derive from back detector

COLUMN COMP 2
Derive from back detector

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description:
Initial temp: 280 'C (On)
Initial time: 0.00 min
Rate Final temp Final time
1 0.0 (Off)

POST RUN
Post Time: 0.00 min

TIME TABLE
Time Specifier Parameter & Setpoint

GC Injector

Front Injector:
Sample Washes 0
Sample Pumps 3
Injection Volume 1.00 microliters
Syringe Size 10.0 microliters
PreInj Solvent A Washes 0
PreInj Solvent B Washes 0
PostInj Solvent A Washes 0
PostInj Solvent B Washes 0
Viscosity Delay 0 seconds
Plunger Speed Fast
PreInjection Dwell 0.00 minutes
PostInjection Dwell 0.00 minutes

Back Injector:
No parameters specified

Column 1 Inventory Number : AB006
Column 2 Inventory Number : AB002

MS ACQUISITION PARAMETERS

General Information

Tune File : atune.u
Acquisition Mode : Scan

MS Information

Solvent Delay : 0.00 min
EM Absolute : False
EM Offset : 0

Resulting EM Voltage : 1541.2

[Scan Parameters]

Low Mass : 5.0
High Mass : 500.0
Threshold : 150
Sample # : 2 A/D Samples 4
Plot 2 low mass : 40.0
Plot 2 high mass : 550.0

[MSZones]

MS Quad : 150 C maximum 200 C
MS Source : 230 C maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\MSDCHEM\1\METHODS\MSDRUGS60NIT.M

Percent Report Settings

Sort By: Signal

Output Destination

Screen: No
Printer: Yes
File: No

Integration Events: AutoIntegrate

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search Minimum Quality

C:\Database\TBI_DRUG.L 0
C:\Database\PMW_Tox3.1 0
C:\Database\NIST05a.L

Integration Events: AutoIntegrate

Report Type: Summary

Output Destination

Screen: No
Printer: No
File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination

Screen: Yes
Printer: No
File: No

Generate Report During Run Method: No

Calibration Last Updated:

Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent

Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

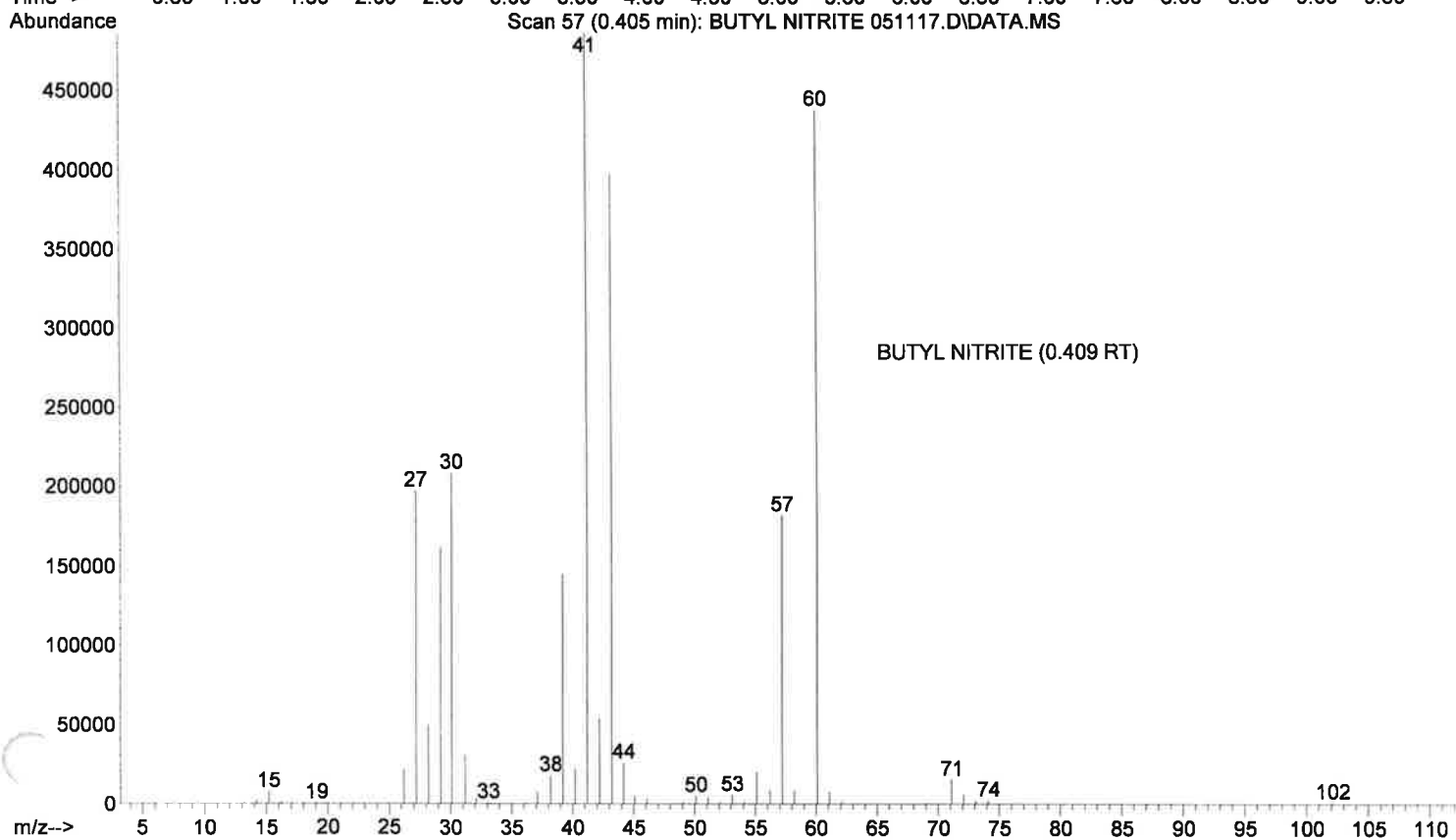
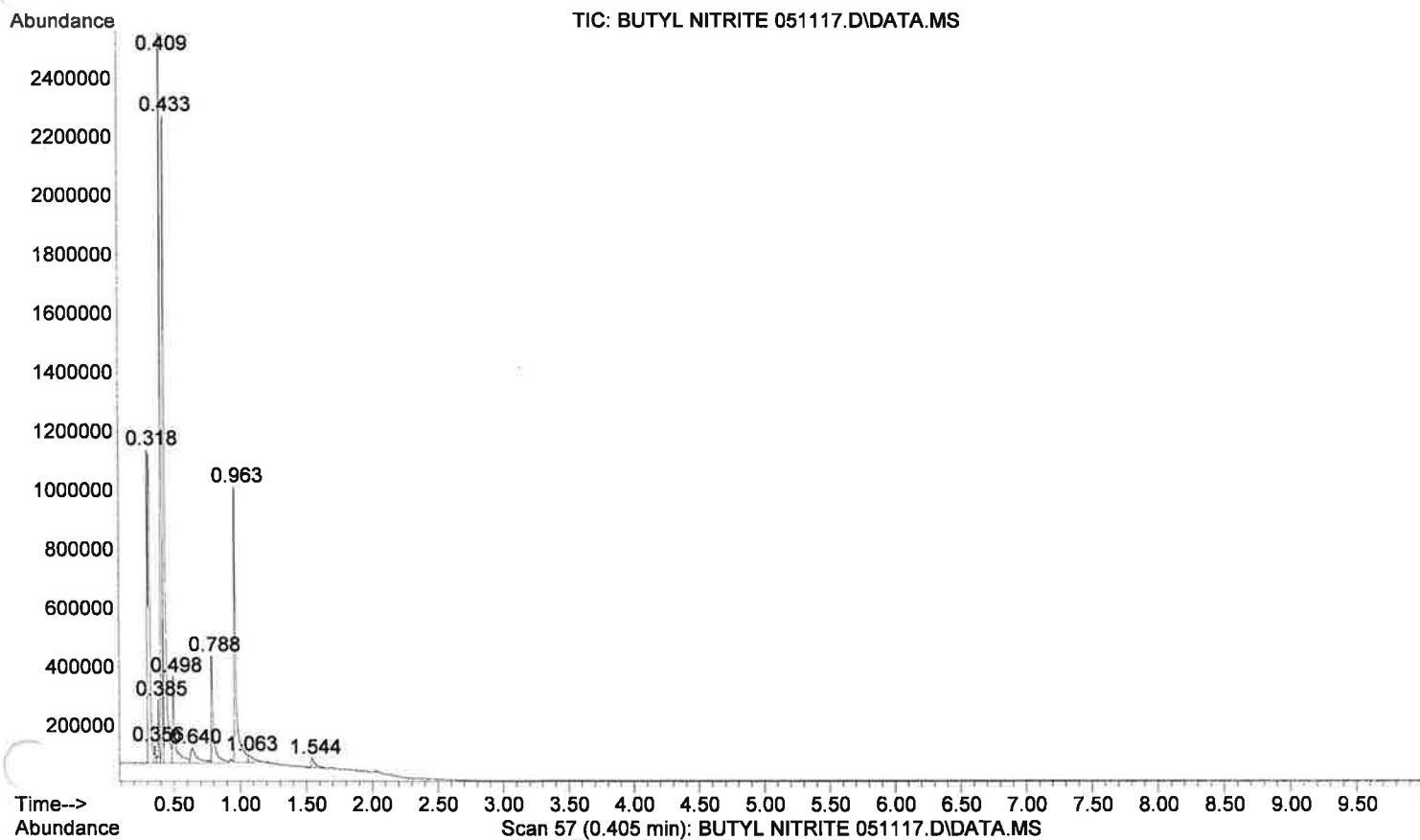
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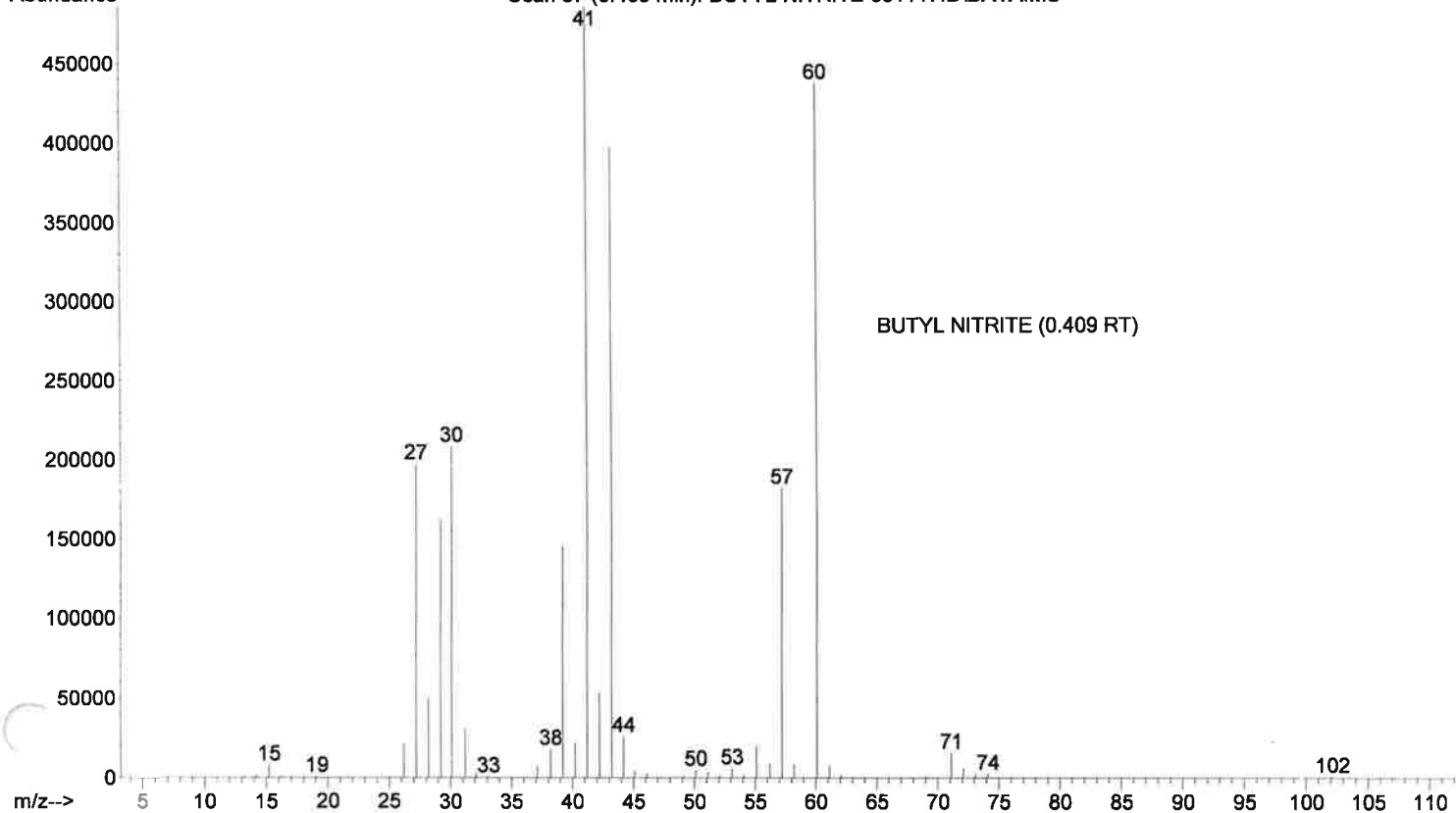
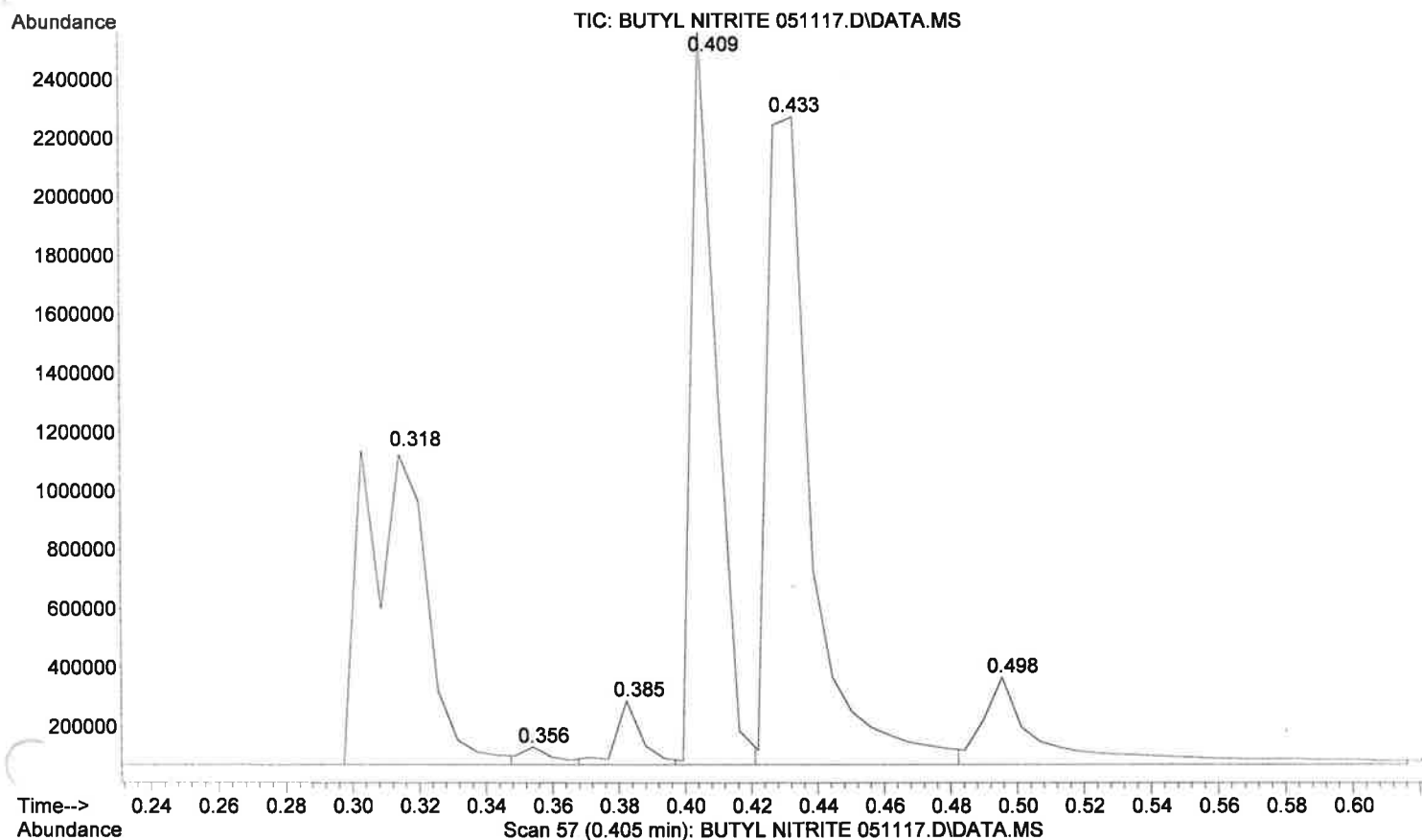
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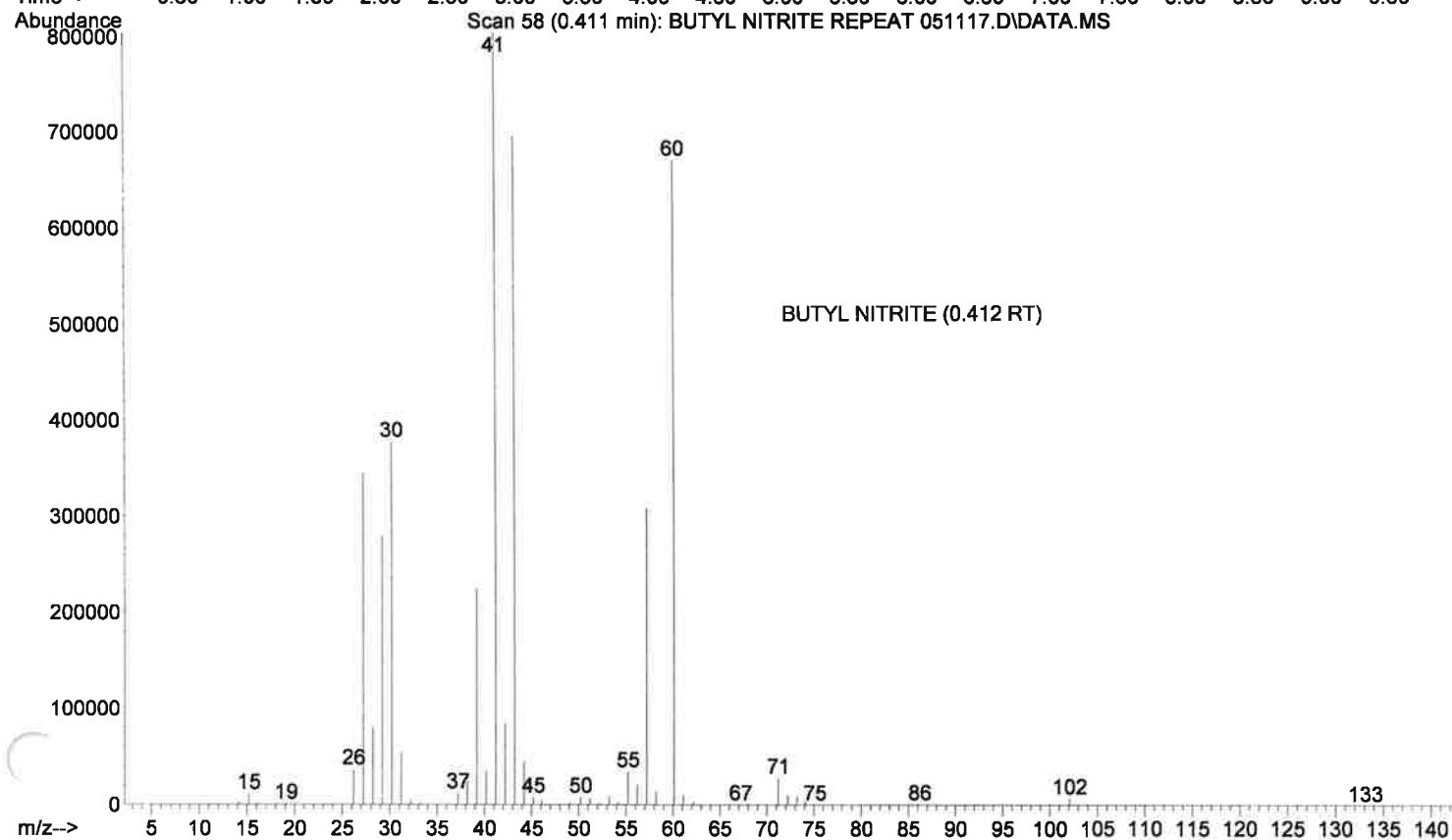
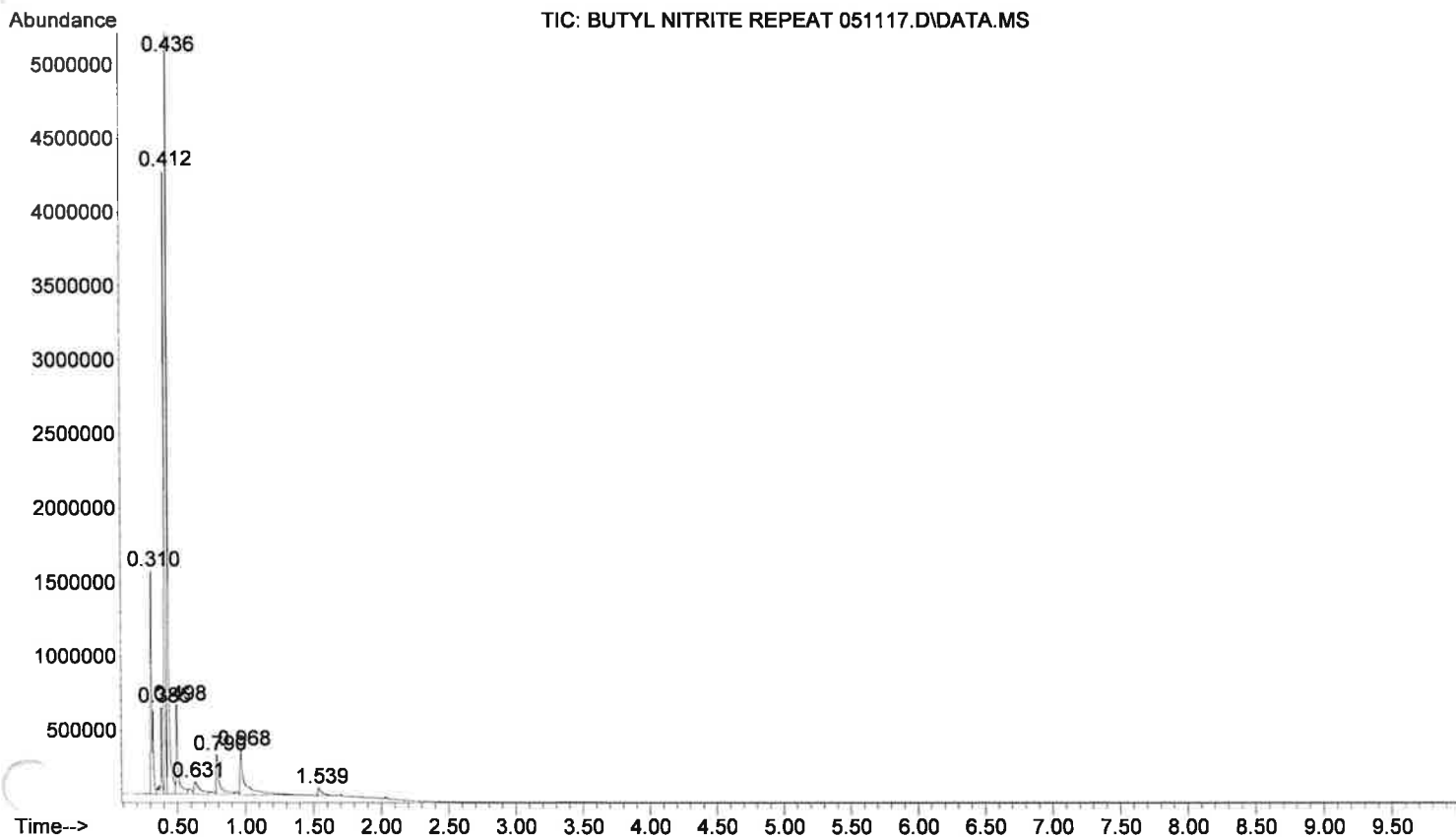
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Instrument : Espresso
Acquired : 11 May 2017 8:55 using AcqMethod MSDRUGS60NIT.M
Sample Name: BUTYL NITRITE 5/11/17
Misc Info : HEADSPACE



