Michigan State Police Laboratory Report

STATE OF MICHIGAN
DEPARTMENT OF STATE POLICE
FORENSIC SCIENCE DIVISION

Lansing Laboratory
7320 N. Canal Rd
Lansing, MI 48913
Phone: (517) 322-6600 Fax: (517) 322-5508

SUPPLEMENTAL REPORT

Laboratory No.	:		Record No.	:	2
Delivered By	:	First Class Mail	Date Received	:	July 14, 2011
Agency	:	Michigan Department of State Police 9301 Red Arrow Highway Bridgman, MI 49106	Time Received	:	9:00 a.m.
			File Class	:	5400-2
			Date Completed	:	November 16, 2011
Incident Number	:				

Subject:

Evidence Received:

Container #1	1 - Sealed Michigan State Police Specimen kit (Tri-Tech) containing:
Item #1	1 - 10 mL grey top tube with approx. 7 mL blood
Item #2	1 - 10 mL grey top tube with approx. 6 mL blood

Results of Analysis:

Item #1:

Detected (quantified):

THC 3 ng/mL
THC-COOH 10 ng/mL

The sample was screened by immunoassay for amphetamines, barbiturates, benzodiazepines, cannabinoids, cocaine metabolites, methadone and opiates.

Cannabinoid results confirmed by GC/MS. Uncertainty of measurement for quantitative results is approximately 7.98% at the 99.7% confidence level.

At the request of the submitting agency, no further testing will be performed on the submitted specimens.

Item #2:

This item was not analyzed.

Samantha Beauchamp

Samantha Beauchamp
Forensic Scientist
Toxicology Unit

Instrumental output and data, library match for spectra data, calibrator and control data are case specific and may not be applicable in every case.

The relevant supporting data upon which the expert opinion or inference was made are available for review/inspection.

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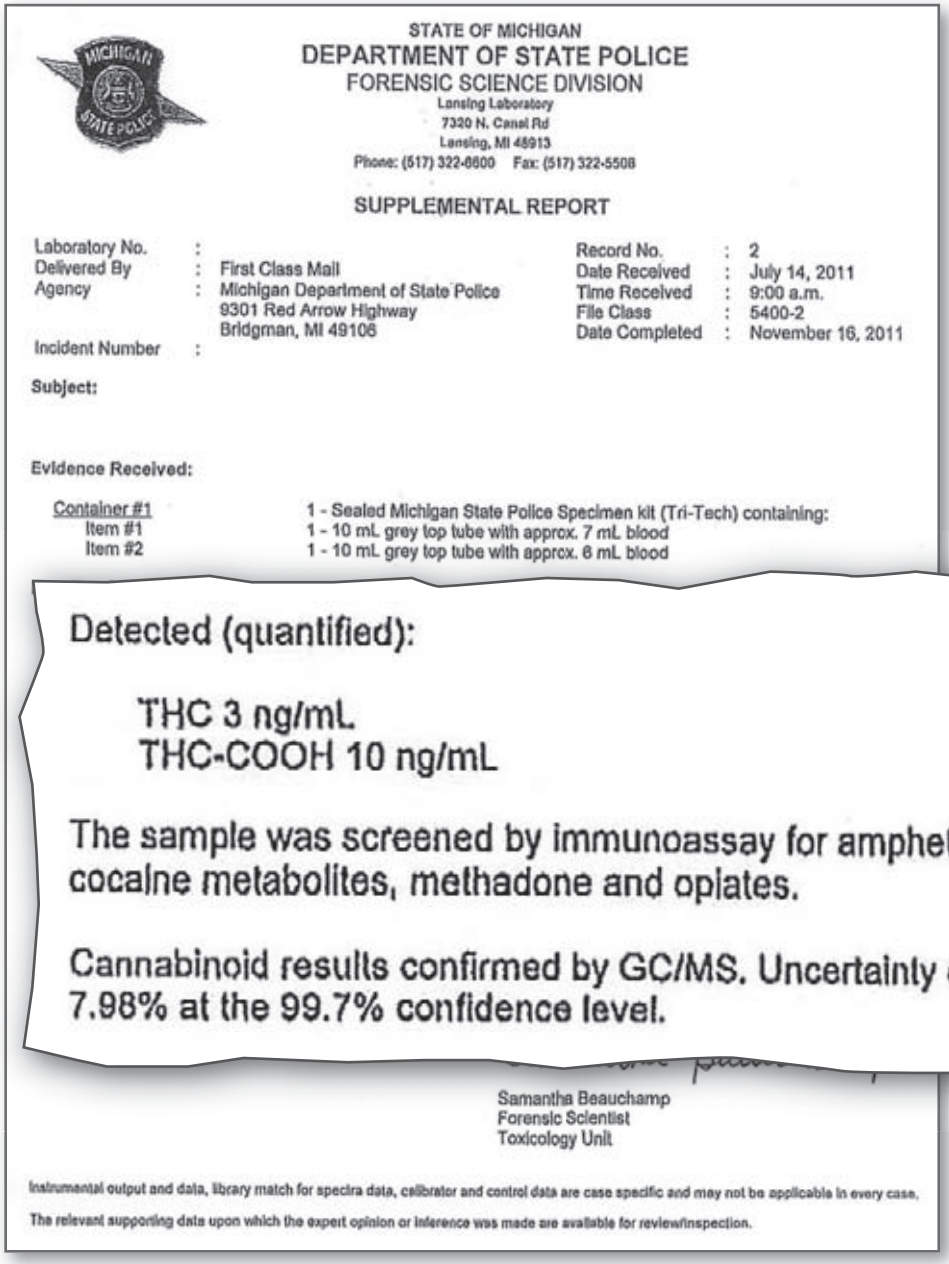

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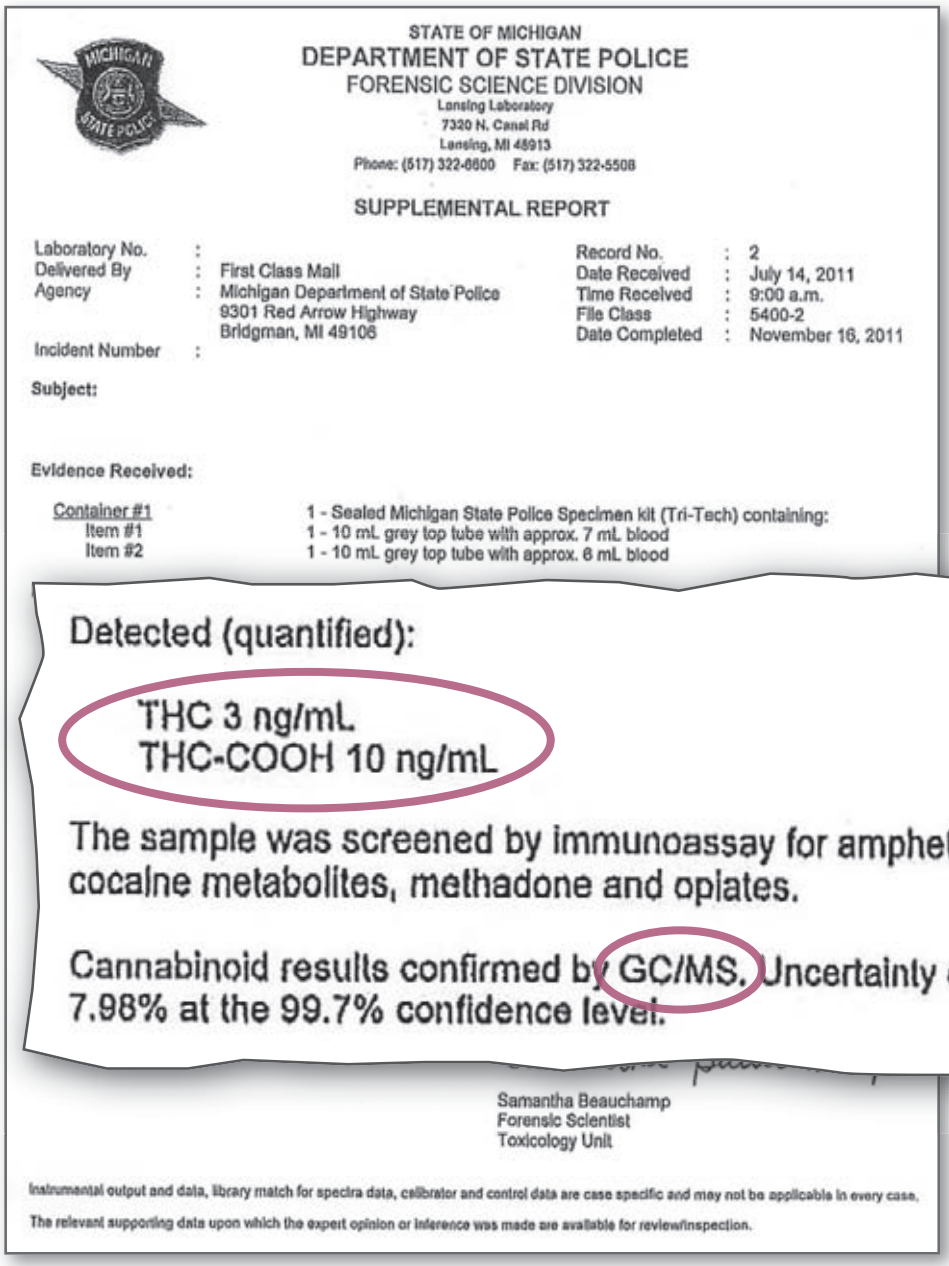

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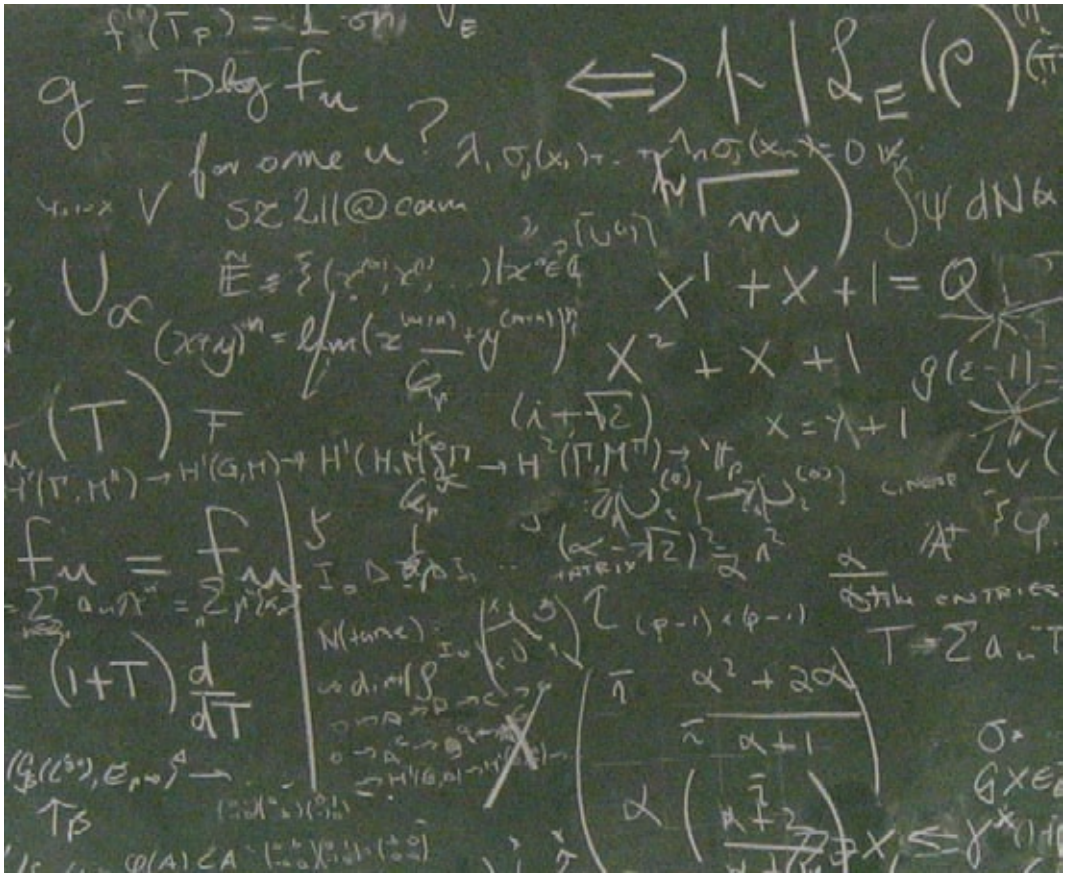
THC = Tetrahydrocannabinol
main psychoactive constituent of the cannabis plant

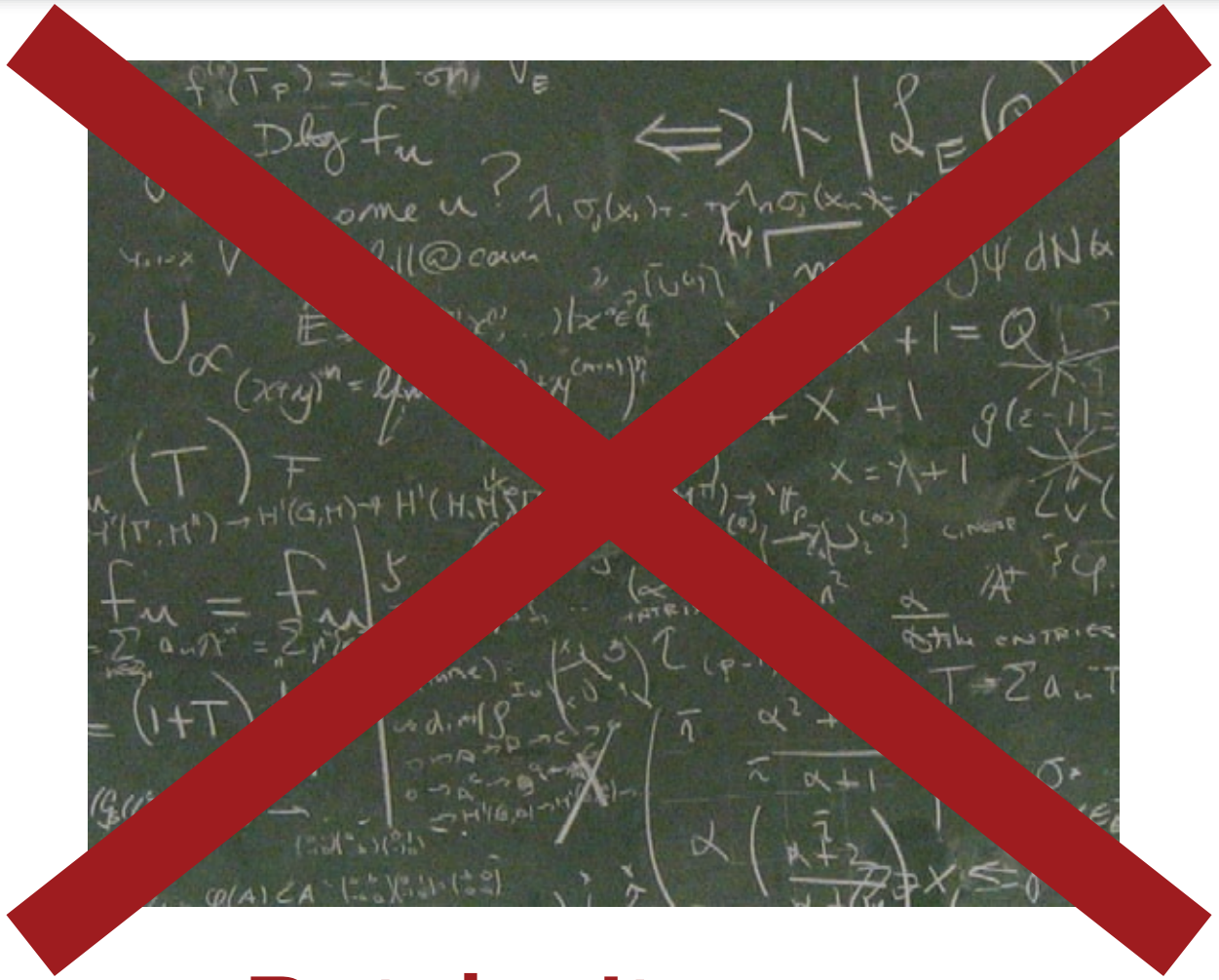
THC-COOH = Carboxy-THC
metabolite of THC formed after cannabis consumption

GC/MS = Gas Chromatography / Mass Spectrometry
method to identify the presence of a substance

SCIENCE


$g = D\log f_u$
for some u ?
$\Leftrightarrow$
$X^2 + X + 1$
$X^1 + X + 1 = 0$
$f_u = f_{uu}$
$(1+T)\frac{d}{dT}$
$T \to \Sigma a_i T$

SCIENCE



But don’t worry:

No equations needed

Gas Chromatography

A gas chromatograph is an analytical instrument used for separation and identification of different components of a mixture of substances.

Substances are separated by the time it takes them to pass through a thin, long tube (column), flushed with a carrier gas. Each substance will extract at a characteristic time (retention time), that can be used to identify the substance.

A detector at the end of the column converts the presence of a substance into an electrical signal.

