

Validation Review

Procedure Split vs. splitless Injection mode study with the
Gas Chromatograph - Infrared Spectrometers

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

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

Examiner (if applicable) [Signature] Rachel Strandquist Date 10-28-16

Unit Supervisor Glenn B. Everett Date 10-26-16

Technical Leader (if different than Supervisor) Glenn B. Everett Date 10-26-16

Crime Laboratory Regional Supervisor [Signature] Date 10/31/16

Quality Assurance Manager CUD [Signature] Date 10/28/16

*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.

Validation Study: Split vs. Splitless Injection Mode with the Gas Chromatograph-Infrared Spectrometers

Nashville Lab, 2016

1. Introduction:

The purpose of this validation study is to verify the performance of forensic chemistry drug identification with different injection modes: split (current mode—Split flow ratio of 2 currently) vs. splitless (new mode). This study will verify the performance and accuracy of identifications by acquiring a standard mix of cocaine and hydrocodone with both modes and conducting a side-by-side comparison of the data. This study will show that utilizing the splitless injection mode increases sample signal intensity while still allowing for baseline resolution and identification of compounds.

2. Objective:

Demonstrate the operation of the gas chromatograph column and infrared detector and the accuracy of the infrared spectra obtained using splitless mode.

3. Materials:

Standard	Company	Lot Number
Standard Mix (Prepared by forensic scientist) • Cocaine HCl • Hydrocodone bitartrate	• Sigma • Merck	• 50k1124 • V1526

Table 1: Standards Utilized during study and corresponding information about standards

4. Methods/Procedures:

GC/IR #1 (Short Column)	
Method name	Method Parameters
"7s"	90-270C for 10 mins. @70C/min. rate

Table 2: Previously validated method used on GC/IR#1

GC/IR #2 (Long Column)	
Method name	Method Parameters
"5L"	120-270C for 10 mins. @70C/min. rate

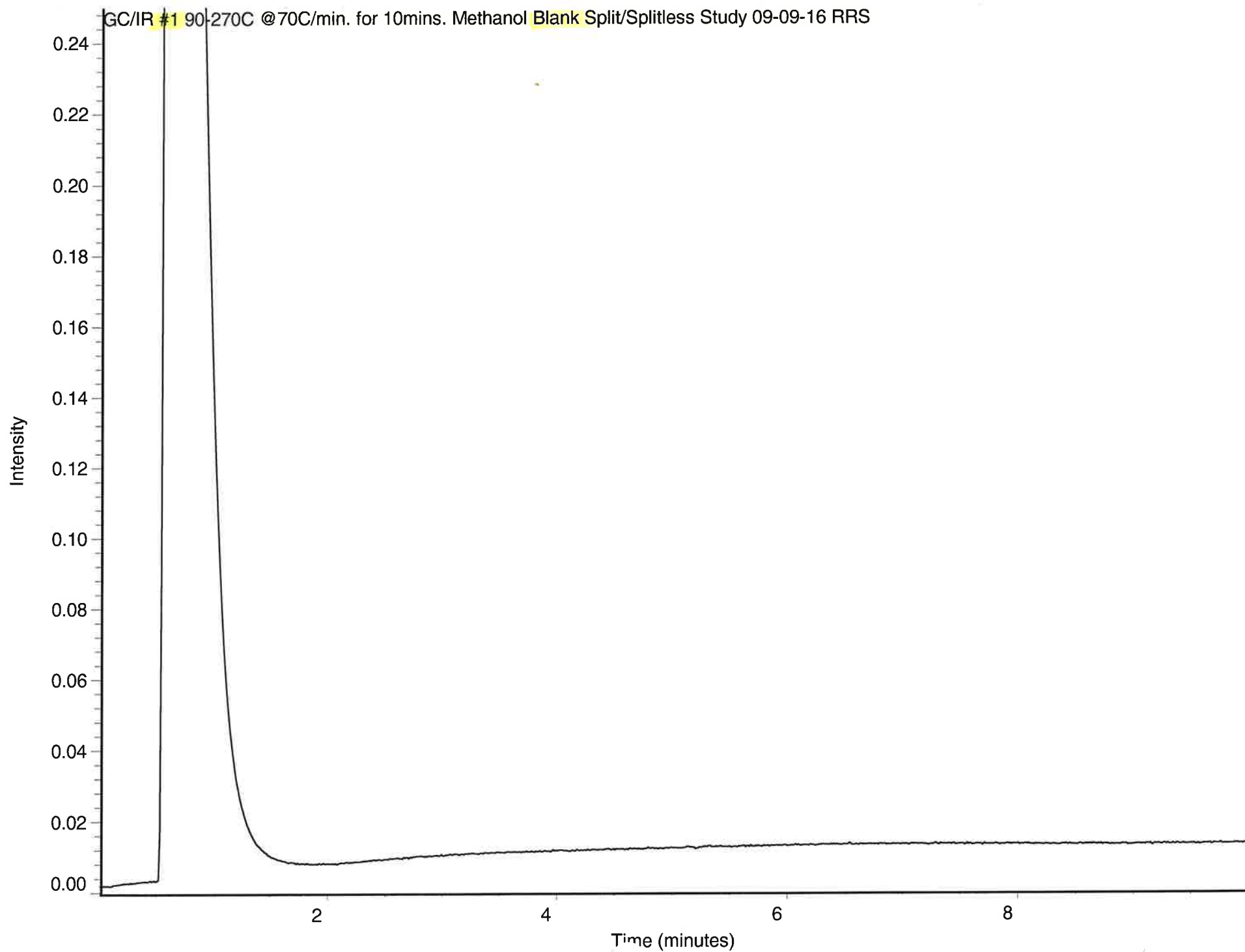
Table 3: Previously validated method used on GC/IR#2

GC/IR Hogwarts	
Method name	Method Parameters
"MAN120TO270"	90-270C for 10 mins. @70C/min. rate—Split
"SLMAN120TO270"	90-270C for 10 mins. @70C/min. rate—Splitless

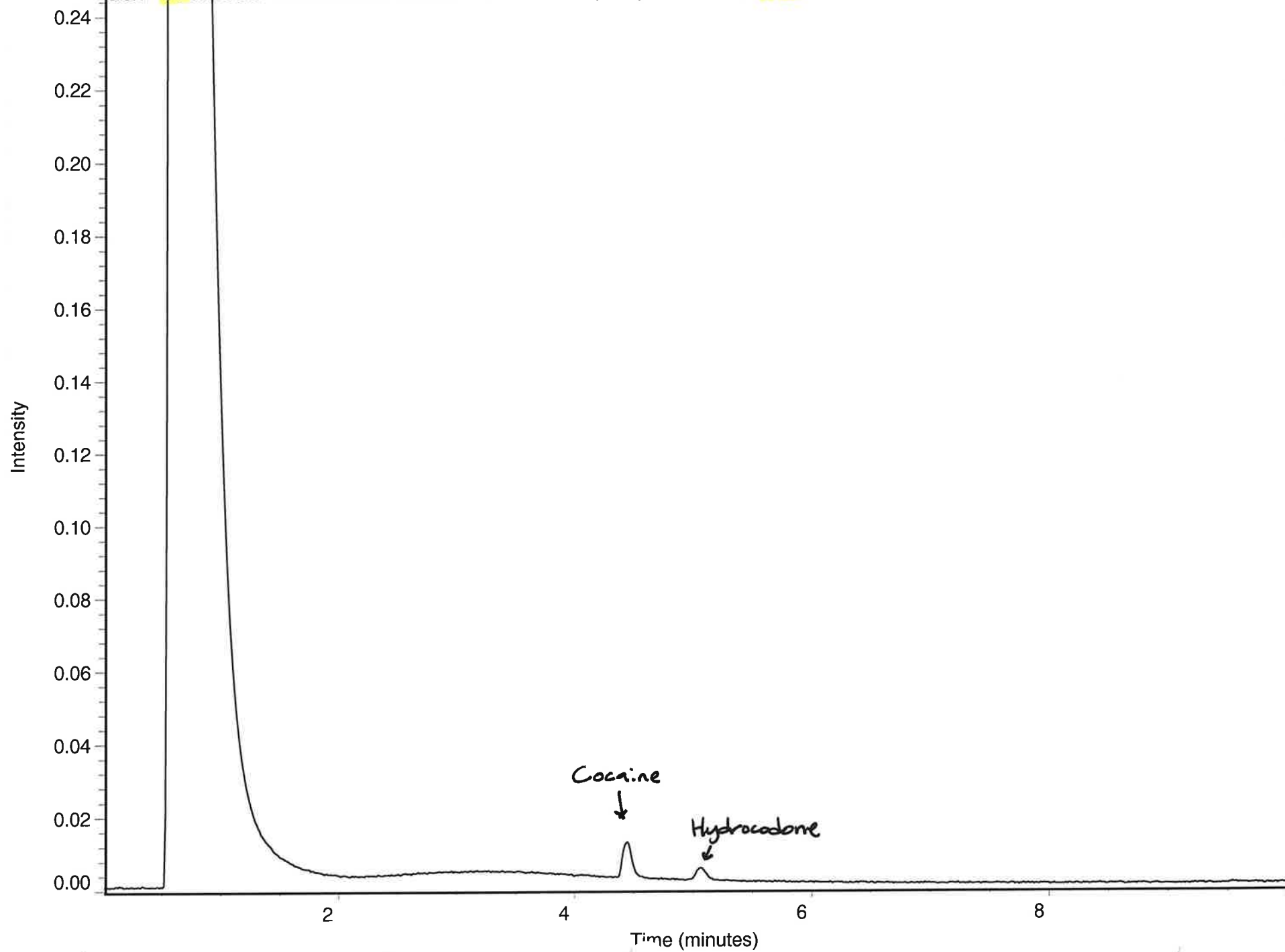
Table 4: Previously validated methods used on Hogwarts

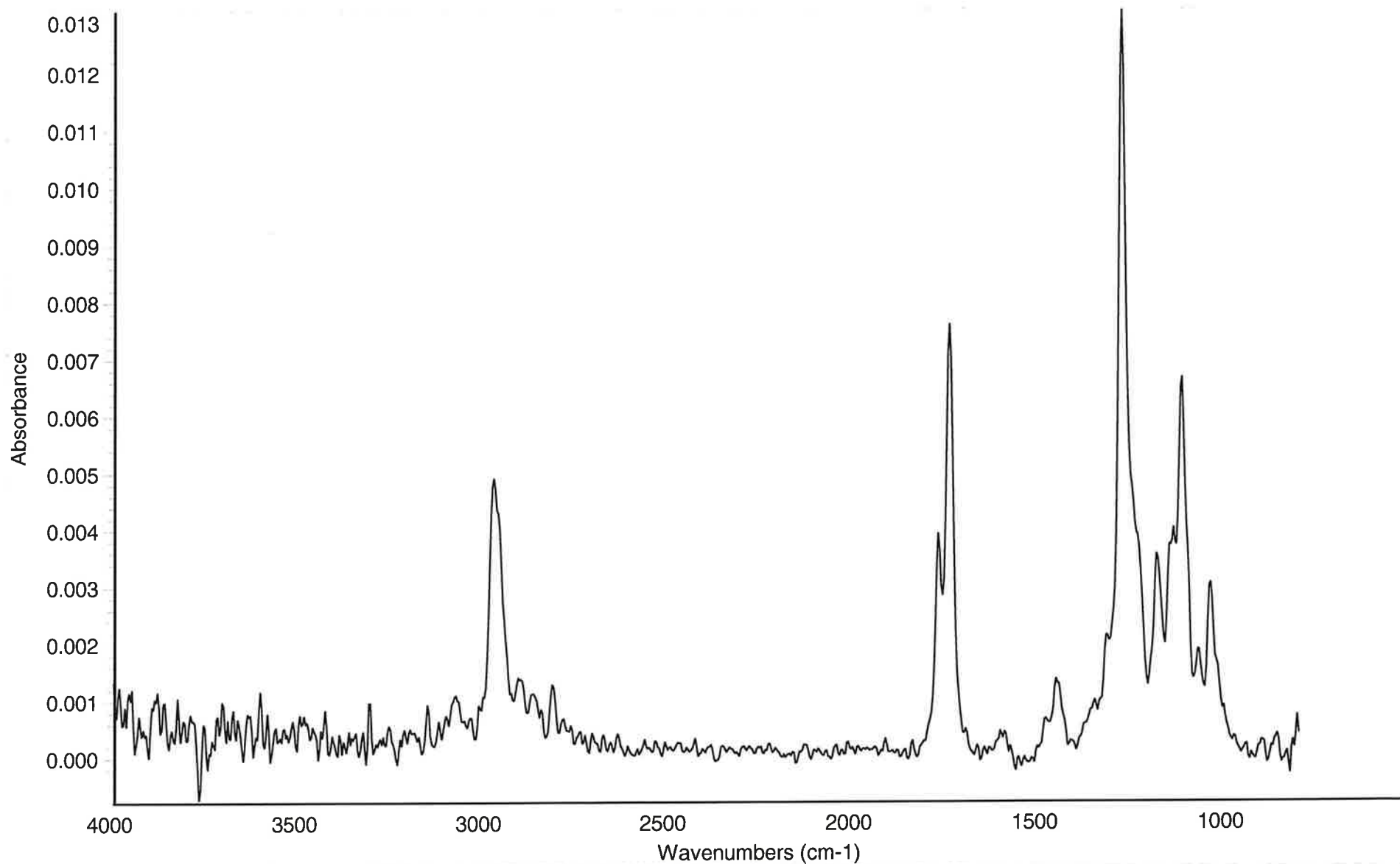
Procedure: Two (2) microliters of the drug standard mix identified in the above table were injected on the respective instrument with the respective method. The standard mix was first acquired with the

