Introducing DiscovIR-GC®

DANI

The Solid Phase FTIR
for Complex Mixtures

IR Detection System
A Beam of Confidence

Flavours & Fragrances

Agriculture

Pharmaceutical

Food & Beverage

Forensic Drugs

Environment

Chemicals

Material Science

History of DANI

1975

1980's

1990's

2000's

2010's

2013

1975

DANI Instruments SpA is established in 1975 with the vision of becoming a premiere innovator in the gas chromatographic market

1975

DANI develops its first Gas Chromatograph – GC 3200

1977

DANI launches its first Head Space Sampler – HSS 3640 and develops the Capillary Gas Chromatograph – GC 3900

1981

DANI presents the first ever Programmable Temperature Vaporizer (PTV)

HSS 39.50

1985

Signs a manufacturing agreement with Hewlett Packard for HSS 39.50

1990

Develops Natural Gas Analyzer product line – PGC 90.25

PGC 90.50

1993

DANI updates its Head Space Sampler – HSS 86.50

HSS 86.50

1996

Releases new digital gas chromatograph – GC1000

1998

Updates its Natural Gas Analyzer – PGC 90.50

2002

Releases Thermal Desorber STD1000

Master TD

2007

Releases GC Capillary Columns – DN Series and Thermal Desorber Master TD

2006

Launches the new Master Line: Master GC (Fast), Liquid Autosampler Master AS

Master AS

2009

Launches Dynamic Headspace Sampler – Master DHS and Time of Flight GC/MS – Master TOF

Master DHS

2010

Establishes DANI Instruments India Private Limited in Mumbai

2011

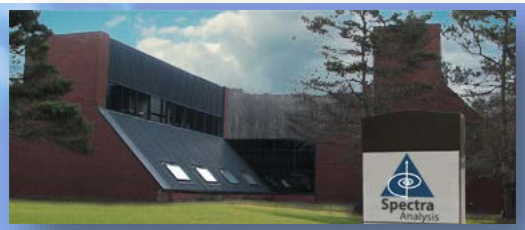
Establishes DANI Instruments France SARL; Further expands in India with two sales offices in Calcutta and Jaipur

Master SHS

2012

Launches Static Headspace Sampler – Master SHS

DANI INSTRUMENTS, INC. (JANUARY 2015)



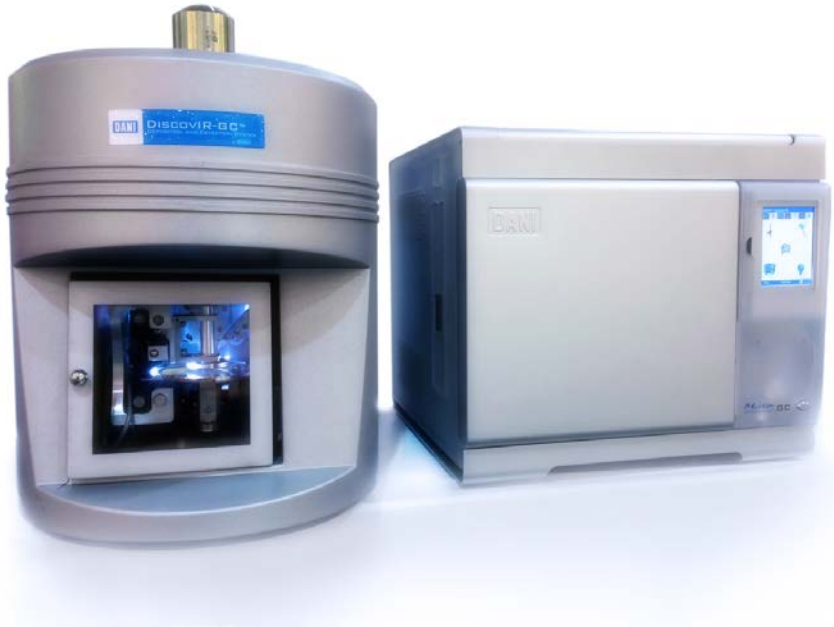
DANI Instruments Inc. is the US-based Company with headquarters in Marlborough (Boston)

DANI Instruments Inc. is the direct result of the recent acquisition of Spectra Analysis, a world leader in FTIR technology, which complete the DANI product line with a highly specialized detector for the identification of a large variety of components in complex mixtures.

DANI Instruments Inc. is responsible for R&D, manufacturing and worldwide commercialization of GC-FTIR as well as commercialization of DANI core portfolio of products for North America.

DANI 40 Years

DiscovIR-GC System Applications



Data Processing ← DiscovIR ← GC

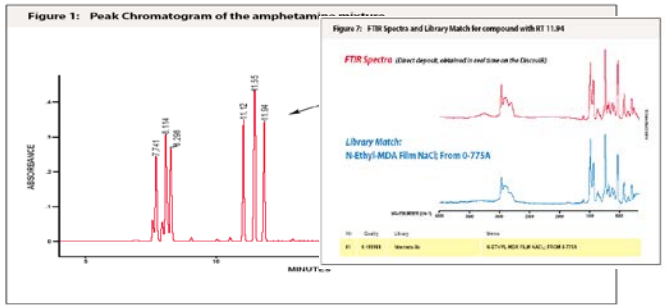
Primary Applications

- Forensic Drug Analysis ***
  - Designer Drugs
  - New/Novel Psychoactive Substances (NPS)
- Chemical Weapons Analysis:
  - Solids, e.g. Anthrax
  - Explosives, e.g. dust analysis
  - Contaminated soils
- Agriculture / Crop Sciences:
  - Pesticides
  - Chemical Ecology
  - Pheromones
- Flavors / Fragrances / Cosmetics

DiscovIR-GC™ The Solution for Emerging Drugs

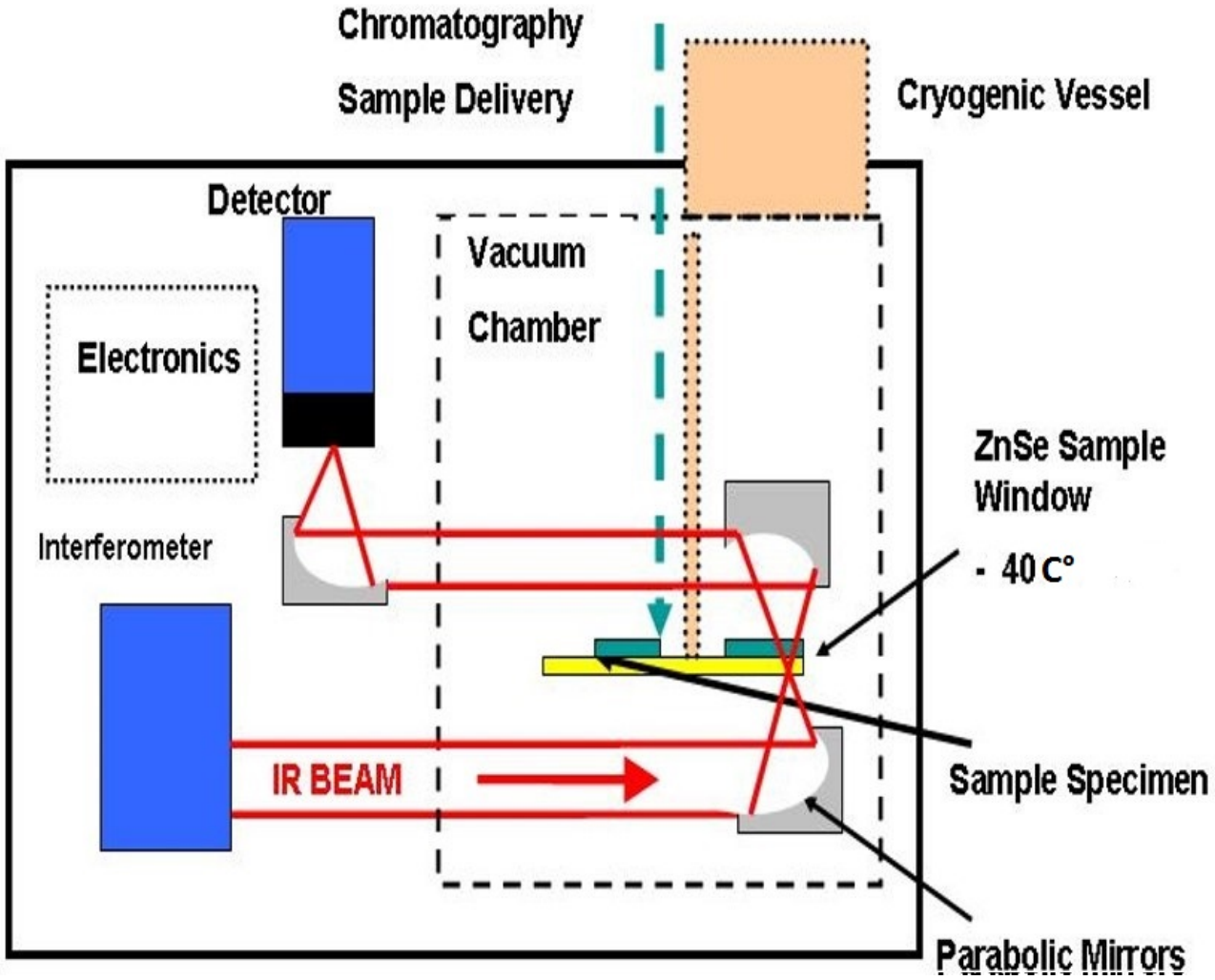
Step #1 Separation by Gas Chromatography

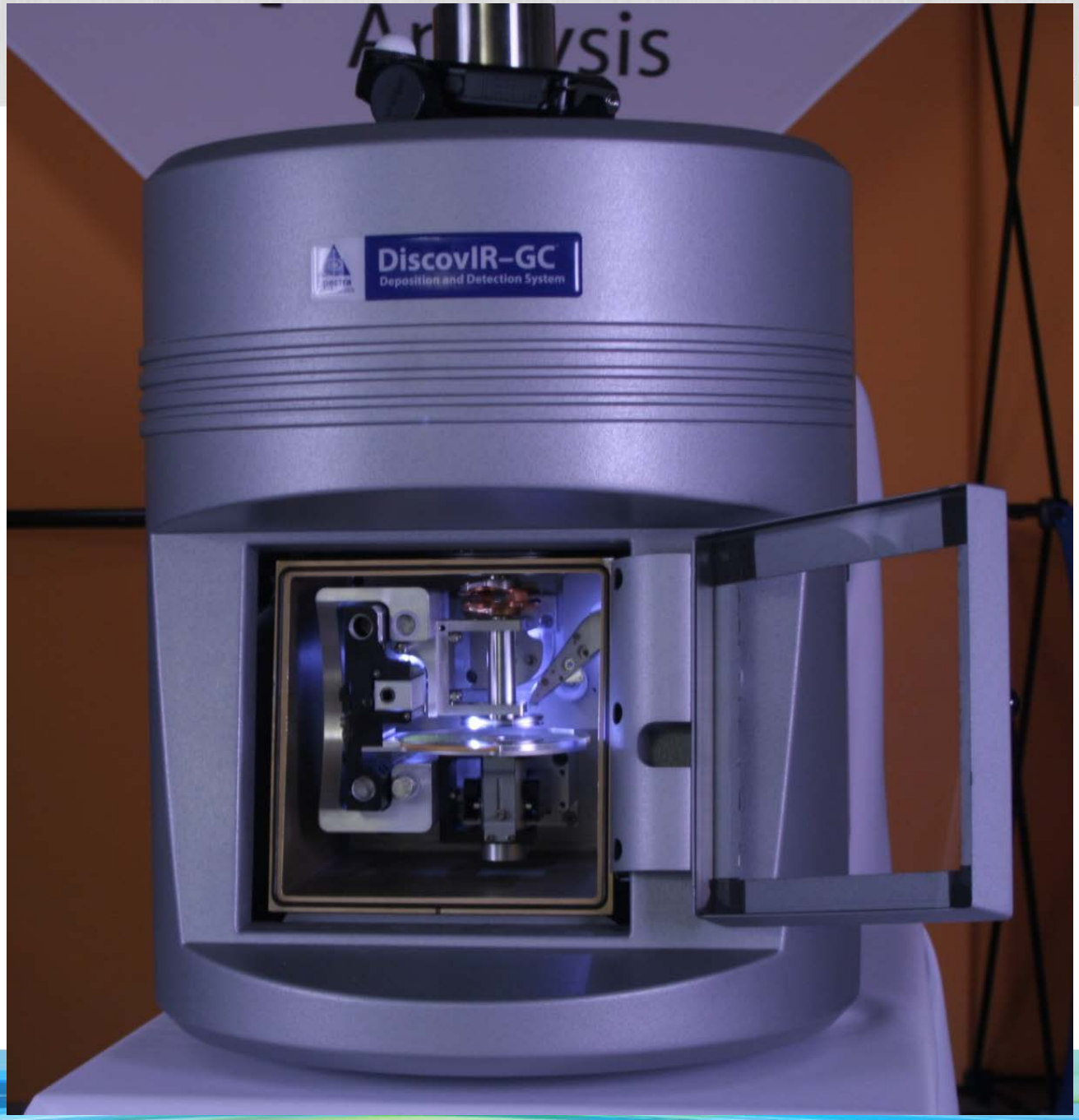
Step #2 Controlled Deposition - unique solid phase deposition.