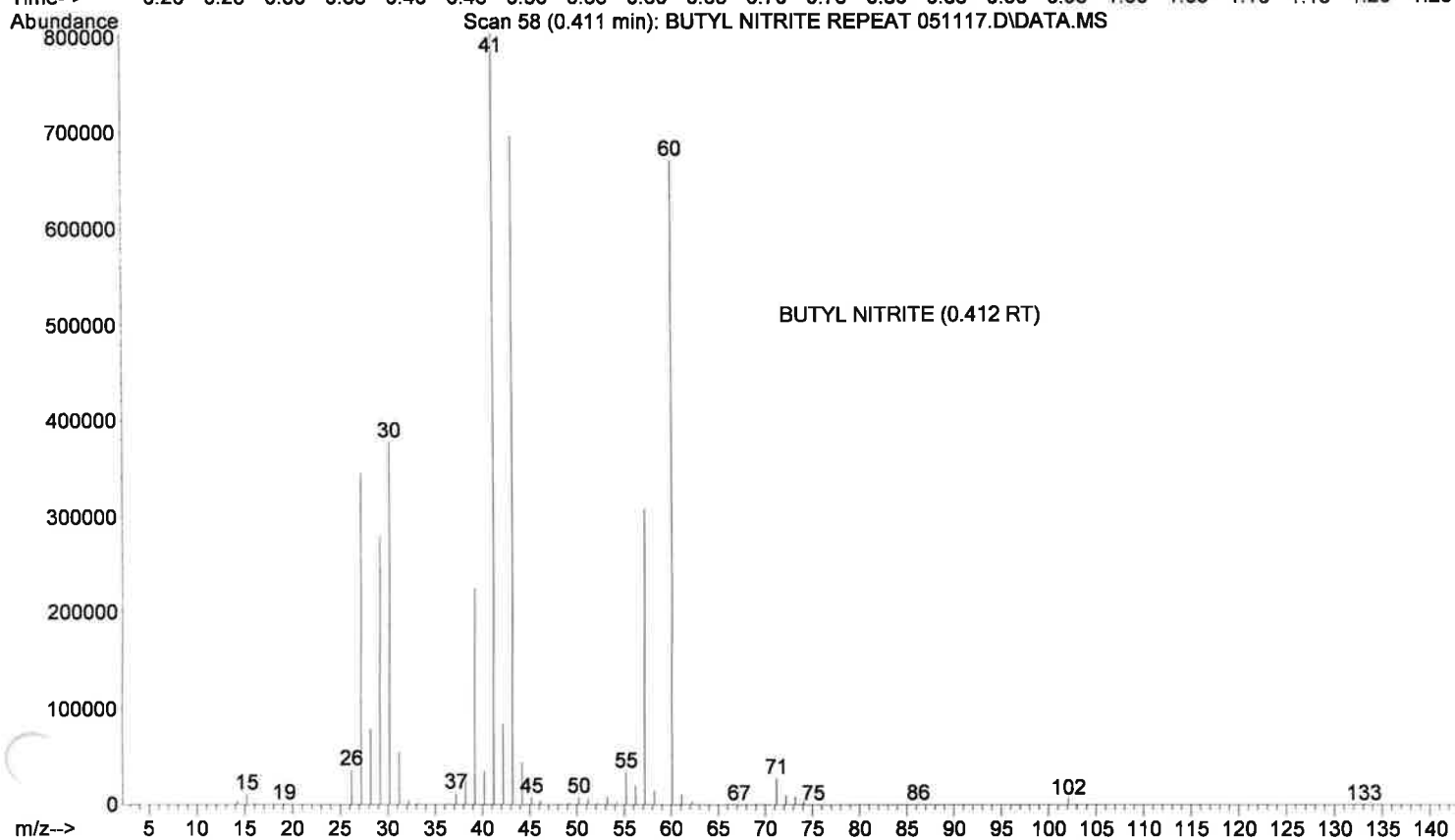
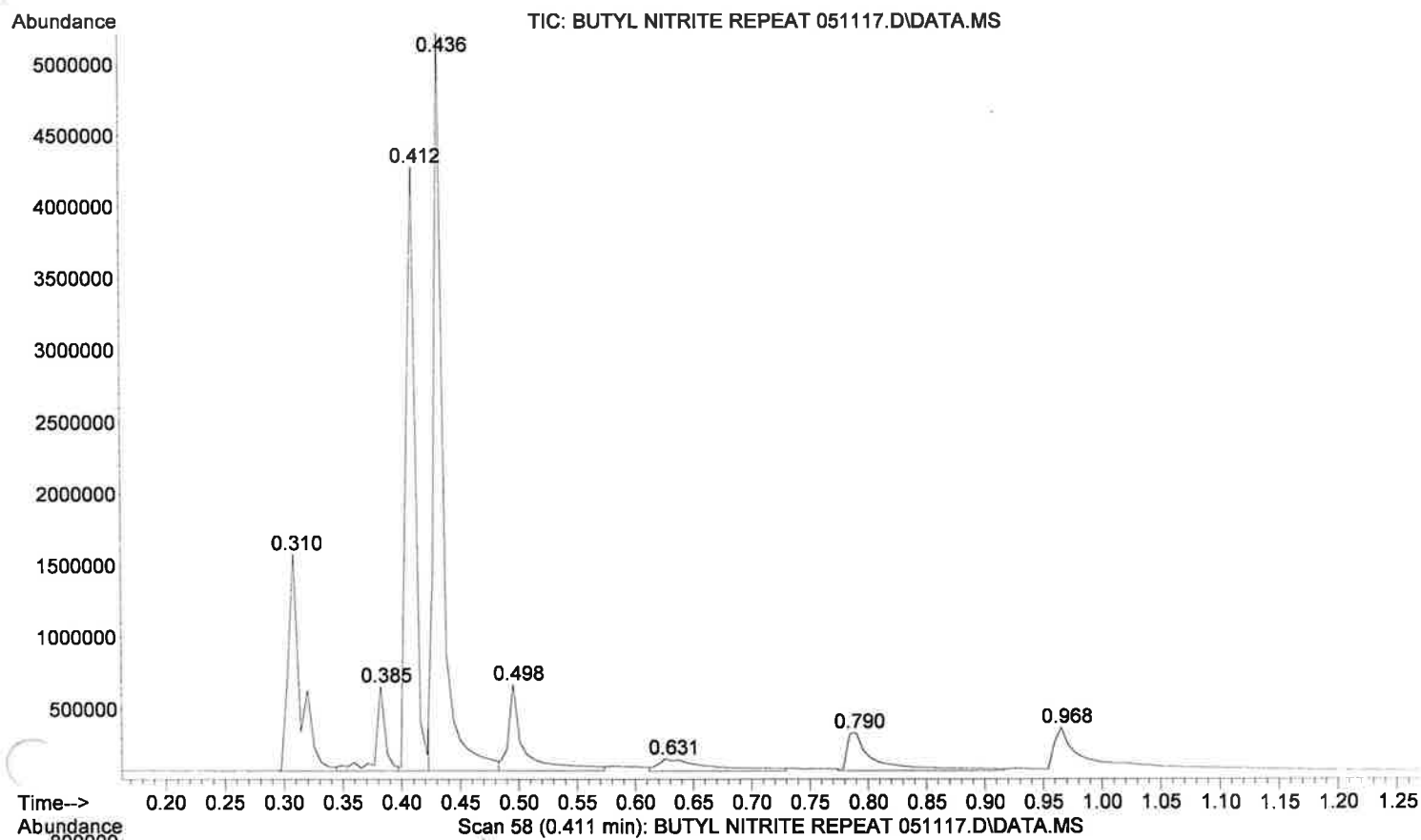
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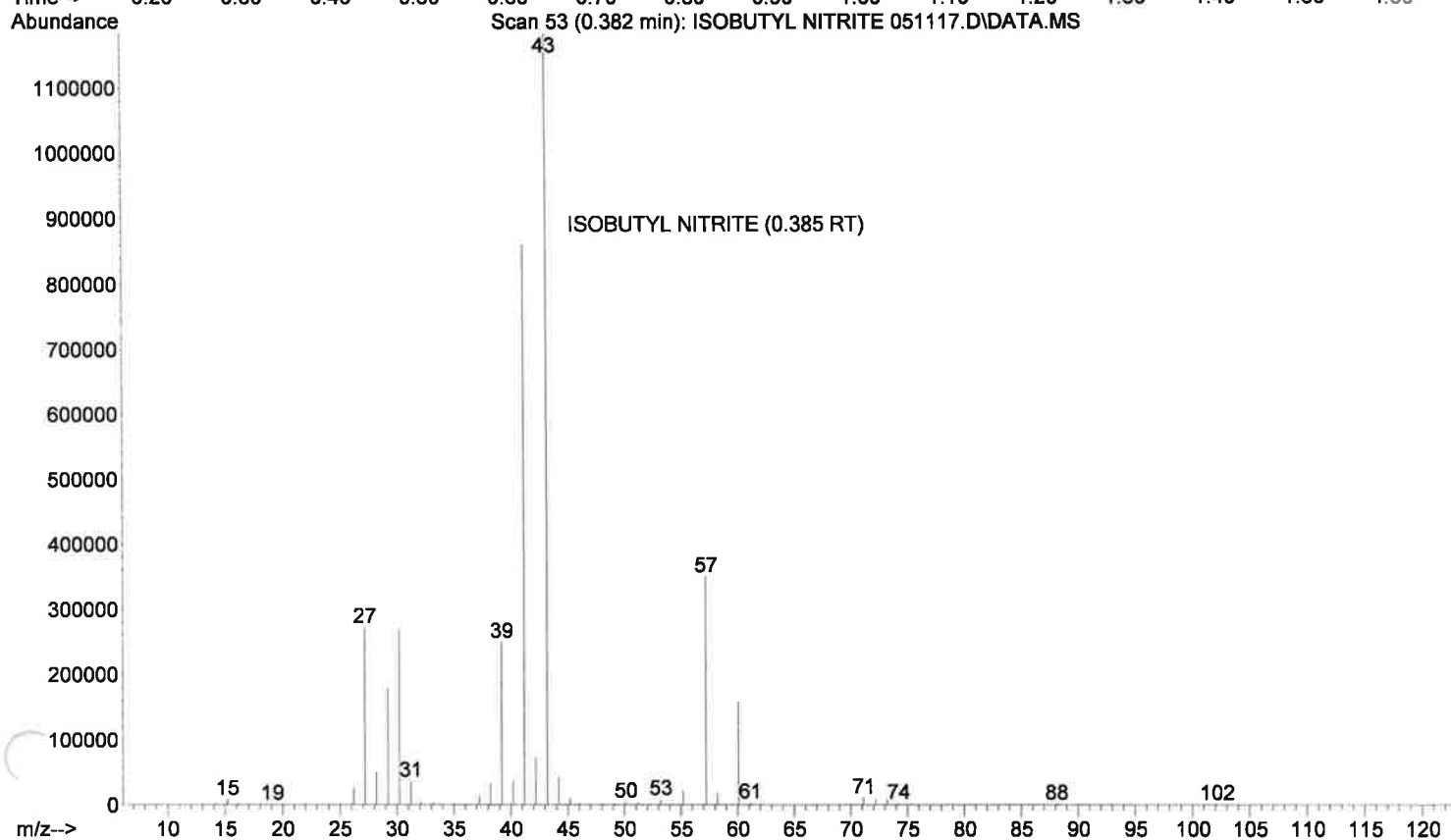
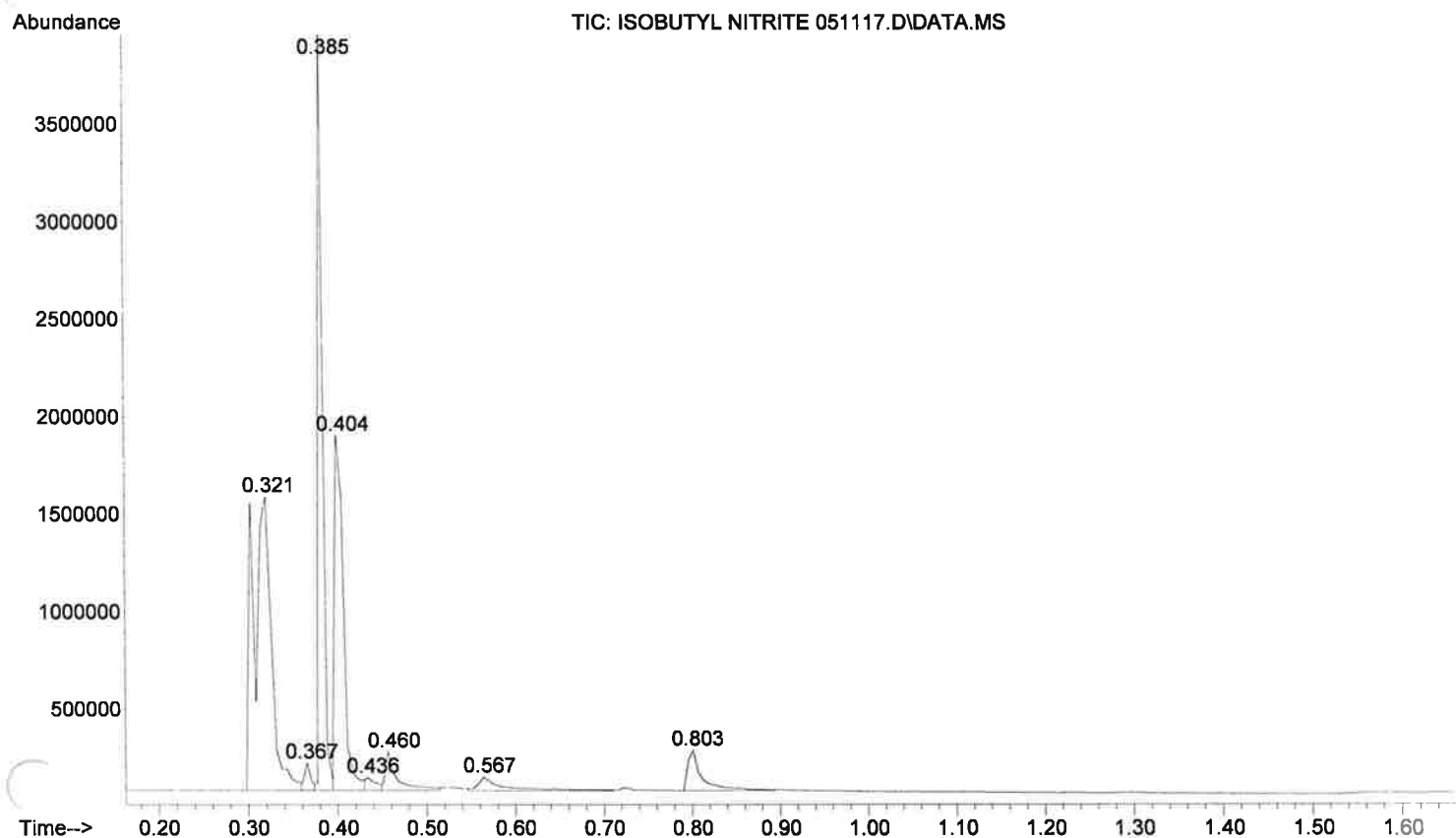
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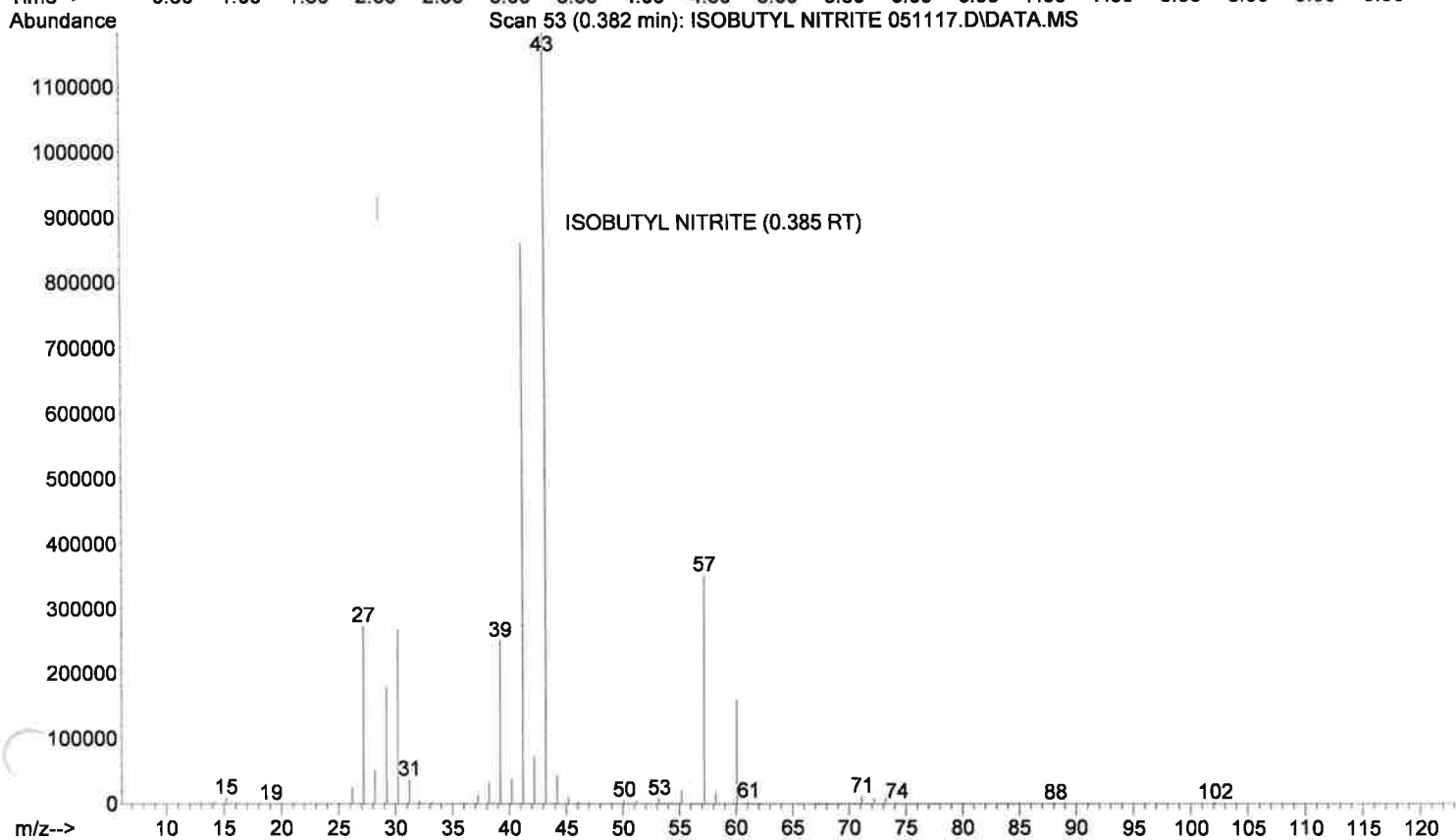