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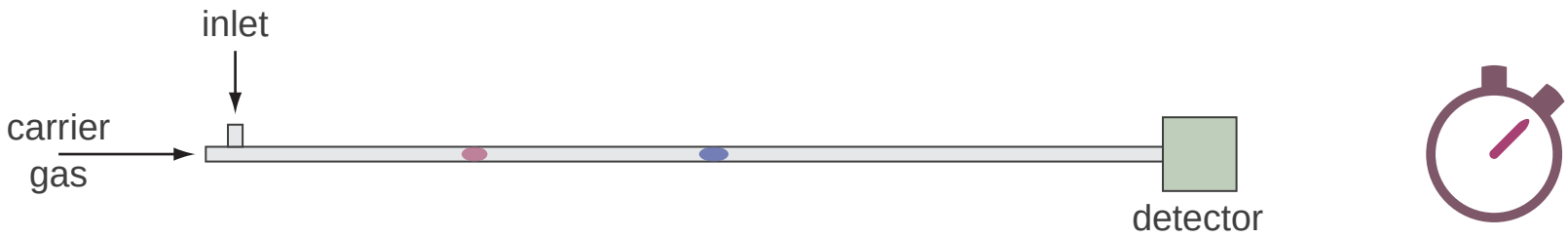

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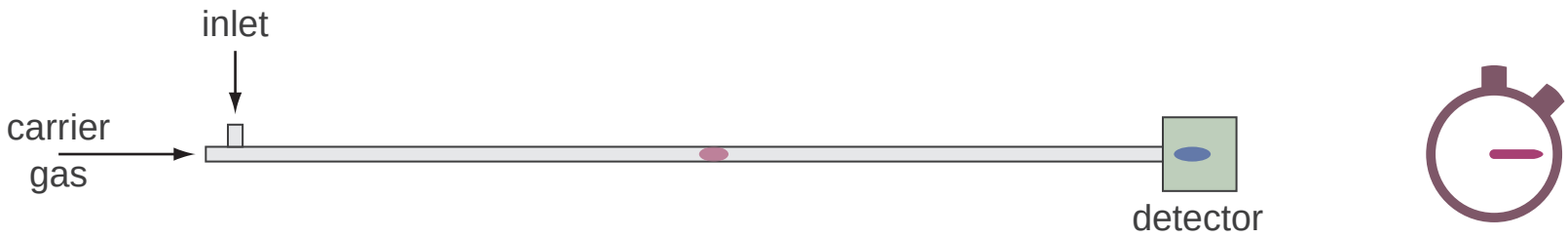

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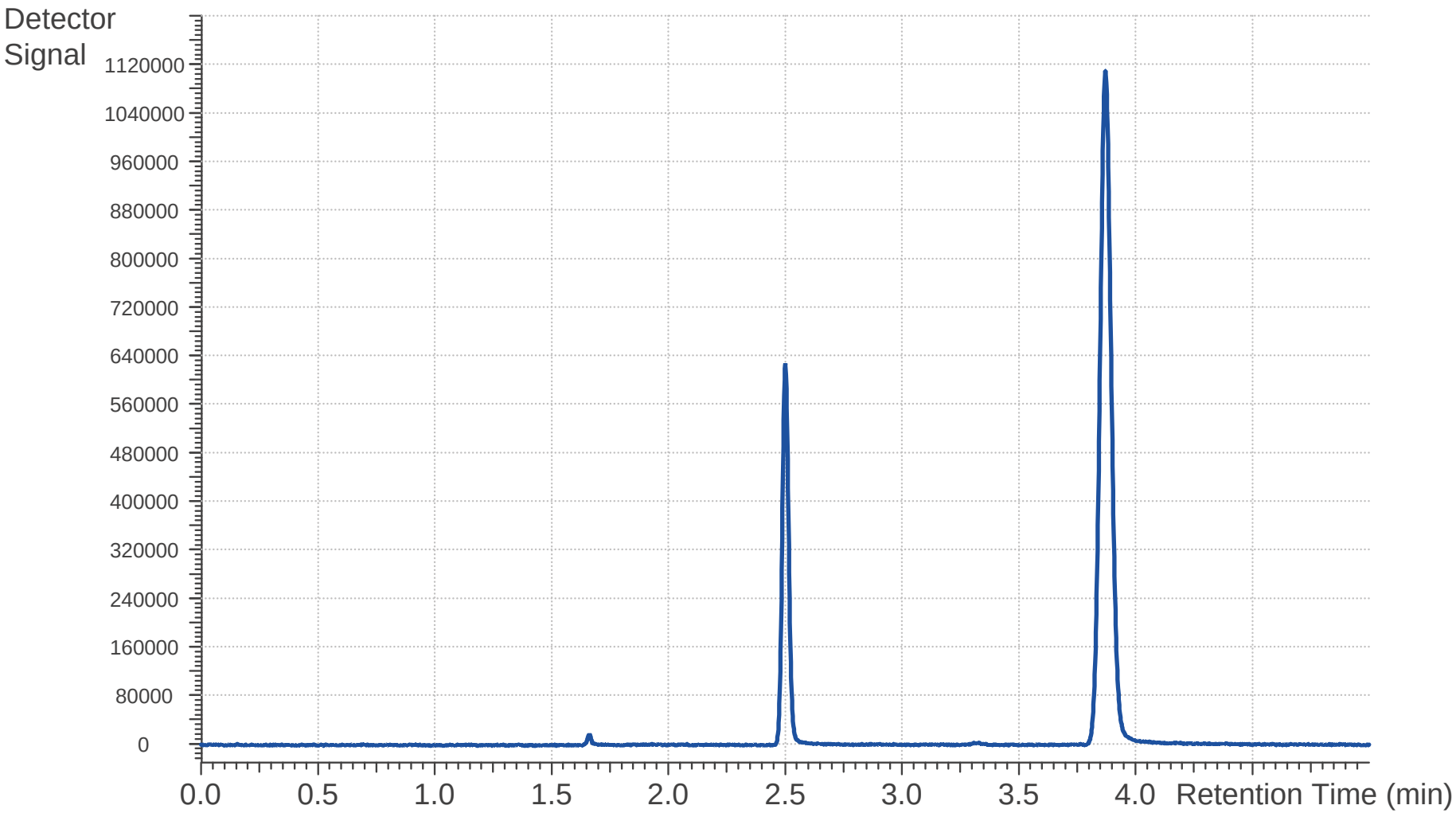
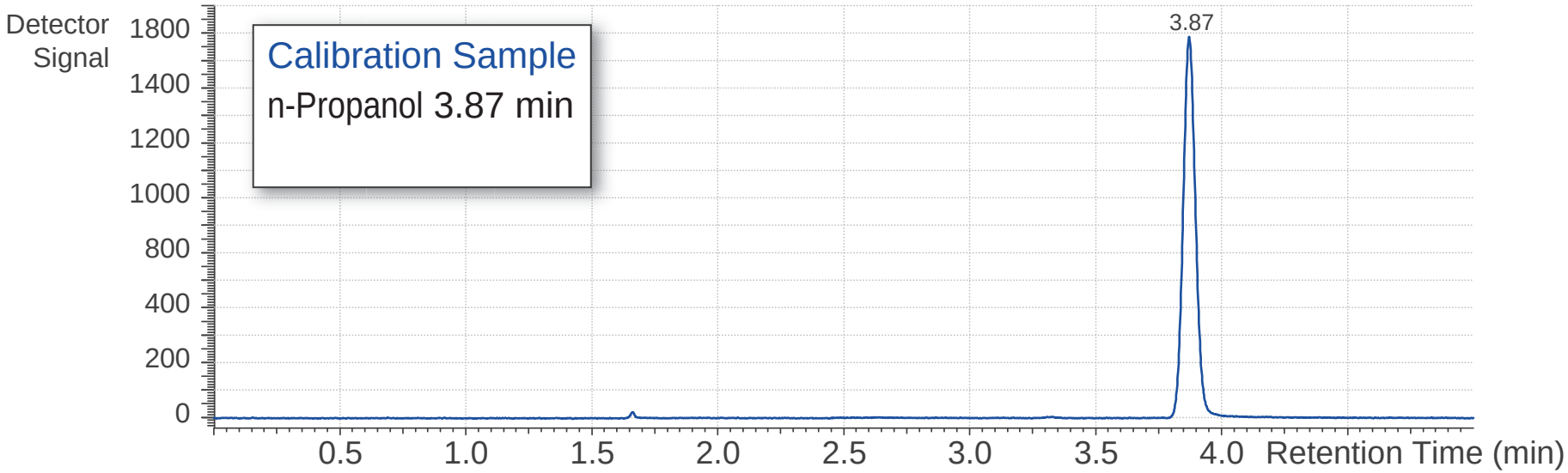
Gas Chromatography

A chromatogram is a graph with the detector response as a function of time. The signal is higher for larger amounts of a substance.



Gas Chromatography – Calibration

To determine the characteristic retention times samples with known composition are analyzed.



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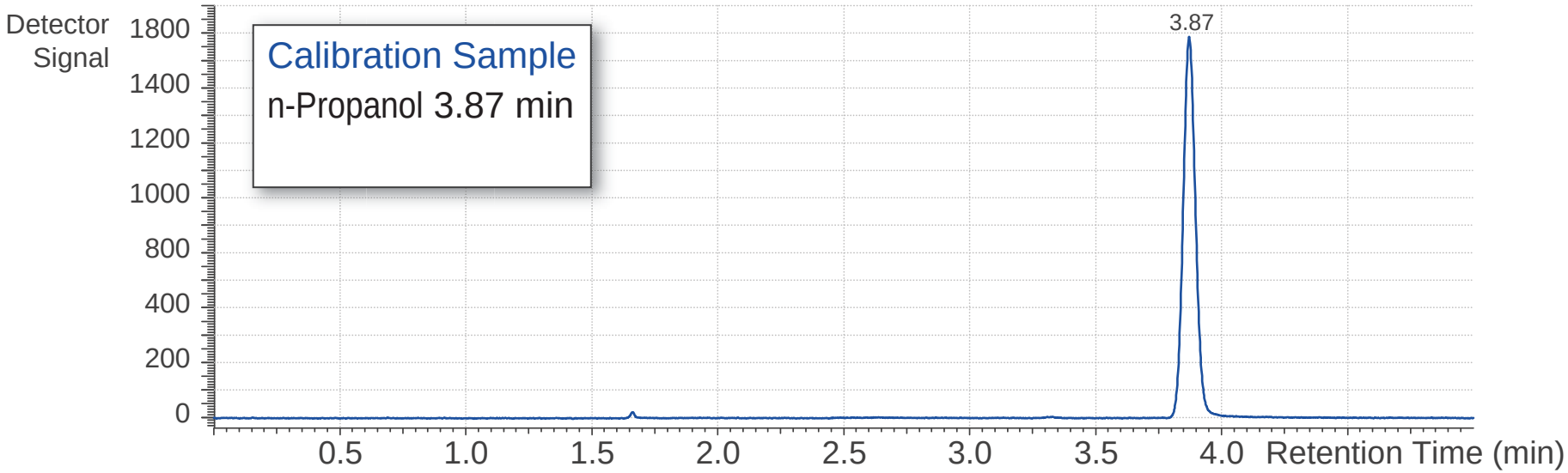

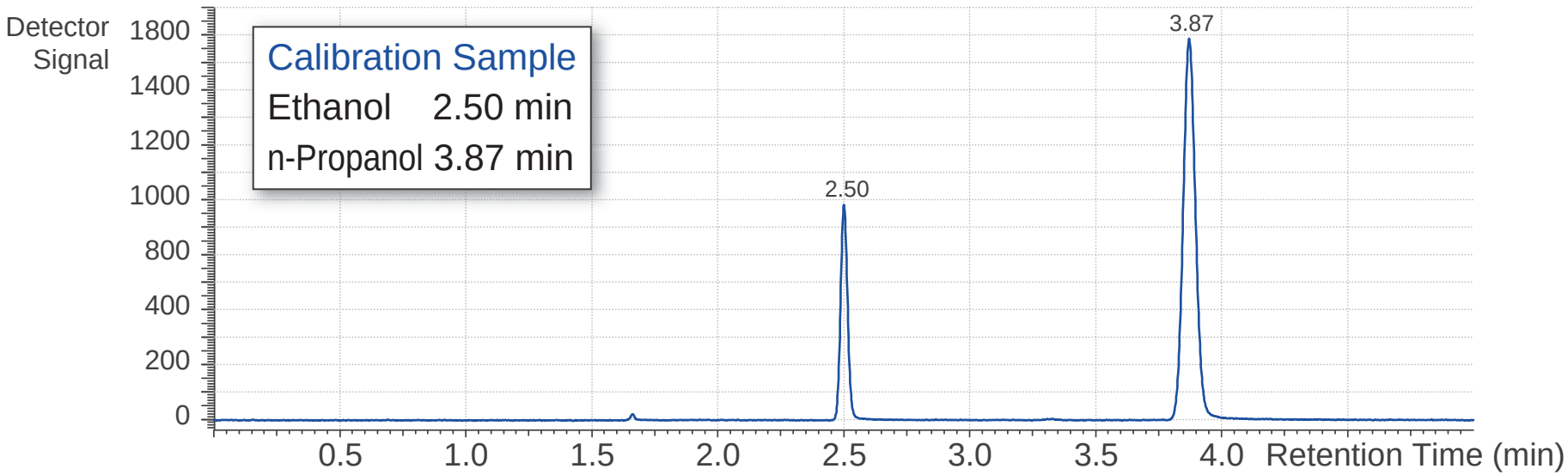

Gas Chromatography – Calibration

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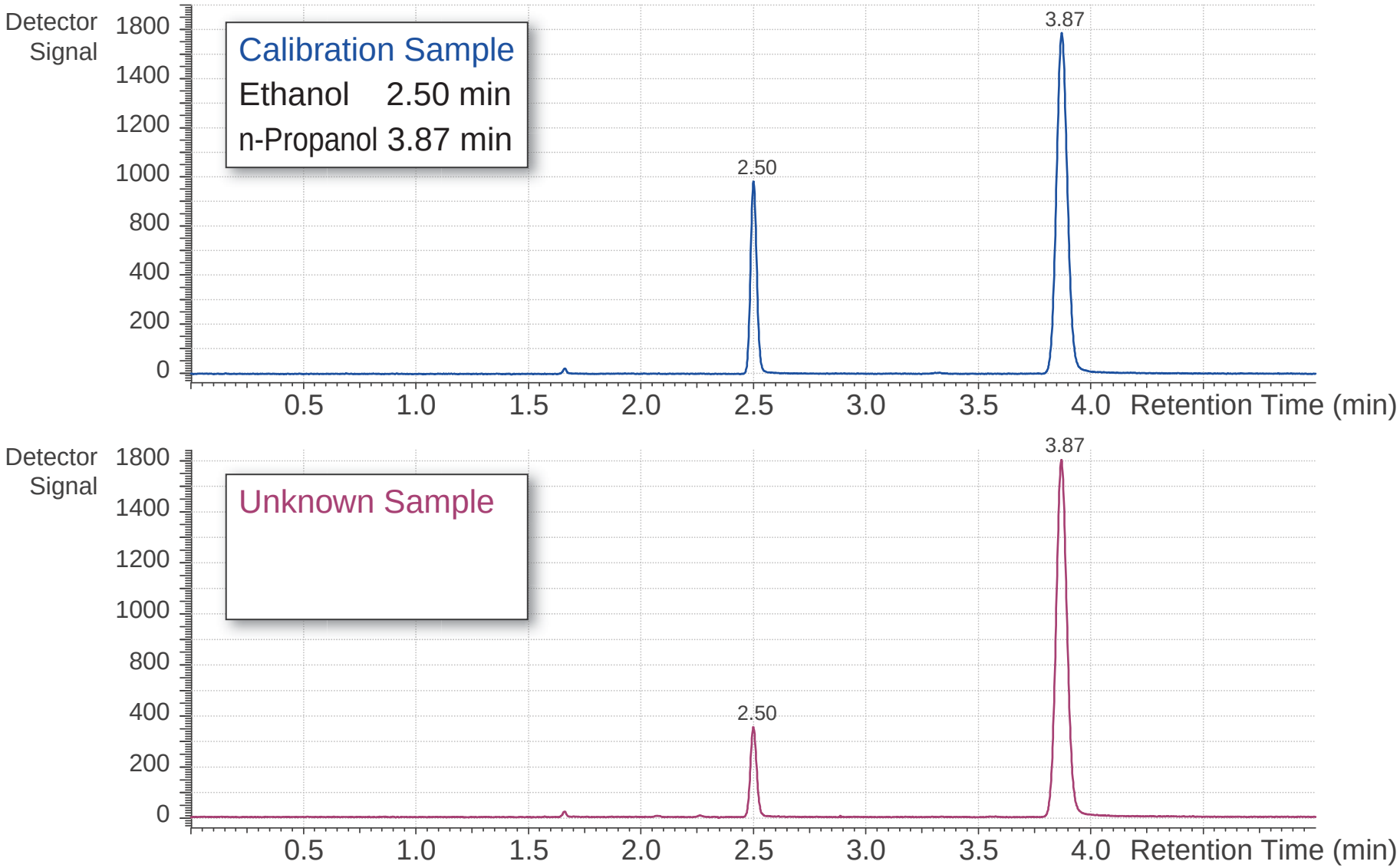
An unknown sample can then be compared with the calibration sample.



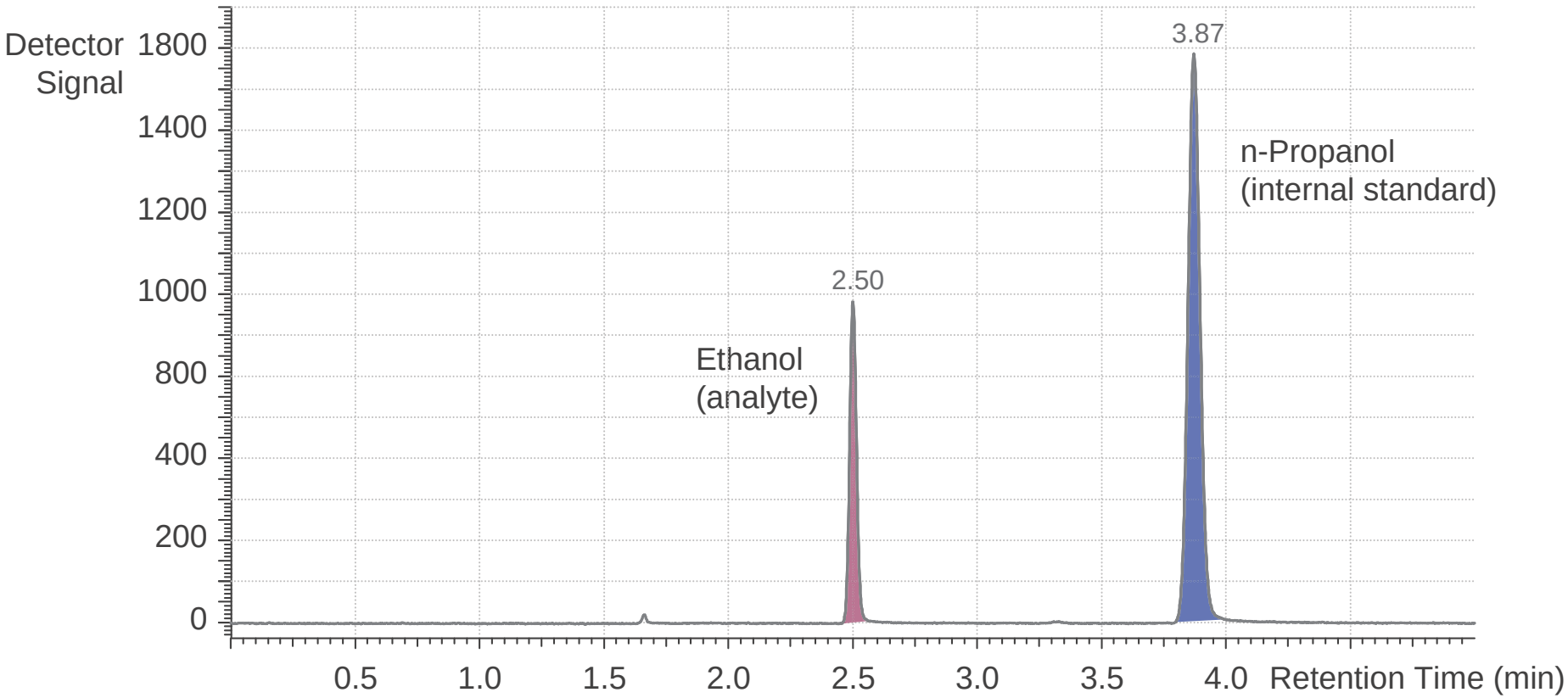
Gas Chromatography – Quantitative Analysis

To determine the concentration of a substance of interest (analyte) a measurement relative to an internal standard with similar chemical structure is performed.