GC/IR #1 90-270C @70C/min. for 10mins. Methanol Blank Split/Splitless Study 09-09-16 RRS



GC/IR #1 90-270C @70C/min. for 10mins. Methanol Mix Split/Splitless Study- Split Mode 09-09-16 RRS





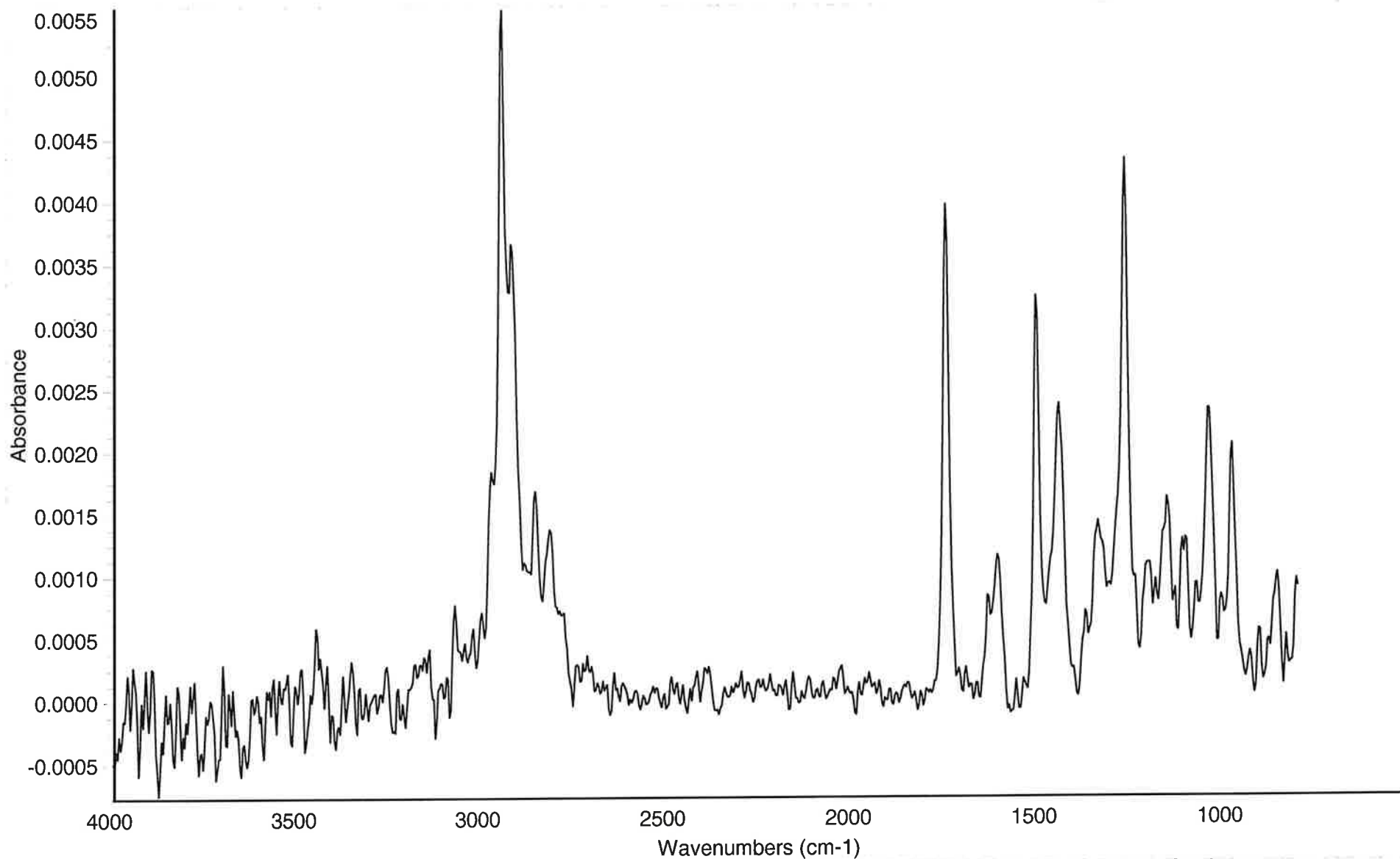
GC/IR #1 90-270C @70C/min. for 10mins. Methanol Mix Split/Splitless Study- Split Mode 09-09-16 RRS Cocaine

Column 19091J-413 5 Meters
Column flow 1.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT-High D* Number of sample scans: 20
Beamsplitter: KBr Number of background scans: 10
Source: IR Resolution: 8.000
Sample gain: 2.0
Mirror velocity: 3.1647
Aperture: 100.00

GC/FTIR 1

Collection time: Fri Sep 09 15:21:46 2016 (GMT-05:00)



GC/IR #1 90-270C @70C/min. for 10mins. Methanol Mix Split/Splitless Study- Split Mode 09-09-16 RRS Hydrocodone

Column 19091J-413 5 Meters
Column flow 1.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT-High D* Number of sample scans: 24
Beamsplitter: KBr Number of background scans: 10
Source: IR Resolution: 8.000
Sample gain: 2.0
Mirror velocity: 3.1647
Aperture: 100.00

GC/FTIR 1

Collection time: Fri Sep 09 15:29:54 2016 (GMT-05:00)

GC/IR #1 90-270C @70C/min. for 10mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-09-16 RRS

Intensity

0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00

Cocaine



Hydrocodone



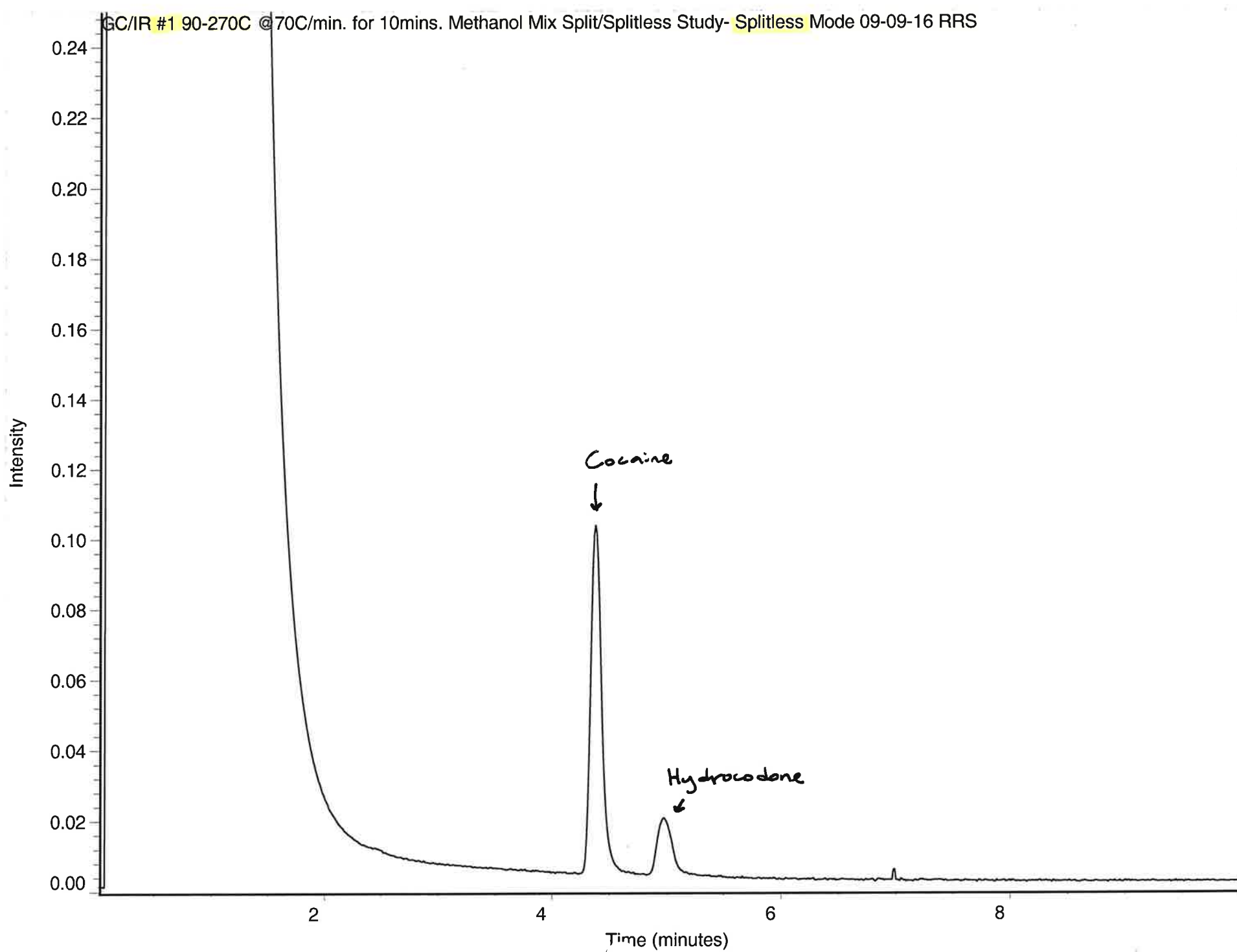
Time (minutes)

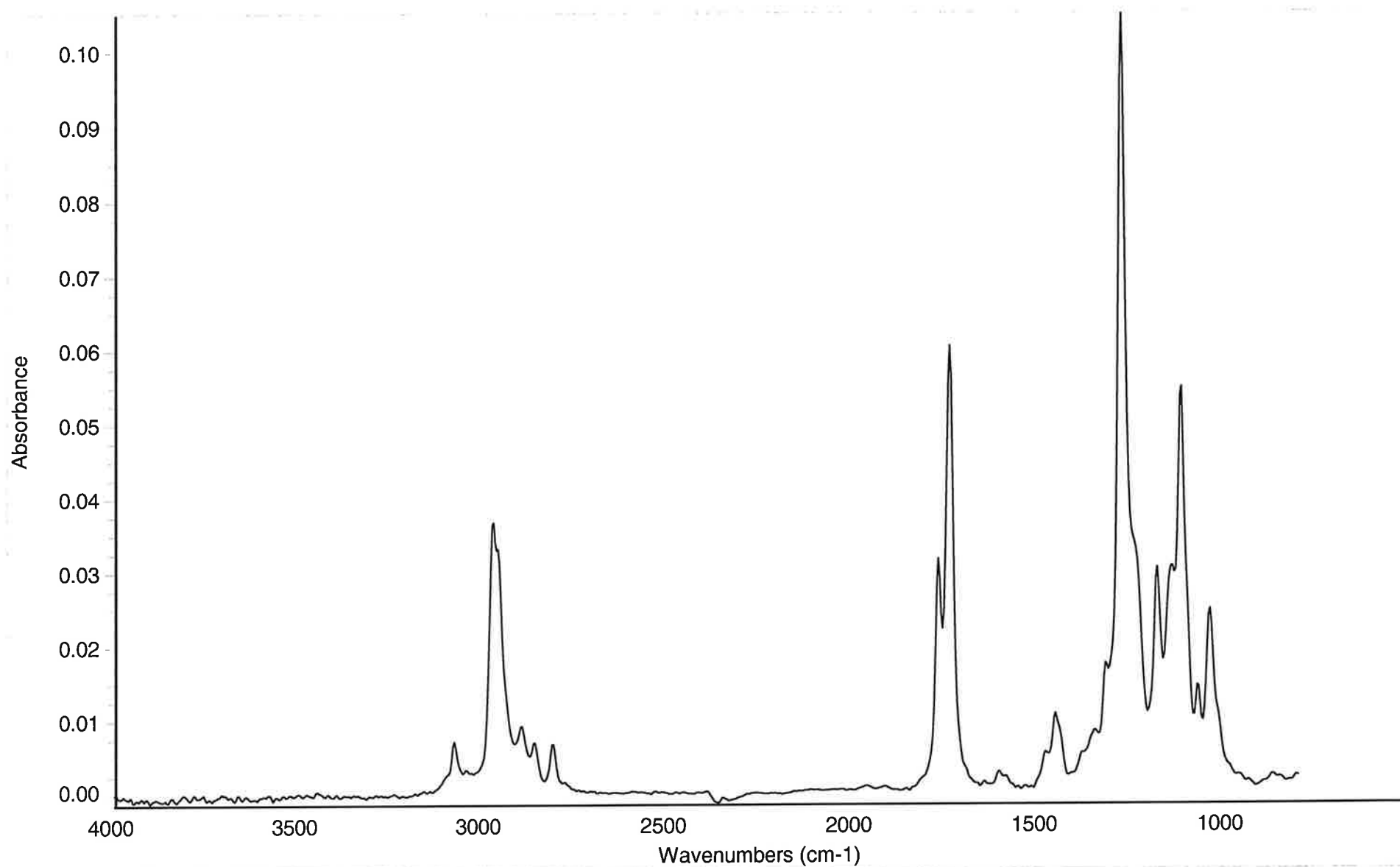
2

4

6

8





GC/IR #1 90-270C @70C/min. for 10mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-09-16 RRS Cocaine