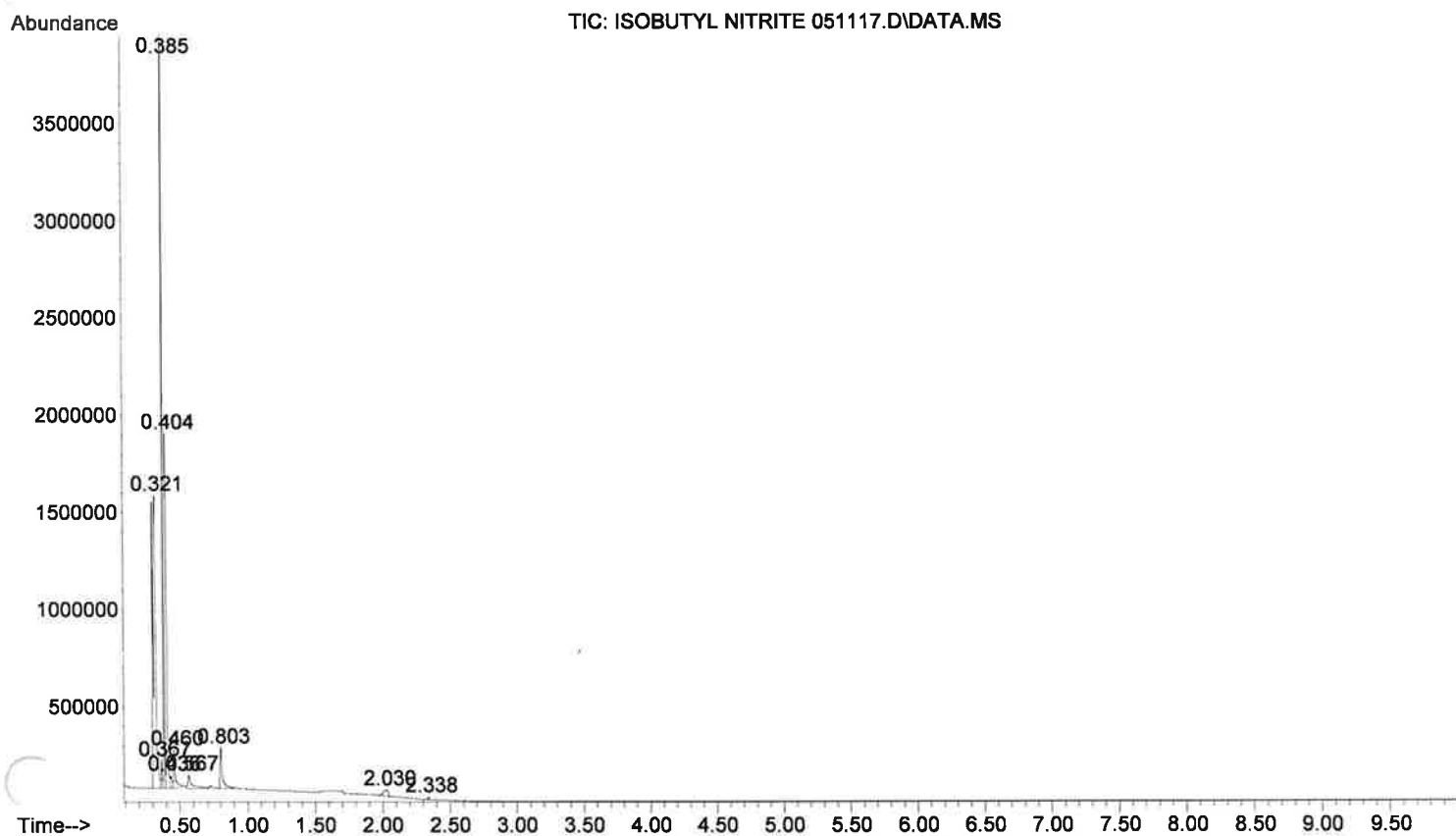
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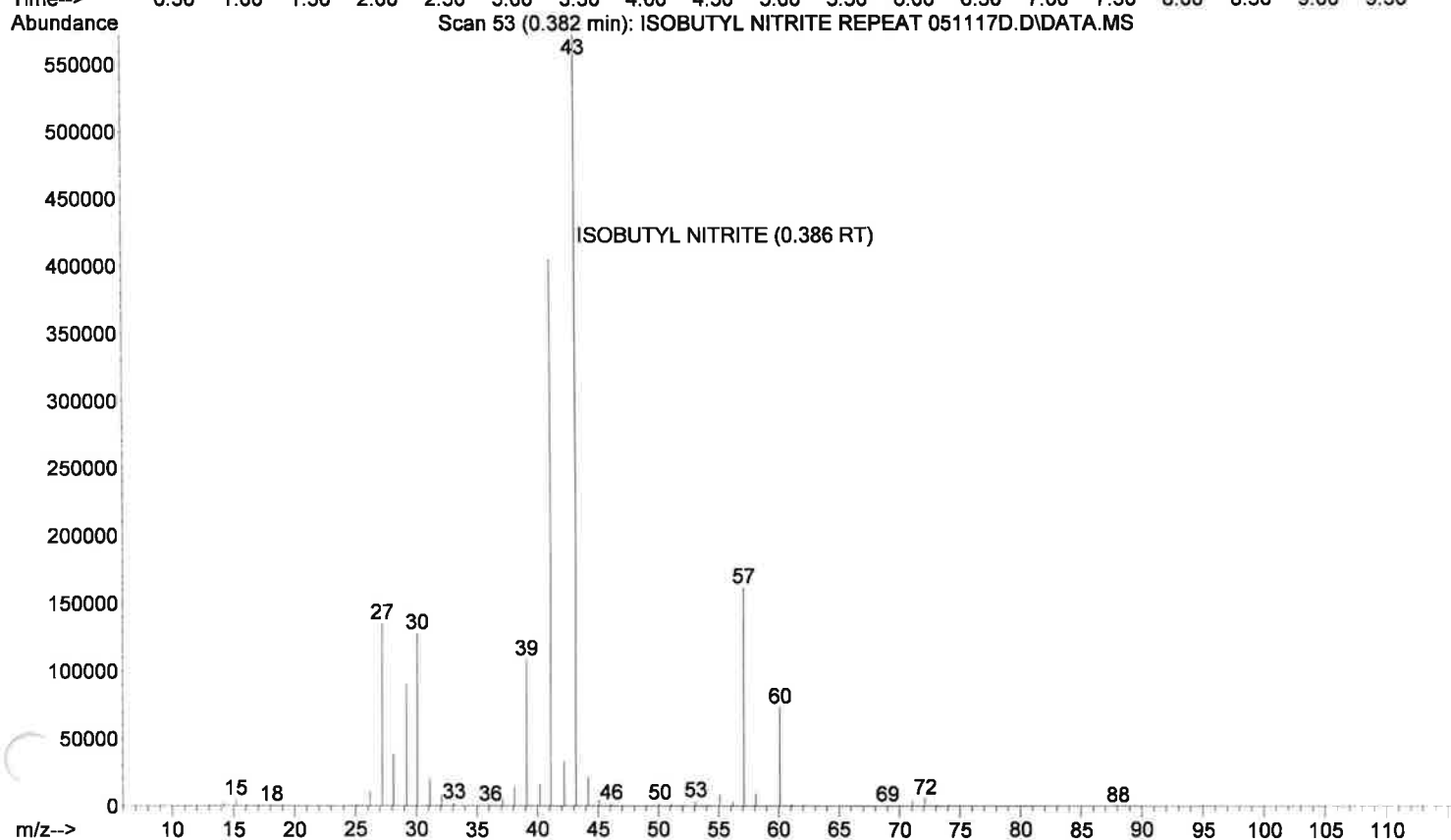
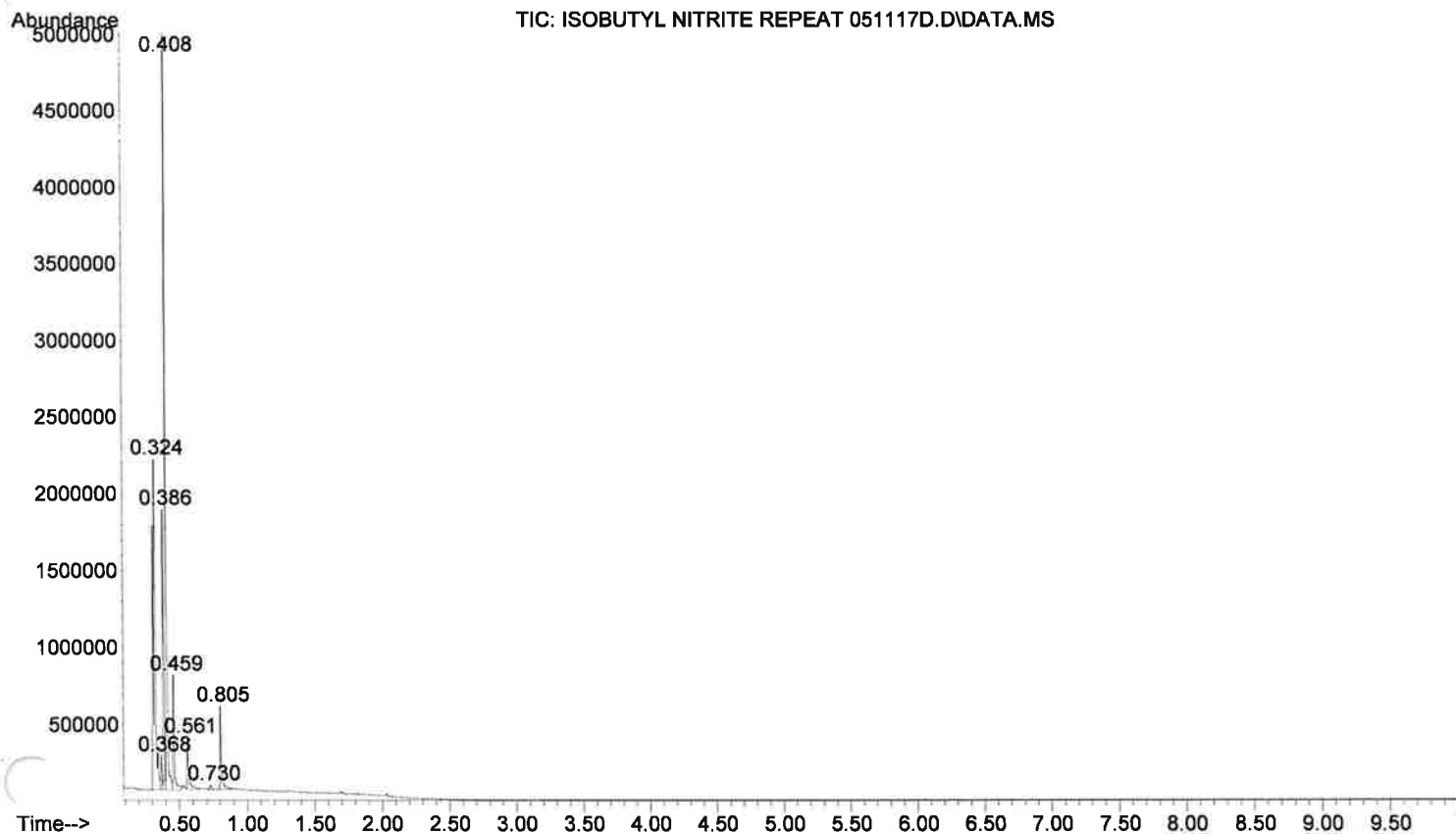
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Mi Info : HEADSPACE