The area underneath the detector signal (peak) is proportional to the amount of the substance.

The analyte concentration is measured as the area ratio between the **analyte area** and the **internal standard area**.

A calibration curve is then used to translate the the area ratio into a concentration value.



Gas Chromatography

Gas Chromatography is used for the analysis of alcohol (ethanol) in blood.

Typical unit of interest for ethanol concentration in blood: 0.01 g / 100 ml

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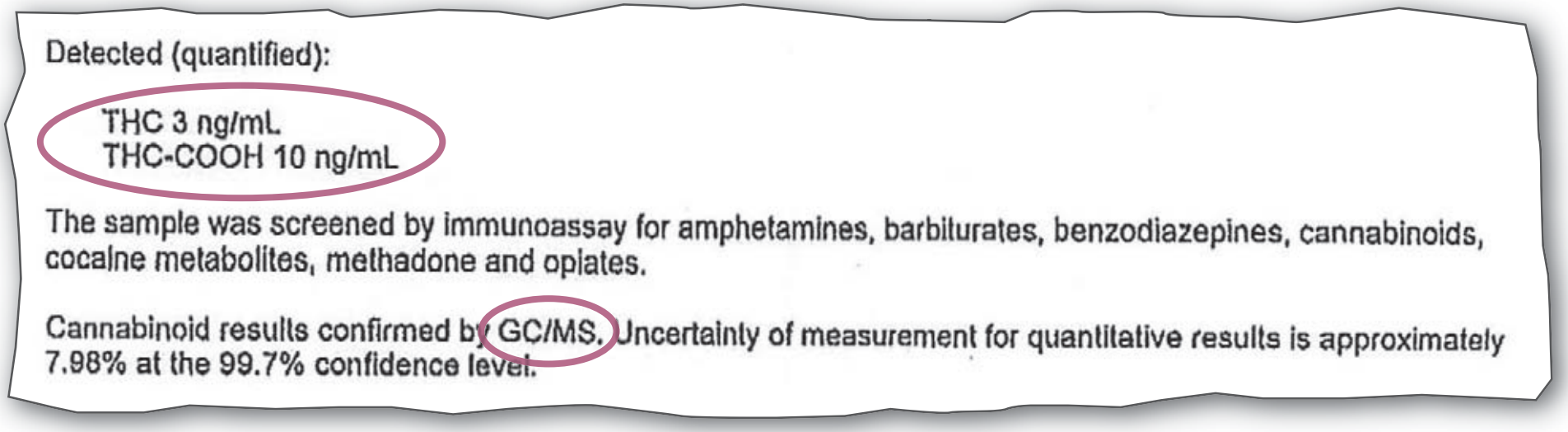

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For the analysis of THC in blood the combination of Gas Chromatography and Mass Spectrometry (GC/MS) is used.

Typical unit of interest for THC concentration in blood: 1 ng / ml

This concentration is 100,000 times smaller.

What is 0.01 g / 100 ml ?

Typical unit of interest for ethanol concentration in blood: 0.01 g / 100 ml

Half a shot glass of pure alcohol mixed into a bath tub full of water

What is 1 ng / ml ?

Typical unit of interest for THC concentration
in blood: 1 ng / ml

Half a tea spoon of sugar mixed
into a olympic-size swimming pool

What is 1 atom out of 100 quadrillion atoms?

At the National Superconducting Cyclotron Laboratory at Michigan State University it is possible to detect a single atom out of 100 quadrillion atoms (a number with 17 zeros).

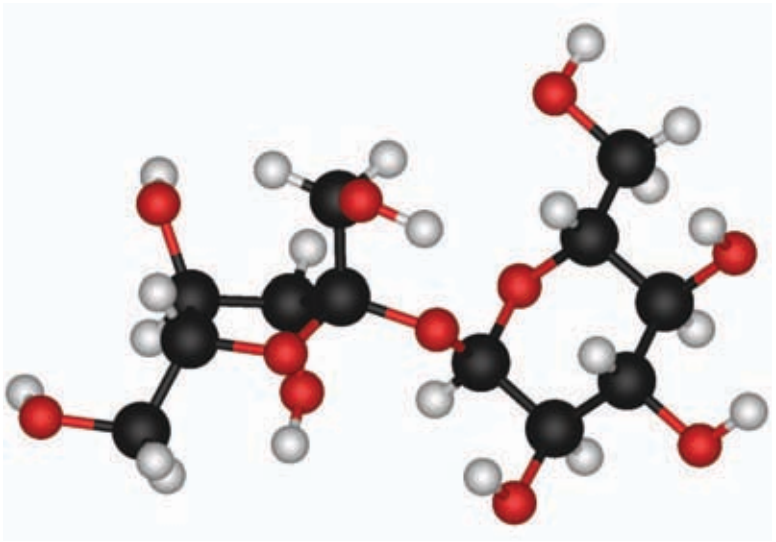
Half a tea spoon of sugar mixed into Lake Erie

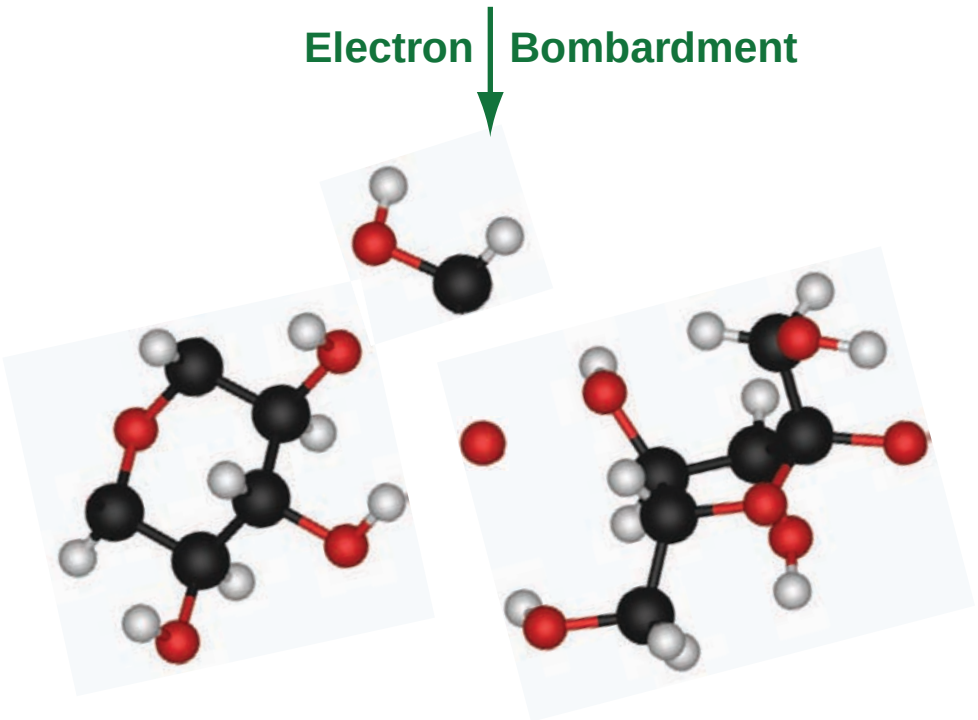
Gas Chromatography / Mass Spectrometry (GC/MS)

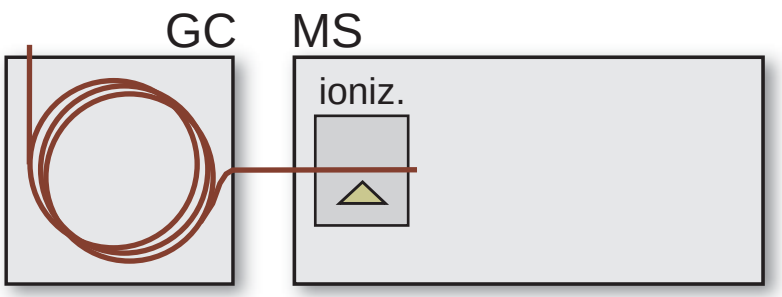
Gas Chromatography / Mass Spectrometry is an analytic method that combines the features of gas chromatography with mass spectrometry.

After a substance leaves the gas chromatograph column, it is bombarded with an electron beam in an ionizer. The molecules of the substance will break into smaller pieces.

The weight (or masses) of these smaller pieces can be used to identify the substance as molecules break into fragments of a characteristic mass pattern.







Gas Chromatography / Mass Spectrometry

Mass Spectrometry is like looking at the pieces of broken china and figuring out what it was.

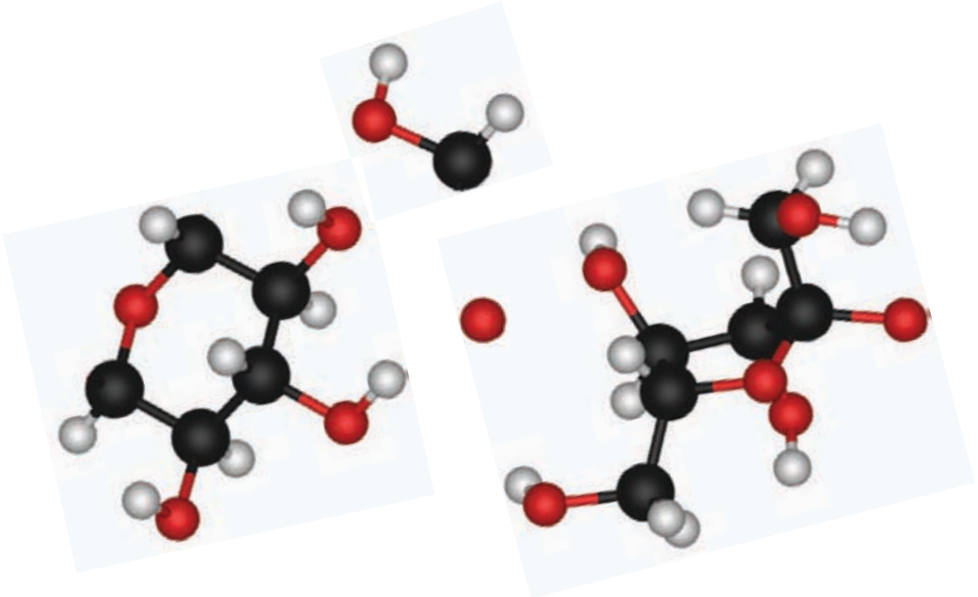
Gas Chromatography / Mass Spectrometry

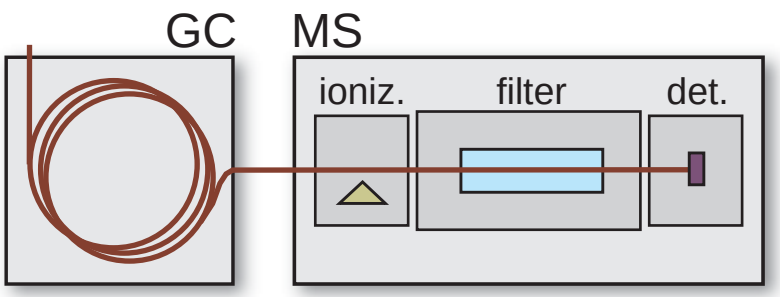
Mass Spectrometry

The molecule fragments are analyzed by an adjustable scanning mass filter.

Only fragments with a specific mass can pass through the filter at a given time. But the selected mass can be changed very quickly many times per second (e.g. full scan over a mass range 40 to 500 within 0.25 seconds).

Molecule fragments are detected by a detector at the end of the filter.





The weight of molecules is measured in atomic mass units (amu).

Typical masses (in amu) for single atoms are:

Hydrogen:	1
Carbon:	12
Nitrogen:	14
Oxygen:	16

Gas Chromatography / Mass Spectrometry

Gas Chromatography (GC)

Selection by retention time through GC column.

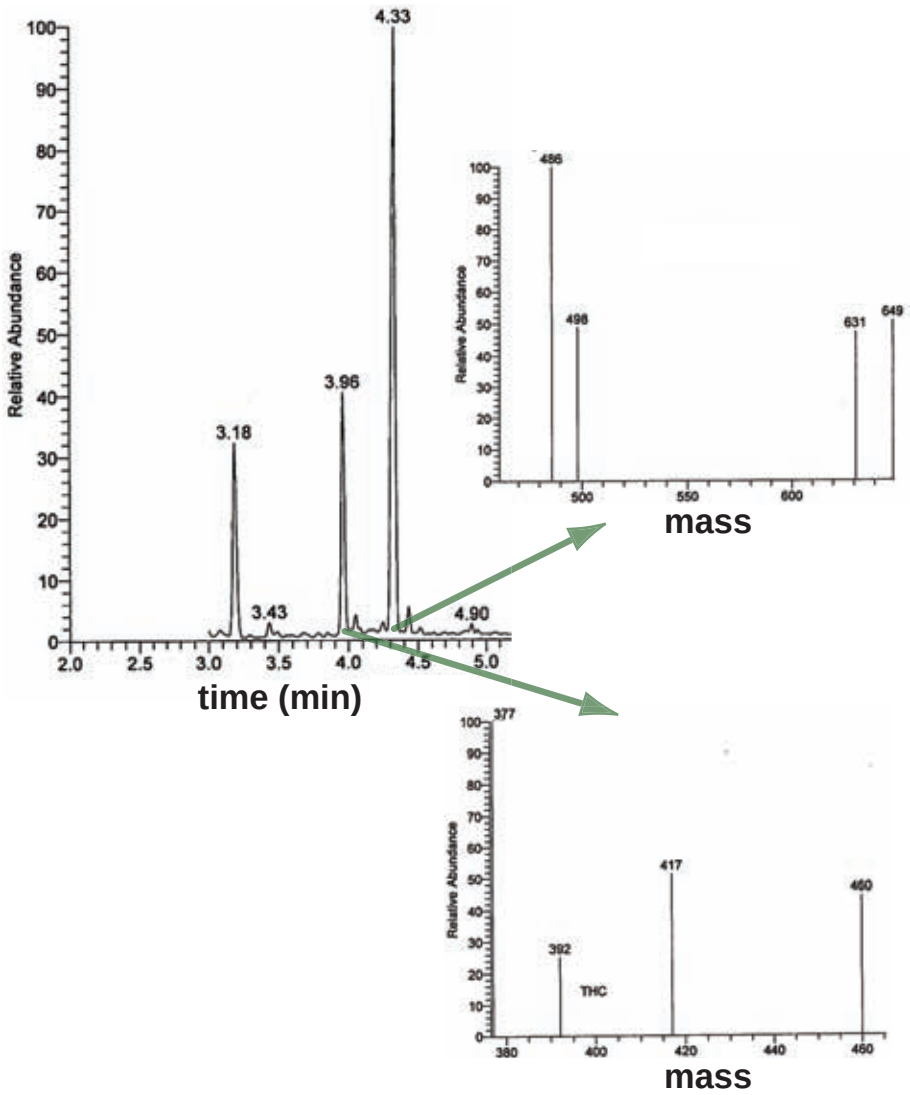
Mass Spectrometry (MS)

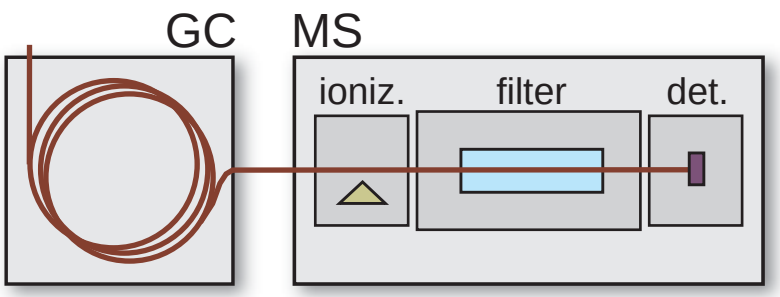
Selection by mass through adjustable scanning mass filter.

GC/MS

The measured data is the detector response as a function of time and selected mass.

A computer records this 3-dimensional data set, we usually see a 2-dimensional spectrum with a selecting criteria.





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Gas Chromatography / Mass Spectrometry is generally accepted in the scientific community as an reliable method for the analysis of small amounts of substances.

But an expensive instrument itself doesn’t guarantee a scientifically valid result.

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...if you can't drive a stick-shift then
this car won't get you anywhere...