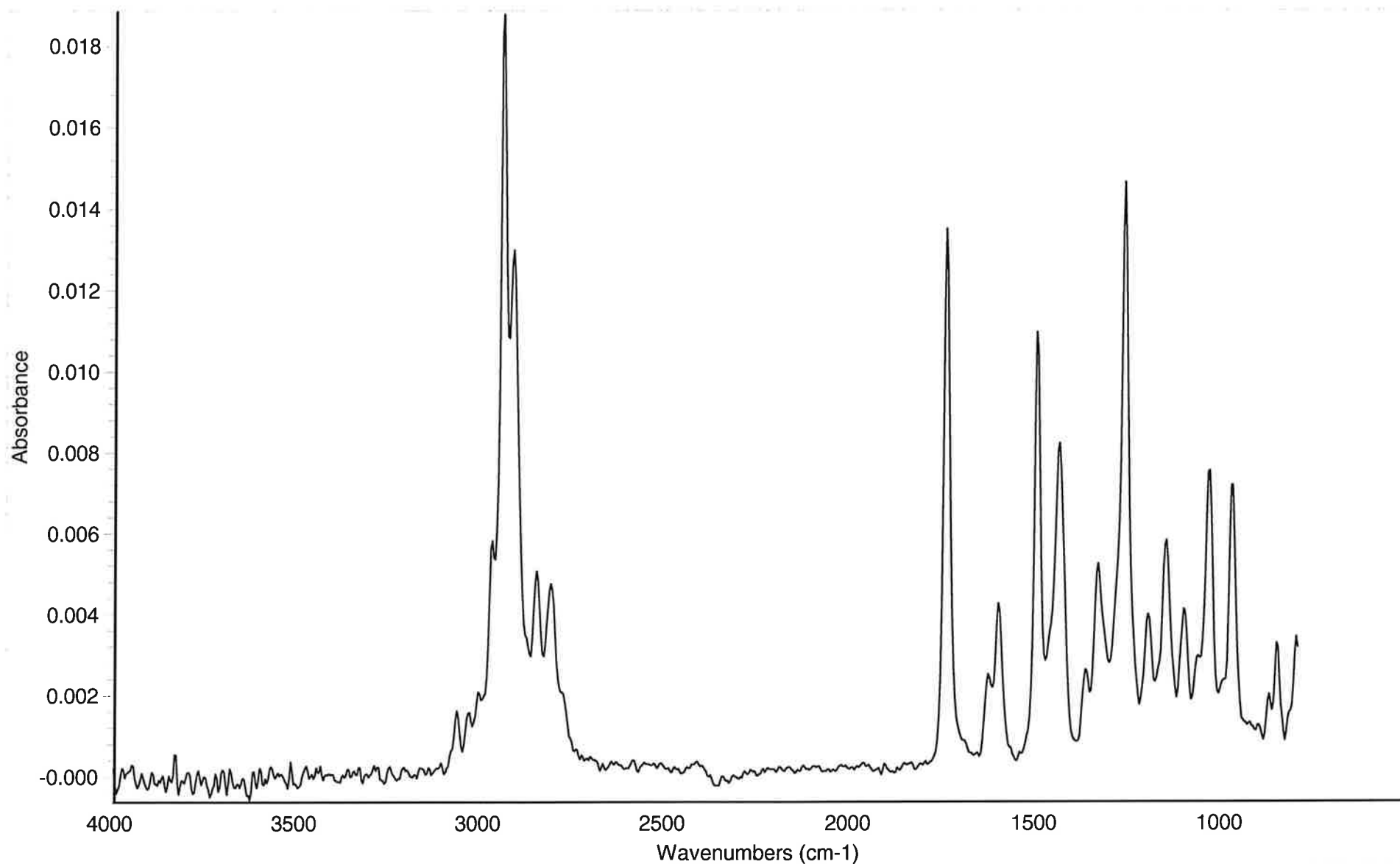
Column 19091J-413 5 Meters
Column flow 1.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT-High D*
Beamsplitter: KBr
Source: IR

Number of sample scans: 32
Number of background scans: 10
Resolution: 8.000
Sample gain: 2.0
Mirror velocity: 3.1647
Aperture: 100.00

GC/FTIR 1

Collection time: Fri Sep 09 15:49:58 2016 (GMT-05:00)



GC/IR #1 90-270C @70C/min. for 10mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-09-16 RRS Hydrocodone

Column 19091J-413 5 Meters
Column flow 1.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

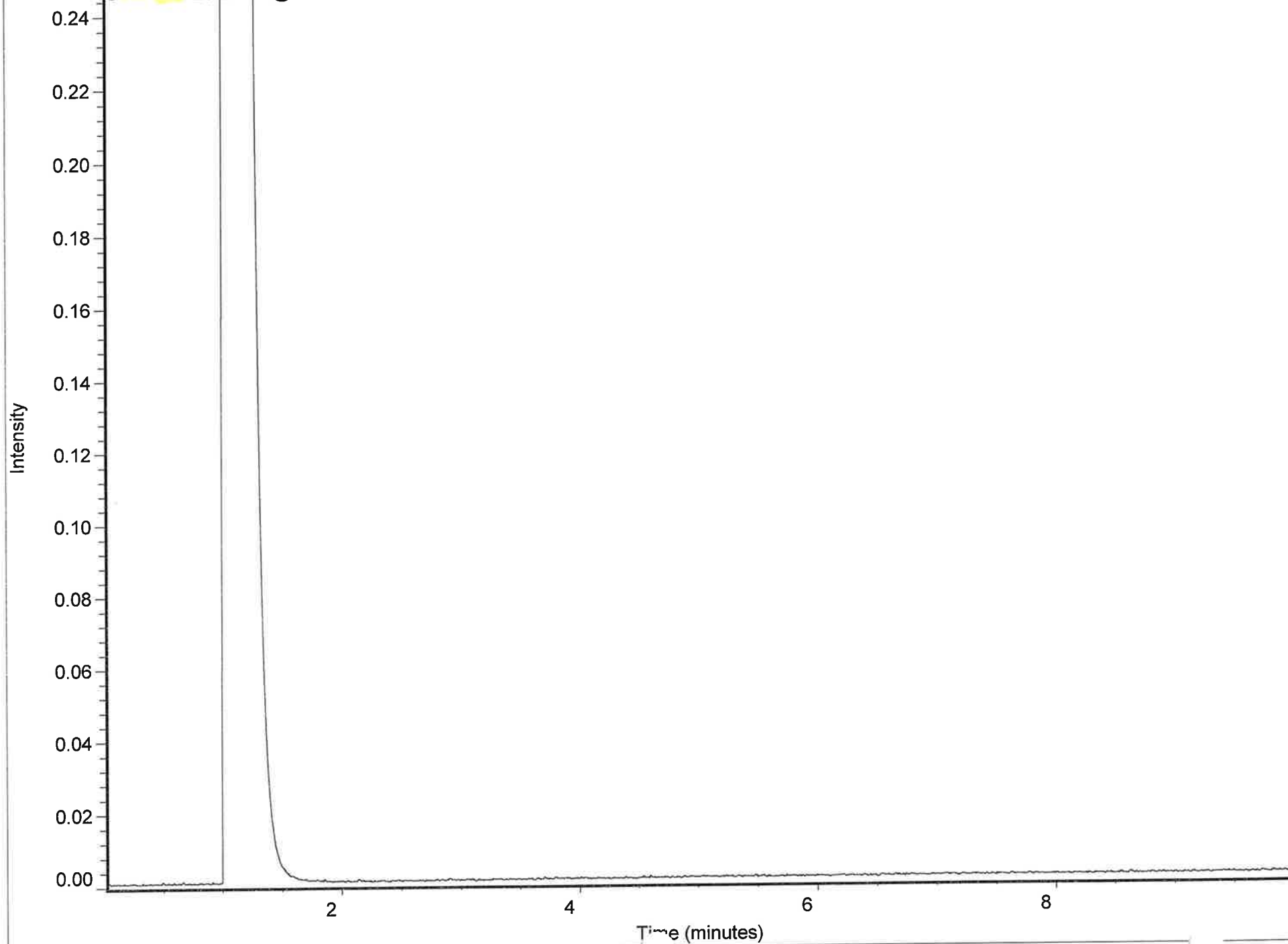
Detector: MCT-High D*
Beamsplitter: KBr
Source: IR

Number of sample scans: 44
Number of background scans: 10
Resolution: 8.000
Sample gain: 2.0
Mirror velocity: 3.1647
Aperture: 100.00