Step #3 Chemical Identification - using FTIR specificity (positional and stereoisomers)

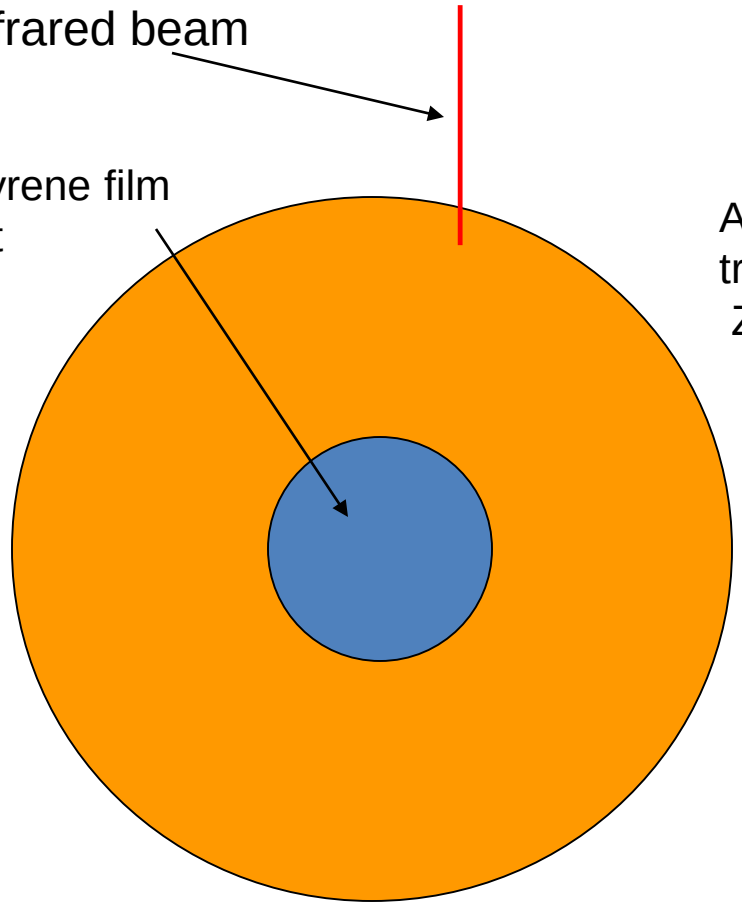
How DiscovIR-GC System operates



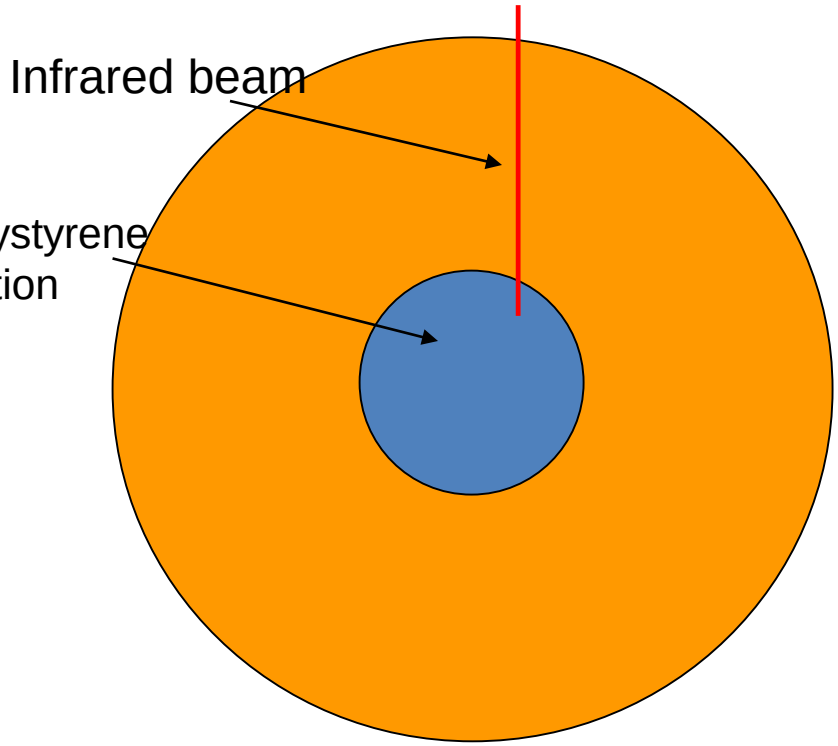

DiscovIR–GC
Deposition and Detection System

Polystyrene Calibration

Recommended by SWGDRG



Polystyrene Calibration



Acquisition of solid phase transmission spectra through ZnSe disc

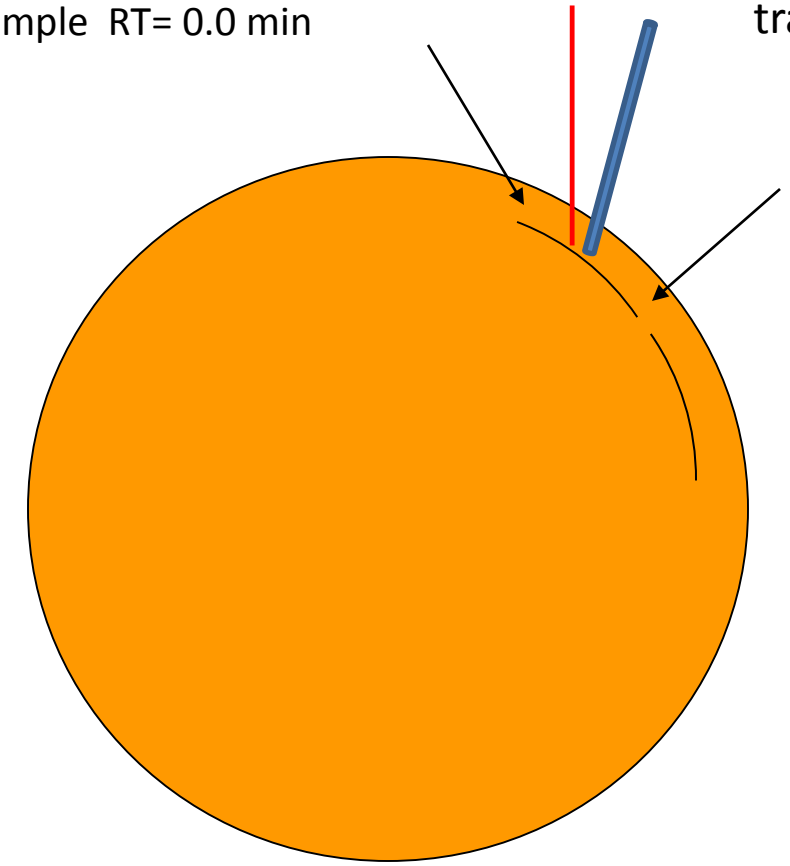
ZnSe Disc

Infrared beam fixed in position generates solid phase transmission spectra

Start Sample RT= 0.0 min

End Sample RT= 20.0 min

ZnSe Disc rotates counterclockwise

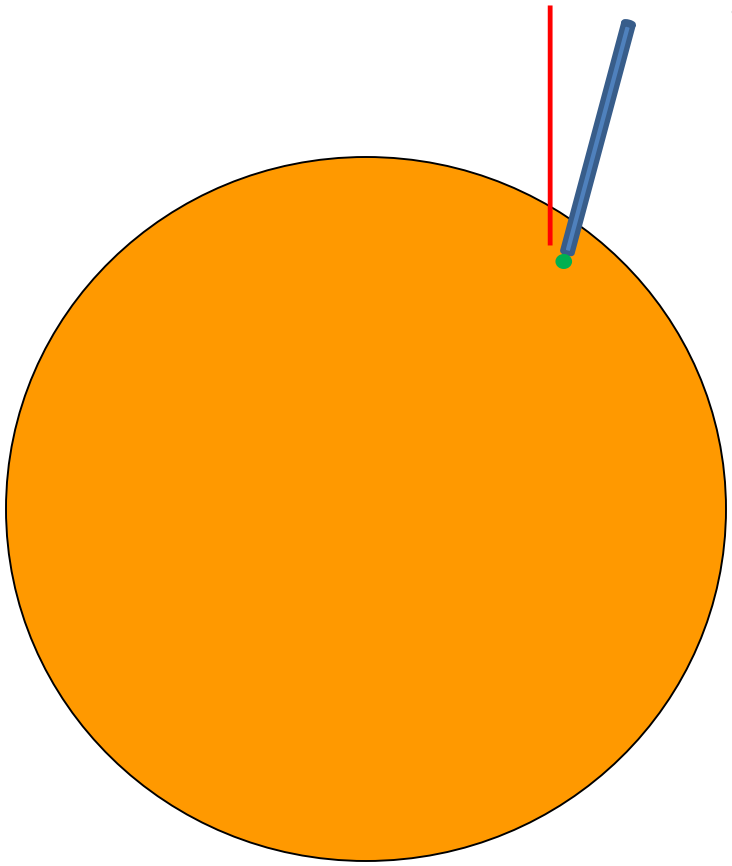


Records linear position and rotary position for For every spectra

ZnSe Disc

Infrared beam fixed in position generates solid phase transmission spectra

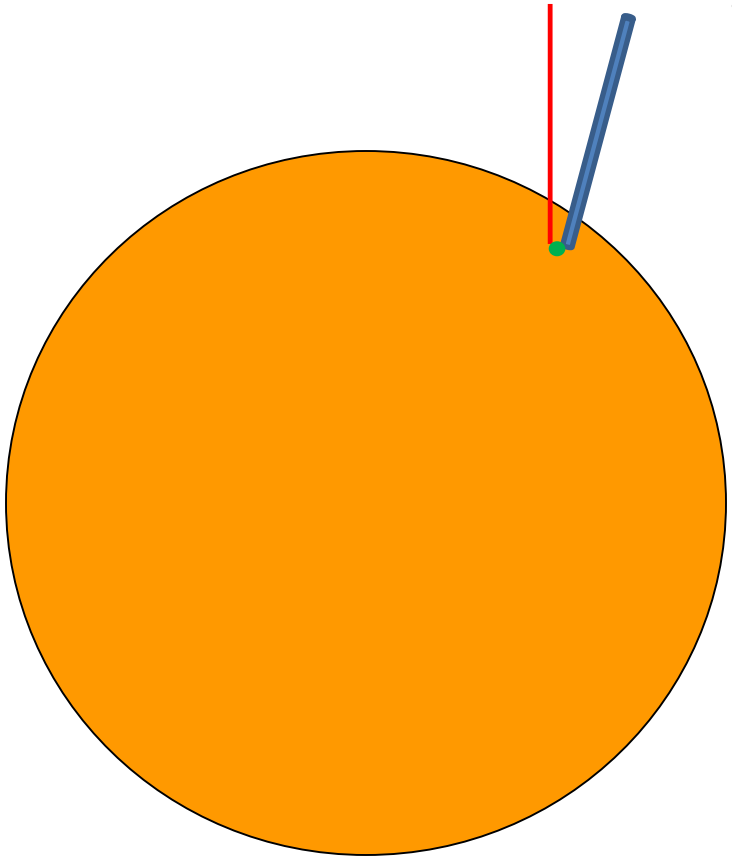
ZnSe Disc rotates counterclockwise



ZnSe Disc

Infrared beam fixed in position generates solid phase transmission spectra

ZnSe Disc rotates counterclockwise



ZnSe Disc

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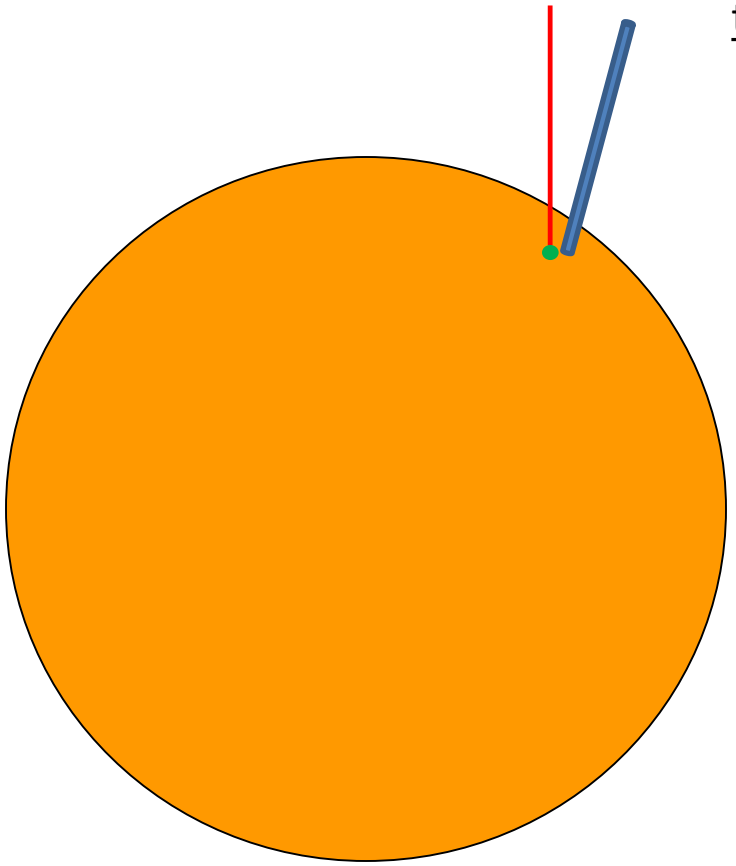