Gas Chromatography / Mass Spectrometry

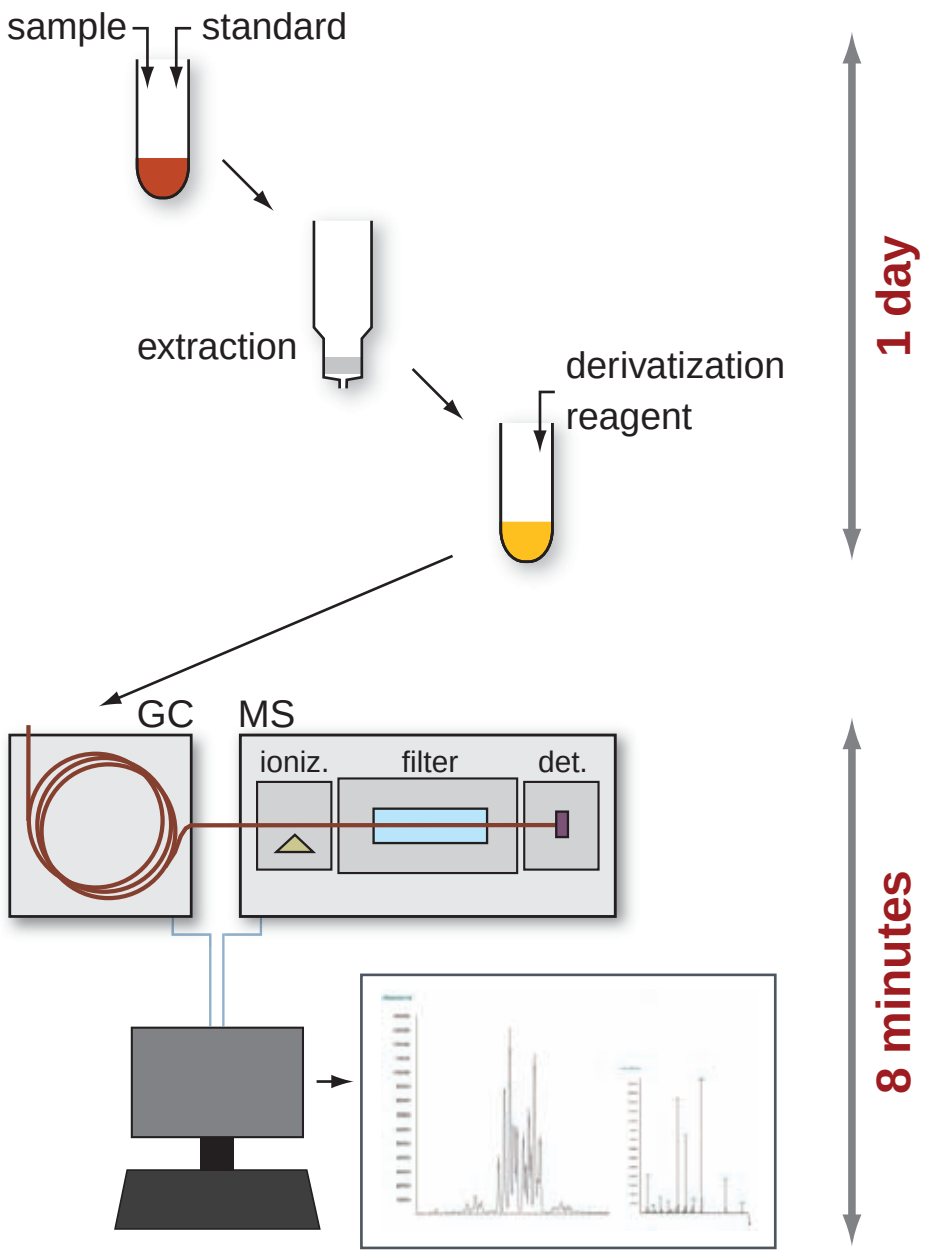
Multi-step Measurement Process

Sample Preparation
Mix sample + internal standard (isotope labeled version of substance)

Solid Phase Extraction
Extraction of substance from blood matrix

Derivatization
Chemical reaction to enhance selectivity and detectability

Gas Chromatography / Mass Spectrometry
Analysis of prepared sample



Michigan State Police Toxicology Lab

http://www.youtube.com/watch?v=ZxgzumkTEeQ

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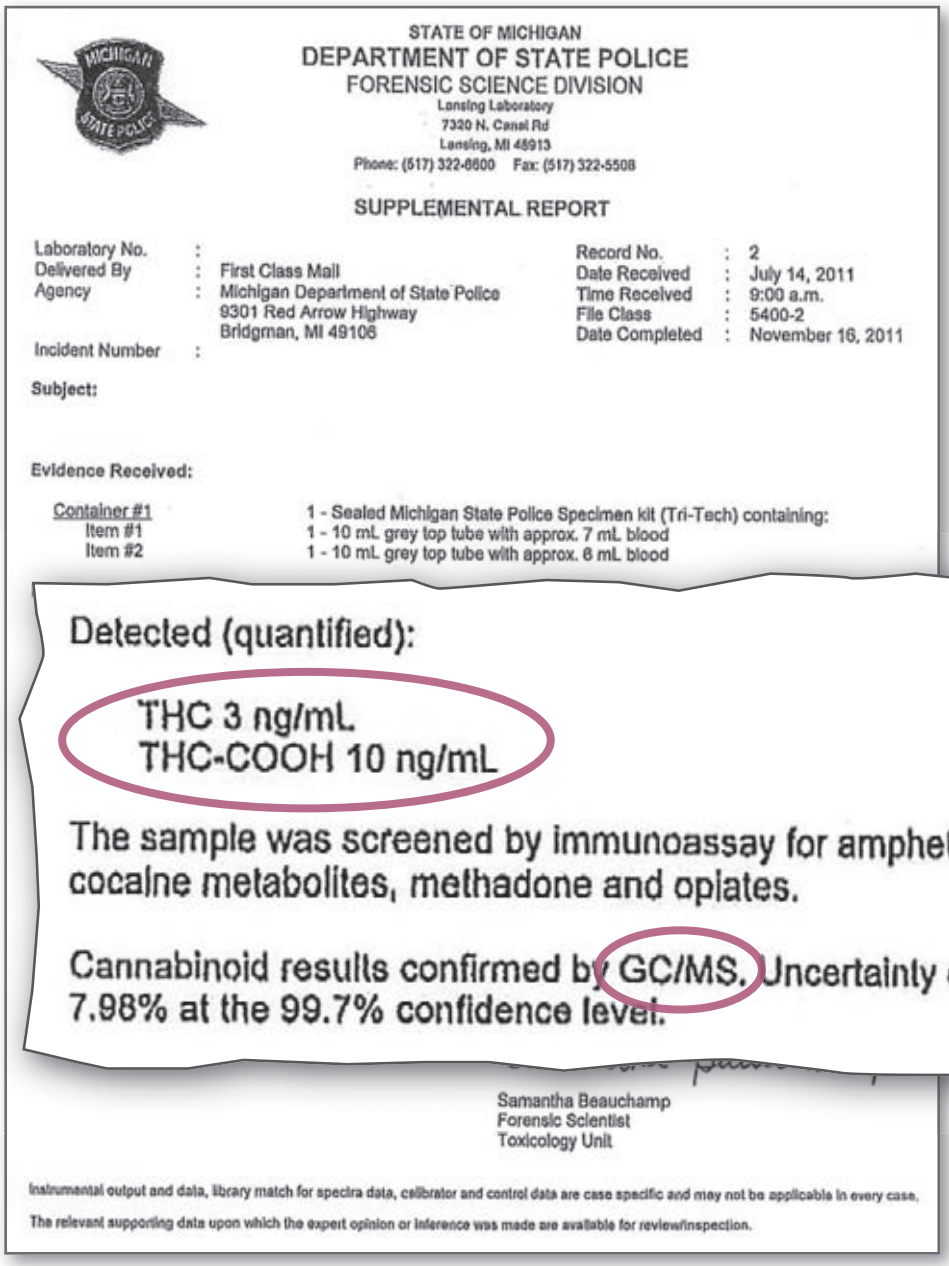

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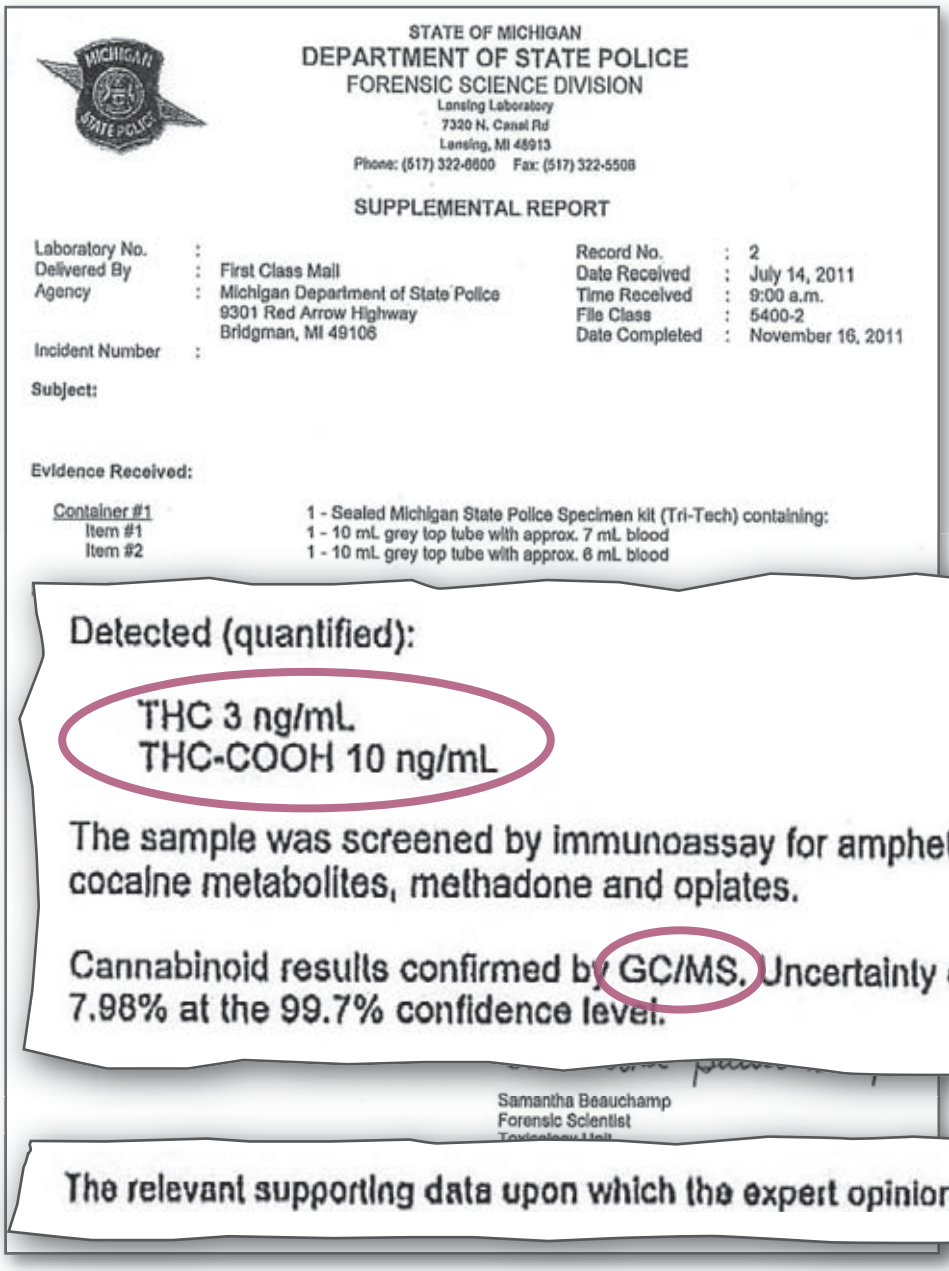

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A single number is not a scientific proof

The validity of a scientific analysis can only be judged by the supporting data.

Supporting Data

Michigan State Police Toxicology Cannabinoid Report

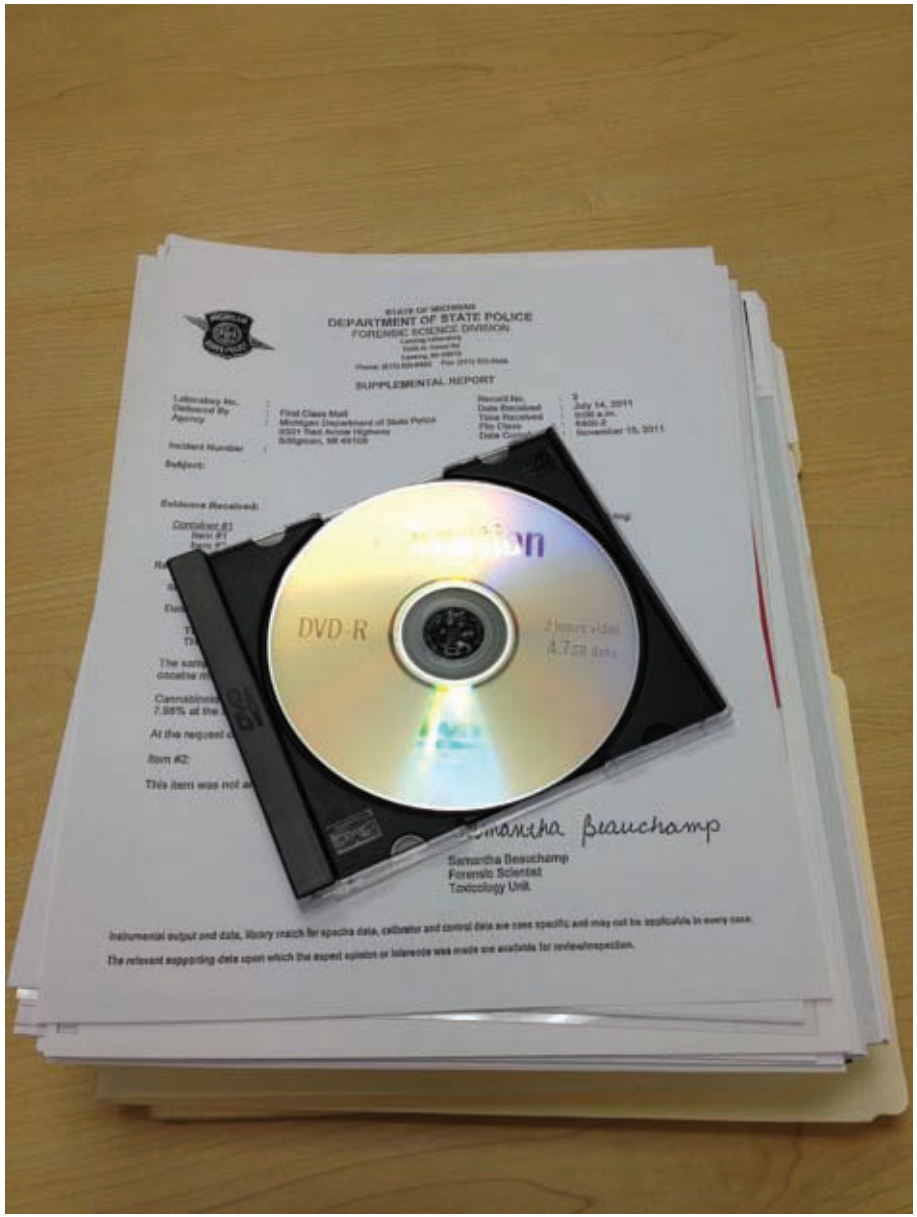
Procedure for “Quantitative Confirmation for Marihuana Metabolite in Blood”

Certificates for calibration standards

Maintenance and repair log for instruments

Validation studies to establish detection and quantification limits

Raw data files from GC / MS instruments

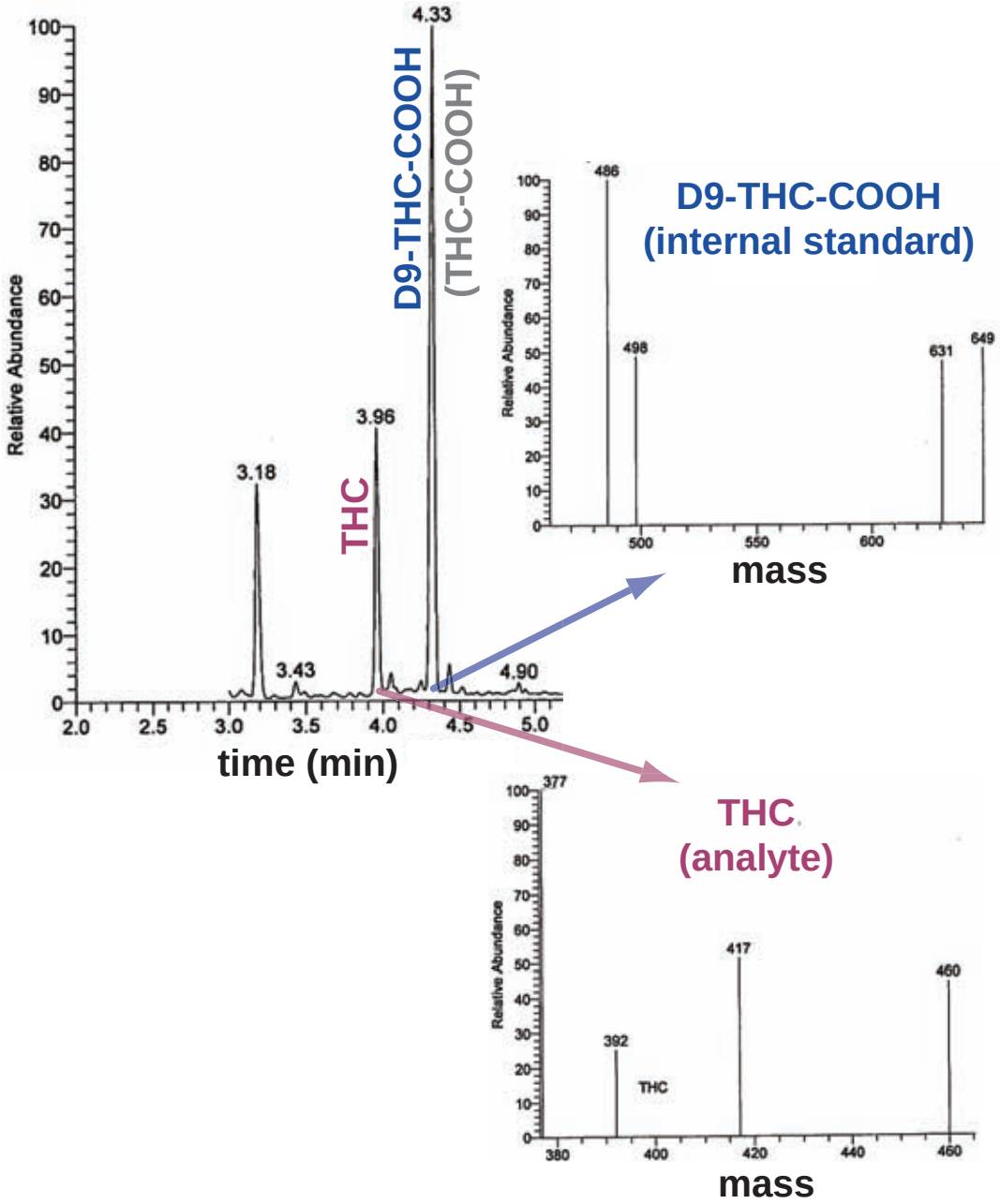


Gas Chromatography / Mass Spectrometry for THC

The Michigan State Police Toxicology Cannabinoid Report contains spectra for two analytes: THC and THC-COOH (Carboxy-THC, a THC metabolyte).

Deuterated THC-COOH is used as an internal standard. This is an isotope-labeled version of THC-COOH that doesn't occur in nature.

Only four characteristic masses are analyzed for each analyte or internal standard.