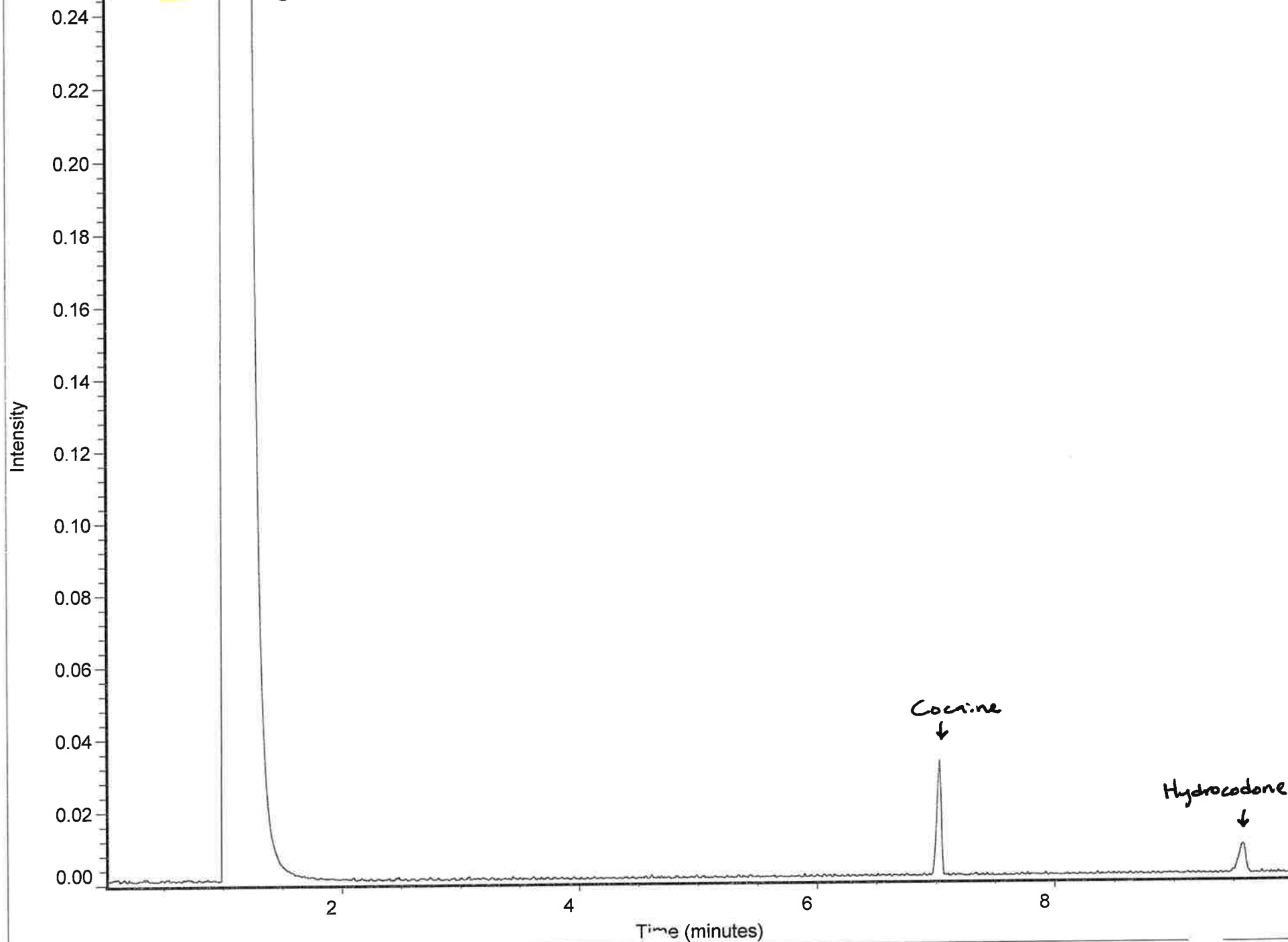
GC/FTIR 1

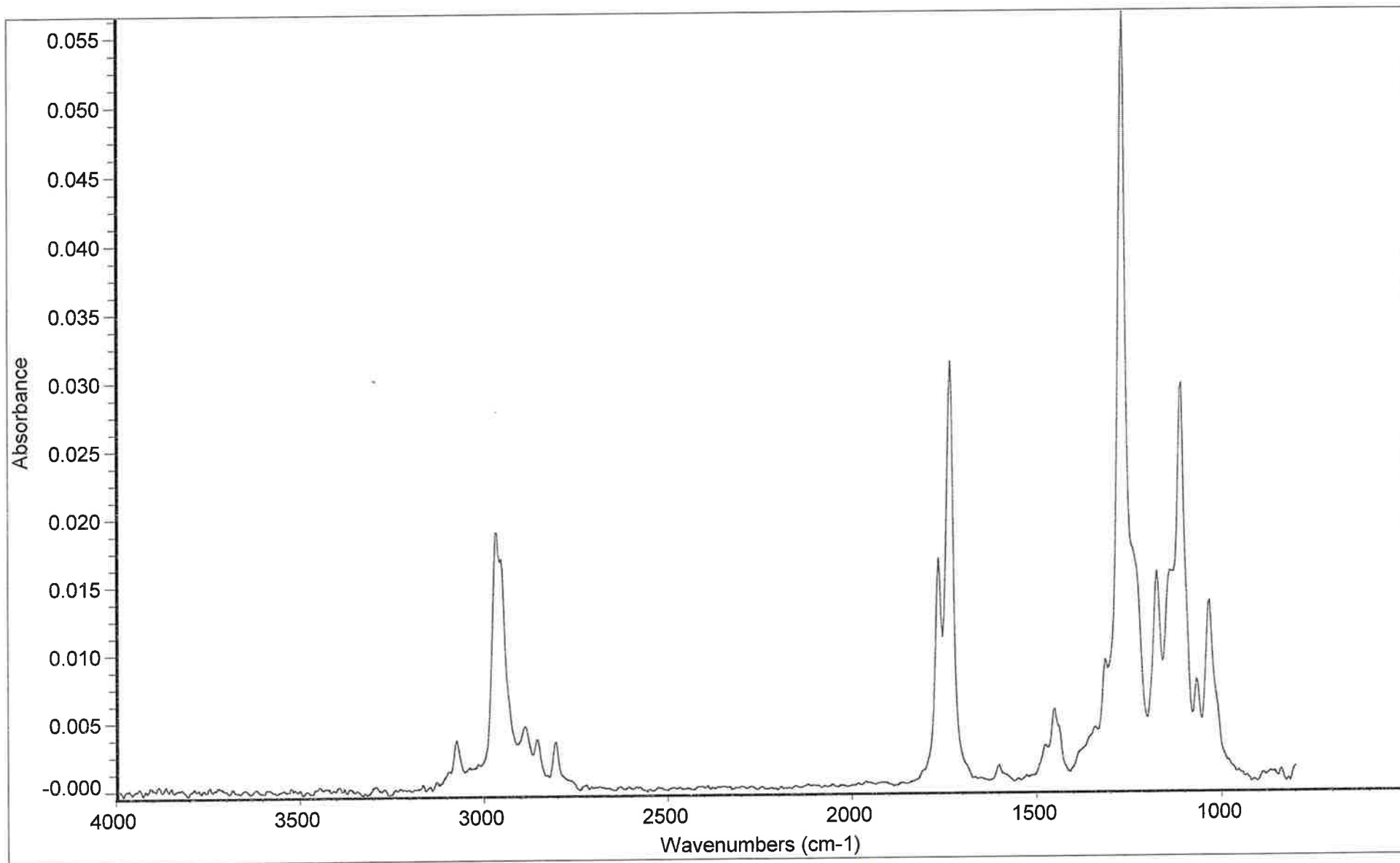
Collection time: Fri Sep 09 16:09:18 2016 (GMT-05:00)

GC/IR#2 120-270C @70C/min. for 10 mins. Methanol Blank Split/Splitless Study 09-09-16 RRS



GC/IR#2 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Split Mode 09-09-16 RRS





GC/IR#2 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Split Mode 09-09-16 RRS Cocaine

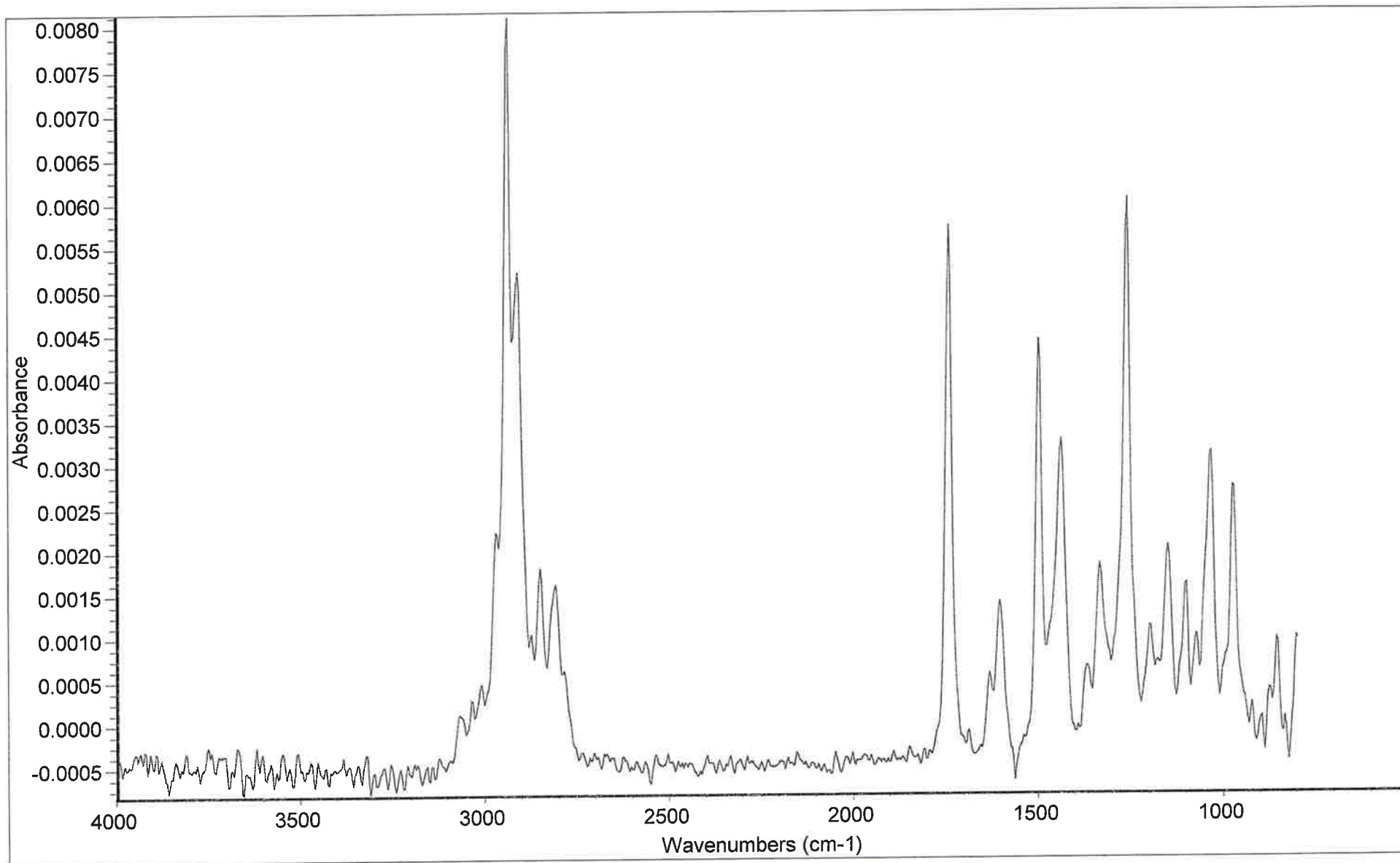
Column 19091Z-213 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT/A
Beamsplitter: KBr
Source: IR

Number of sample scans: 12
Number of background scans: 10
Resolution: 8.000
Sample gain: 2.0
Mirror velocity: 3.1647
Aperture: 100.00

GC/FTIR 2

Collection time: Fri Sep 09 14:35:47 2016 (GMT-06:00)



GC/IR#2 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Split Mode 09-09-16 RRS Hydrocodone

Column 19091Z-213 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

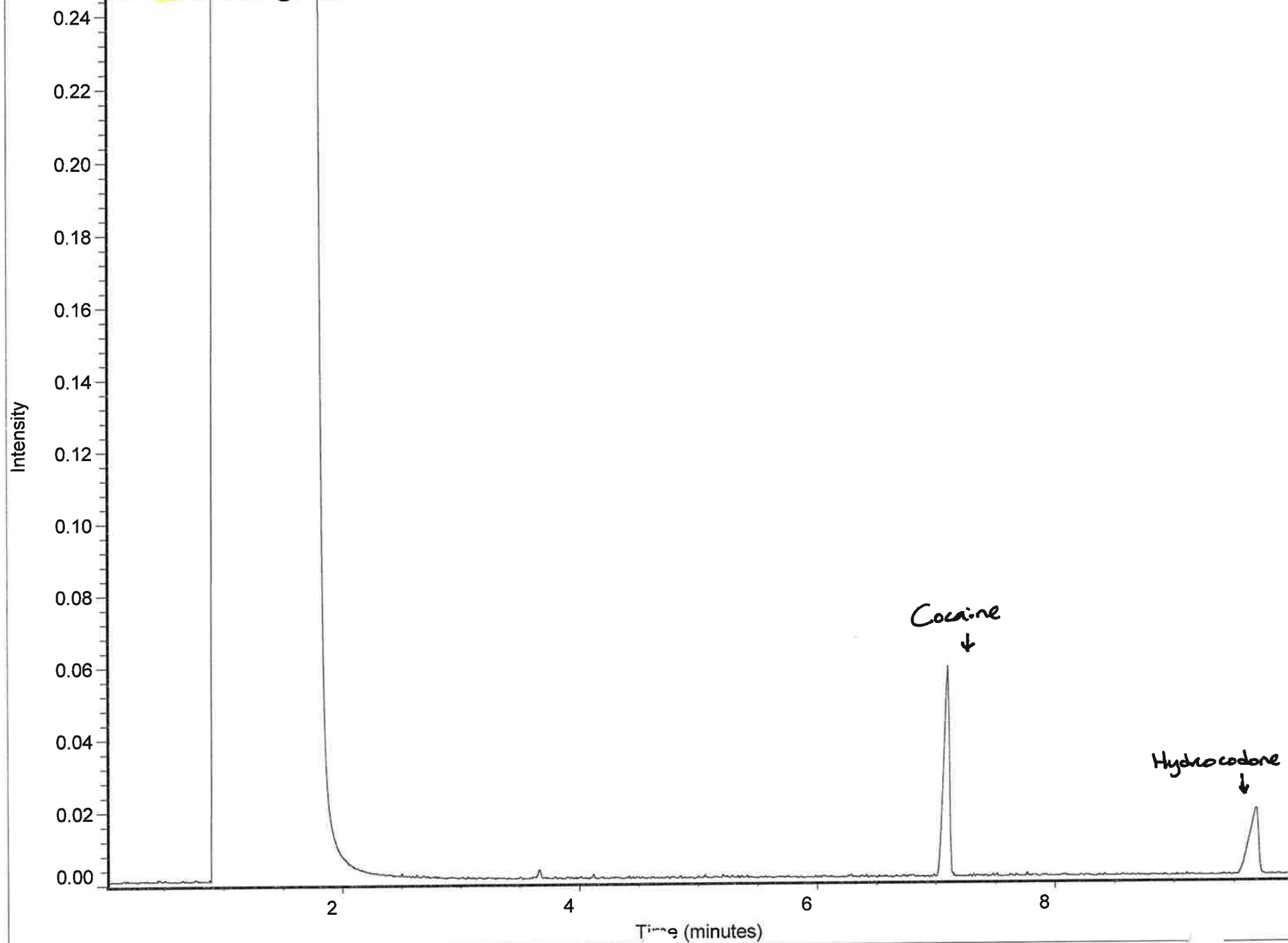
Detector: MCT/A
Beamsplitter: KBr
Source: IR