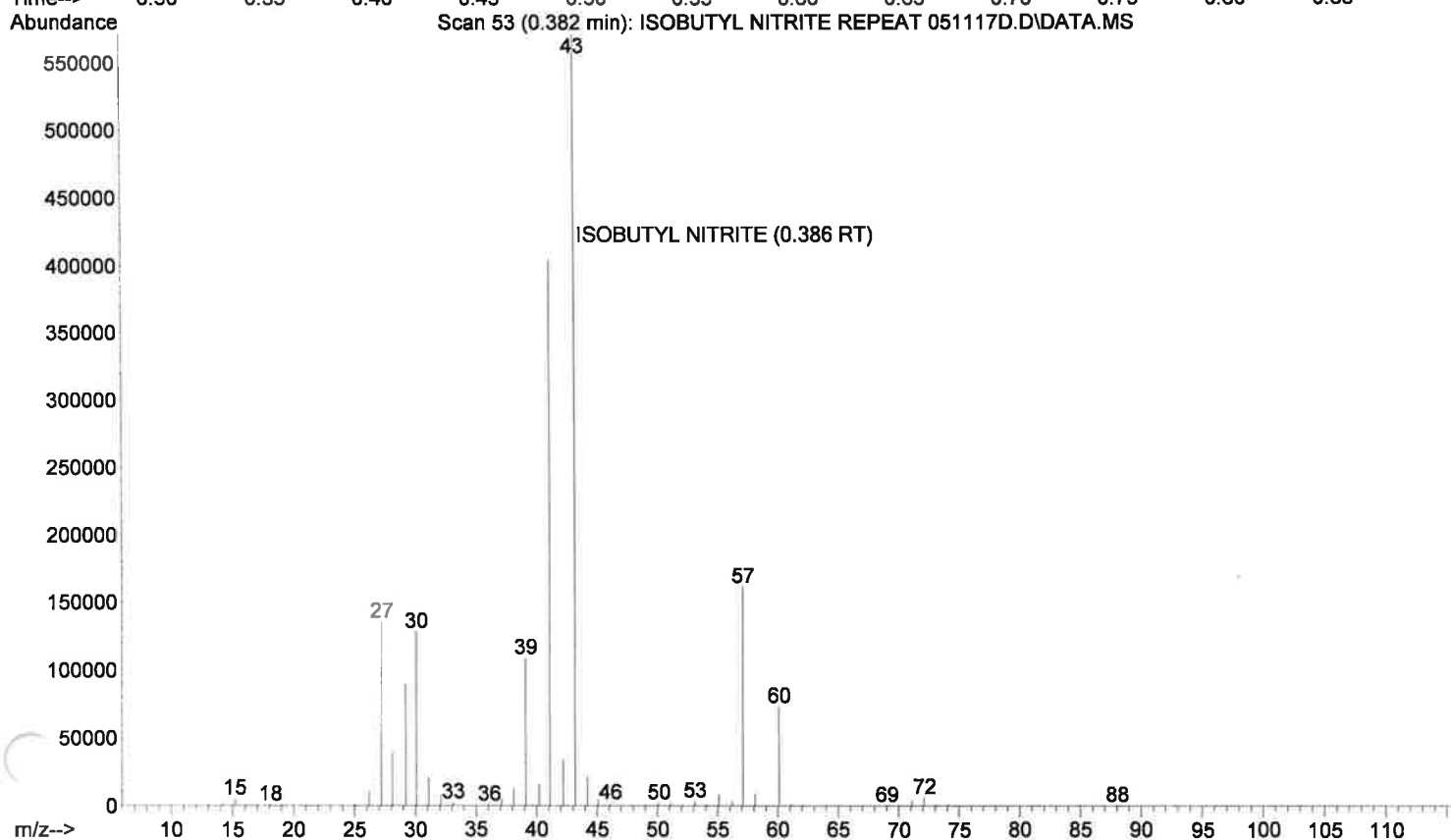
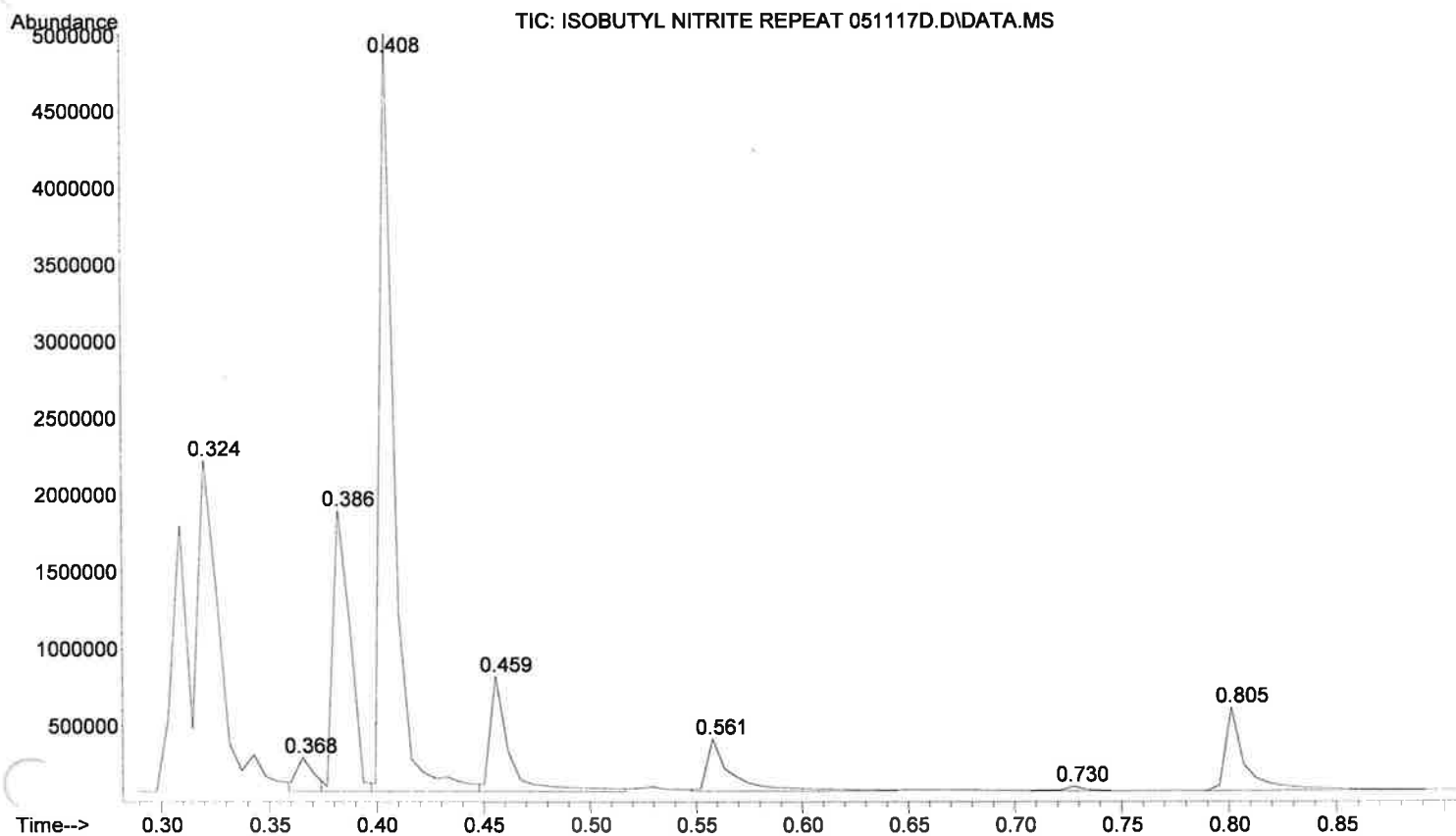
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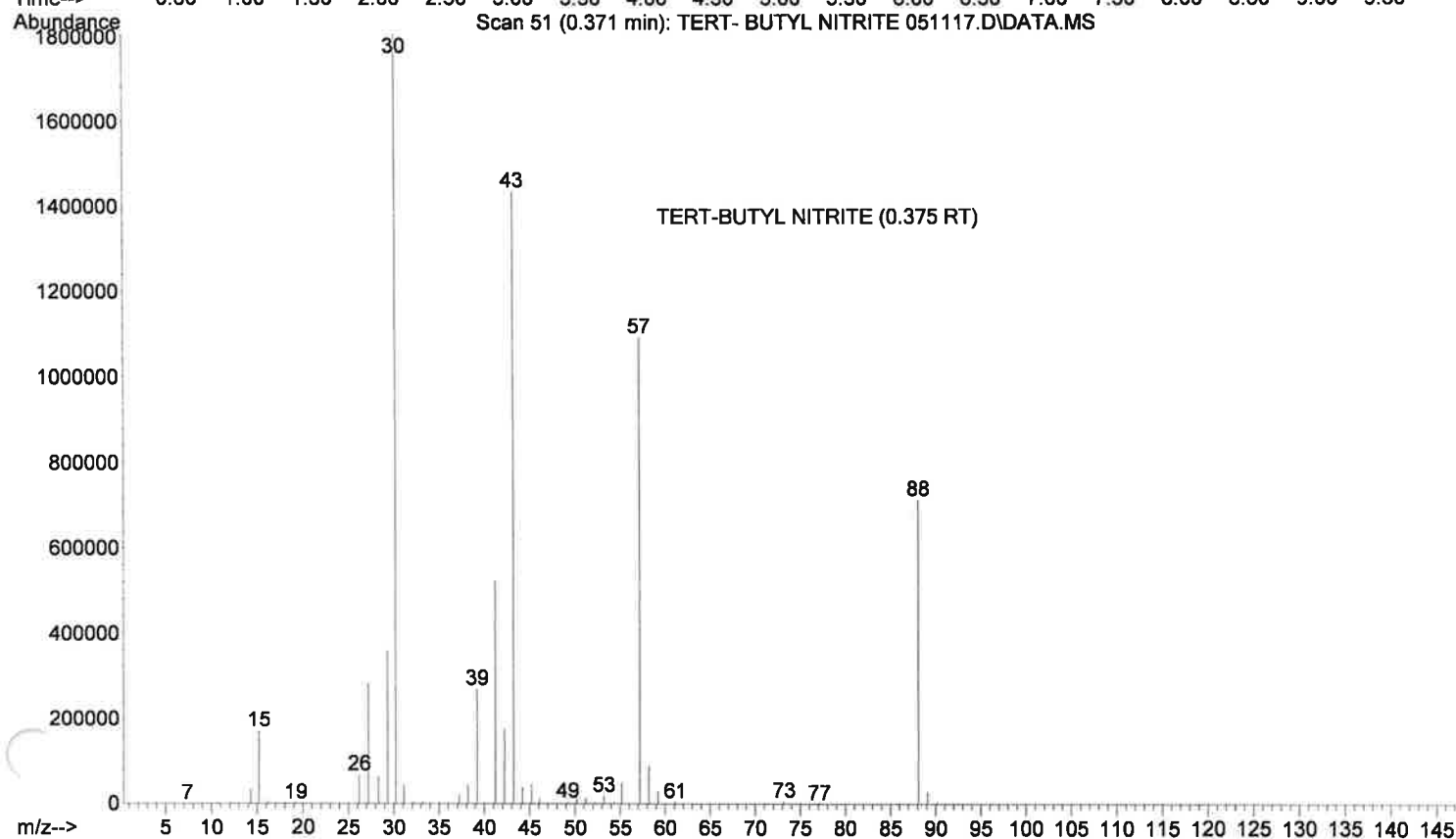
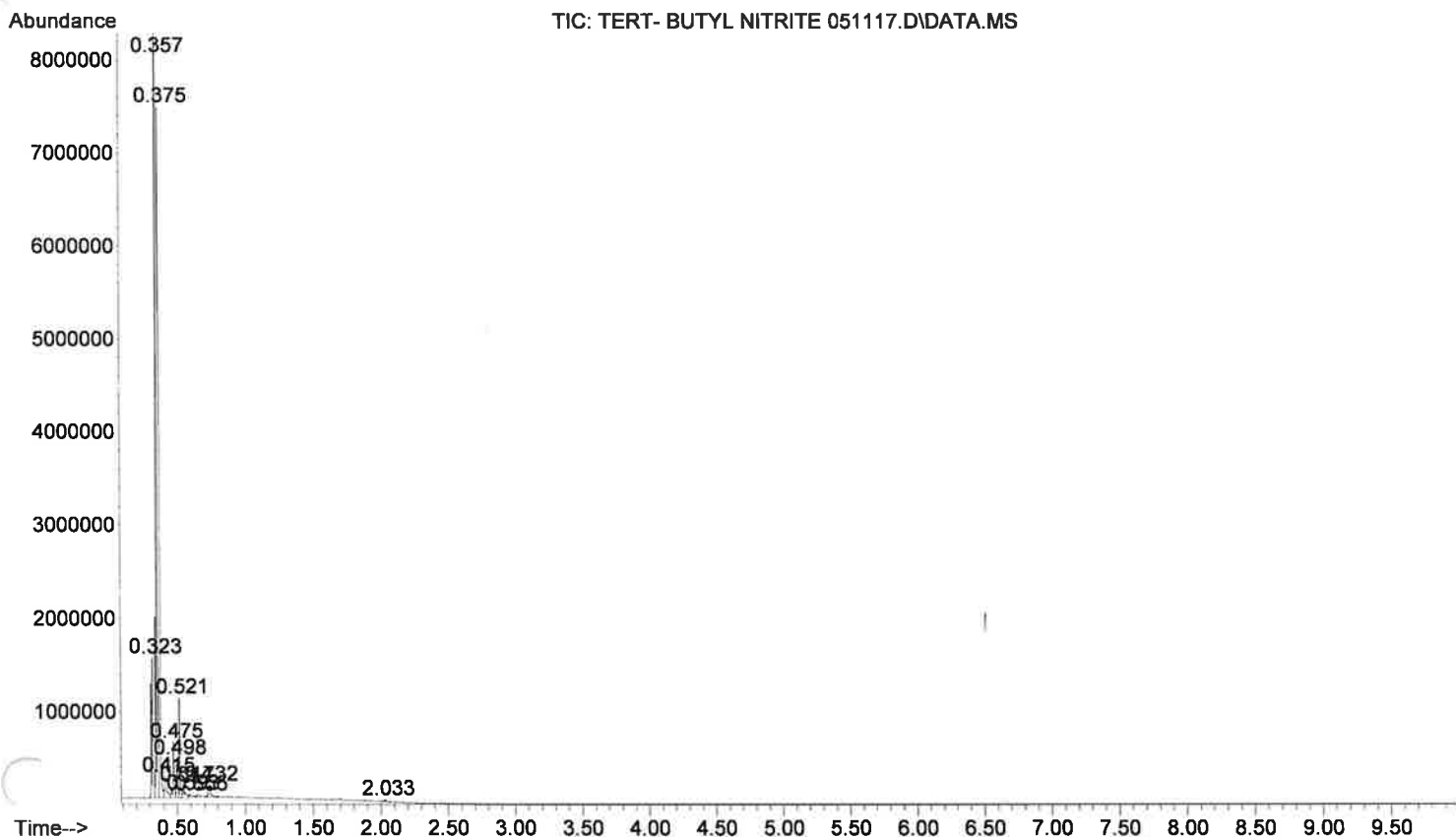
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Mi Info : HEADSPACE