ZnSe Disc

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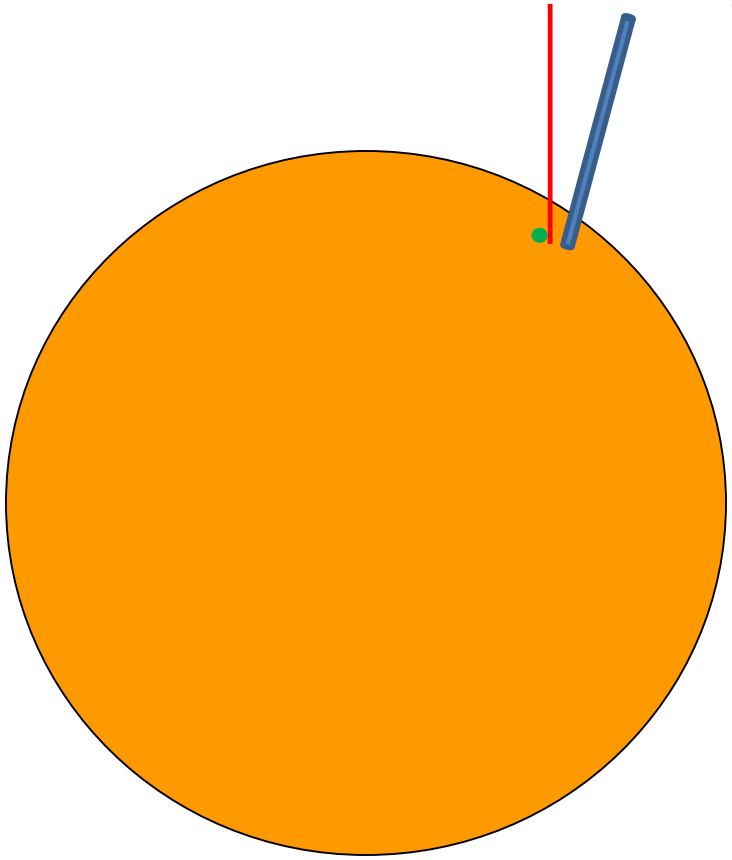
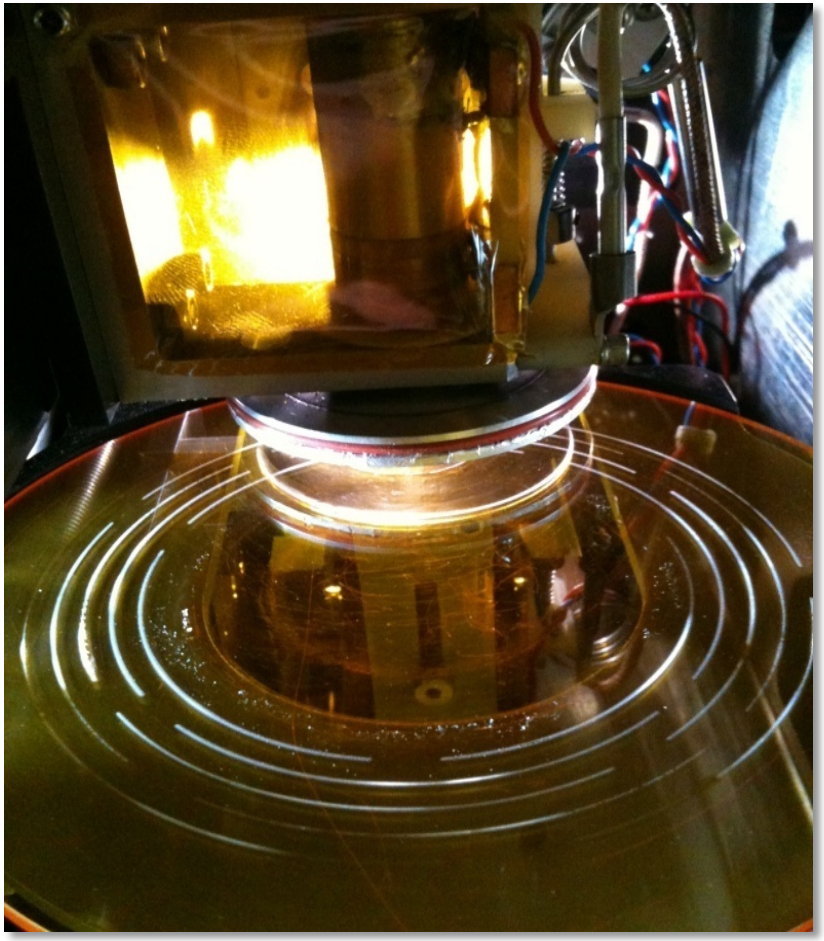


Records linear position and rotary position for For every spectra

Direct Deposition - Sample Disk Under Vacuum

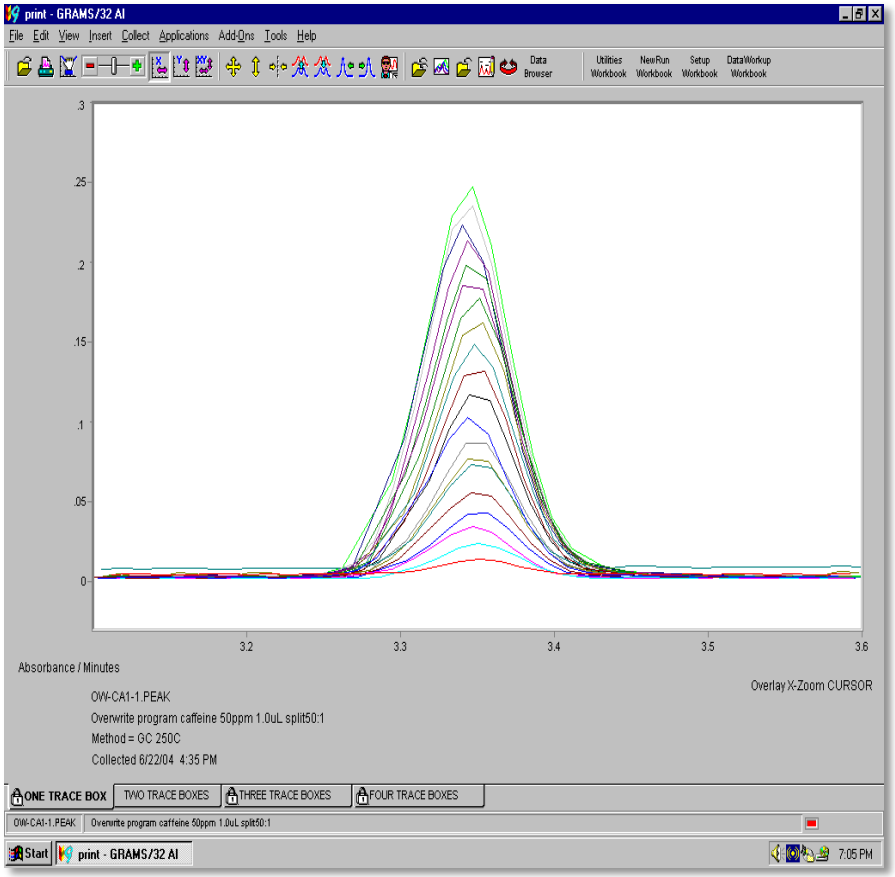
View inside Chamber



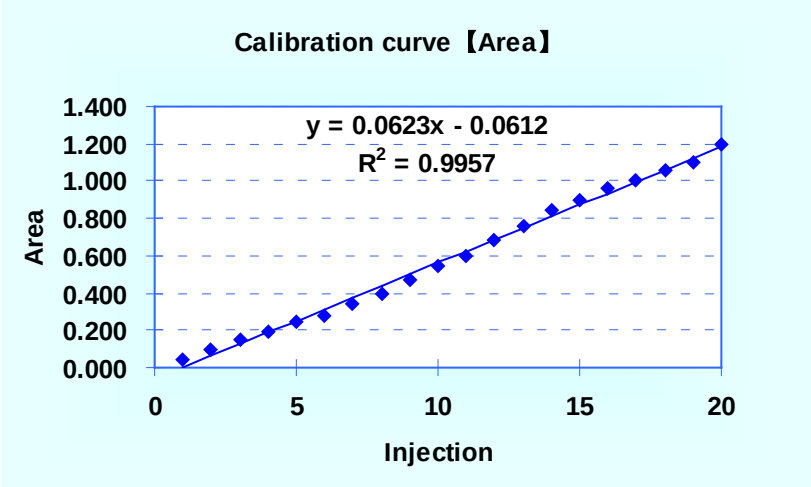
Key Features

1) Disk rotates at 3 mm/min
2) Disk loads in a spiral, holds 40 hours of chromatography
3) Re-usable after solvent cleaning
4) The disk is under vacuum without moisture or CO_2 interference
5) Disk cooled to - 40C, which makes solid deposit.
6) Transmission IR analysis is done on the solid deposit.
7) ZnSe Disk is transparent in Mid-IR range
8) Embedded polystyrene standard for easy daily Operational Quality (OQ) verification.

DiscovIR-GC Precision & Reproducibility for Caffeine Sample



- **Twenty consecutive runs overlaid on the disk.**



Benefits of condensed phase GC-IR

- Sample is concentrated in small spot
- Distortion of spectra is eliminated in solid phase
- Excellent Spectral Resolution provides unique IR spectra for all isomers
- Standard GC columns and injectors may be used ie. 0.2micron ID columns, 1 µl injections, 1mg/ml solutions

Infrared Techniques for Substance Analysis

	Spectrum Type	Mixture Analysis
ATR	Solid Phase Reflectance	De-Convolution*
Gas Phase GC-IR	Gas Phase Absorbance	Limited GC
DiscovIR GC-IR	Solid Phase Transmission	Standard GC

SWGDRUG Minimum Standards:

*The classification of a technique may be lower, if the sample, analyte or mode of operation diminishes its discriminating power.