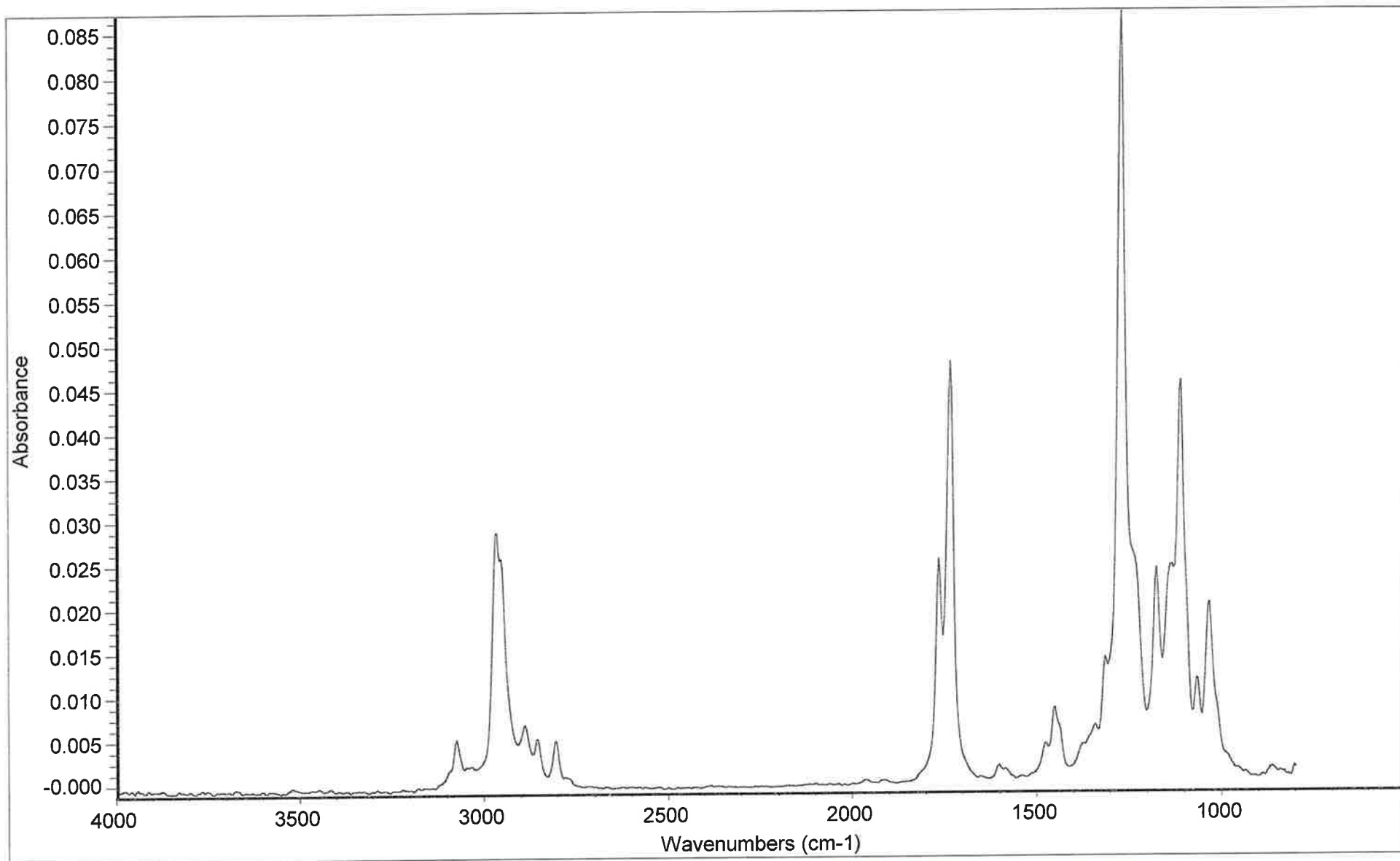
Number of sample scans: 24
Number of background scans: 10
Resolution: 8.000
Sample gain: 2.0
Mirror velocity: 3.1647
Aperture: 100.00

GC/FTIR 2

Collection time: Fri Sep 09 15:11:58 2016 (GMT-06:00)

GC/IR#2 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-09-16 RRS





GC/IR#2 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-09-16 RRS Cocaine

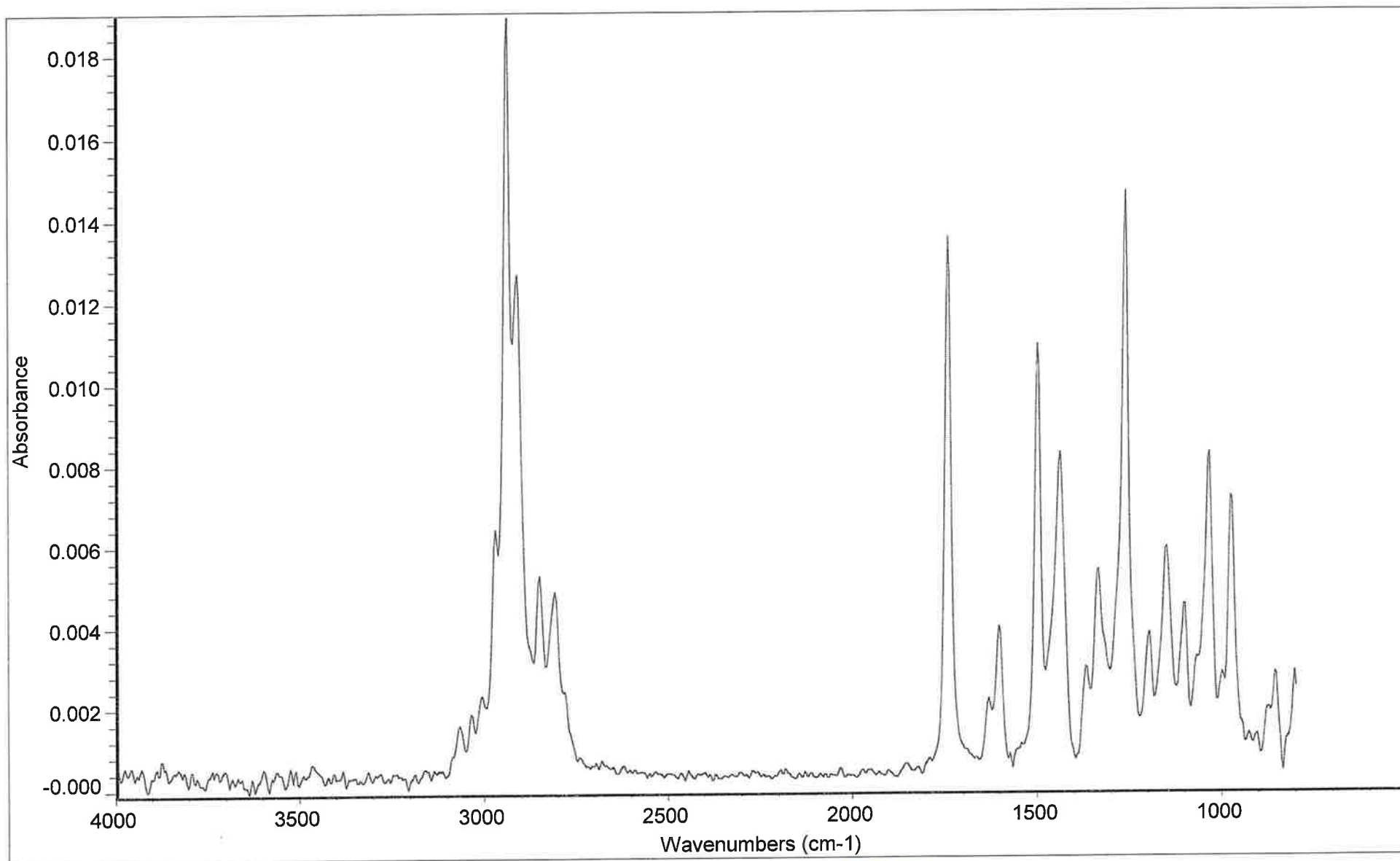
Column 19091Z-213 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT/A
Beamsplitter: KBr
Source: IR

Number of sample scans: 24
Number of background scans: 10
Resolution: 8.000
Sample gain: 2.0
Mirror velocity: 3.1647
Aperture: 100.00

GC/FTIR 2

Collection time: Fri Sep 09 15:12:22 2016 (GMT-06:00)



GC/IR#2 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-09-16 RRS Hydrocodone

Column 19091Z-213 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

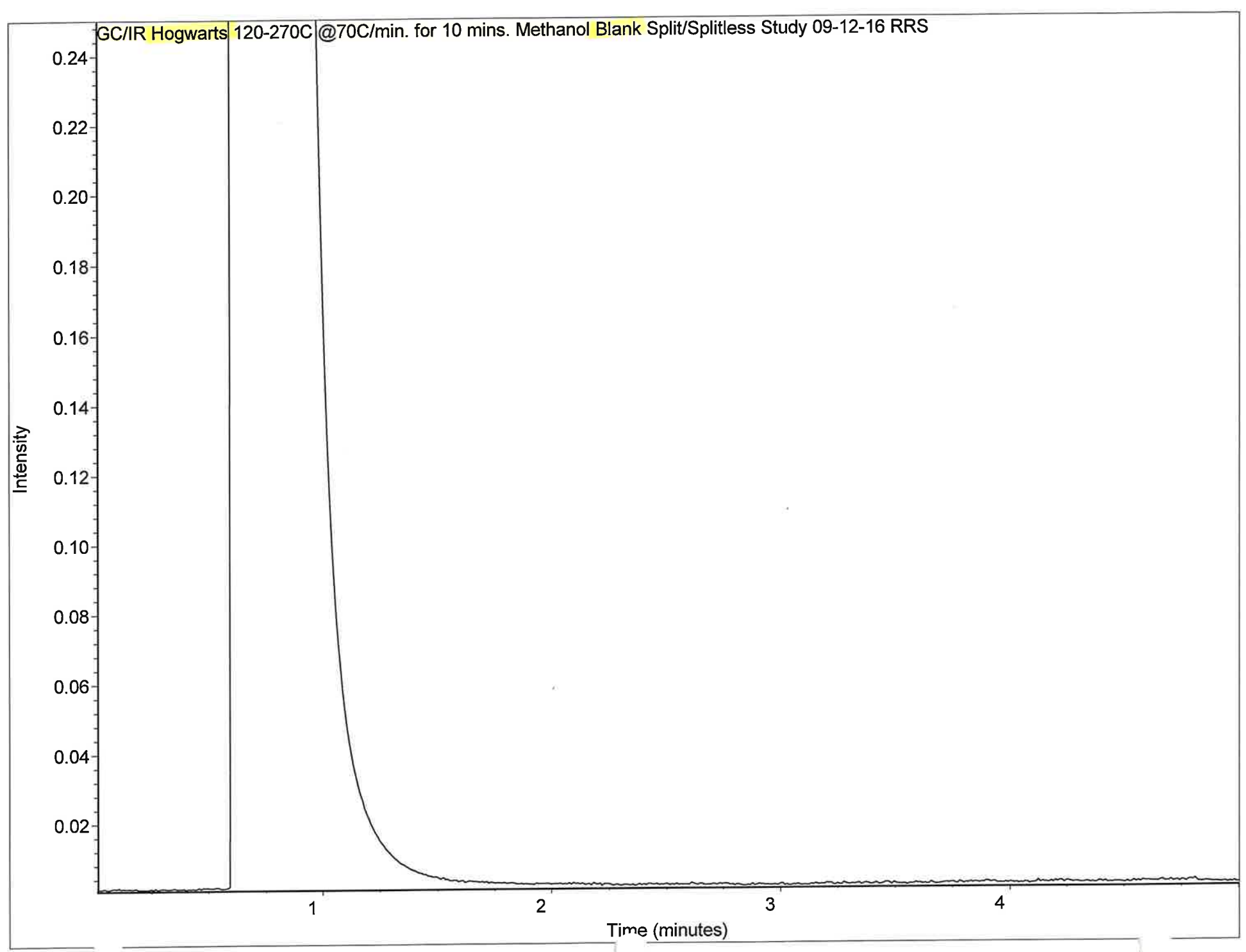
Detector: MCT/A
Beamsplitter: KBr
Source: IR