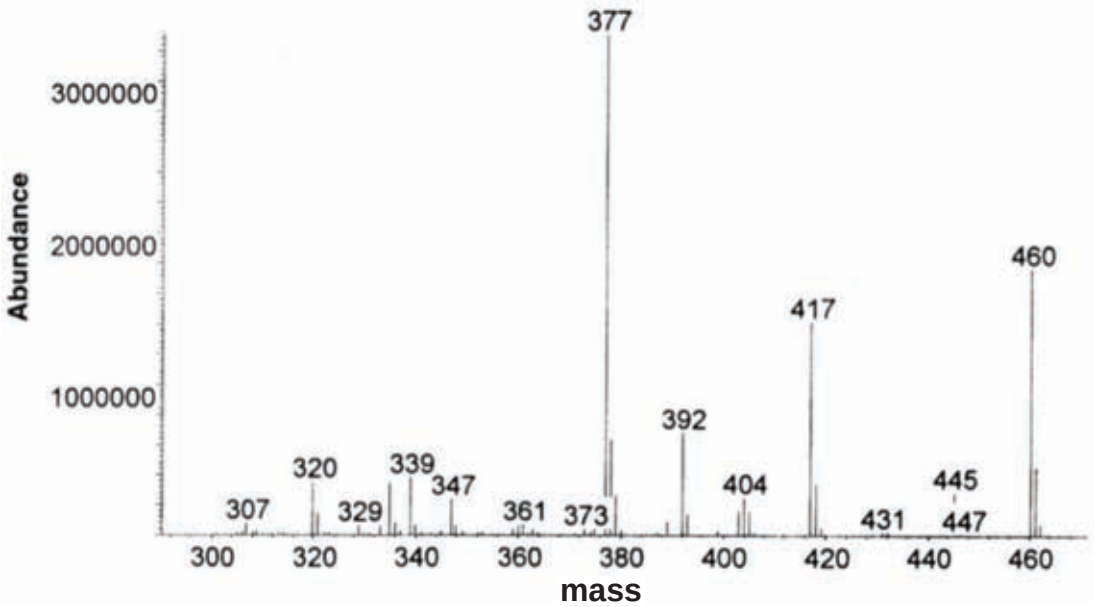


Gas Chromatography / Mass Spectrometry for THC

Full Mass Scan

mass filter scans through the full mass range

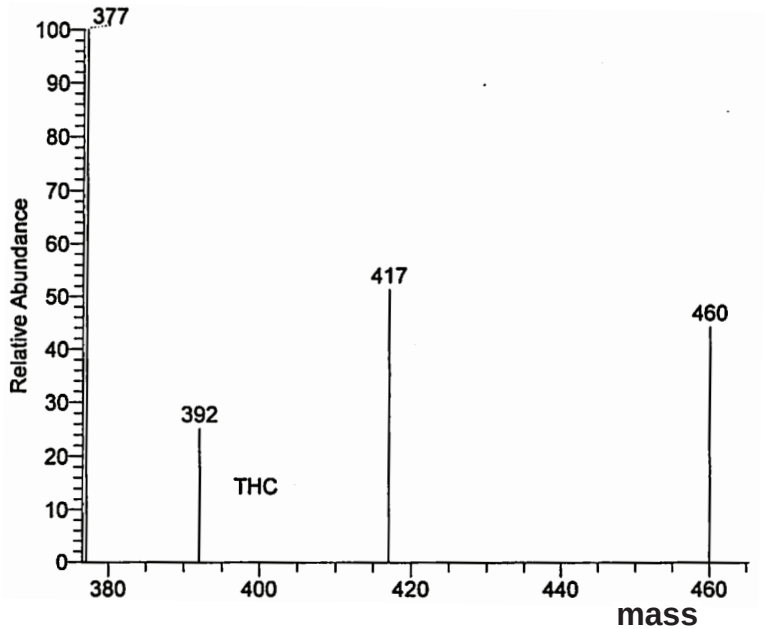
The abundance of each measured mass is represented as a bar graph. The abundance spectrum can be used for identification of a substance like a “fingerprint”.



Selective Ion Monitoring

mass filter steps through a small number of preselected masses

This mode is more sensitive as more time is spent on a single mass.

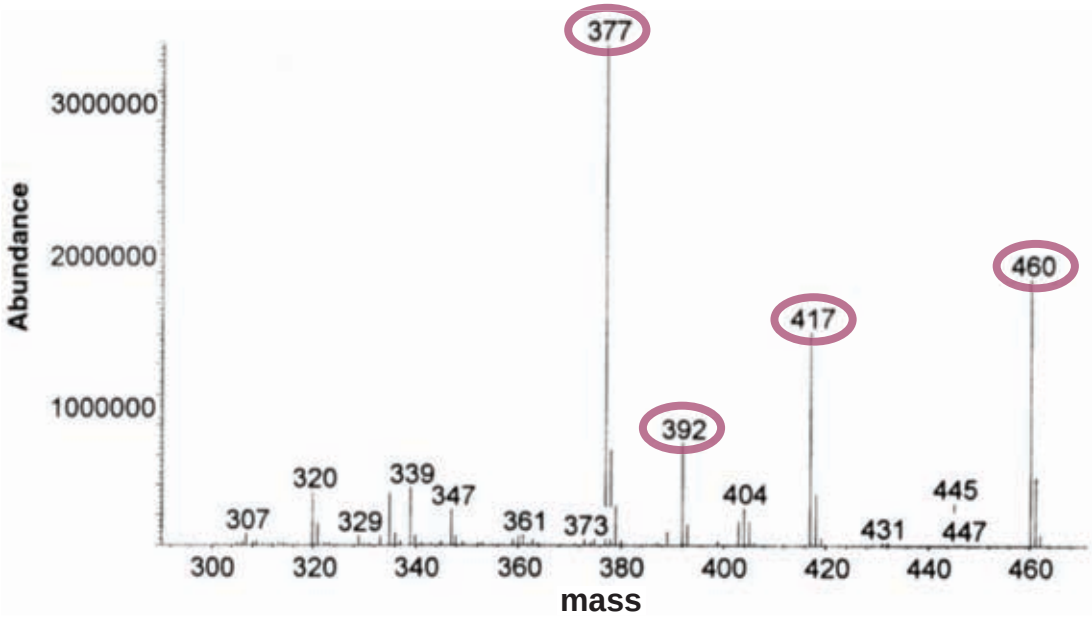


Gas Chromatography / Mass Spectrometry for THC

Full Mass Scan

mass filter scans through the full mass range

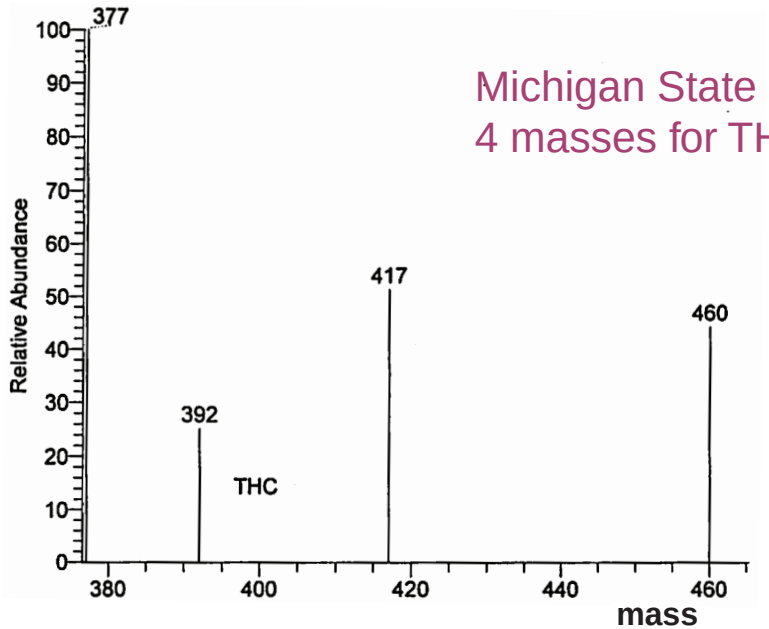
The abundance of each measured mass is represented as a bar graph. The abundance spectrum can be used for identification of a substance like a “fingerprint”.



Selective Ion Monitoring

mass filter steps through a small number of preselected masses

This mode is more sensitive as more time is spent on a single mass.



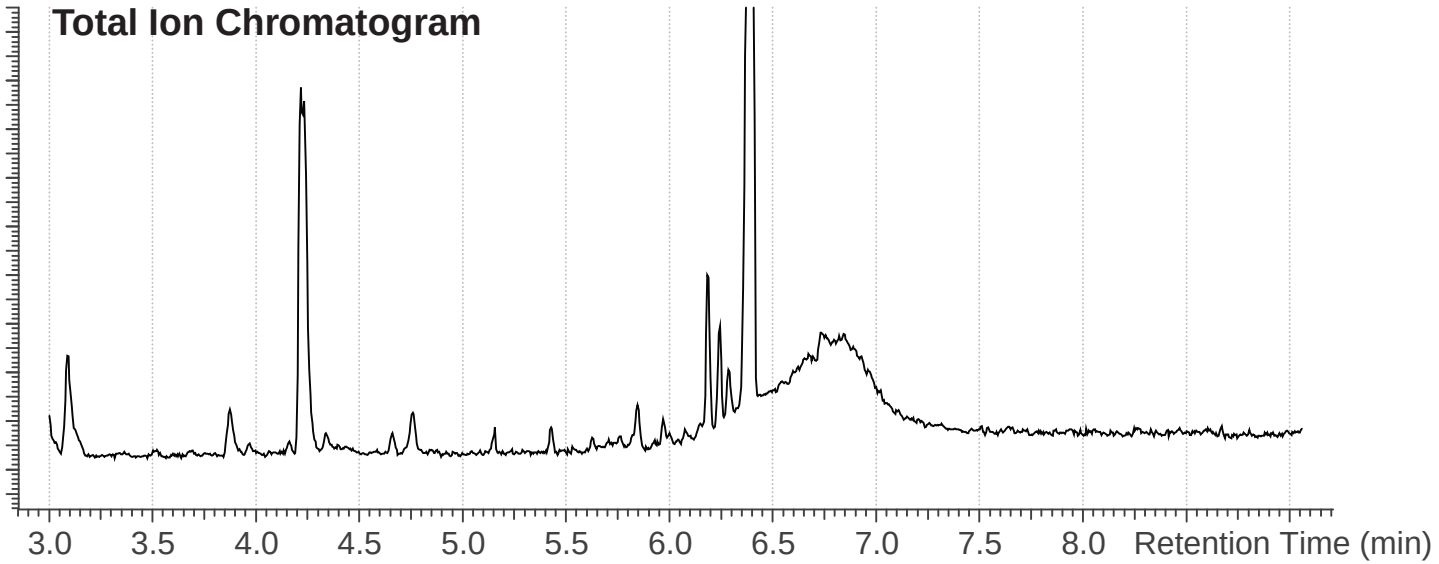
Michigan State Police uses 4 masses for THC analysis

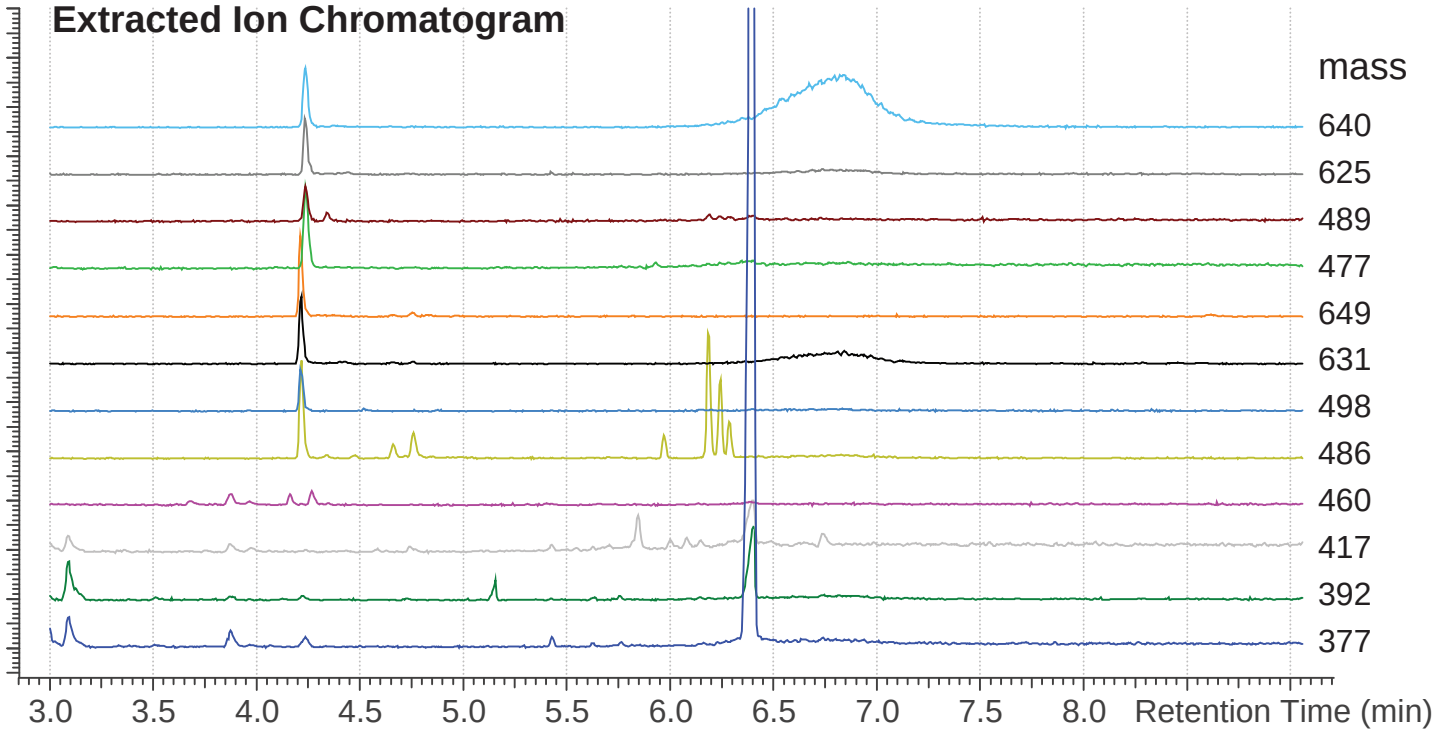
Gas Chromatography / Mass Spectrometry for THC

Calibration Sample STD3

Sample spiked with
THC 25 ng/ml
THC-COOH 100 ng/ml

raw data from MSP
taken on 06/20/2011



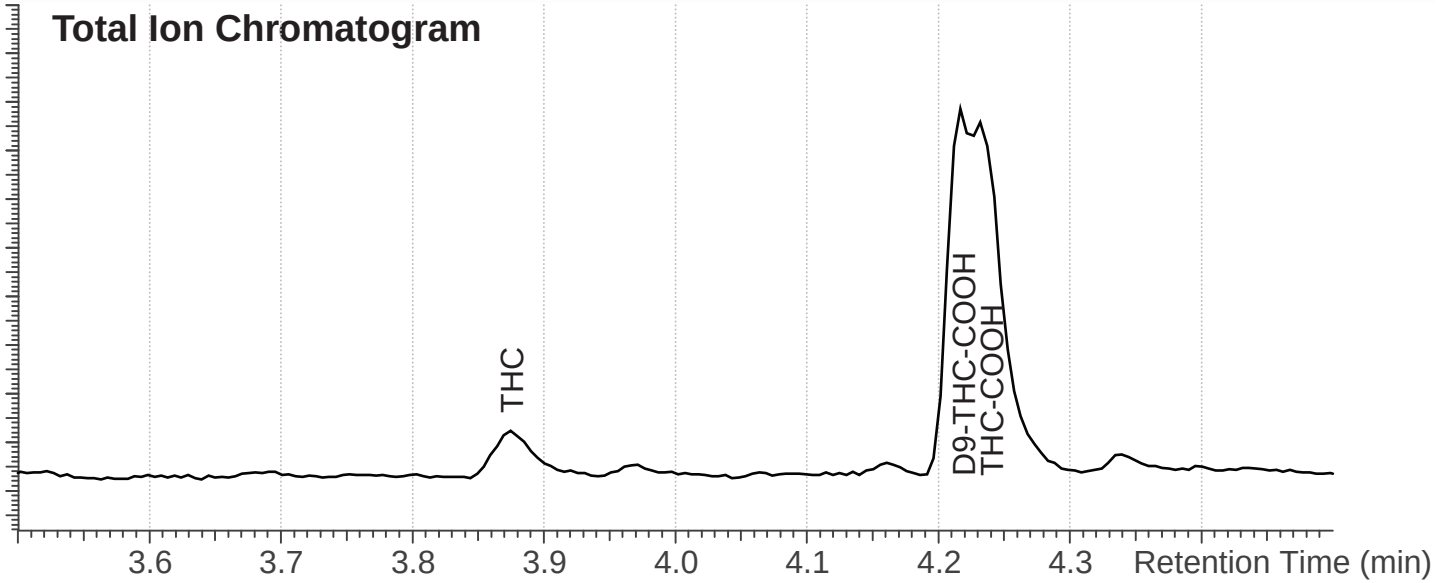


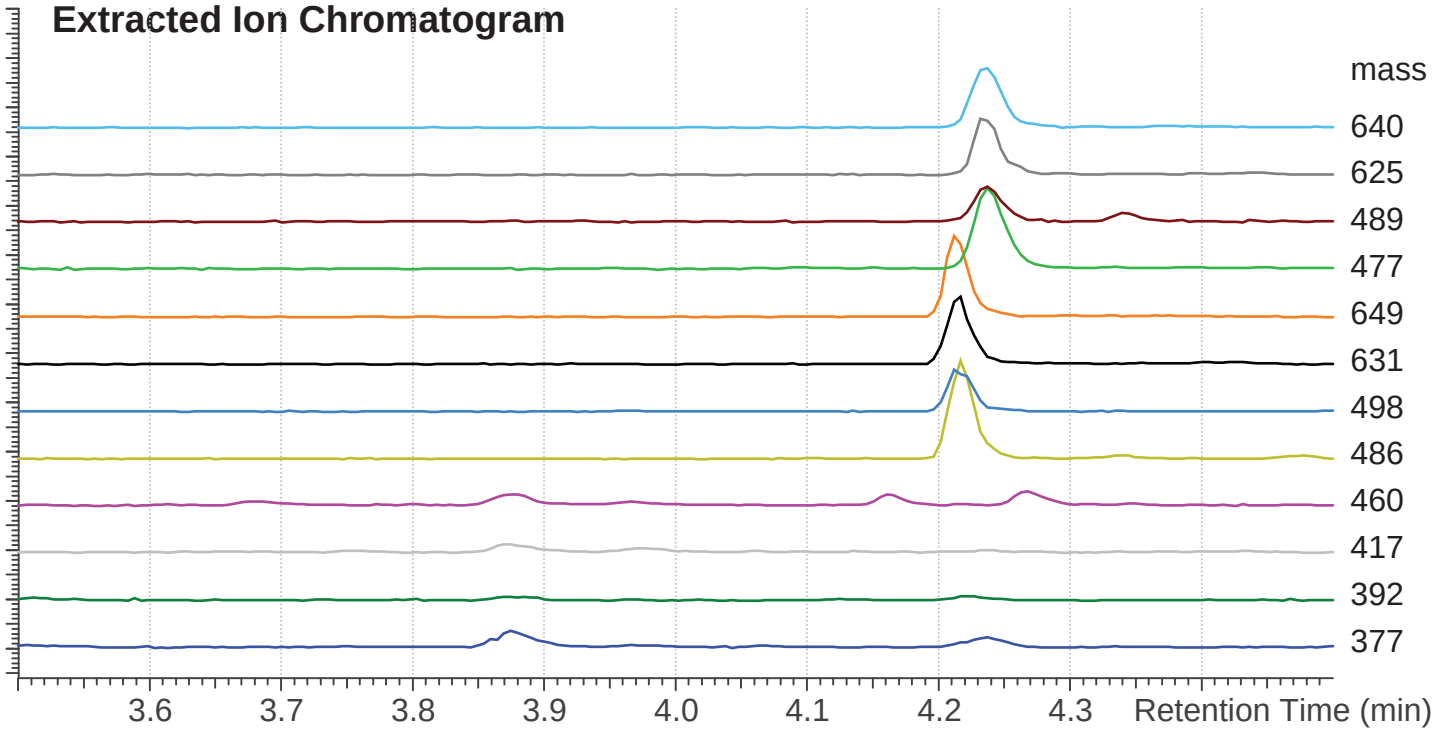
Gas Chromatography / Mass Spectrometry for THC