Examples of diminished discriminating power may include:

- an infrared spectroscopy technique applied to a mixture which produces a combined spectrum

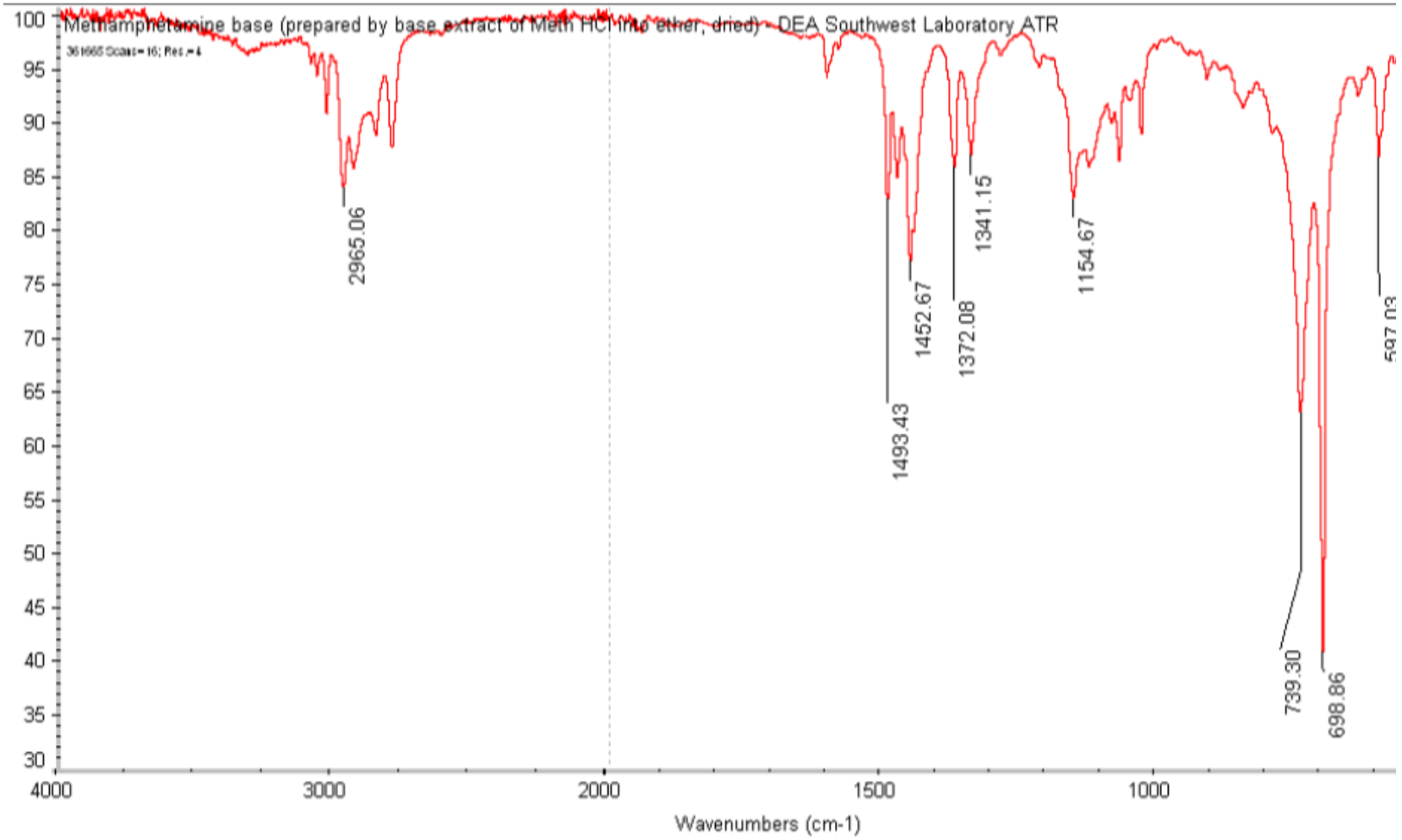
Infrared Techniques for Substance Analysis

	Spectrum Type	Mixture Analysis
ATR	Solid Phase Reflectance	De-Convolution
Gas Phase GC-IR	Gas Phase Absorbance*	Limited GC**
DiscovIR GC-IR	Solid Phase Transmission	Standard GC

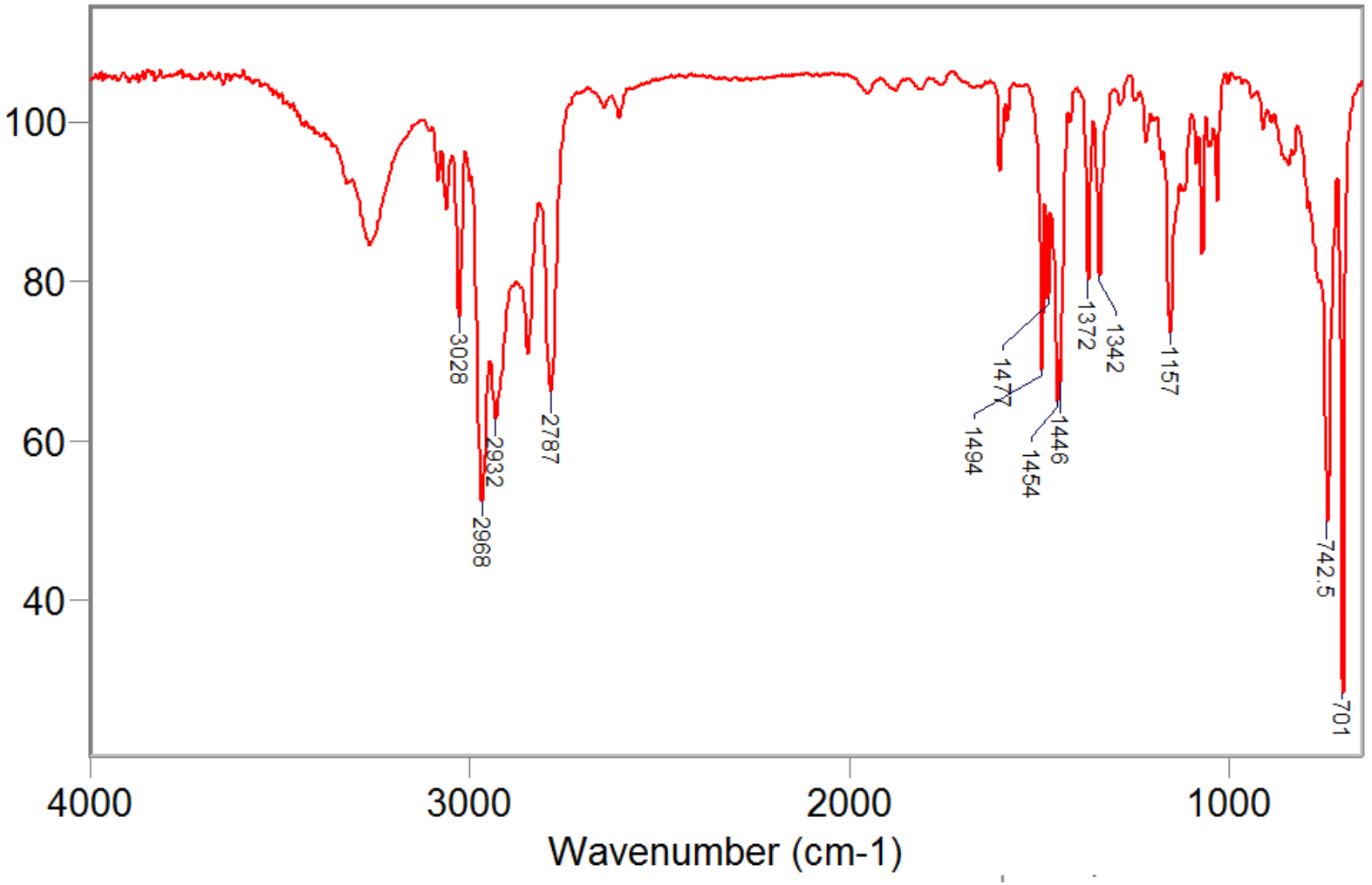
*Gas phase GC-IR spectra suffer from centrifugal distortion in the gas phase.

**Large volume injections are used to compensate for lack of sensitivity, but can overload columns and alter retention times. Gas phase GC-IR has limited temperature range, oven Temperatures above 280^{o}C compromise sensitivity.

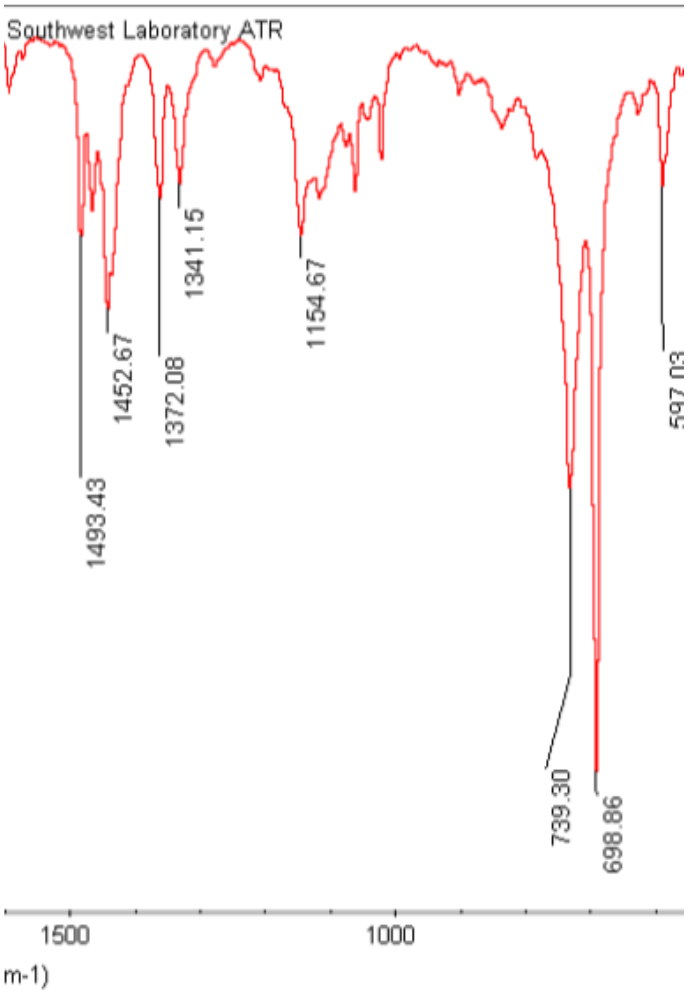
ATR spectra of pure methamphetamine

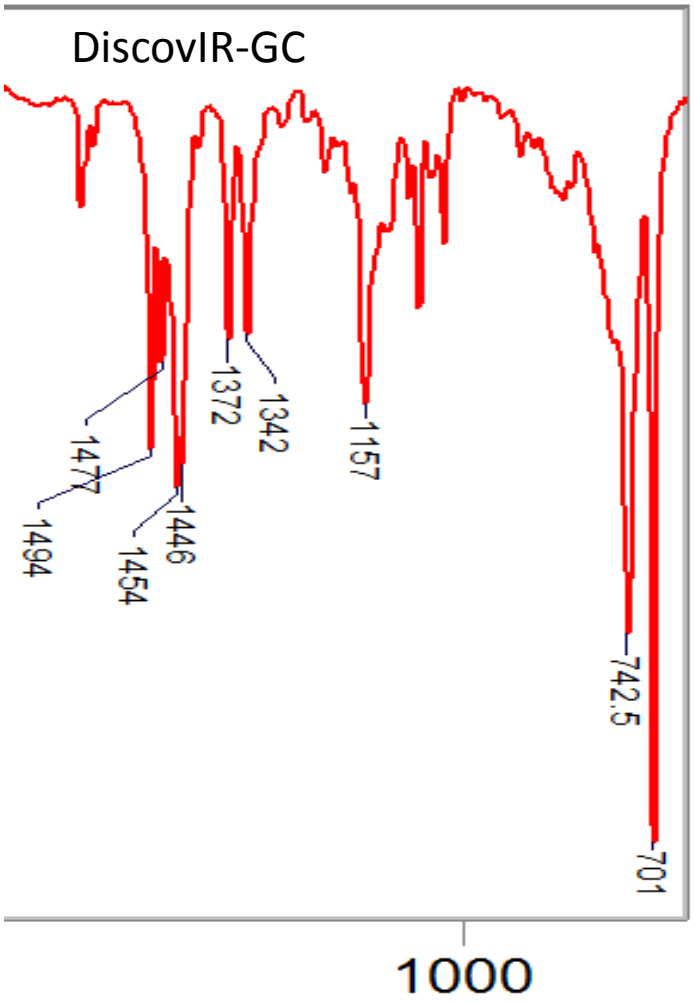


Methamphetamine DiscovIR spectra



Similarity of DiscovIR and ATR spectra
Pure Methamphetamine comparison

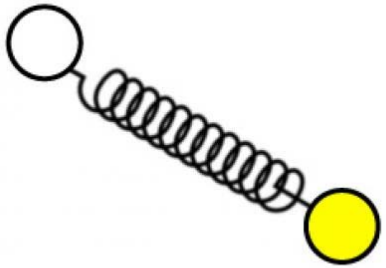




Dissimilarity between DiscovIR spectra and Gas phase spectra: IR Theory

- Infrared Spectra result from transitions between quantized vibrational energy states