Number of sample scans: 24
Number of background scans: 10
Resolution: 8.000
Sample gain: 2.0
Mirror velocity: 3.1647
Aperture: 100.00

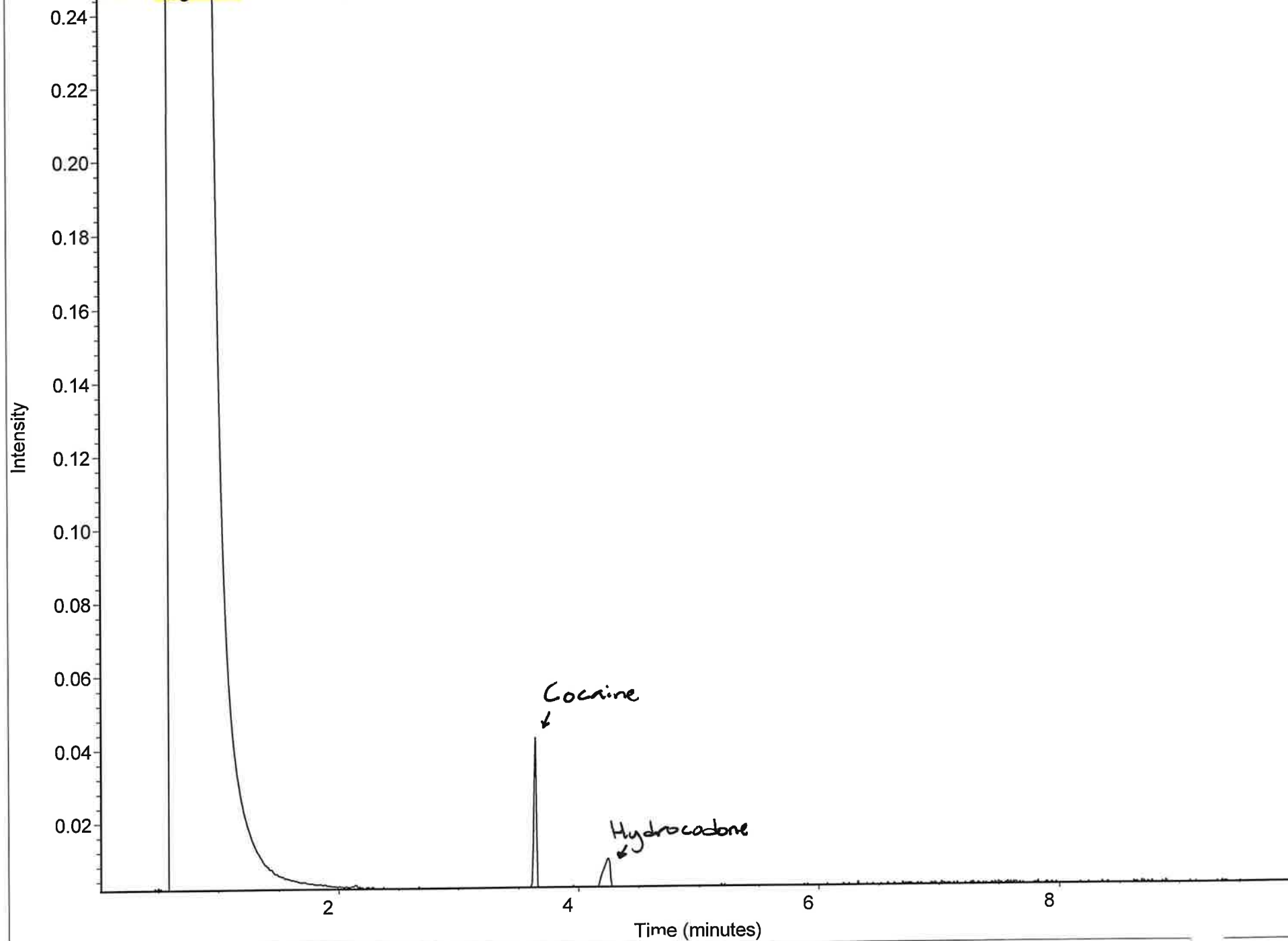
GC/FTIR 2

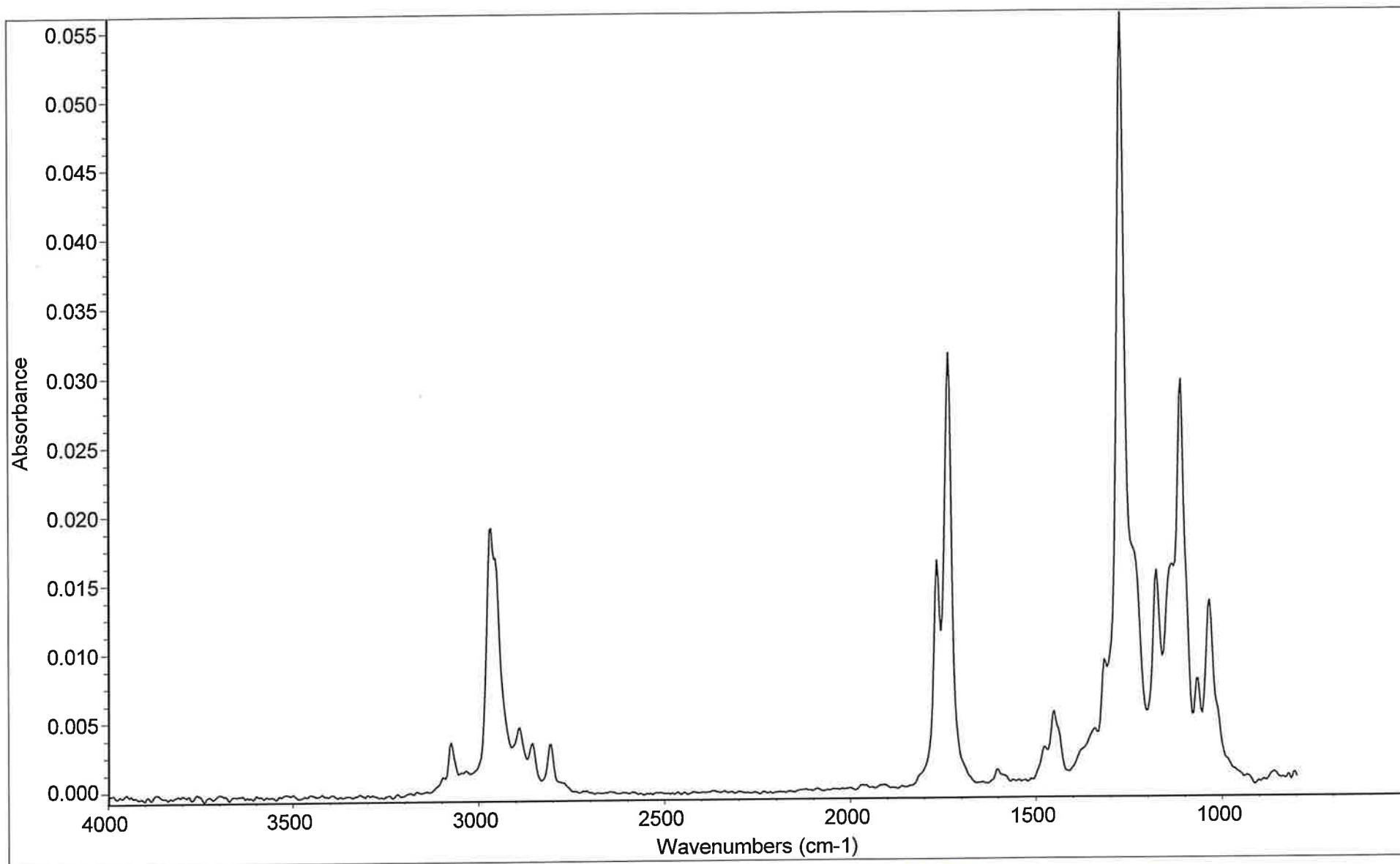
Collection time: Fri Sep 09 15:27:56 2016 (GMT-06:00)

GC/IR Hogwarts 120-270C @70C/min. for 10 mins. Methanol Blank Split/Splitless Study 09-12-16 RRS



GC/IR Hogwarts 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Split Mode 09-12-16 RRS





GC/IR Hogwarts 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Split Mode 09-12-16 RRS Cocaine

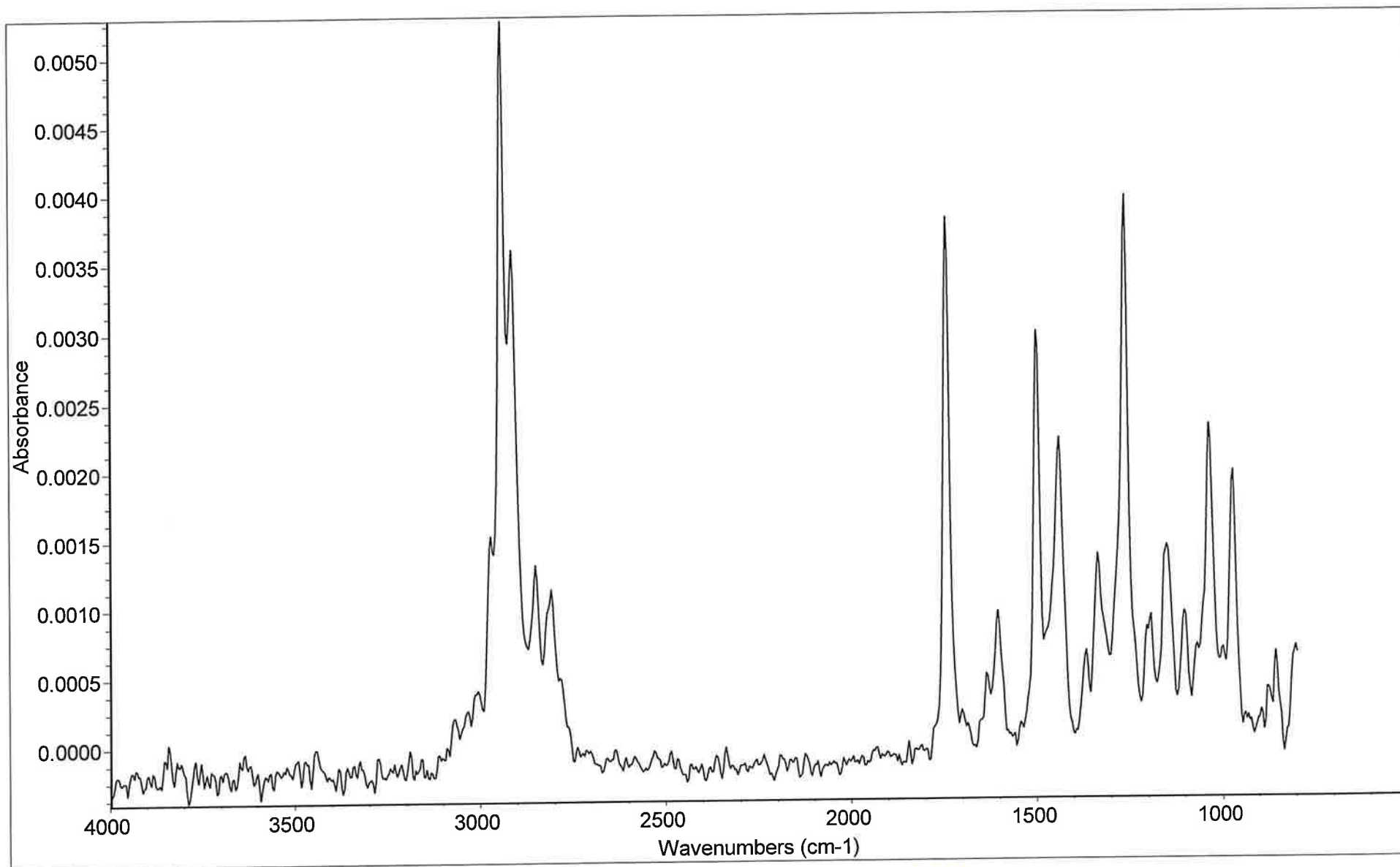
Column USF668212H 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT/A
Beamsplitter: KBr
Source: IR

Number of sample scans: 12
Number of background scans: 64
Resolution: 8.000
Sample gain: 1.0
Optical velocity: 3.7974
Aperture: 100.00

HOGWARTS

Collection time: Mon Sep 12 08:56:16 2016 (GMT-05:00)



GC/IR Hogwarts 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Split Mode 09-12-16 RRS Hydrocodone