Calibration Sample STD3

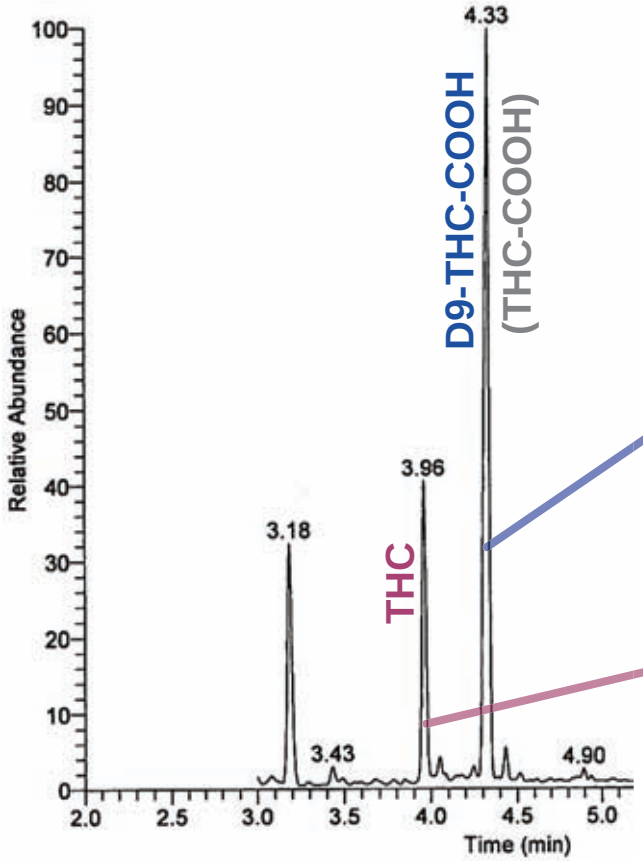
Sample spiked with
THC 25 ng/ml
THC-COOH 100 ng/ml

raw data from MSP
taken on 06/20/2011

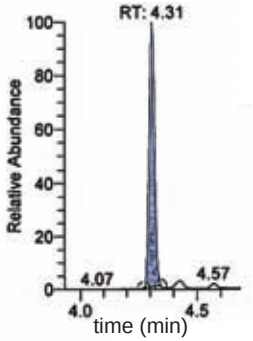


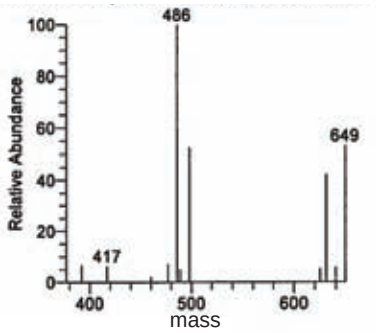


Quantitative Analysis of THC

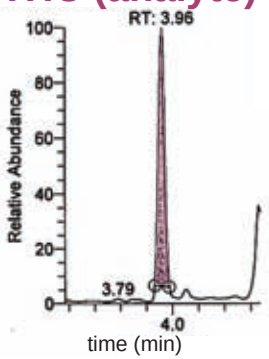


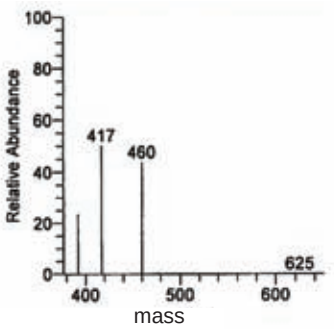
D9-THC-COOH (internal standard)





THC (analyte)





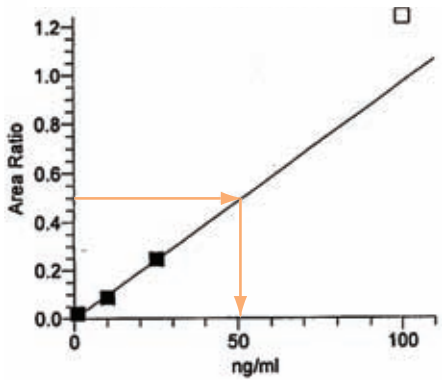
Name	Calculated Amount	RT	Area
D9-THC-COOH-486	N/A	4.31	12508488
THC-377	128.364	3.96	15548062
THC-COOH-477	198.251	4.33	28266727

A measurement relative to an internal standard with similar or identical chemical structure and known concentration allows to compensate for many variabilities in the measurement process.

$$\text{Area Ratio} = \frac{\text{analyte area}}{\text{int. standard area}}$$

The analyte concentration is measured as the area ratio between the analyte area and internal standard area.

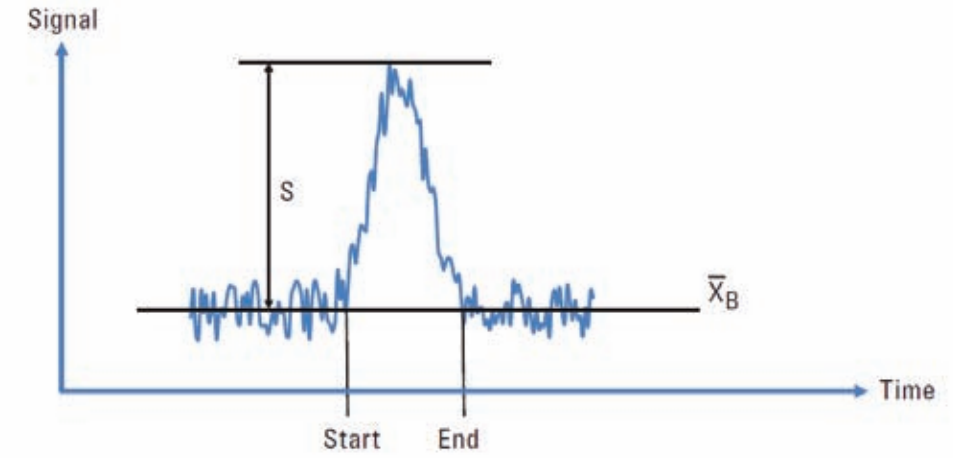
Calibration Curve for THC



Limit of Detection (LOD)

Instrument detection limit (IDL)

Most analytical instruments produce a signal even when a blank sample without analyte is analyzed. The instrument detection limit is the analyte concentration required to produce **a signal that is distinguishable from the noise level** within a particular statistical confidence limit (in many cases 3 – 5 times the standard deviation of a blank sample is chosen).



Method detection limit (MDL)

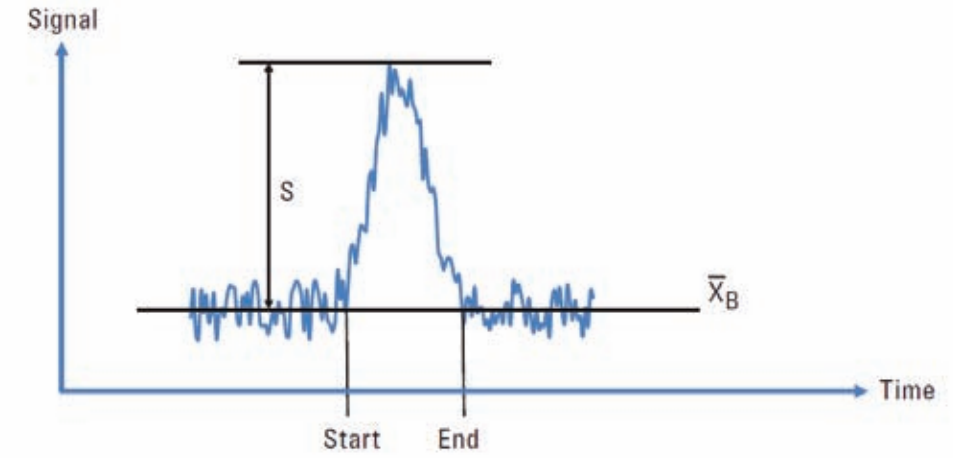
For most applications, there is more to the analytical method than just analyzing a clean analyte. **Additional steps in an analysis** add additional opportunities for error. Since detection limits are defined in terms of error, this **will increase the measured detection limit**.

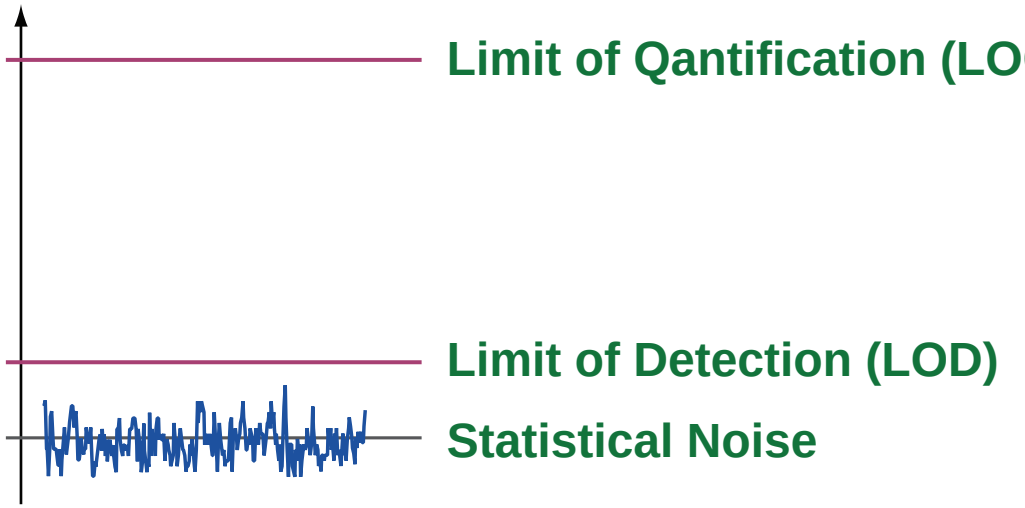
Agilent Technical Note 5990-7651EN:
Signal, Noise, and Detection Limits in Mass Spectrometry; 2011

Limit of Quantification (LOQ)

Limit of Quantification (LOQ)

The limit of quantification is the limit at which we can reasonably tell the difference between two different values of the amount of analyte. The LOQ is often practically defined simply as about 5 – 10 times the method detection limit (MDL).





Agilent Technical Note 5990-7651EN: Signal, Noise, and Detection Limits in Mass Spectrometry; 2011

Limit of Detection (LOD) for THC

Michigan State Police Procedure "Quantitative Confirmation for Marihuana Metabolite in Blood":

"The LOD for both THC and 11-COOH-THC has been determined to be approximately 0.5 ng/ml in full scan mode, 0.1 ng/ml in SIM mode.

The LOQ for both compounds has been designated to be 2 ng/ml in full scan mode, 1 ng/ml in SIM mode".

(SIM stands for selective ion monitoring mode. Confirmatory cannabinoid analysis is performed in SIM mode)

The Michigan Department of State Police could not provide any documents to support these numbers.

Limit of Detection (LOD) for THC

Michigan State Police Procedure "Quantitative Confirmation for Marihuana Metabolite in Blood":

"The LOD for both THC and 11-COOH-THC has been determined to be approximately 0.5 ng/ml in full scan mode, 0.1 ng/ml in SIM mode.

The LOQ for both compounds has been designated to be 2 ng/ml in full scan mode, 1 ng/ml in SIM mode".

Amount	%Diff	Units	RT	Sample ID
6.306	26%	ng/mL	4.47	STD1
26.888	8%	ng/mL	4.47	STD2
97.475	-3%	ng/mL	4.47	STD3
200.994	0%	ng/mL	4.47	STD4
N/F	N/A	ng/mL		0.1 NG/ML A
N/F	N/A	ng/mL		0.1 NG/ML B
N/F	N/A	ng/mL		0.1 NG/ML C
N/F	N/A	ng/mL		0.1 NG/ML D
N/F	N/A	ng/mL		0.1 NG/ML E
N/F	N/A	ng/mL		0.5 NG/ML A
N/F	N/A	ng/mL		0.5 NG/ML B
N/F	N/A	ng/mL		0.5 NG/ML C
N/F	N/A	ng/mL		0.5 NG/ML D
N/F	N/A	ng/mL		0.5 NG/ML E
1.214		ng/mL	4.47	1.0 NG/ML A
1.295		ng/mL	4.47	1.0 NG/ML B
1.580		ng/mL	4.47	1.0 NG/ML C
1.260		ng/mL	4.47	1.0 NG/ML D
1.627		ng/mL	4.46	1.0 NG/ML E

Data from a recent study performed by Michigan State Police in June 2012 shows that the THC content in control samples spiked with 0.1 ng/ml and 0.5 ng/ml THC could not be identified.

Samples containing more than 1.0 ng/ml THC are marked with a measured concentration.

Measured values are up to 60% higher than the nominal concentration.

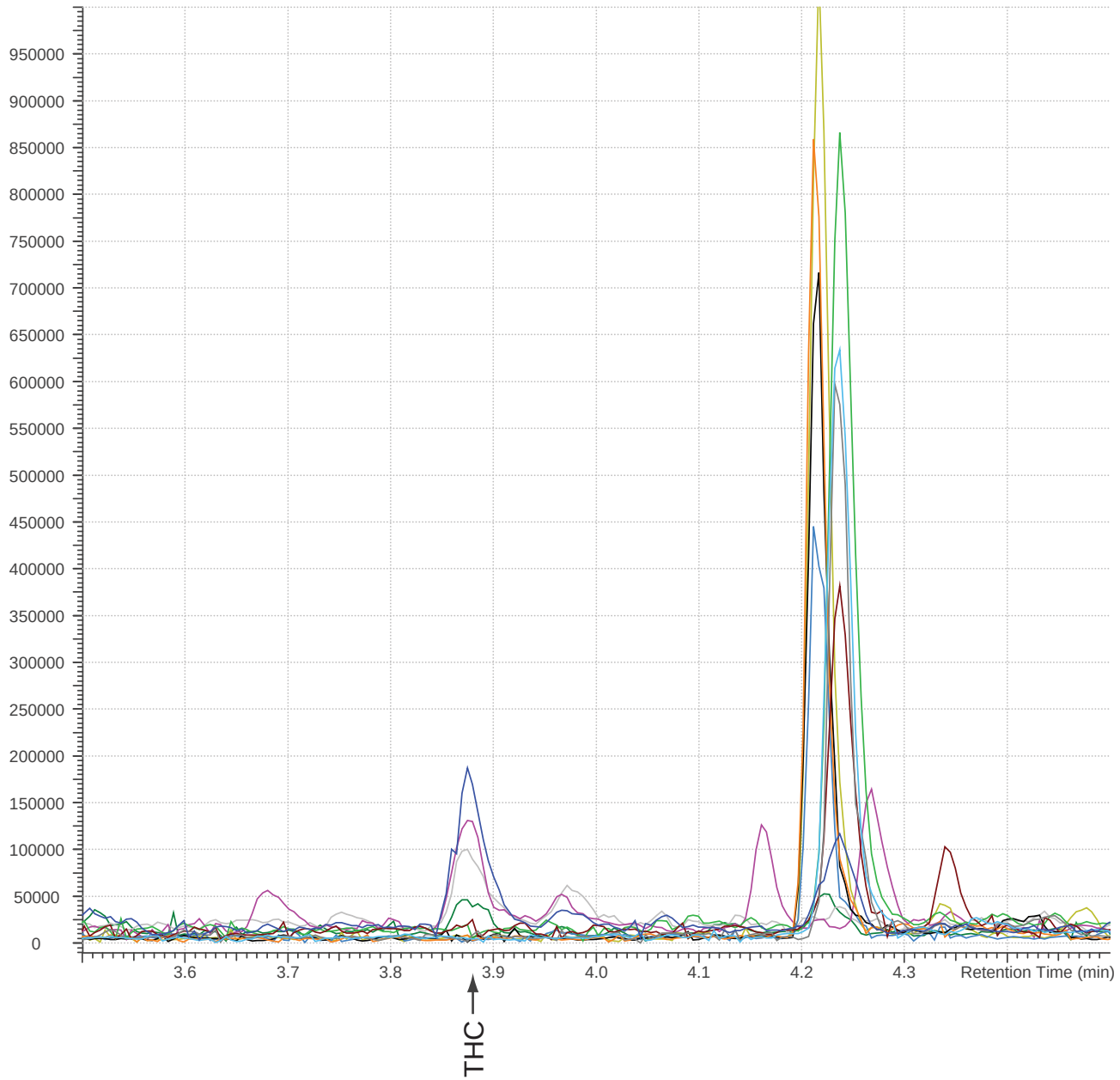
The analysis of the study is ongoing.