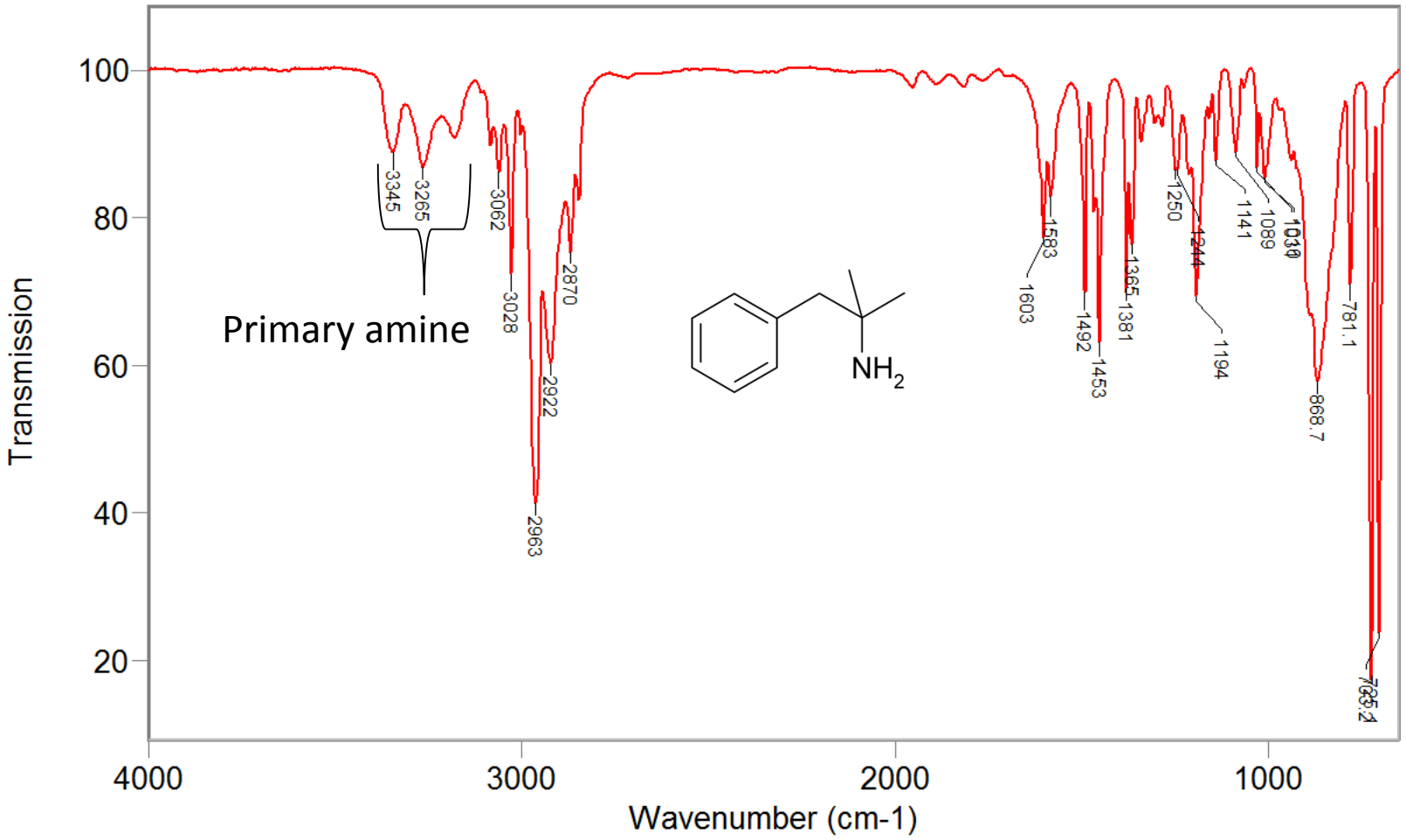
- Molecules with N atoms have 3N degrees of freedom
  - 3N-6 vibrational
  - 3 translational (x,y,z)
  - 3 rotational



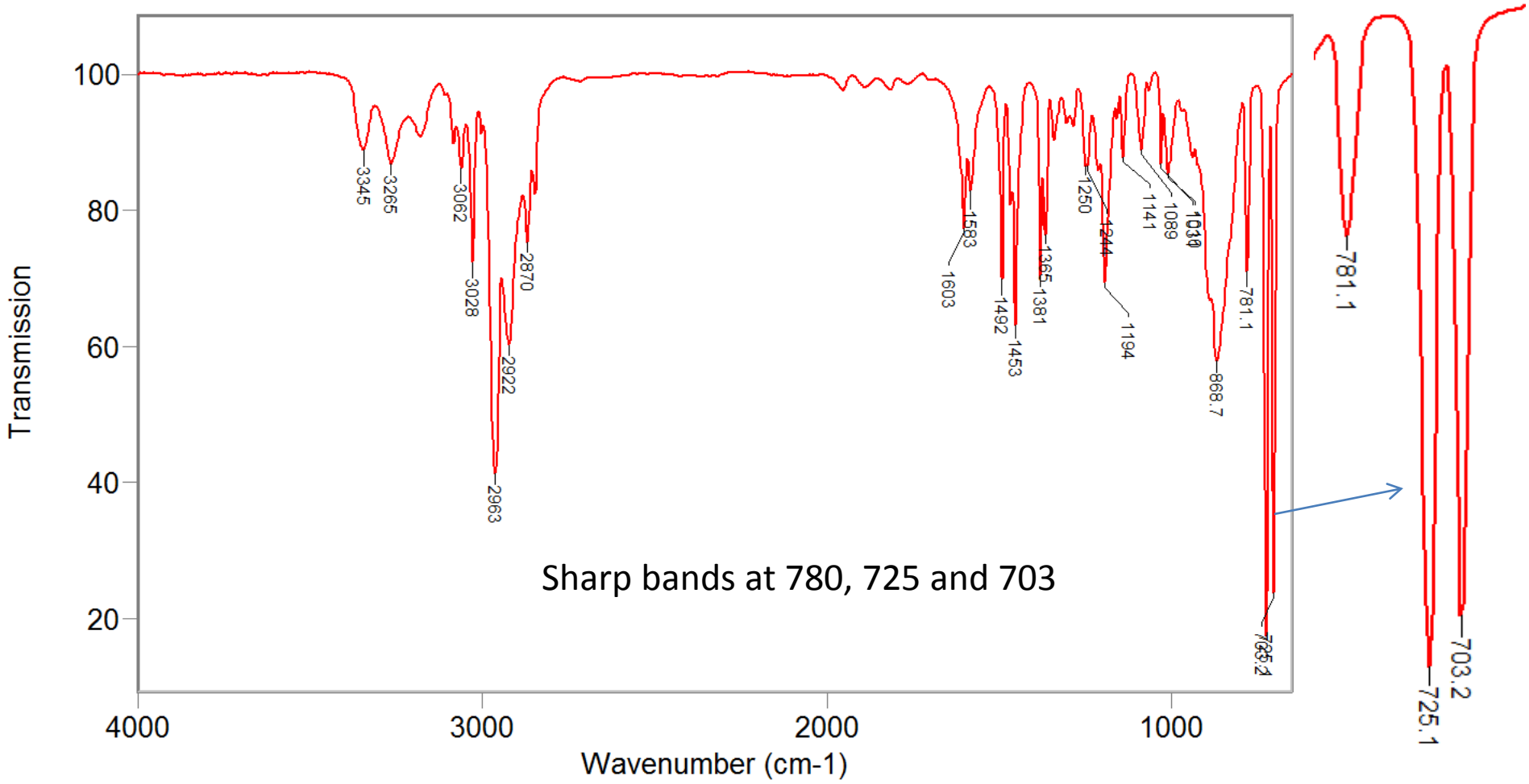
Gas phase GC-IR limitations

- Gas molecules are free to rotate
- Centrifugal Distortion causes diffusion of Infrared bands in gas phase
- Insufficient Spectral Resolution to generate unique IR spectra for all isomers
- Isomer identification is based on inconclusive spectra dependent on retention time (no improvement over GC-MS of isomers)