Column USF668212H 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

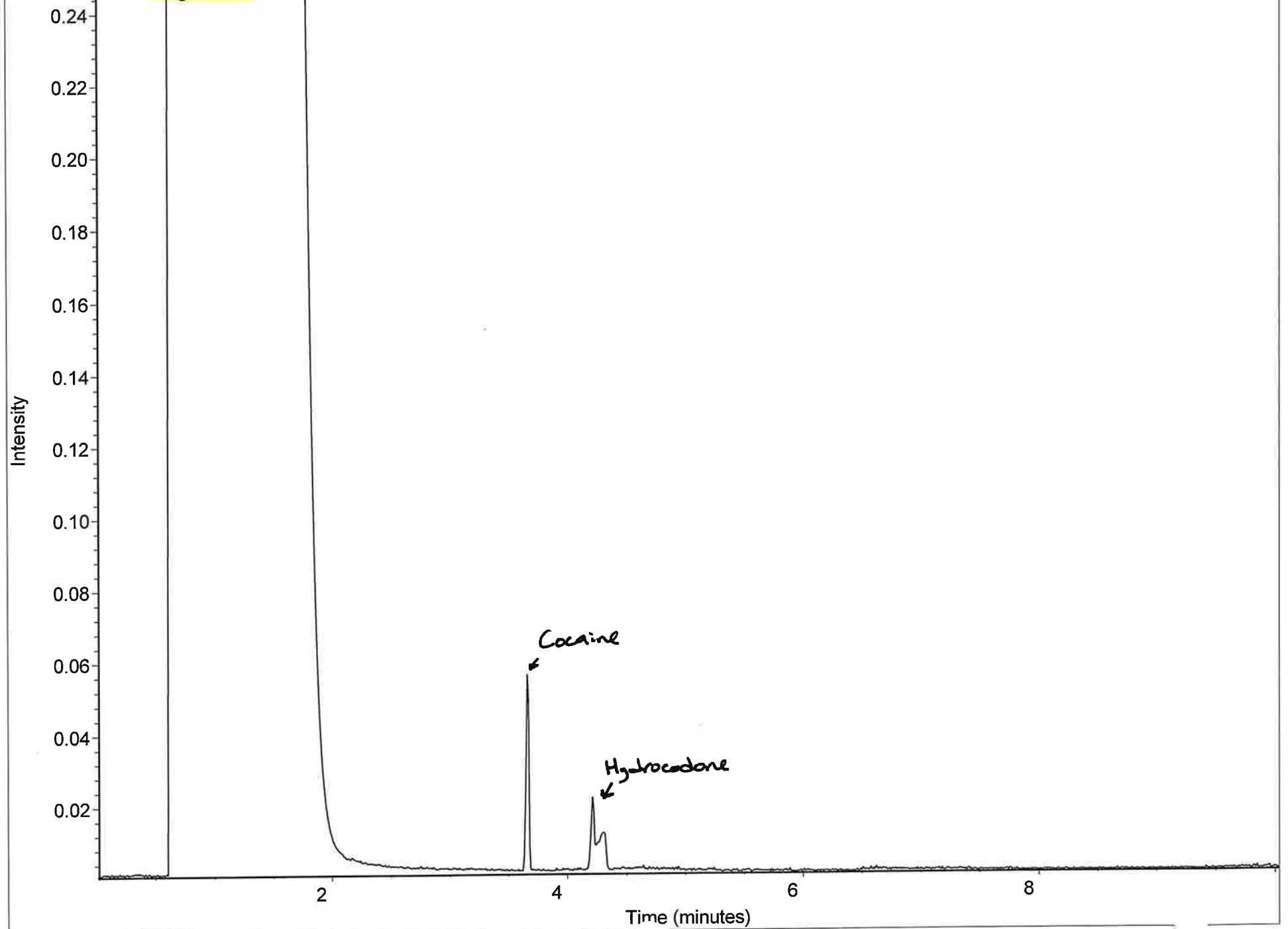
Detector: MCT/A
Beamsplitter: KBr
Source: IR

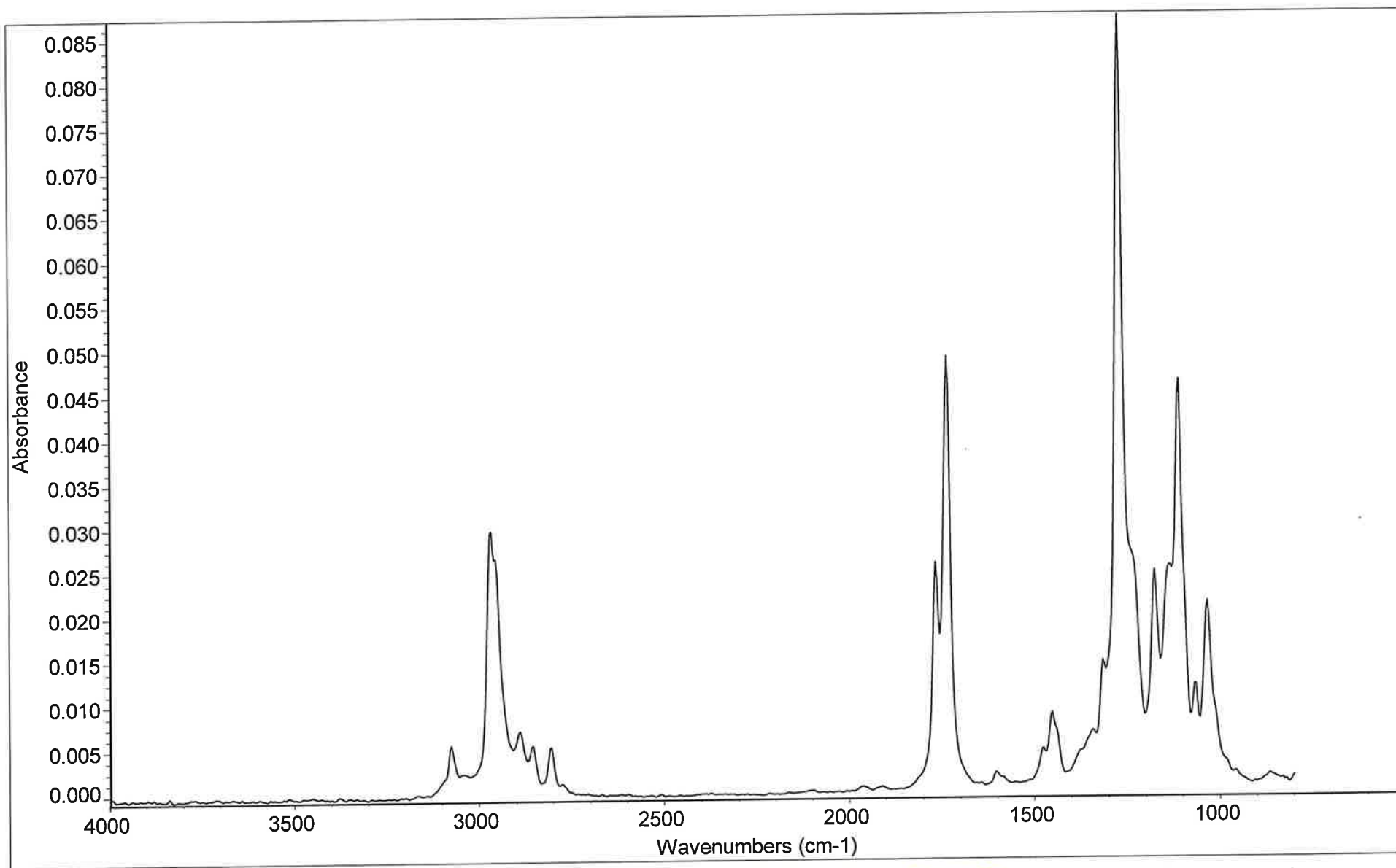
Number of sample scans: 20
Number of background scans: 64
Resolution: 8.000
Sample gain: 1.0
Optical velocity: 3.7974
Aperture: 100.00

HOGWARTS

Collection time: Mon Sep 12 08:56:16 2016 (GMT-05:00)

GC/IR Hogwarts 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-12-16 RRS





GC/IR Hogwarts 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-12-16 RRS Cocaine

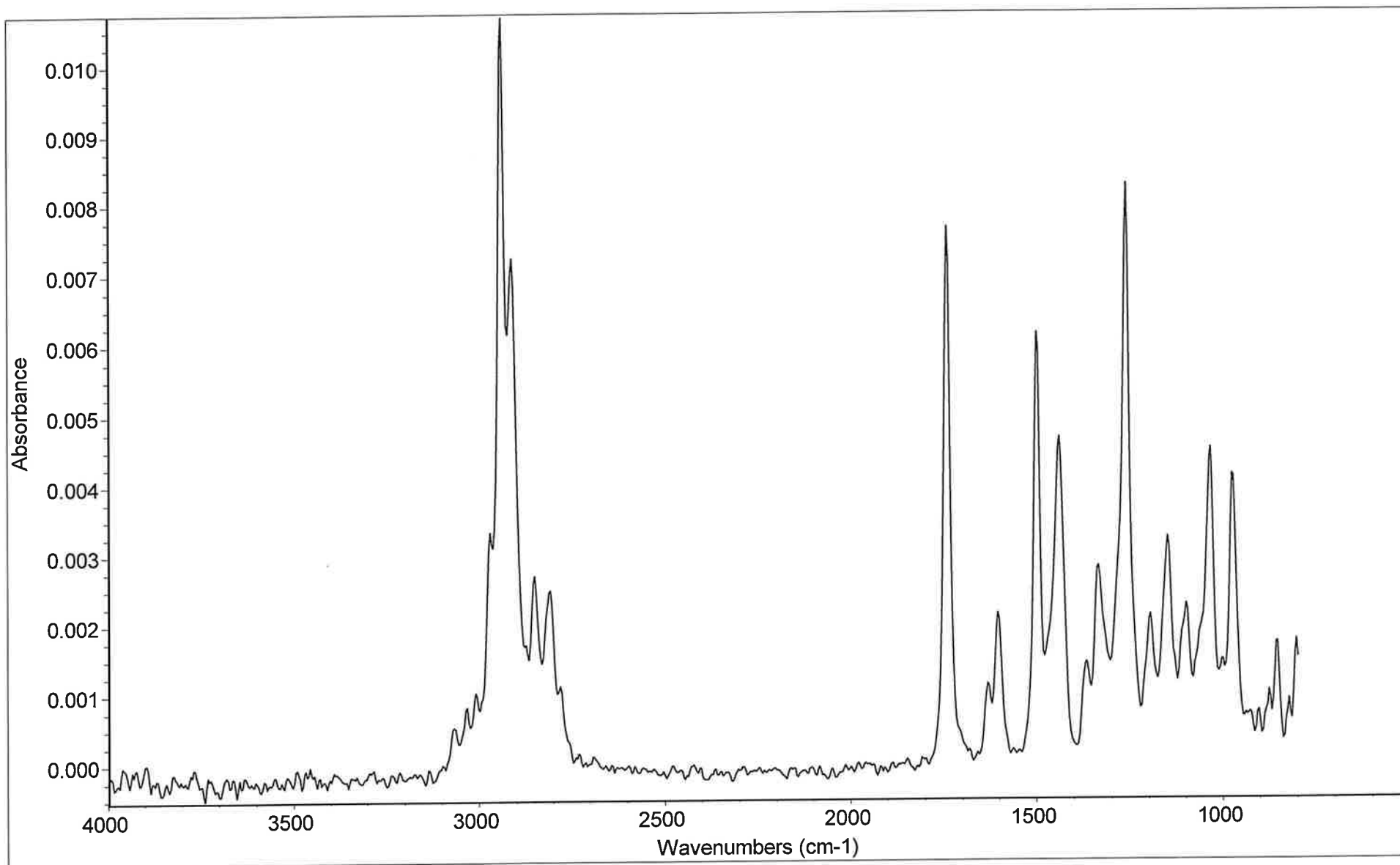
Column USF668212H 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT/A
Beamsplitter: KBr
Source: IR

Number of sample scans: 12
Number of background scans: 64
Resolution: 8.000
Sample gain: 1.0
Optical velocity: 3.7974
Aperture: 100.00

HOGWARTS

Collection time: Mon Sep 12 09:08:20 2016 (GMT-05:00)



GC/IR Hogwarts 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Splitless Mode 09-12-16 RRS Hydrocodone