Gas Chromatography / Mass Spectrometry for THC

Calibration
Sample STD3

Sample spiked with
THC 25 ng/ml
THC-COOH 100 ng/ml

raw data from MSP
taken on 06/20/2011



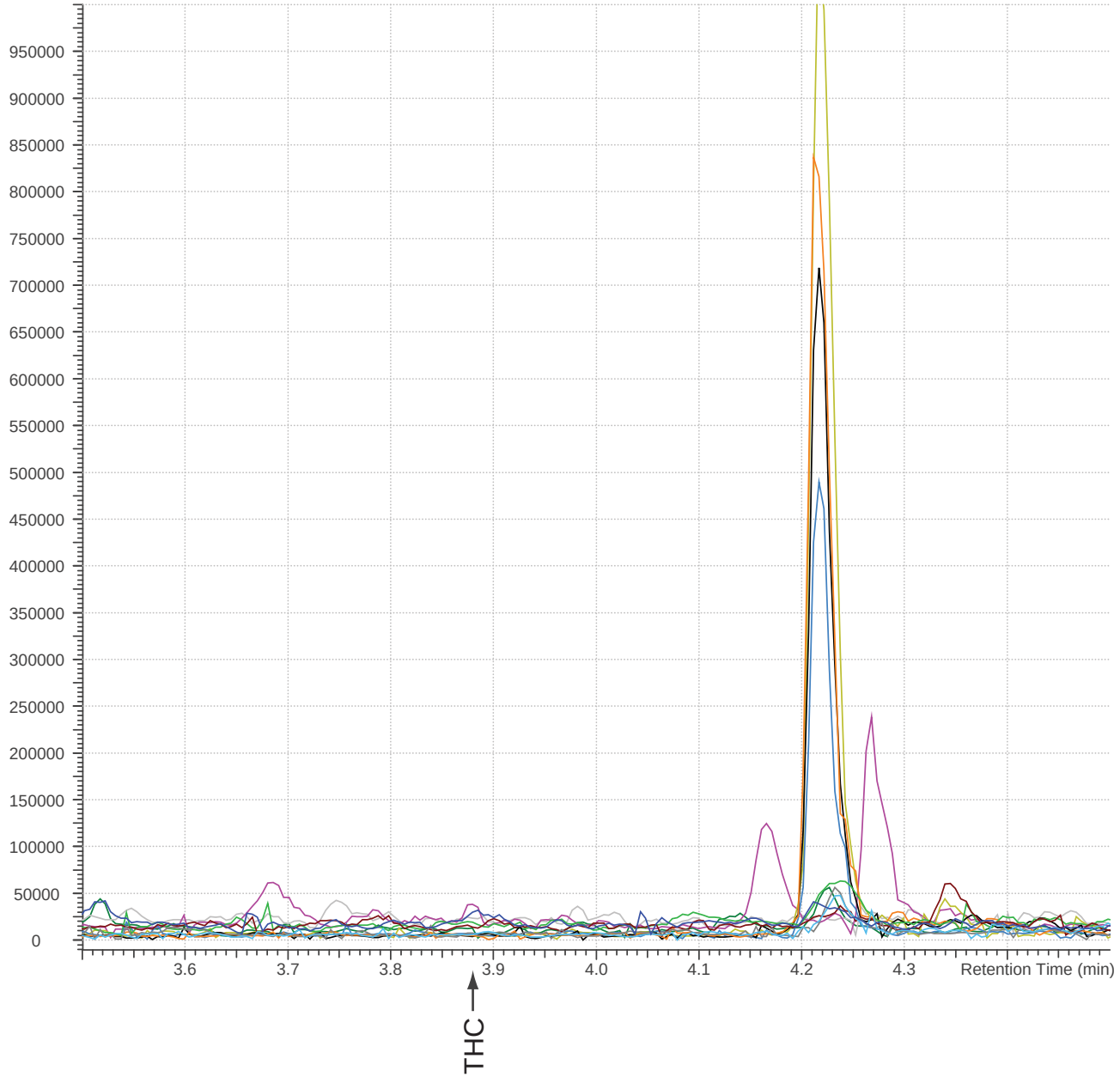
Identification criteria:
retention time
mass abundance ratio

Gas Chromatography / Mass Spectrometry for THC

Calibration
Sample STD1

Sample spiked with
THC 1 ng/ml
THC-COOH 5 ng/ml

raw data from MSP
taken on 06/20/2011



Identification criteria:
retention time
mass abundance ratio

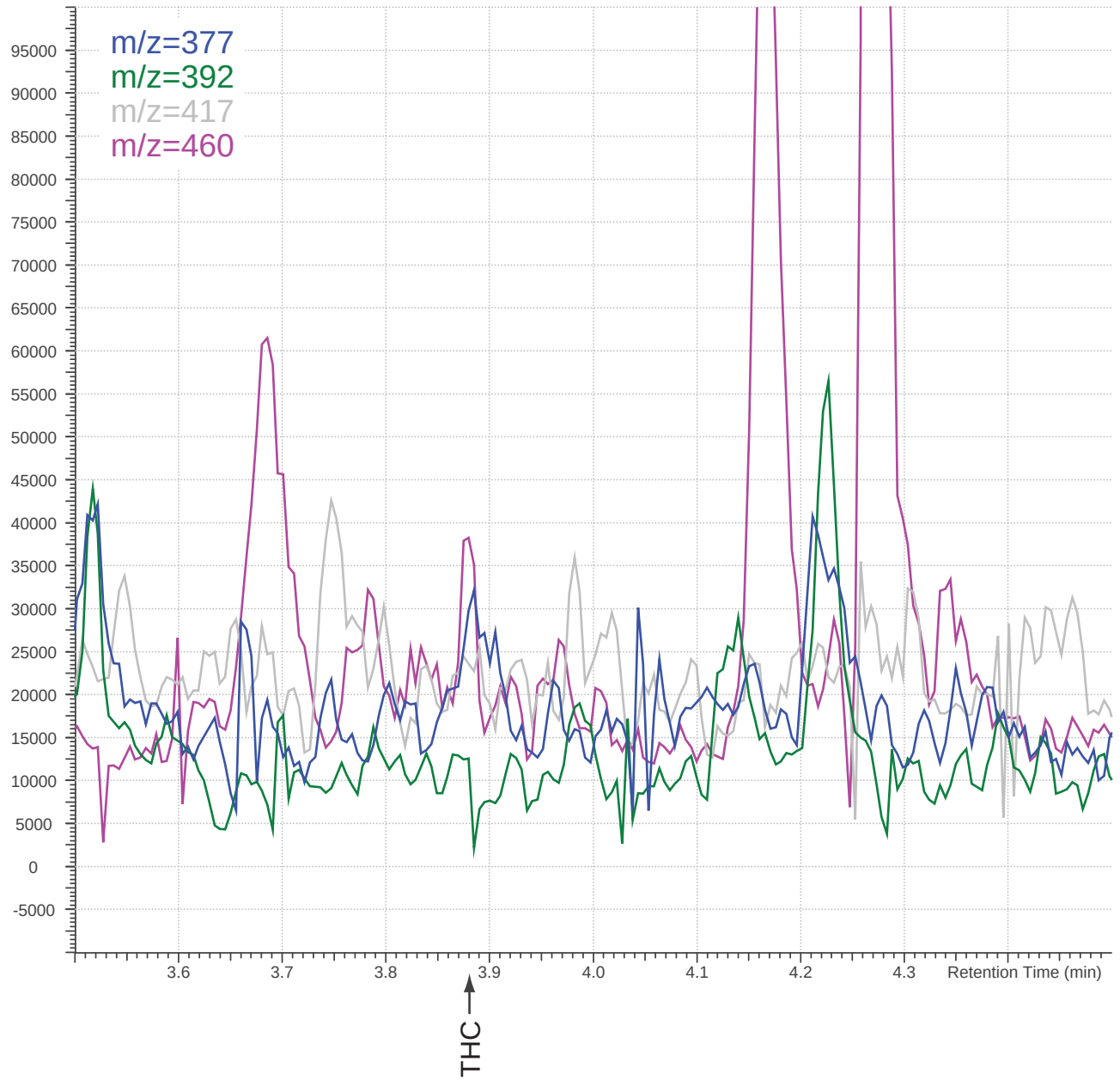
Gas Chromatography / Mass Spectrometry for THC

Calibration
Sample STD1

Sample spiked with
THC 1 ng/ml
THC-COOH 5 ng/ml

raw data from MSP
taken on 06/20/2011

vertical scale x10
only masses for THC



Identification criteria:
retention time
mass abundance ratio

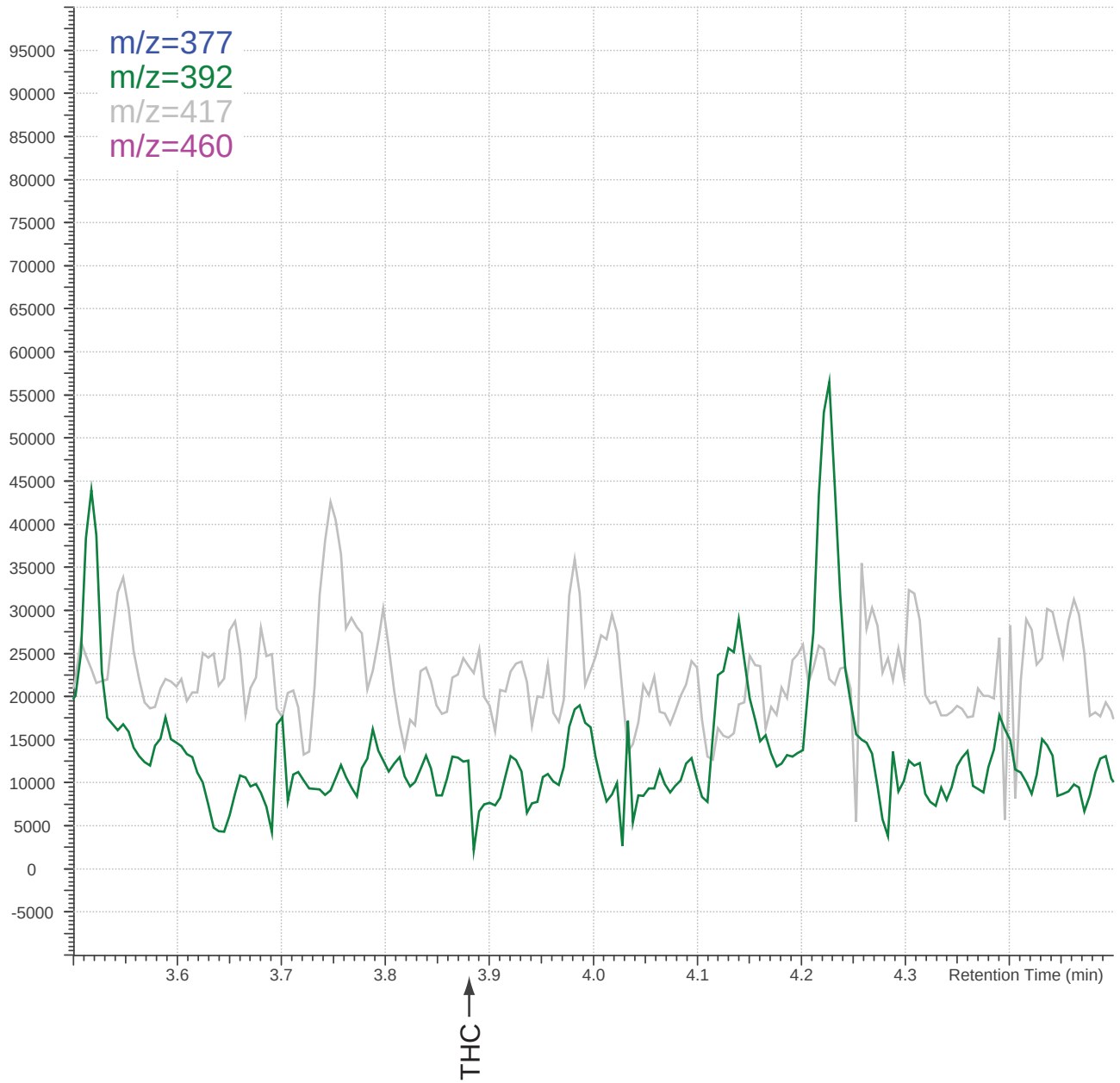
Gas Chromatography / Mass Spectrometry for THC

Calibration
Sample STD1

Sample spiked with
THC 1 ng/ml
THC-COOH 5 ng/ml

raw data from MSP
taken on 06/20/2011

vertical scale x10
only masses for THC



Identification criteria:
retention time
mass abundance ratio

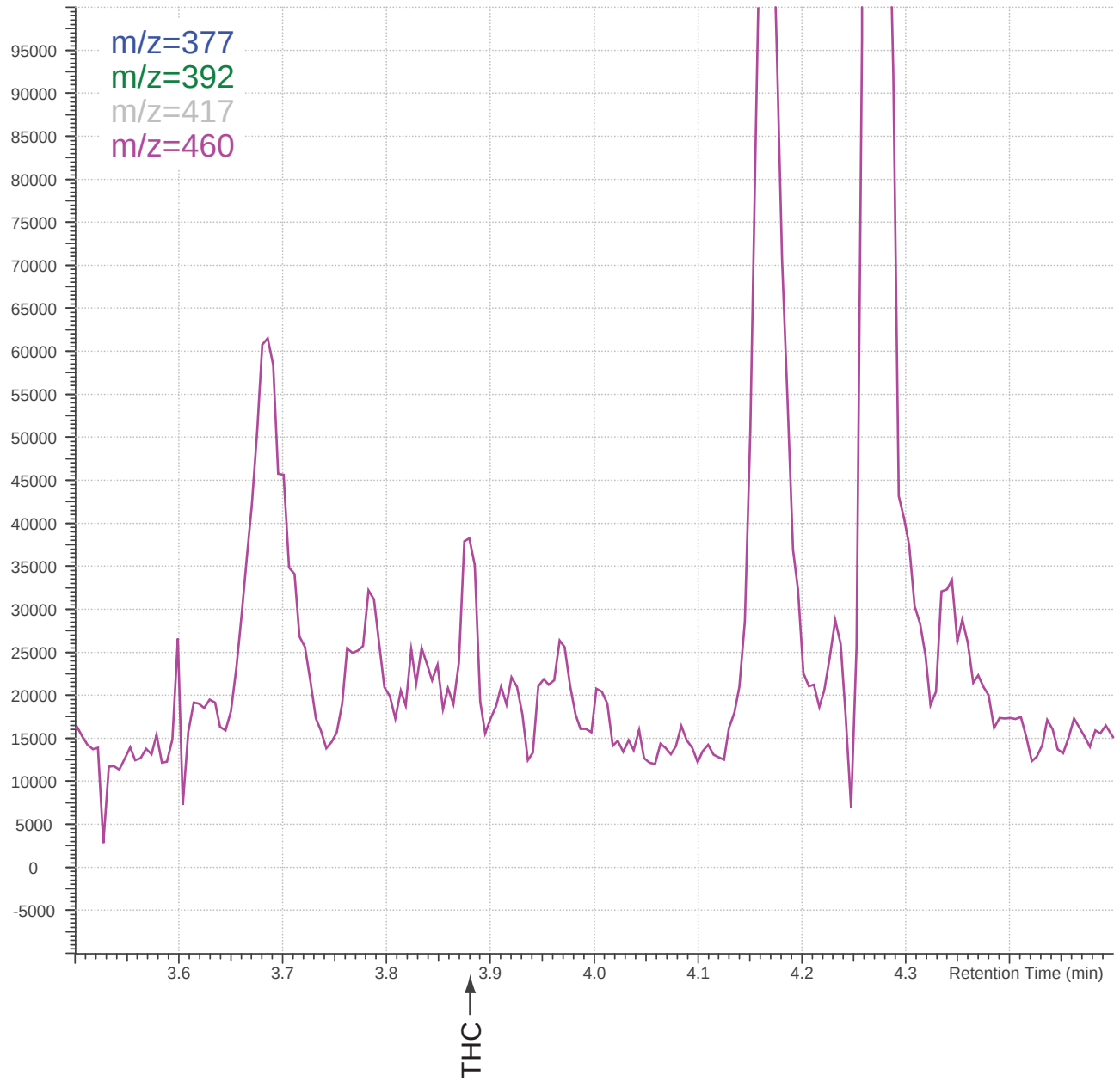
Gas Chromatography / Mass Spectrometry for THC

Calibration
Sample STD1

Sample spiked with
THC 1 ng/ml
THC-COOH 5 ng/ml

raw data from MSP
taken on 06/20/2011

vertical scale x10
only masses for THC



Identification criteria:
retention time
mass abundance ratio

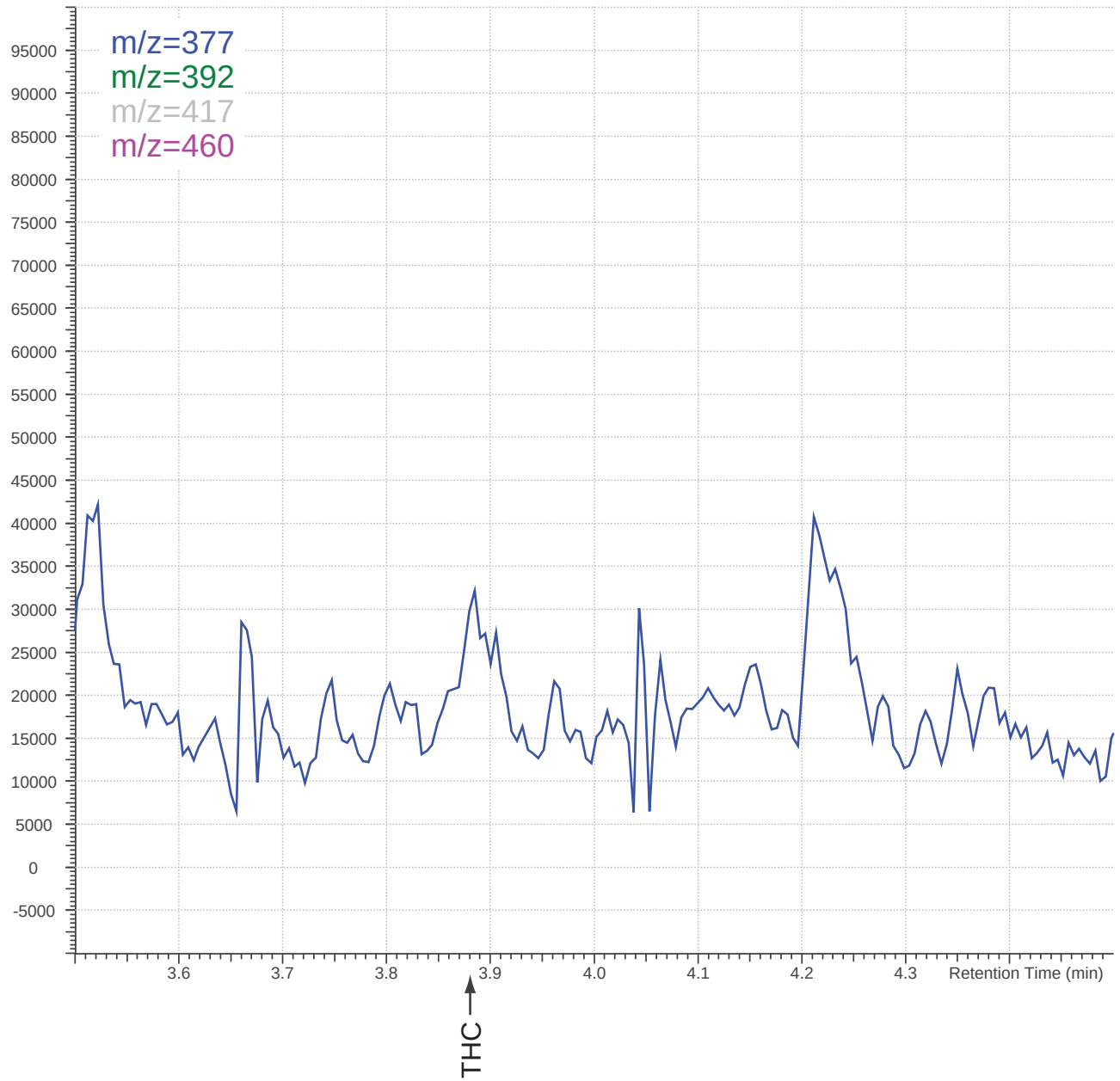
Gas Chromatography / Mass Spectrometry for THC