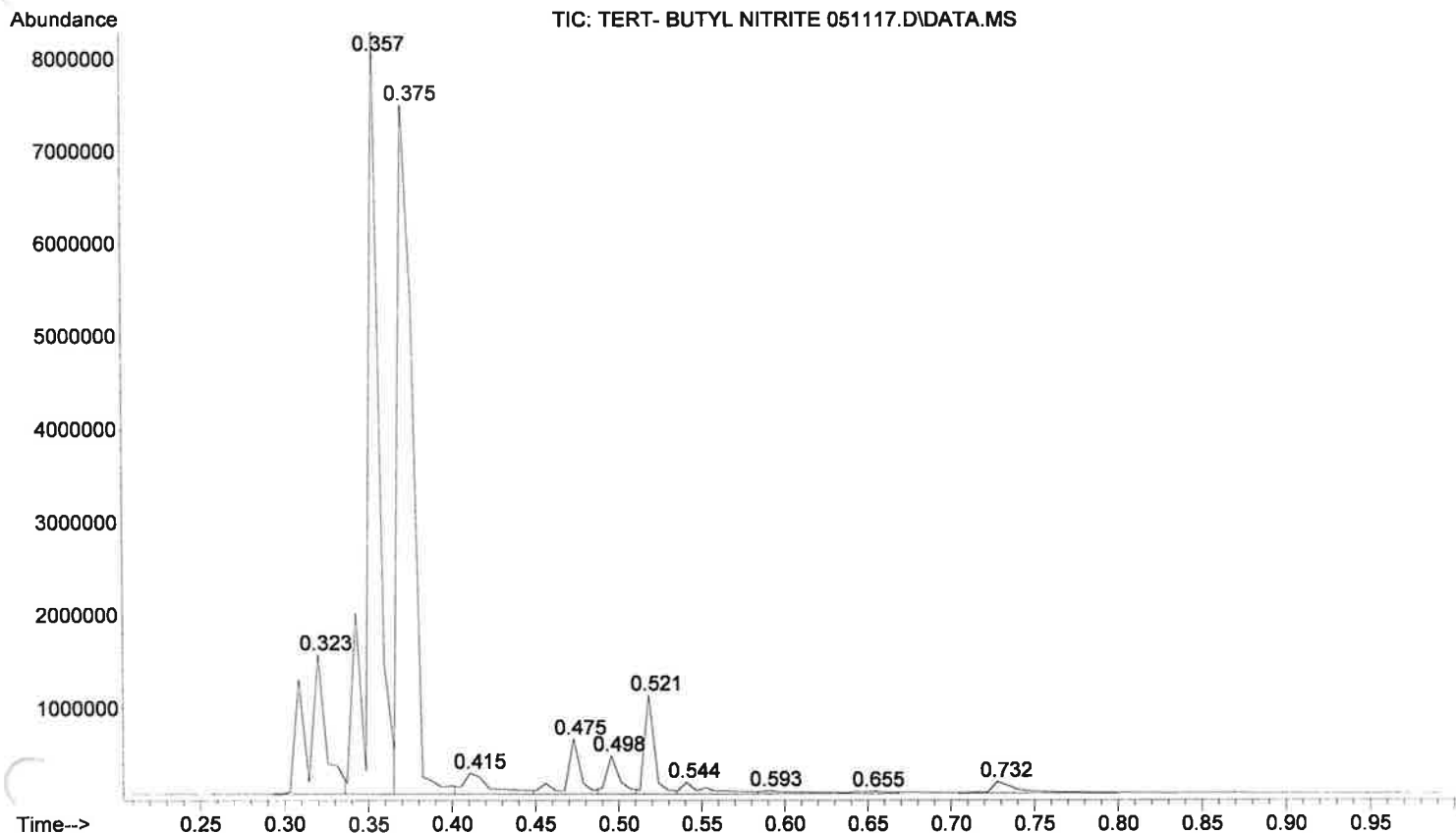
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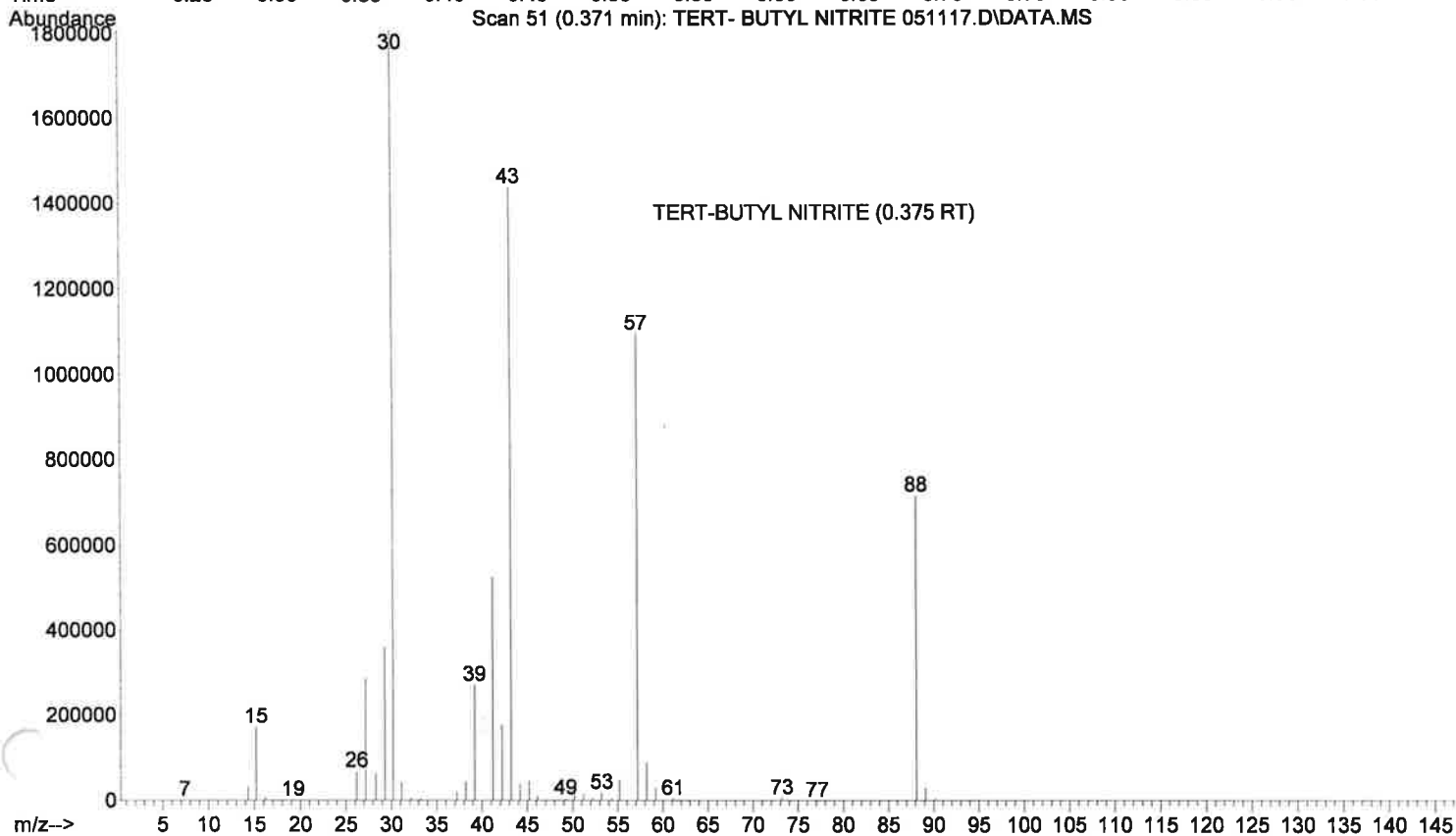
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Mi Info : HEADSPACE