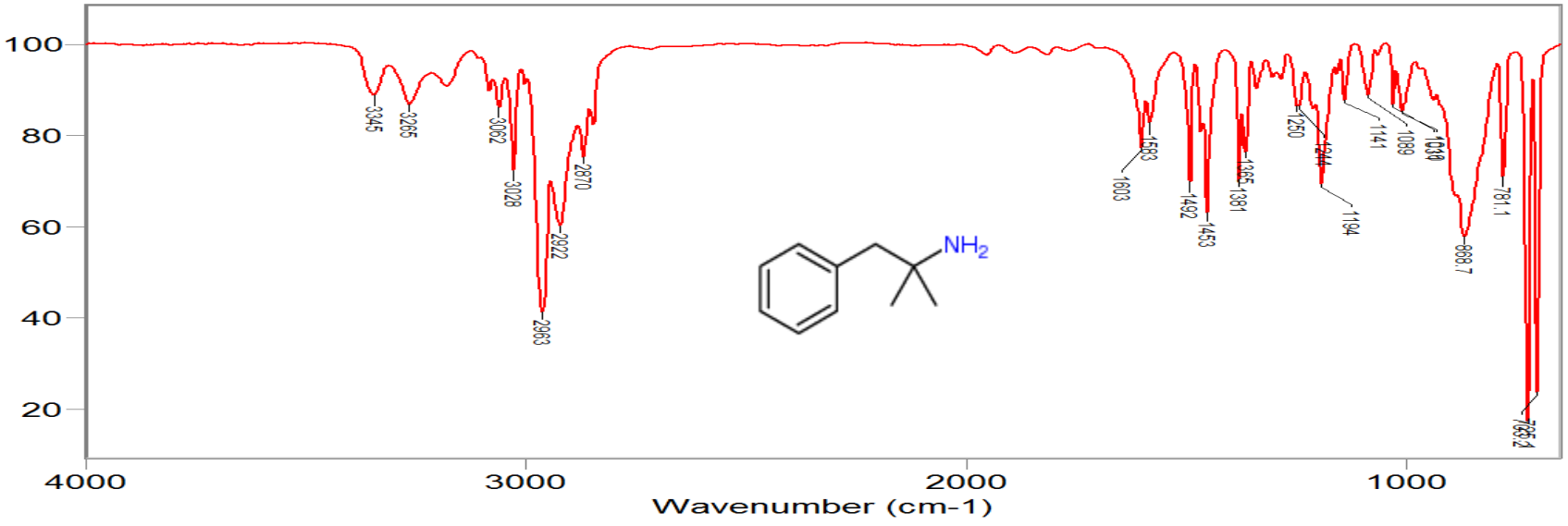
Phentermine DiscovIR spectra

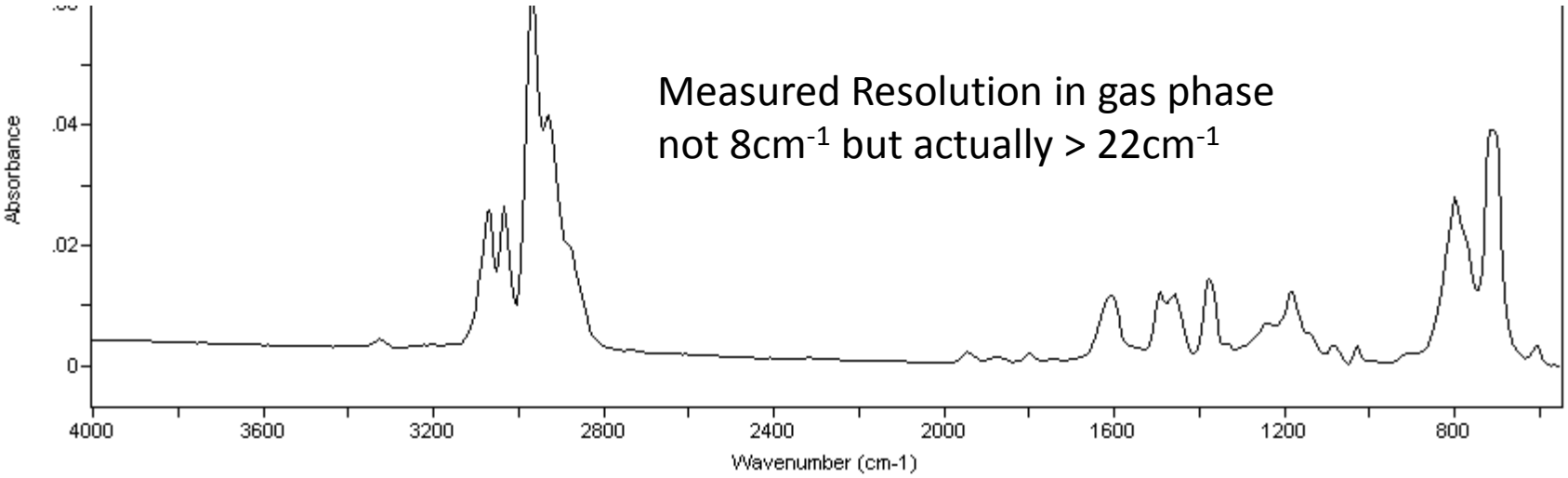


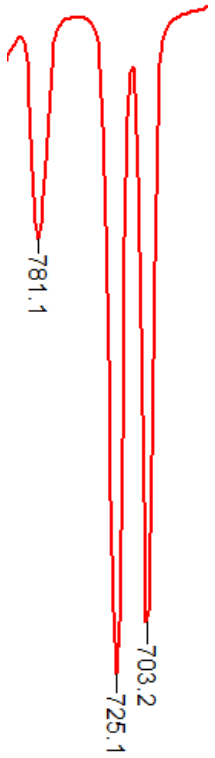
Phentermine DiscovIR spectra



Phentermine DiscovIR vs gas phase





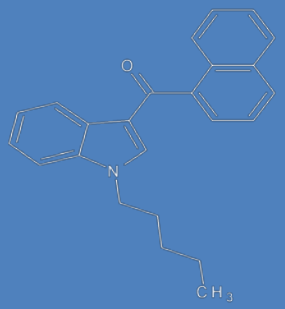


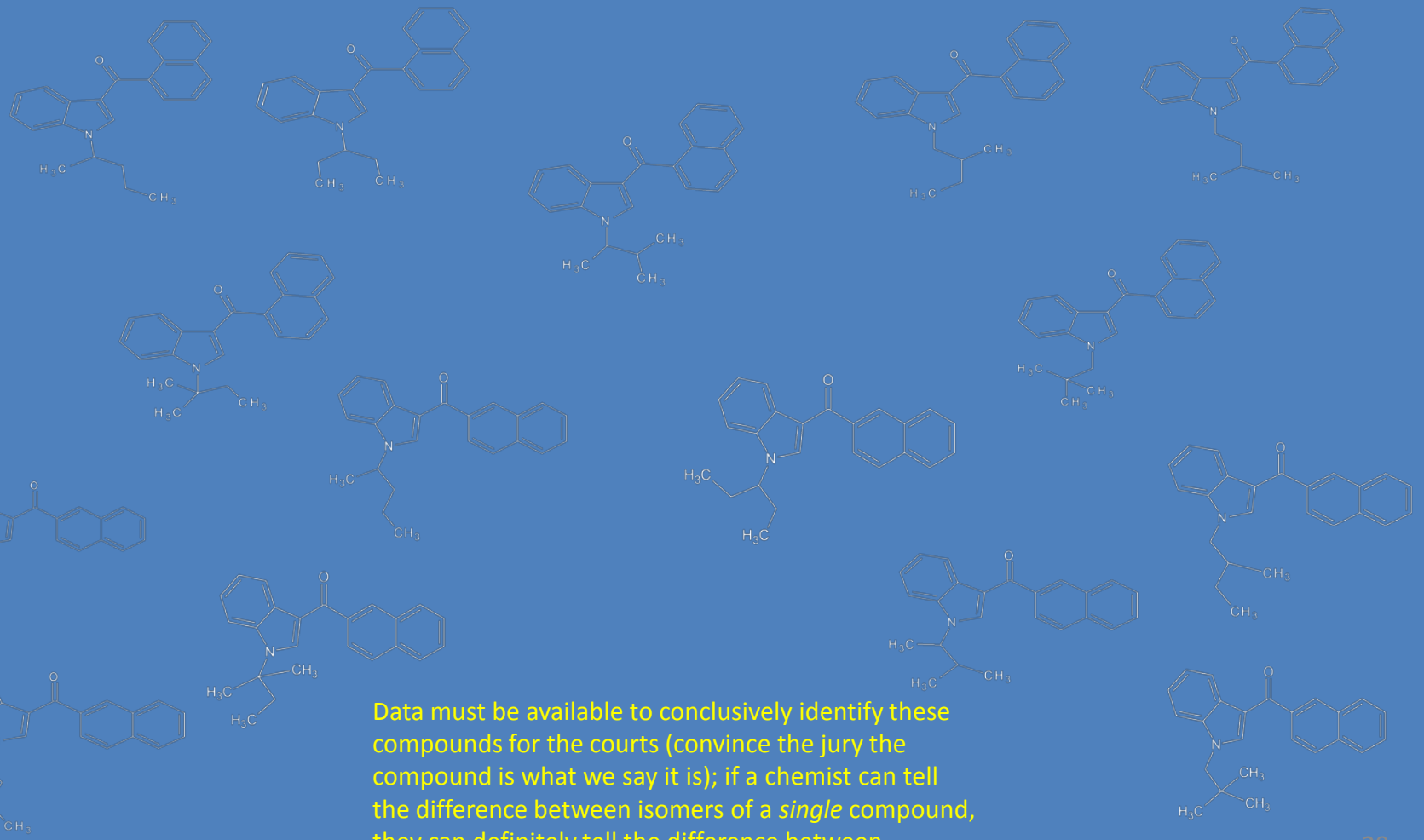
Infrared Techniques for Substance Analysis

	Vibrations	Rotations	Resolution
ATR	Yes	No	≤ 4 cm^{-1}
Gas Phase GC-IR	Yes	Yes	>16 cm^{-1}
DiscovIR GC-IR	Yes	No	4 cm^{-1}

- Molecular rotation in gas phase causes centrifugal distortion of Spectra.
- Severe band broadening takes place in the gas phase. IR bands 20, 30 even 40 cm^{-1} apart are merged together.
- Even though the hardware can be set at 4 cm^{-1}, the spectra still suffer from centrifugal distortion in the gas phase.
- Spectral resolution of 4 cm^{-1} is required to differentiate isomers of synthetic cannabinoids.

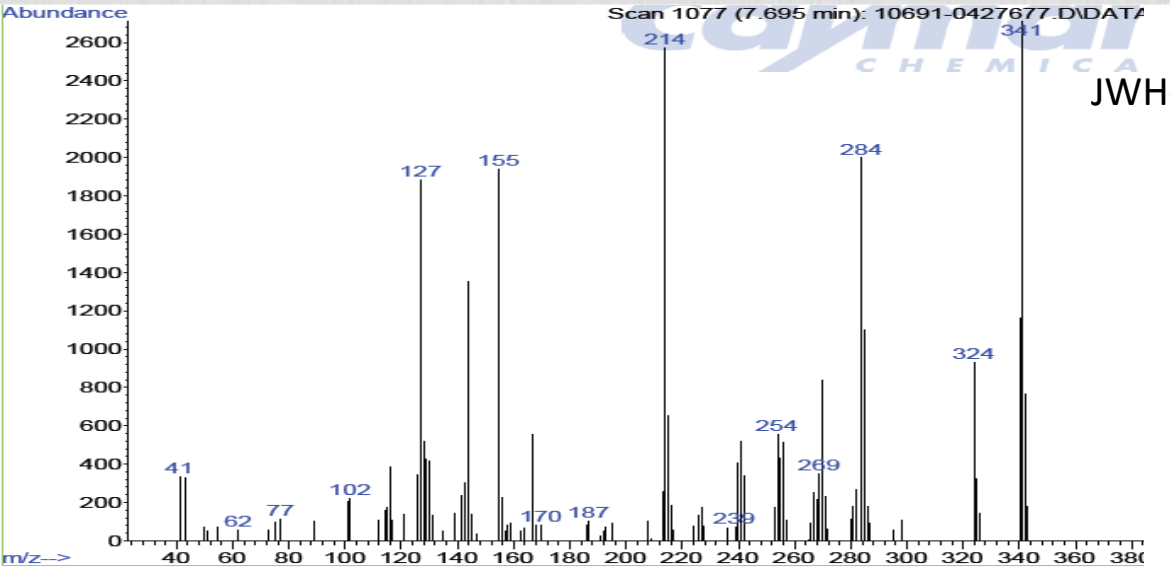
JWH-018



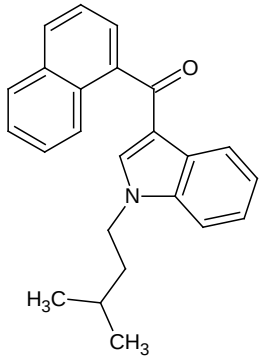


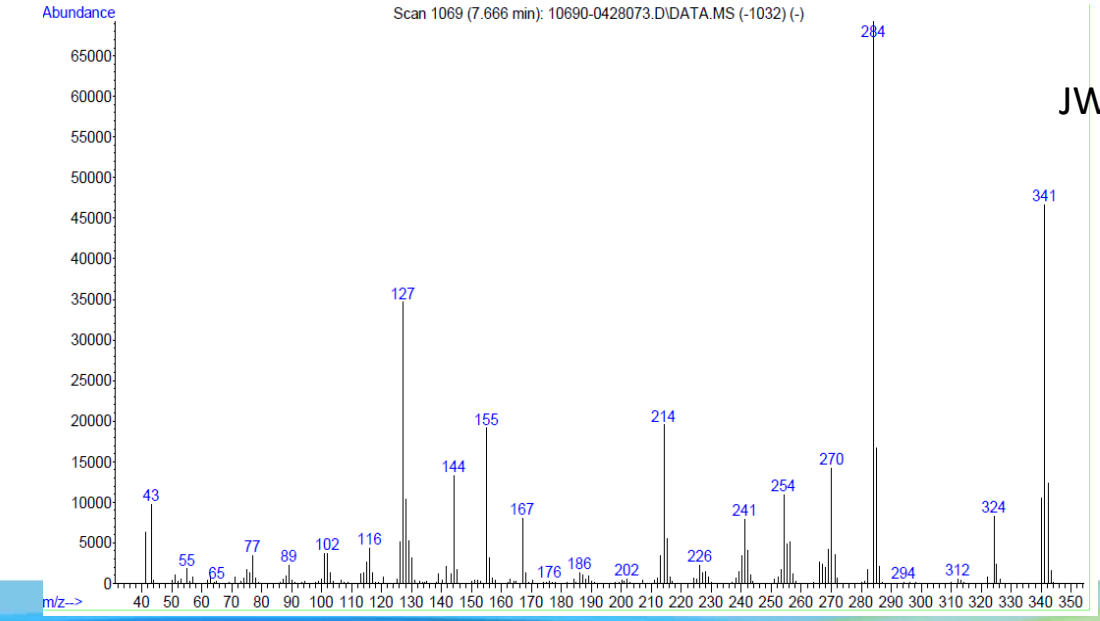
Data must be available to conclusively identify these compounds for the courts (convince the jury the compound is what we say it is); if a chemist can tell the difference between isomers of a *single* compound, they can definitely tell the difference between *different* compounds

Similar Mass Spectra of JWH-018 isomers

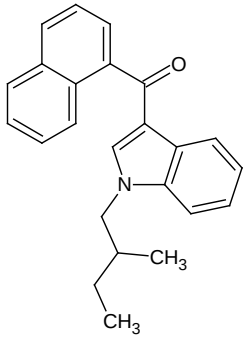


JWH-018-N-(3-methylbutyl) isomer

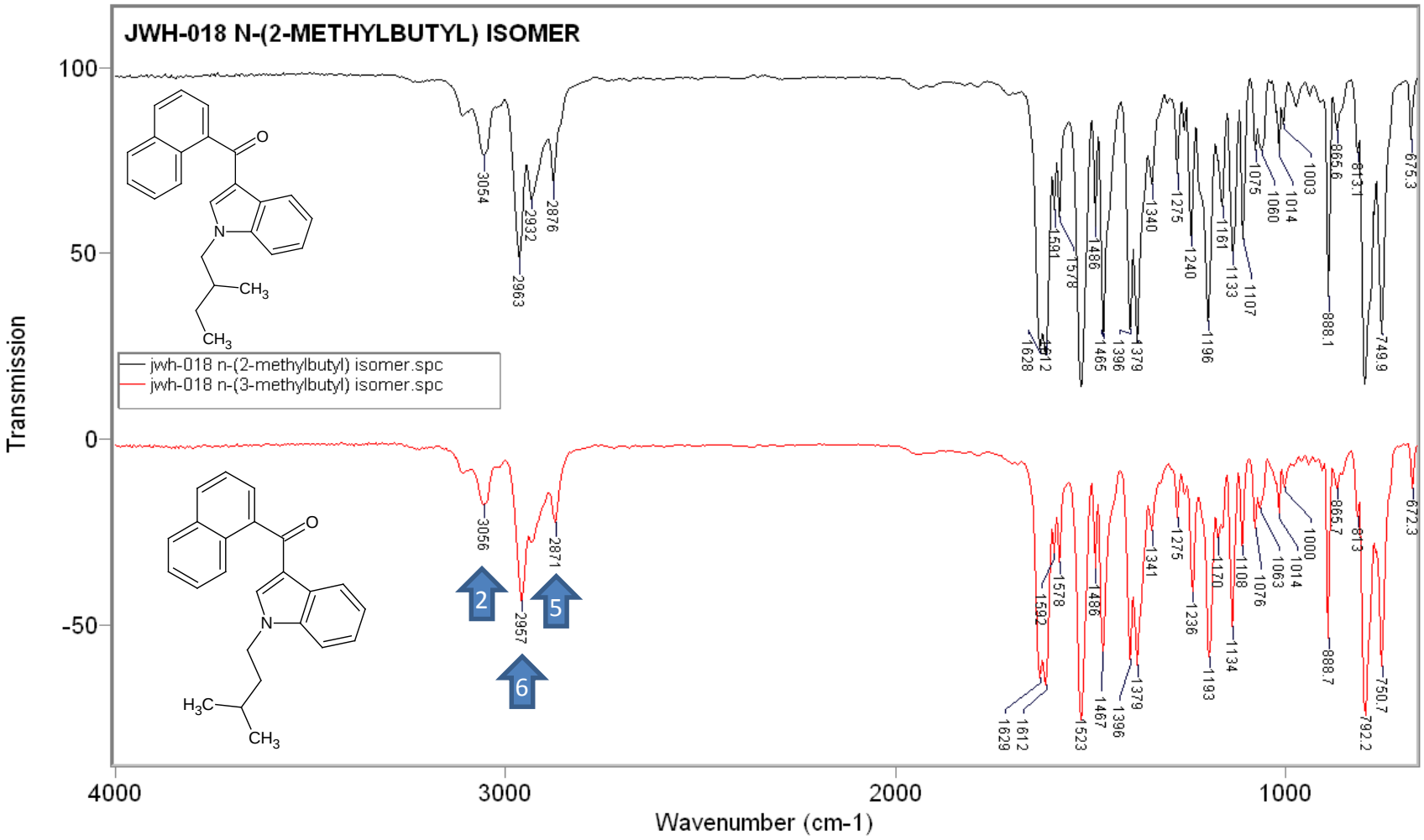




JWH-018-N-(2-methylbutyl) isomer



JWH-018-N-(2-methylbutyl) isomer and JWH-018-N-(3-methylbutyl) isomer co-elute



Fingerprint Region of JWH-018 N-(2-methylbutyl) and JWH-018-N-(3-methylbutyl) isomers