Calibration
Sample STD1

Sample spiked with
THC 1 ng/ml
THC-COOH 5 ng/ml

raw data from MSP
taken on 06/20/2011

vertical scale x10
only masses for THC



Identification criteria:
retention time
mass abundance ratio

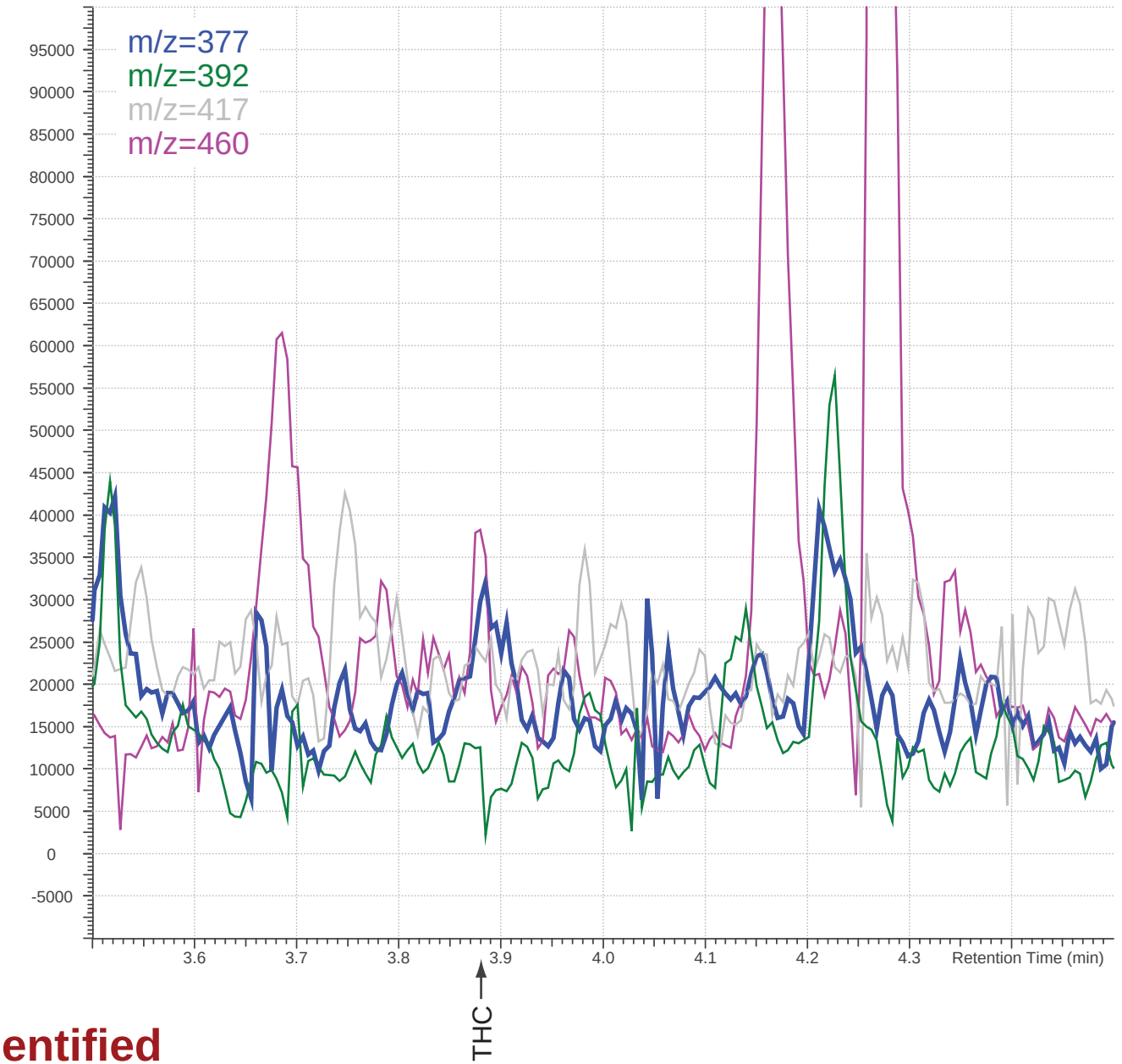
Gas Chromatography / Mass Spectrometry for THC

Calibration
Sample STD1

Sample spiked with
THC 1 ng/ml
THC-COOH 5 ng/ml

raw data from MSP
taken on 06/20/2011

vertical scale x10
only masses for THC



Identification criteria:
retention time
mass abundance ratio

Only 1 mass clearly identified

It’s a Lion!

Be careful if you can’t see the full picture!

It’s a Lion!

Be careful if you can’t see the full picture!

It’s a Lion!?

Be careful if you can’t see the full picture!

It’s a Lion????

Be careful if you can’t see the full picture!

Limit of Detection (LOD) for THC

Amount	Amount	%Diff	Units	RT	Sample ID
5.000	6.306	26%	ng/mL	4.47	STD1
25.000	26.888	8%	ng/mL	4.47	STD2
100.000	97.475	-3%	ng/mL	4.47	STD3
200.000	200.994	0%	ng/mL	4.47	STD4
N/A	N/F	N/A	ng/mL		0.1 NG/ML A
N/A	N/F	N/A	ng/mL		0.1 NG/ML B
N/A	N/F	N/A	ng/mL		0.1 NG/ML C
N/A	N/F	N/A	ng/mL		0.1 NG/ML D
N/A	N/F	N/A	ng/mL		0.1 NG/ML E
N/A	N/F	N/A	ng/mL		0.5 NG/ML A
N/A	N/F	N/A	ng/mL		0.5 NG/ML B
N/A	N/F	N/A	ng/mL		0.5 NG/ML C
N/A	N/F	N/A	ng/mL		0.5 NG/ML D
N/A	N/F	N/A	ng/mL		0.5 NG/ML E
	1.214		ng/mL	4.47	1.0 NG/ML A
	1.295		ng/mL	4.47	1.0 NG/ML B
	1.580		ng/mL	4.47	1.0 NG/ML C
	1.260		ng/mL	4.47	1.0 NG/ML D
	1.627		ng/mL	4.46	1.0 NG/ML E
	1.963		ng/mL	4.47	1.5 NG/ML A
	2.145		ng/mL	4.46	1.5 NG/ML B
	2.101		ng/mL	4.46	1.5 NG/ML C
	2.090		ng/mL	4.46	1.5 NG/ML D
	2.318		ng/mL	4.46	1.5 NG/ML E
	2.580		ng/mL	4.47	2.0 NG/ML A
	2.141		ng/mL	4.47	2.0 NG/ML B
	2.462		ng/mL	4.47	2.0 NG/ML C
	2.108		ng/mL	4.46	2.0 NG/ML D
	2.439		ng/mL	4.46	2.0 NG/ML E

1.0 ng/ml

1.5 ng/ml

2.0 ng/ml

Data from a recent study performed by Michigan State Police in June 2012

Samples containing more than 1.0 ng/ml THC are marked with a measured concentration.

Measured values are up to 60% higher than the nominal concentration.

The analysis of the study is ongoing.

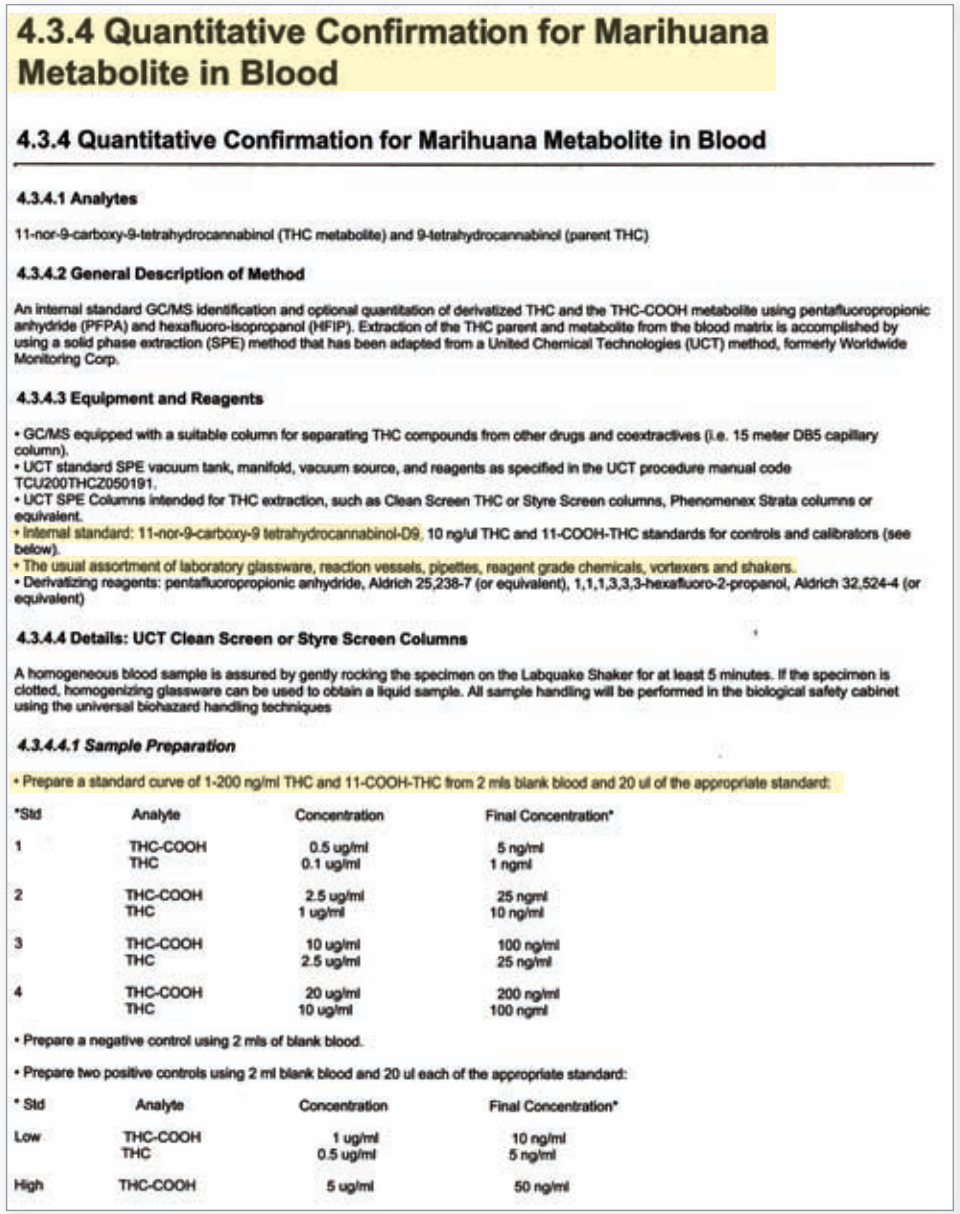
In September 2012 Michigan State Police changed the measurement uncertainty for THC quantification from 7.98% to 35%.

Procedural Deficiencies

The “Quantitative Confirmation for Marihuana Metabolite” **procedure is optimized for the measurement of the metabolite COOH-THC (carboxy-THC) and not for the measurement of THC.** The procedure uses deuterated COOH-THC as internal standard and not deuterated THC.

The procedure states to use 4 different “appropriate standards” to mix the calibrators. Only one concentration for THC (10 ng/ul) is on the reagent list. The provided certificates is for a 1.013 mg/ml (=1013 ng/ul) THC standard from Ceriliant. **There are no instructions how to mix the 4 different standard solutions.**

The statement in the procedure “The usual assortment of laboratory glassware, reaction vessels, pipettes,…” **is not prescriptive enough** and leaves room for error.



4.3.4 Quantitative Confirmation for Marihuana Metabolite in Blood

4.3.4 Quantitative Confirmation for Marihuana Metabolite in Blood

4.3.4.1 Analytes

11-nor-9-carboxy-9-tetrahydrocannabinol (THC metabolite) and 9-tetrahydrocannabinol (parent THC)

4.3.4.2 General Description of Method

An internal standard GC/MS identification and optional quantitation of derivatized THC and the THC-COOH metabolite using pentafluoropropionic anhydride (PFPA) and hexafluoro-isopropanol (HFIP). Extraction of the THC parent and metabolite from the blood matrix is accomplished by using a solid phase extraction (SPE) method that has been adapted from a United Chemical Technologies (UCT) method, formerly Worldwide Monitoring Corp.

4.3.4.3 Equipment and Reagents

- GC/MS equipped with a suitable column for separating THC compounds from other drugs and coextractives (i.e. 15 meter DB5 capillary column).
- UCT standard SPE vacuum tank, manifold, vacuum source, and reagents as specified in the UCT procedure manual code TCU200THCZ050191.
- UCT SPE Columns intended for THC extraction, such as Clean Screen THC or Styre Screen columns, Phenomenex Strata columns or equivalent.
- Internal standard: 11-nor-9-carboxy-9 tetrahydrocannabinol-D9, 10 ng/ul THC and 11-COOH-THC standards for controls and calibrators (see below).
- The usual assortment of laboratory glassware, reaction vessels, pipettes, reagent grade chemicals, vortexers and shakers.
- Derivatizing reagents: pentafluoropropionic anhydride, Aldrich 25,238-7 (or equivalent), 1,1,1,3,3,3-hexafluoro-2-propanol, Aldrich 32,524-4 (or equivalent)

4.3.4.4 Details: UCT Clean Screen or Styre Screen Columns

A homogeneous blood sample is assured by gently rocking the specimen on the Labquake Shaker for at least 5 minutes. If the specimen is clotted, homogenizing glassware can be used to obtain a liquid sample. All sample handling will be performed in the biological safety cabinet using the universal biohazard handling techniques

4.3.4.4.1 Sample Preparation

- Prepare a standard curve of 1-200 ng/ml THC and 11-COOH-THC from 2 mls blank blood and 20 ul of the appropriate standard:

*Std	Analyte	Concentration	Final Concentration*
1	THC-COOH THC	0.5 ug/ml 0.1 ug/ml	5 ng/ml 1 ngml
2	THC-COOH THC	2.5 ug/ml 1 ug/ml	25 ngml 10 ng/ml
3	THC-COOH THC	10 ug/ml 2.5 ug/ml	100 ng/ml 25 ng/ml
4	THC-COOH THC	20 ug/ml 10 ug/ml	200 ng/ml 100 ngml

- Prepare a negative control using 2 mls of blank blood.
- Prepare two positive controls using 2 ml blank blood and 20 ul each of the appropriate standard:

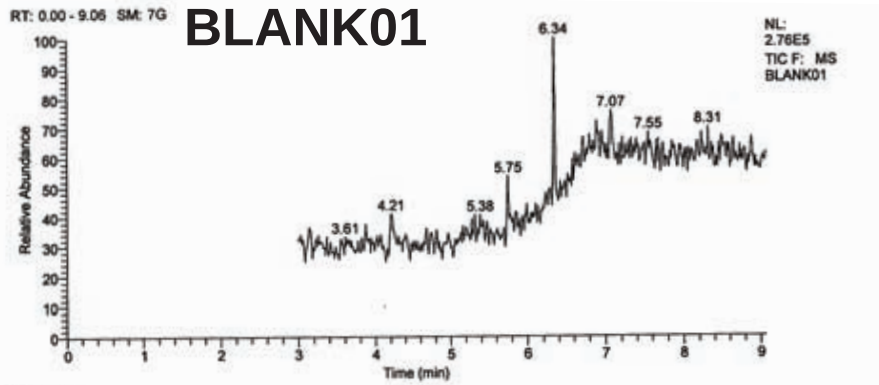
* Std	Analyte	Concentration	Final Concentration*
Low	THC-COOH THC	1 ug/ml 0.5 ug/ml	10 ng/ml 5 ng/ml
High	THC-COOH	5 ug/ml	50 ng/ml

Negative Controls

Negative Controls are used to verify that a sample without analyte will test negative. In order to test for possible contamination a negative control sample needs to be processed exactly like any other unknown sample.

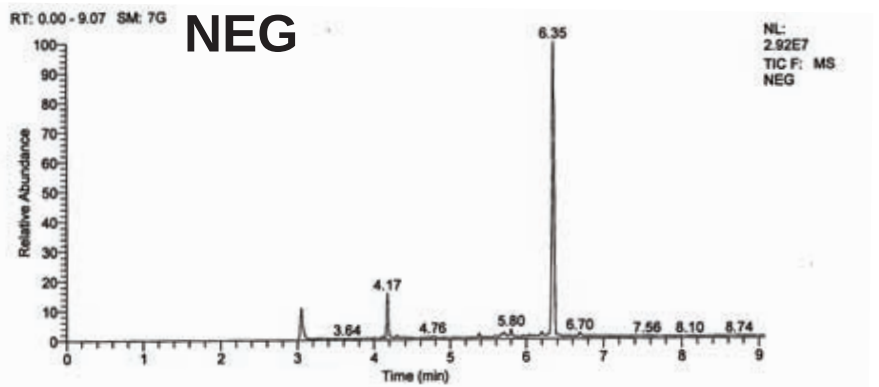
Michigan State Police uses in one analysis batch about 50 samples labelled ‘BLANK..’ and one sample labelled ‘NEG’.

‘BLANK..’ samples do not test for process contamination. They do not contain an internal standard and they are not processed identically.



Total Ion Chromatogram

Name	Calculated Amount	RT	Area	Height
D9-THC-COOH-486	N/A	N/A	N/A	N/A
THC-377	N/A	N/A	N/A	N/A
THC-COOH-477	N/A	N/A	N/A	N/A



Total Ion Chromatogram

Name	Calculated Amount	RT	Area	Height
D9-THC-COOH-486	N/A	4.17	2215716	1406396
THC-377	N/A	N/A	N/A	N/A
THC-COOH-477	N/A	N/A	N/A	N/A

The Bottom Line

Michigan State Police Procedure "Quantitative Confirmation for Marihuana Metabolite in Blood":

"The LOD for both THC and 11-COOH-THC has been determined to be approximately (...) 0.1 ng/ml (...).

The LOQ for both compounds has been designated to be (...) 1 ng/ml (...)".

The Michigan Department of State Police could not provide any documents to support these numbers.

The analysis of data provided by Michigan State Police suggests that the Limit of Detection and the Limit of Quantification for THC is significantly higher.

In absence of the result of a scientific validation study it is not known at what concentration level Michigan State Police can reliably detect the presence of THC.