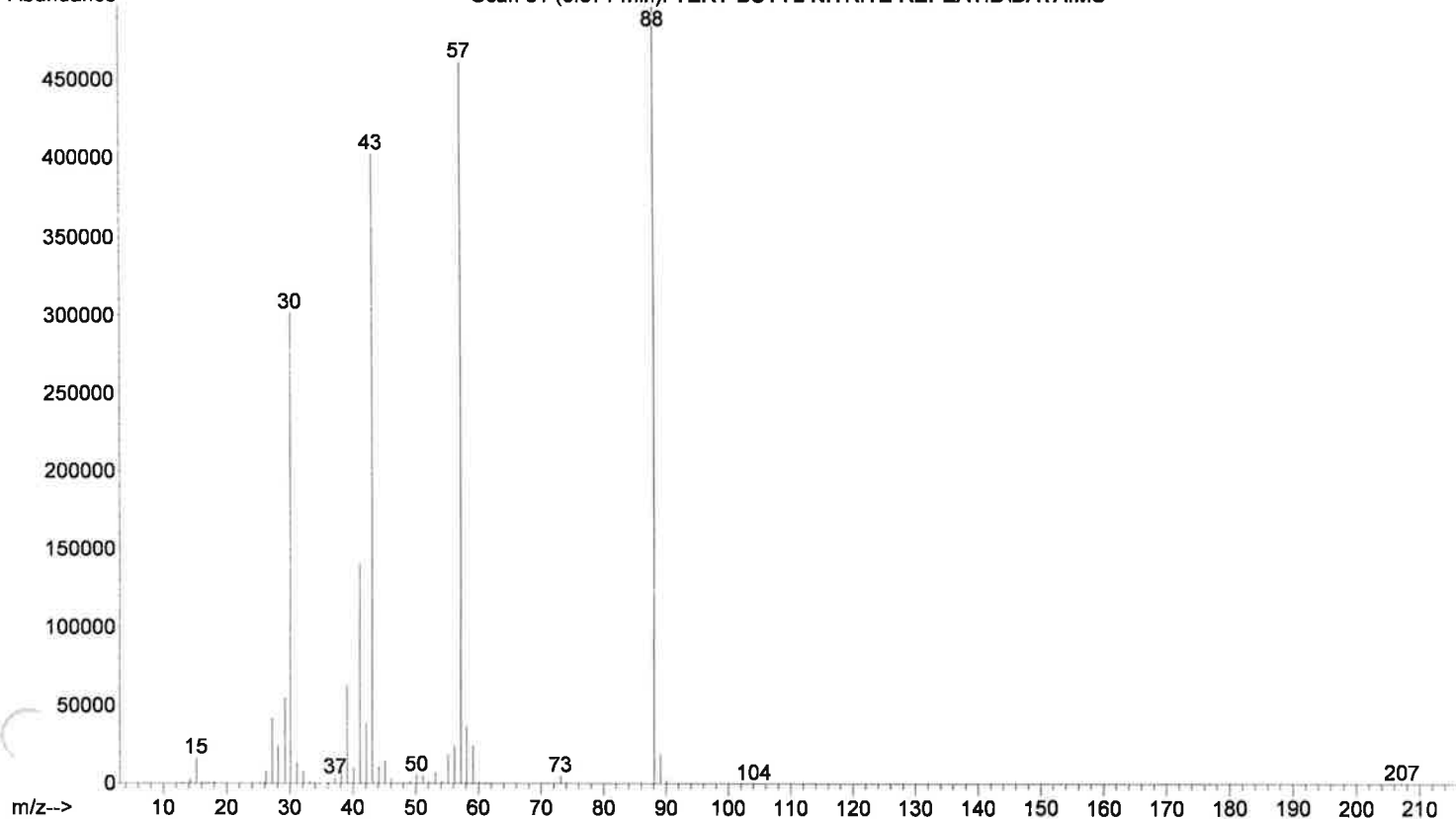
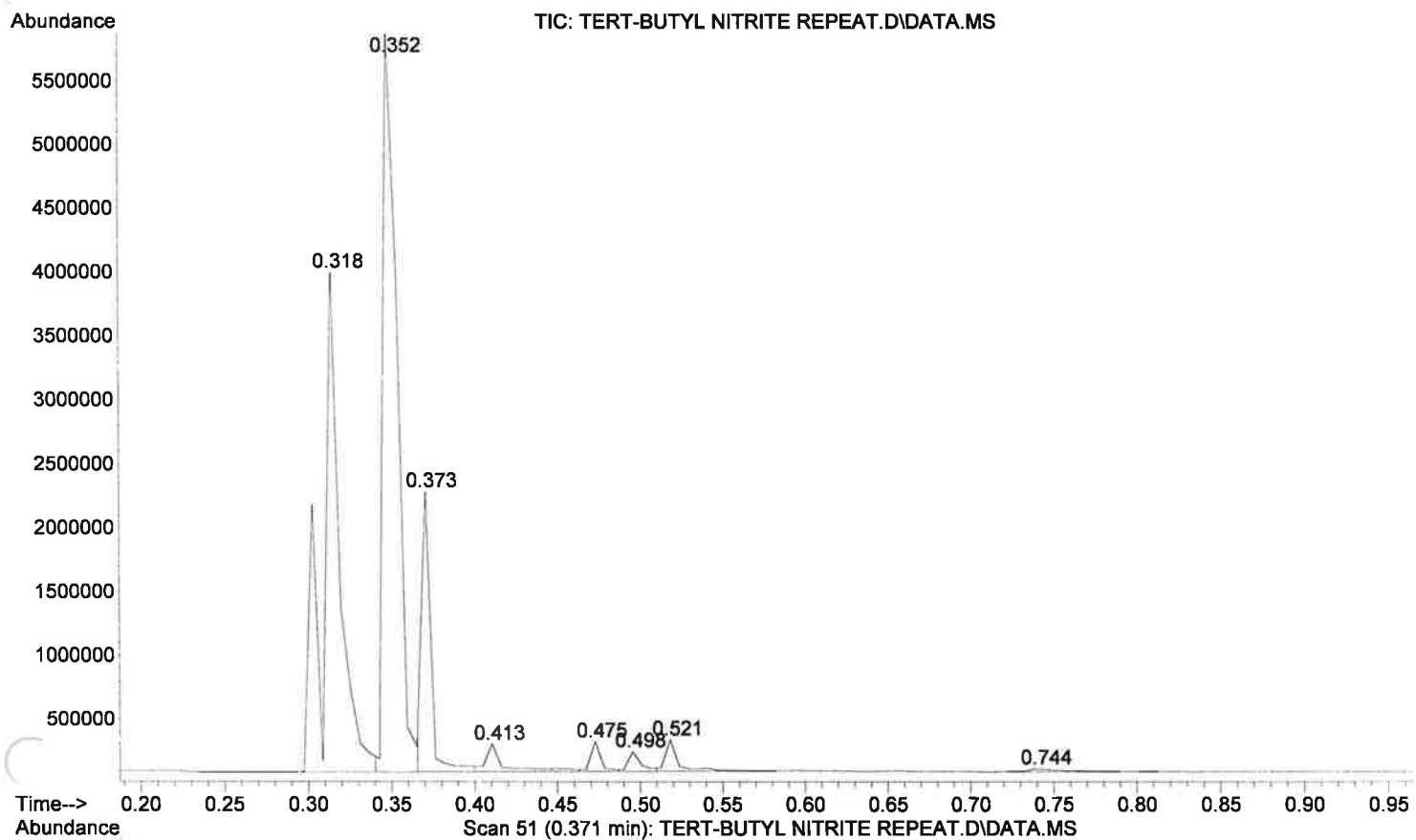
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Sample Name: TERT- BUTYL NITRITE 5/11/17
Mi Info : HEADSPACE