Column USF668212H 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

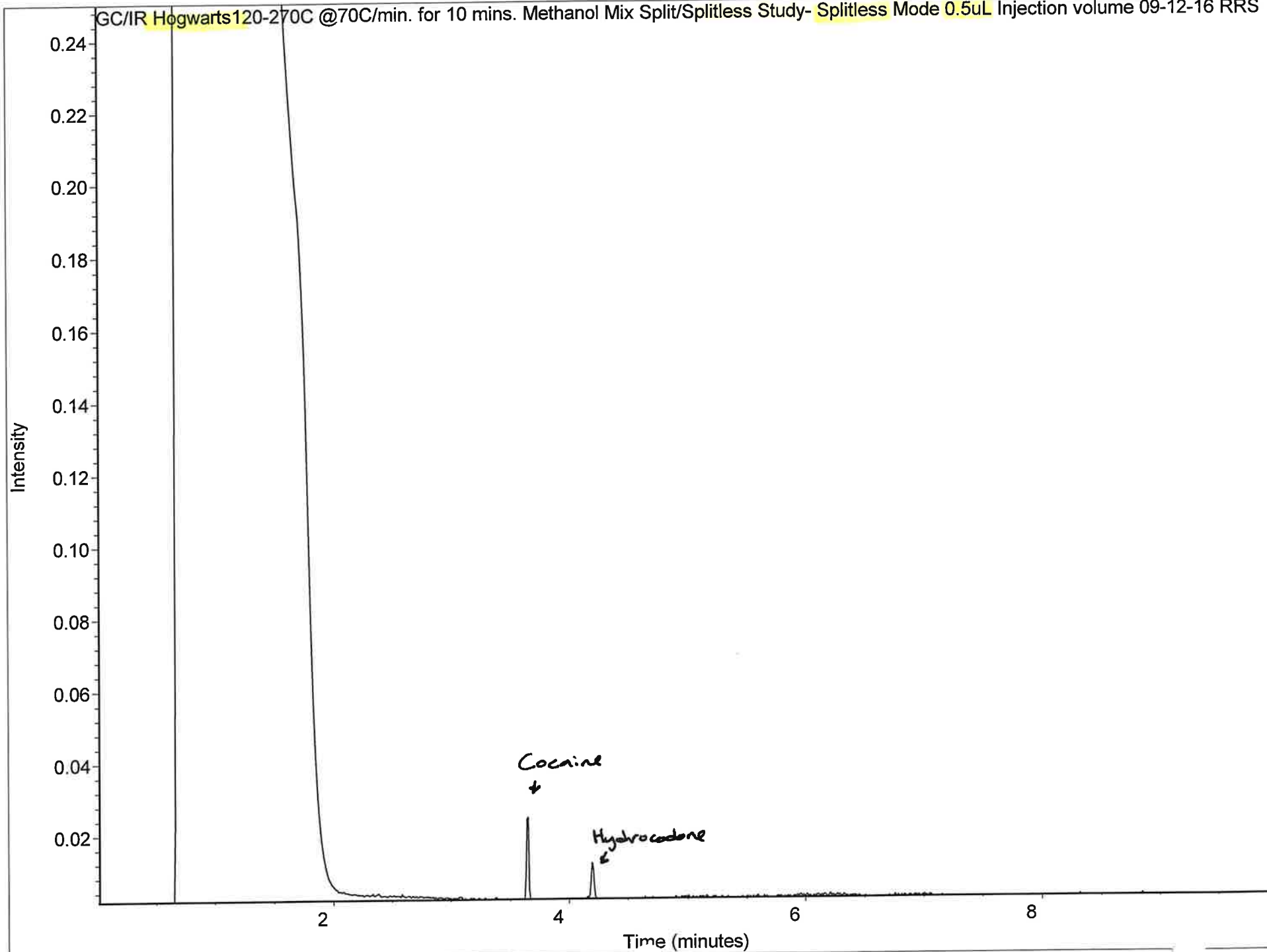
Detector: MCT/A
Beamsplitter: KBr
Source: IR

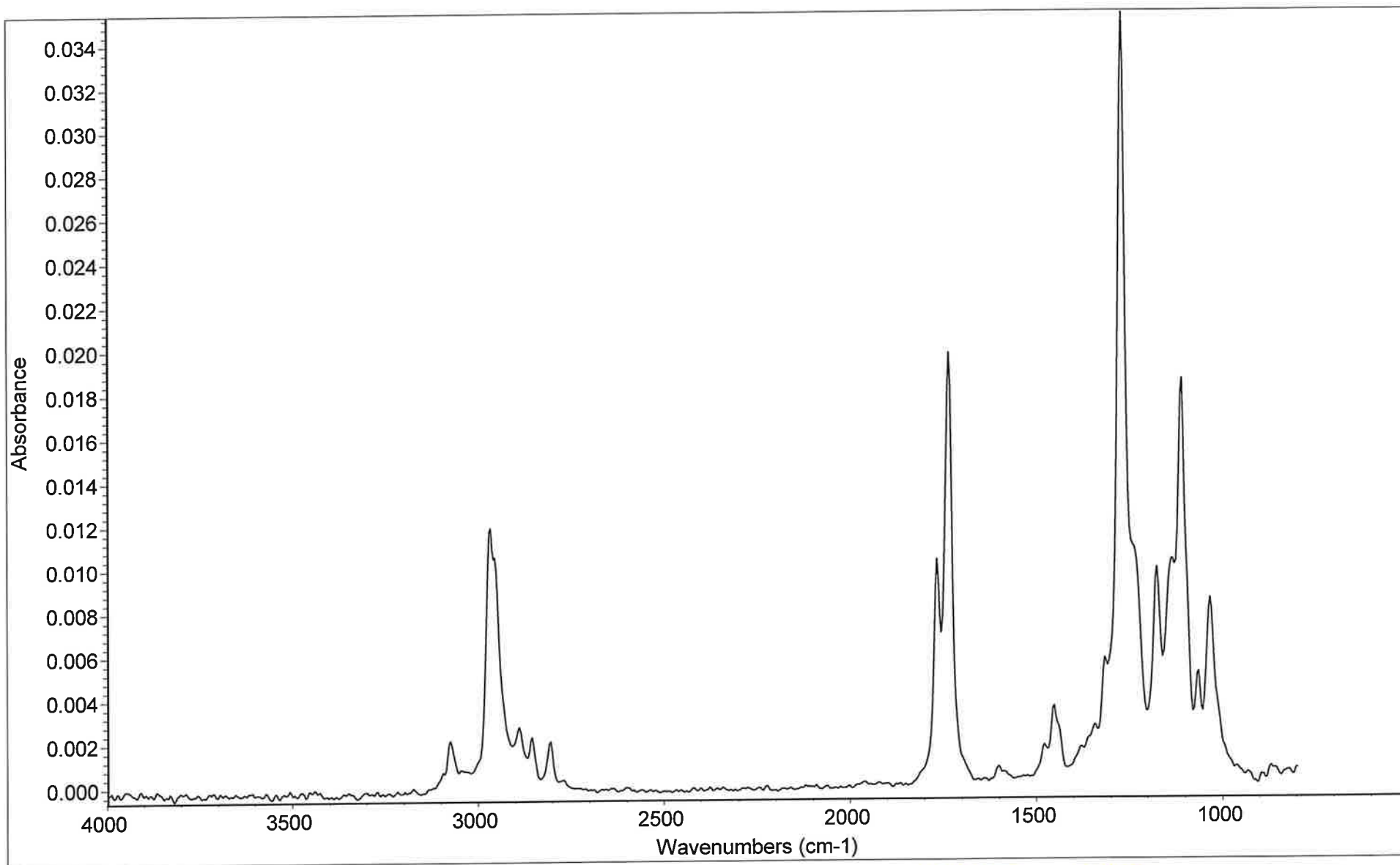
Number of sample scans: 16
Number of background scans: 64
Resolution: 8.000
Sample gain: 1.0
Optical velocity: 3.7974
Aperture: 100.00

HOGWARTS

Collection time: Mon Sep 12 09:08:20 2016 (GMT-05:00)

GC/IR Hogwarts 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Splitless Mode 0.5uL Injection volume 09-12-16 RRS





GC/IR **Hogwarts** 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- Splitless Mode 0.5uL injection volume 09-12-16

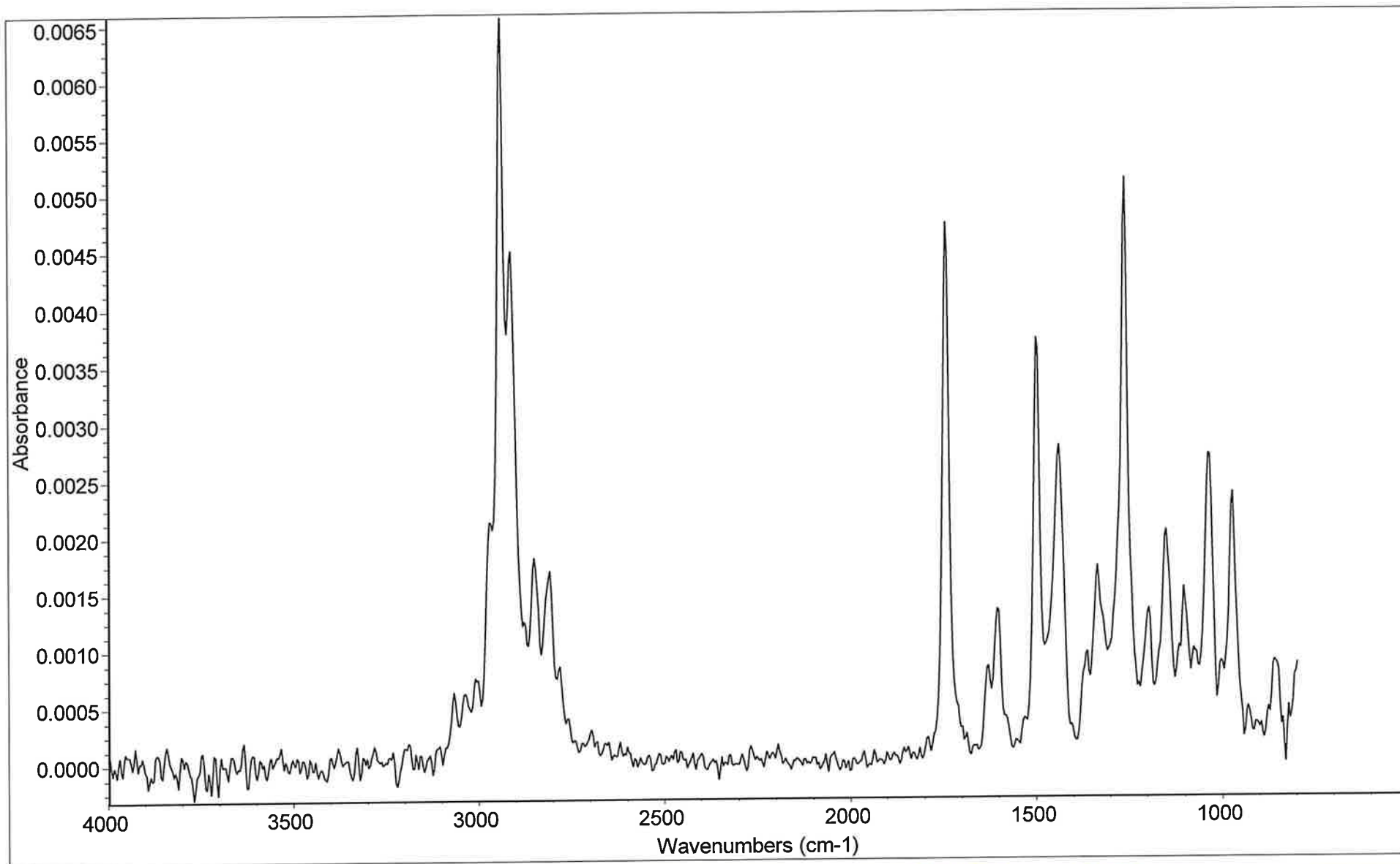
Column USF668212H 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT/A
Beamsplitter: KBr
Source: IR

COCAINE PKs
Number of sample scans: 12
Number of background scans: 64
Resolution: 8.000
Sample gain: 1.0
Optical velocity: 3.7974
Aperture: 100.00

HOGWARTS

Collection time: Mon Sep 12 10:03:47 2016 (GMT-05:00)



GC/IR **Hogwarts** 120-270C @70C/min. for 10 mins. Methanol Mix Split/Splitless Study- **Splitless Mode** 0.5uL injection volume 09-12-16

Column USF668212H 30 Meters
Column flow 3.0
Split ratio 2.0
Transfer Line 280 degrees C
Light Pipe 280 degrees C

Detector: MCT/A
Beamsplitter: KBr
Source: IR

Hydrocodone mes
Number of sample scans: 12
Number of background scans: 64
Resolution: 8.000
Sample gain: 1.0
Optical velocity: 3.7974
Aperture: 100.00

HOGWARTS

Collection time: Mon Sep 12 10:03:47 2016 (GMT-05:00)