4 cm^{-1} Resolution

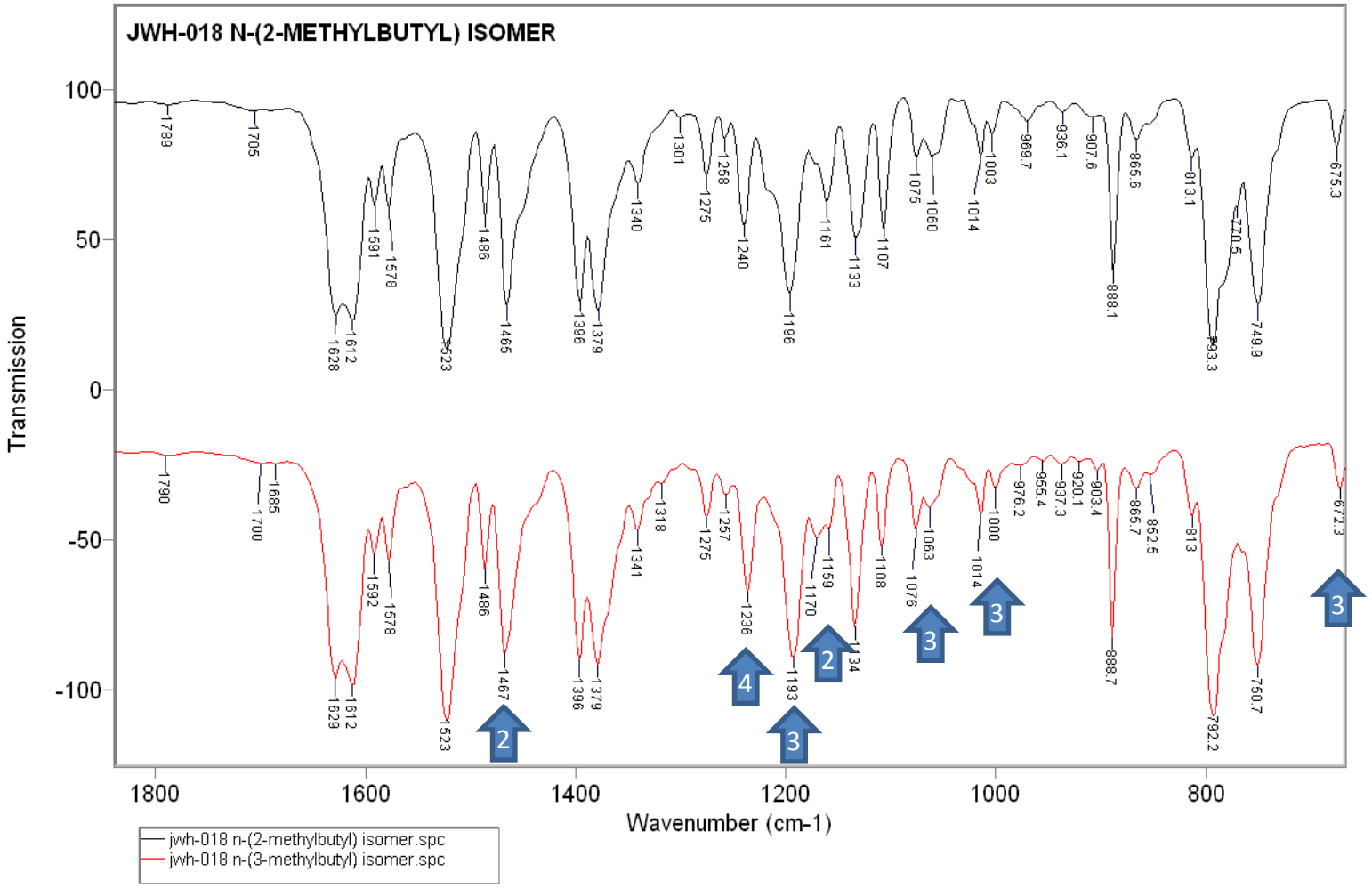
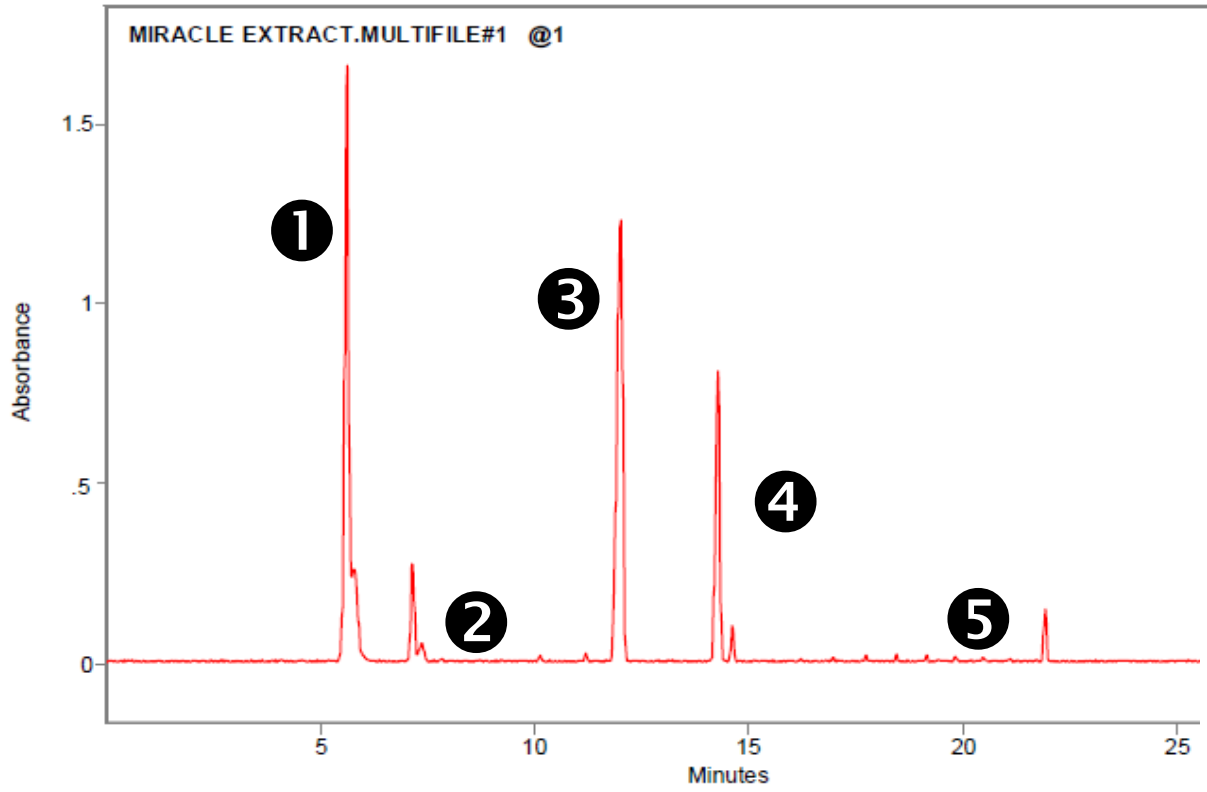


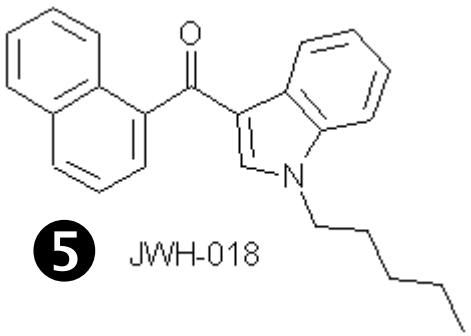
Table of Differentiating Infrared Bands

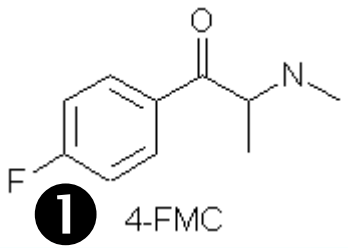
	RT	Wavenumber of differentiating bands								
JWH-018-N-(2-methylbutyl) isomer	7.667	2963	2876	1465	1240	1196	1060	1003	969.7	675
JWH-018-N-(3-methylbutyl) isomer	7.697	2957	2871	1467	1236	1193	1063	1000	955.4	672

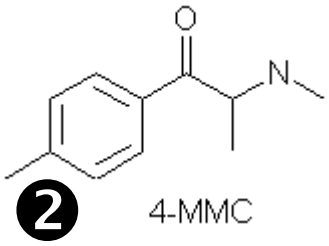
AT-5 Column 30m x 0.32mm, 240°C-300°C, 30°C/min