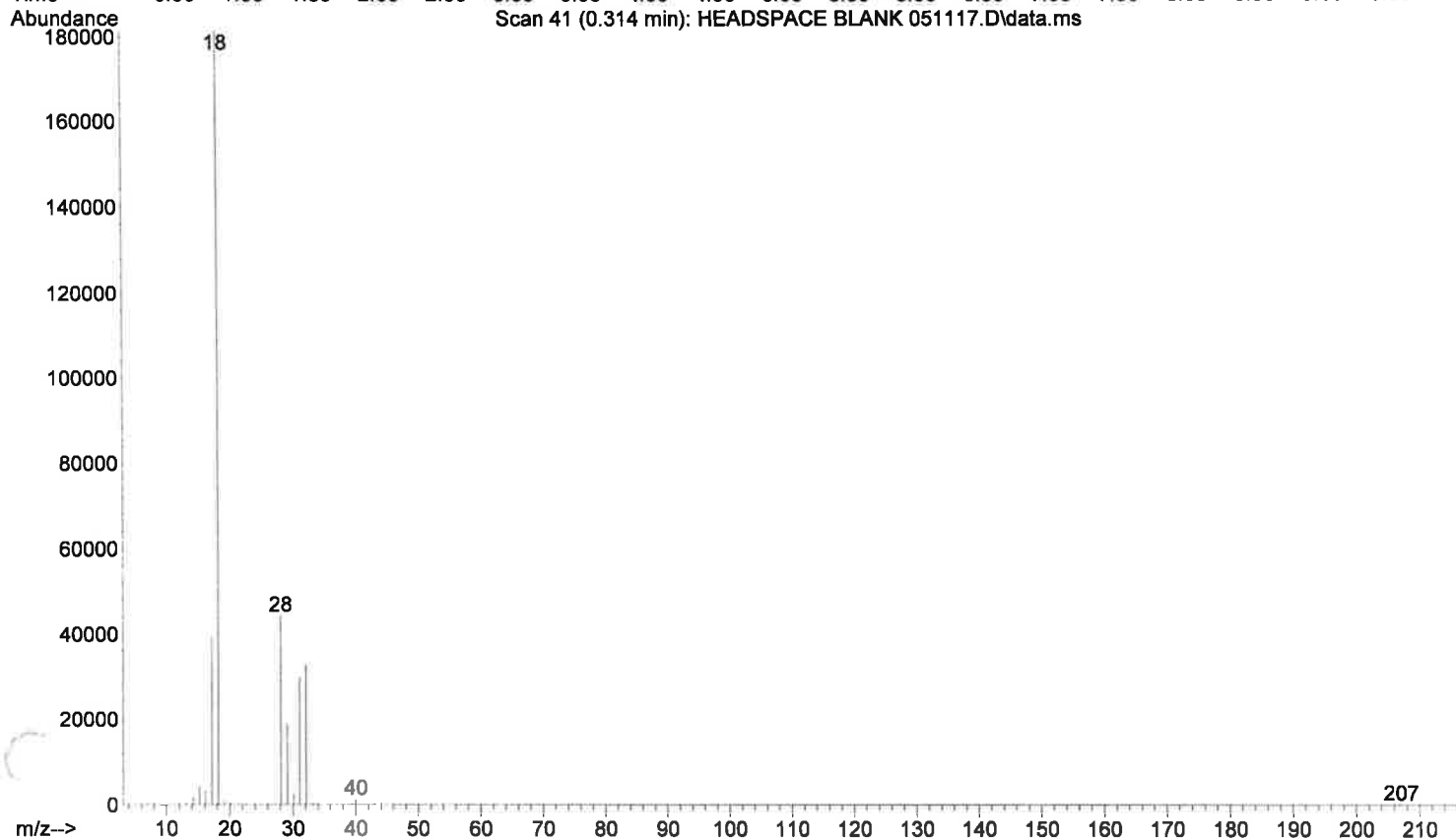
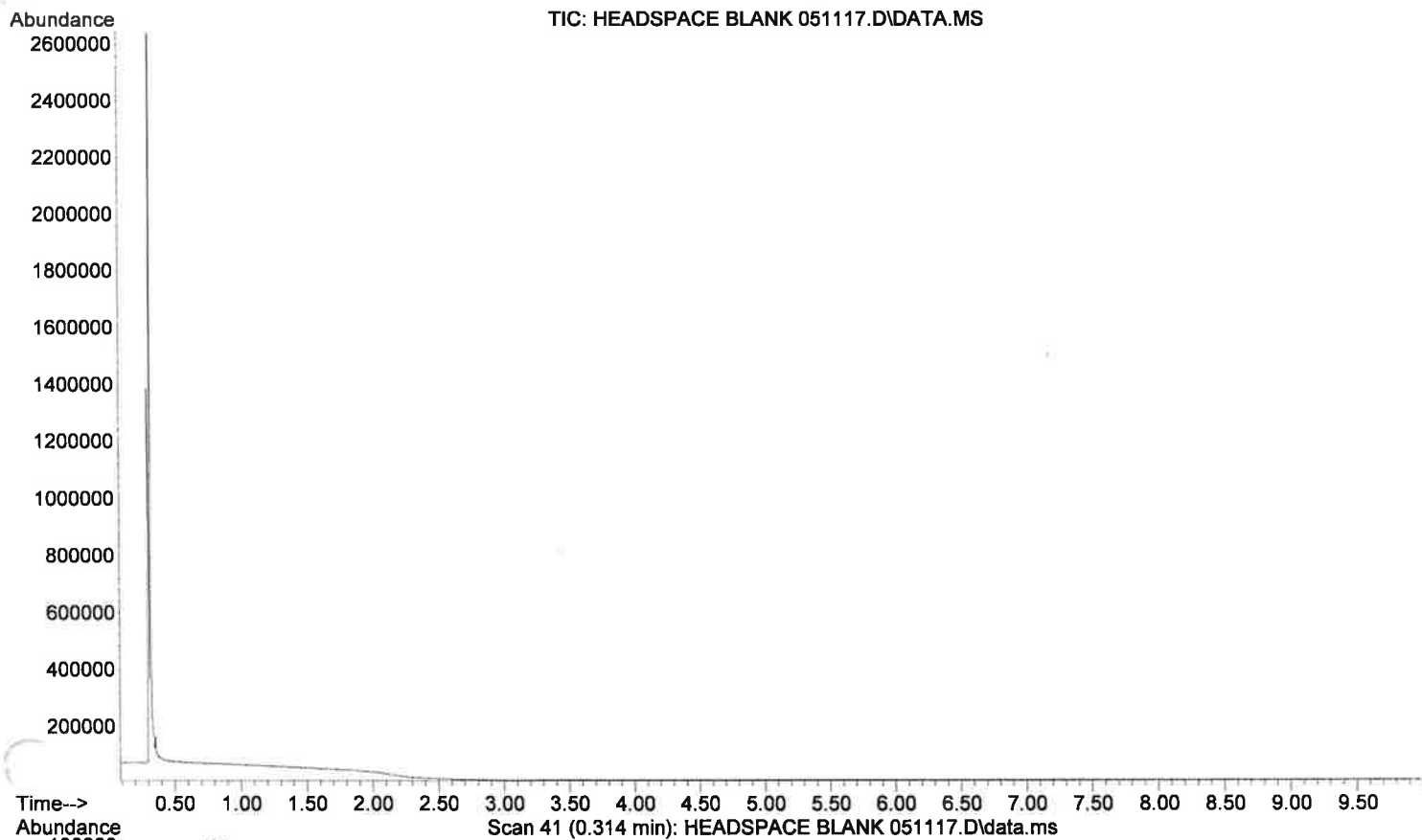
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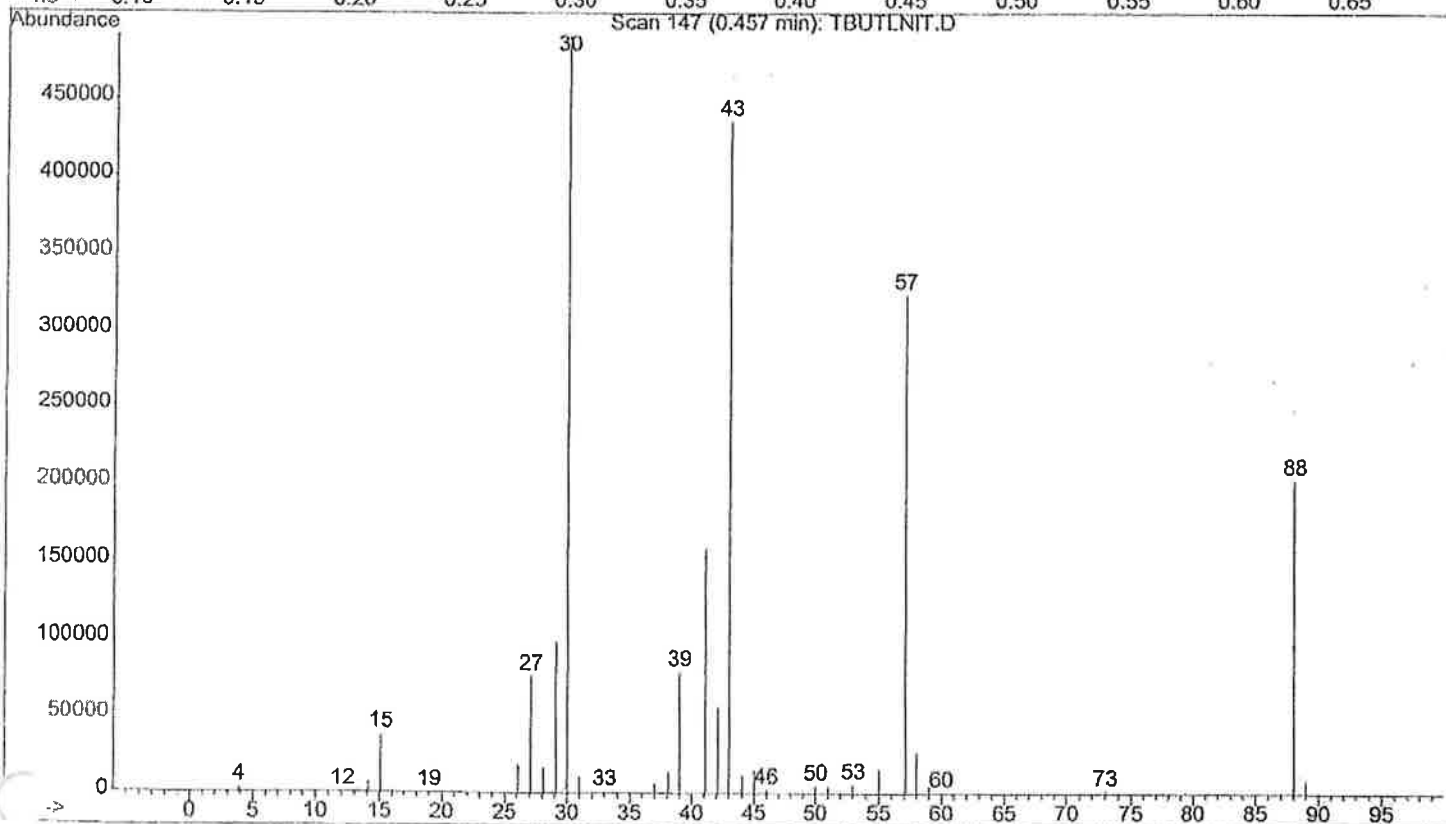
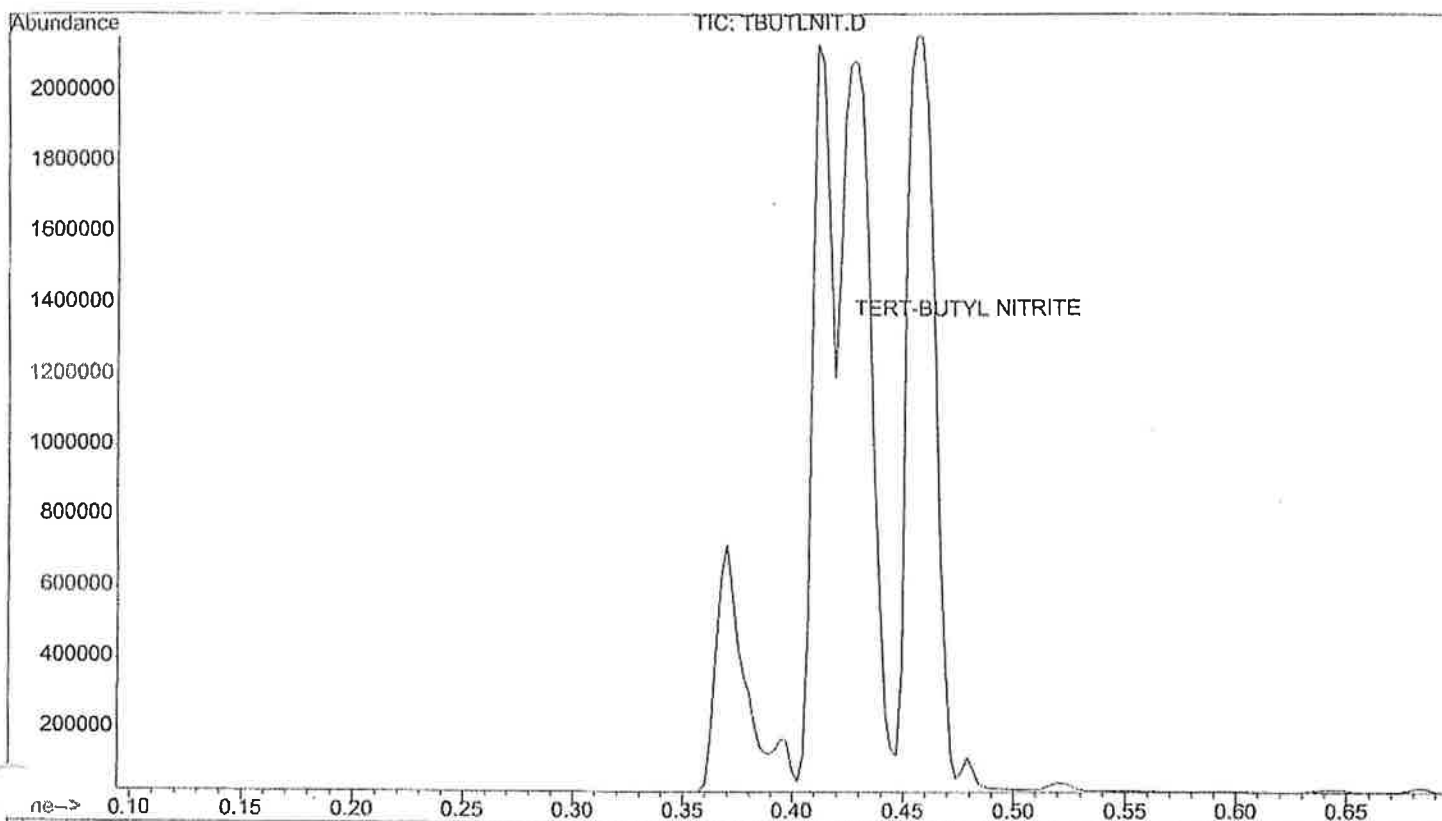
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Mi Info : HEADSPACE



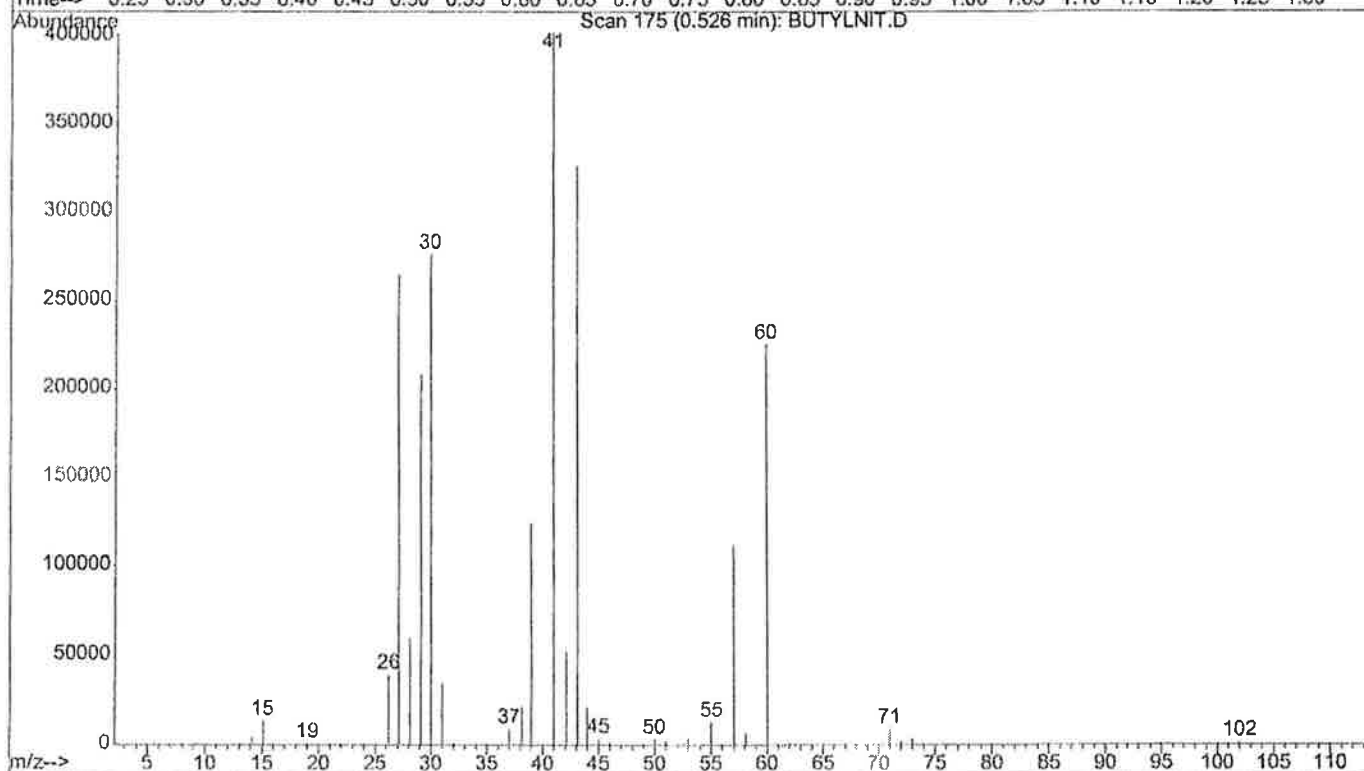
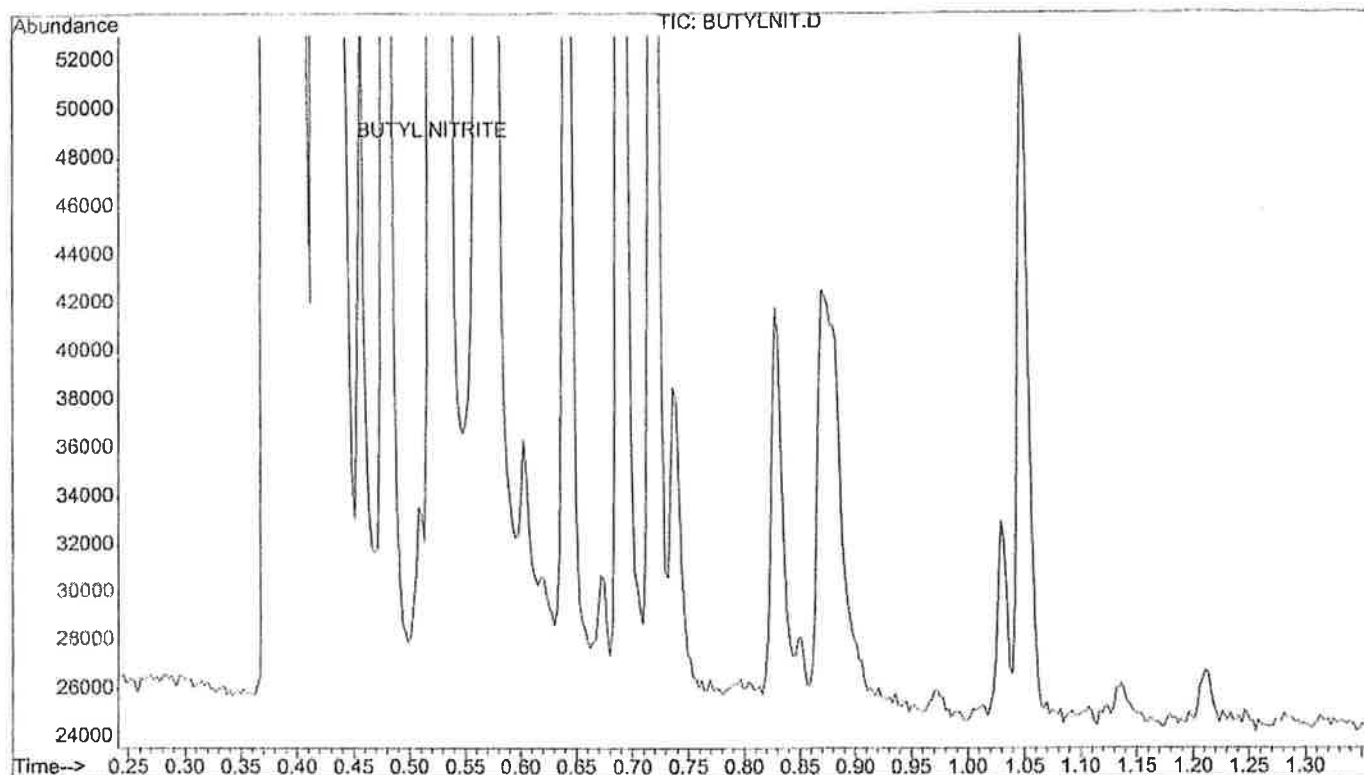
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Instrument : GC/MS Ins
Sample Name: TERT-BUTYL NITRITE (ALDRICH LOT 0108910AI)
Misc Info : VAPOR RAN NITRITE PROGRAM START @ 60C
Run Number: 1



File : C:\HPCHEM\1\DATA\TB\BUTYLNIT.D
Operator : TIFANY BARR
Acquired : 12 Feb 2003 13:46 using AcqMethod MSNIT
Instrument : GC/MS Ins
Sample Name: BUTYL NITRITE (ALDRICH LOT TU00708TS) ^{ABE}
Scan Info : VAPOR RAN NITRITE PROGRAM START @ 60C
Sample Number: 1



File : C:\HPCHEM\1\DATA\TB\IBUTLNIT.D
Operator : TIFFANY BARR
Acquired : 12 Feb 2003 13:56 using AcqMethod MSNIT
Instrument : GC/MS Ins
Sample Name: ISOBUTYL NITRITE (ALDRICH LOT 08311CI)
Scan Info : VAPOR RAN NITRITE PROGRAM START @ 60C
Vial Number: 1