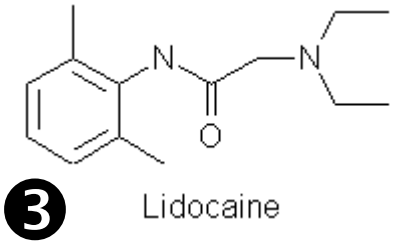
Bath salt sample Analysis

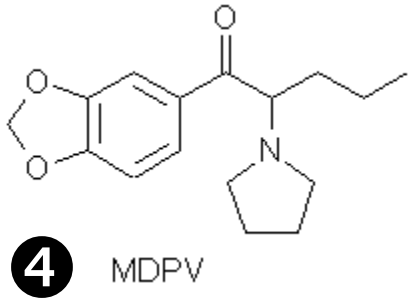






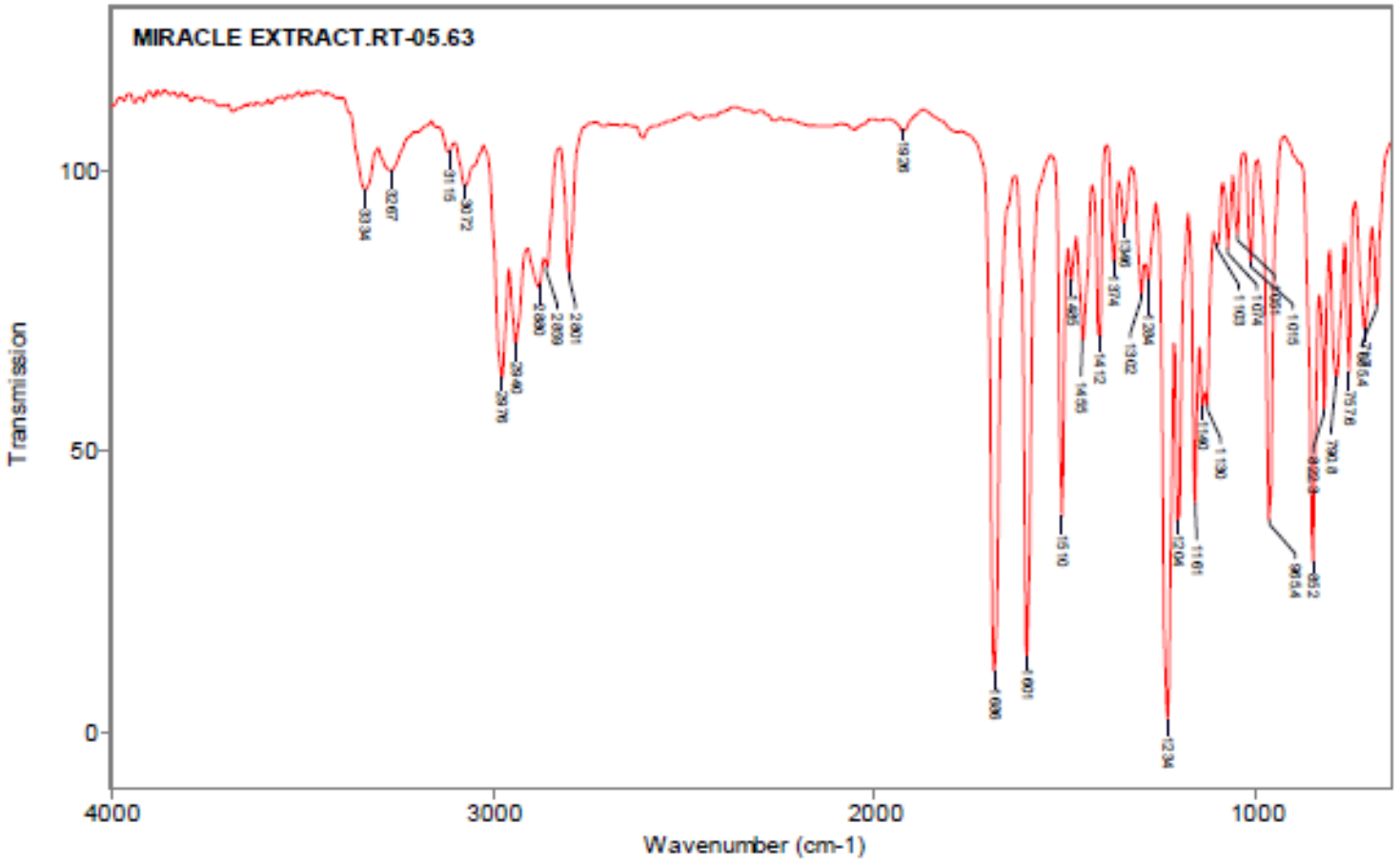






Spectra of 4-Fluoromethcathinone (4-FMC)

❶



Solid Phase Mid-IR Spectrum of Mephedrone

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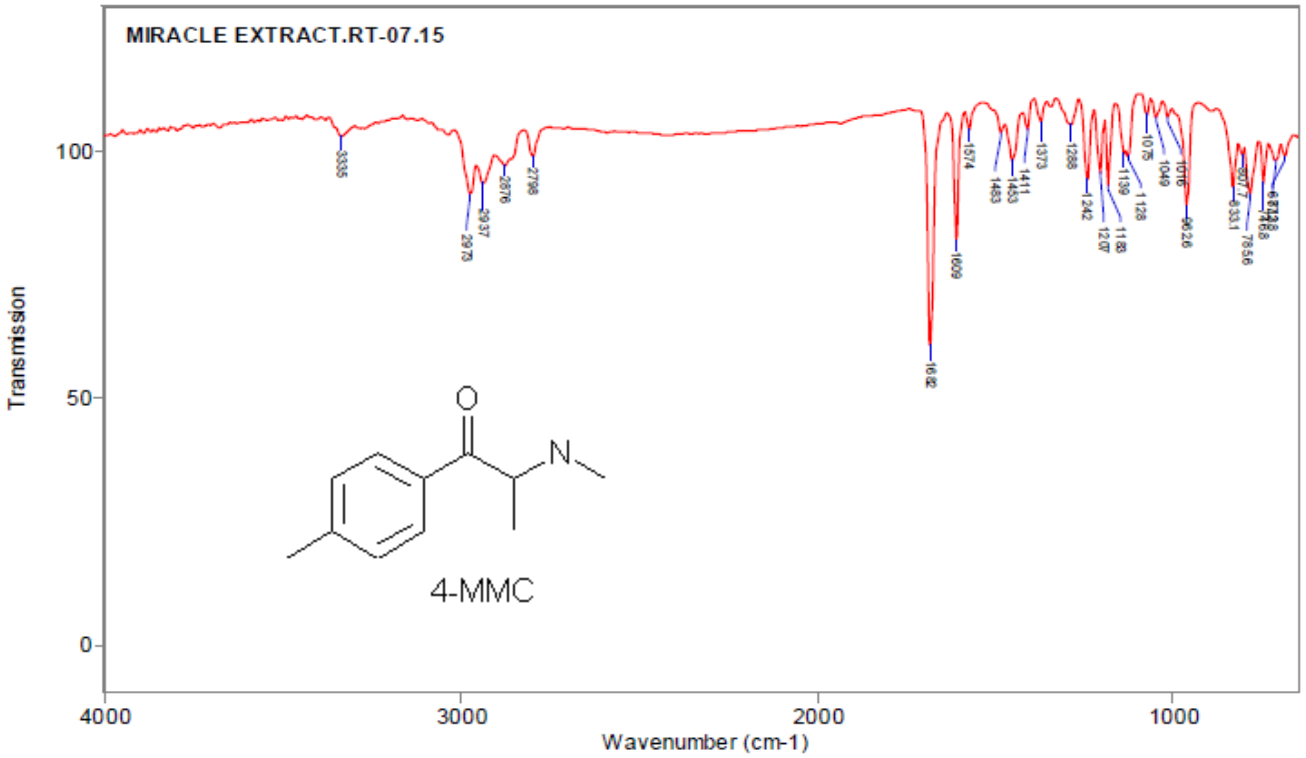
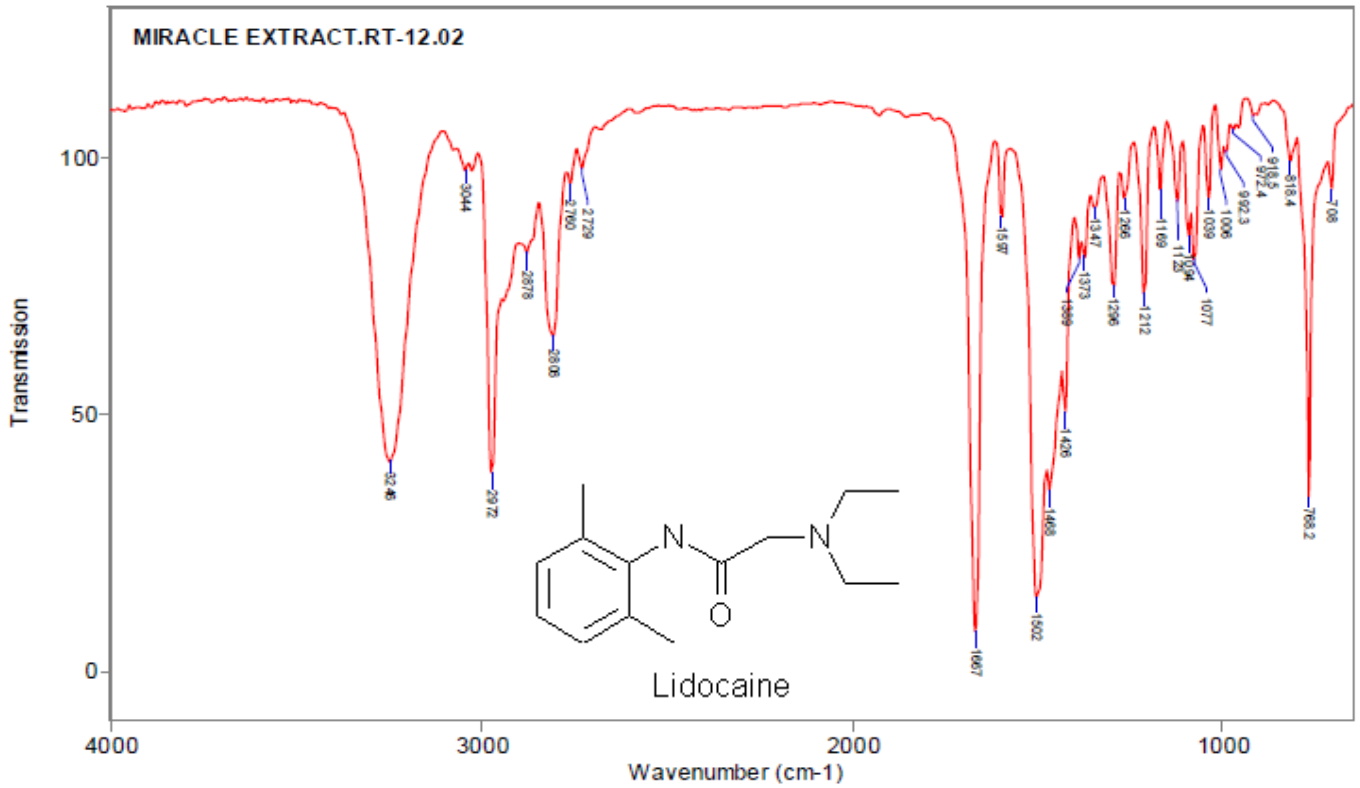


Solid Phase Mid-IR Spectrum of Lidocaine

❸

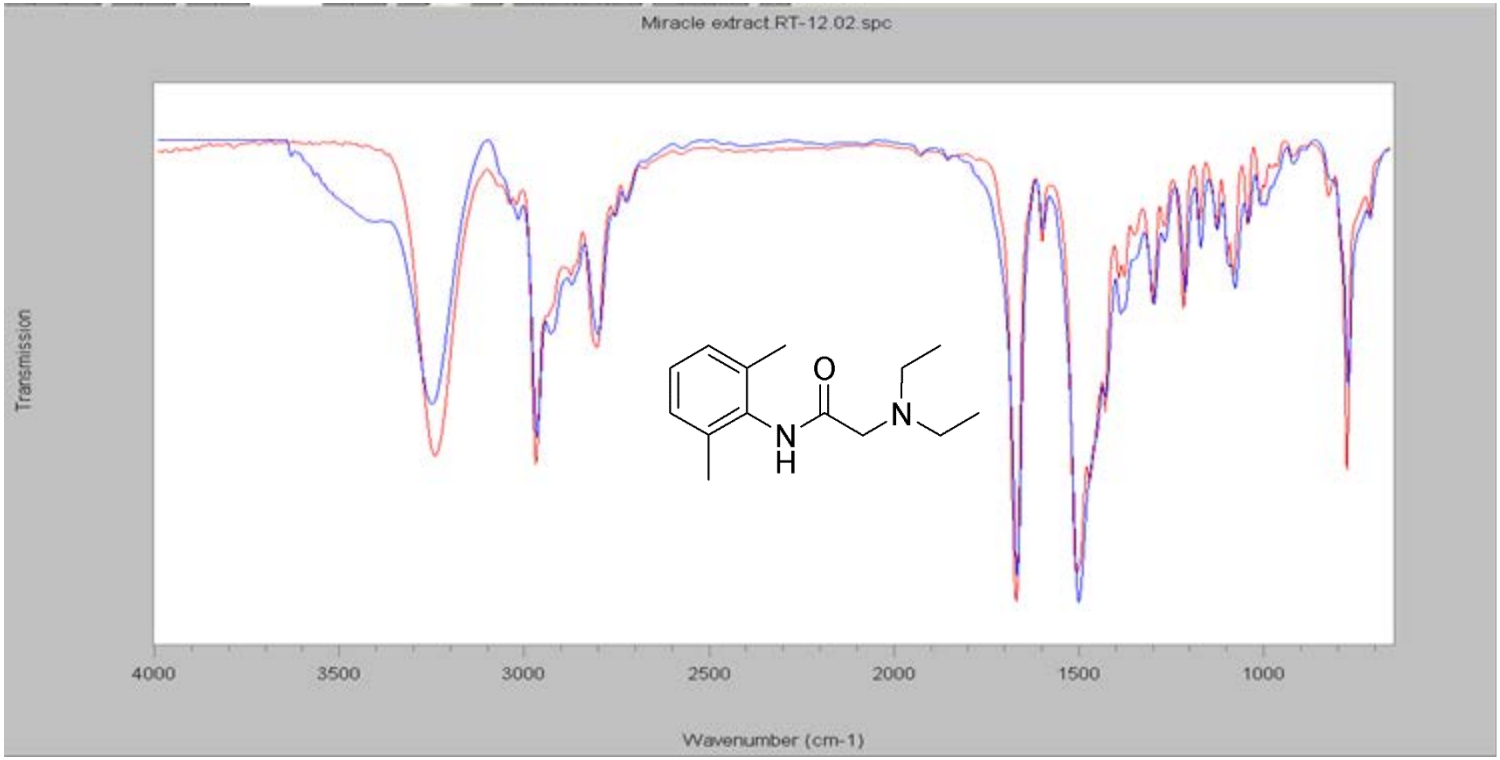


Overlay of DiscovIR GC-IR spectra (red) with Lidocaine FTIR Spectra (blue)

Library spectra is not from the DiscovIR GC-IR library.

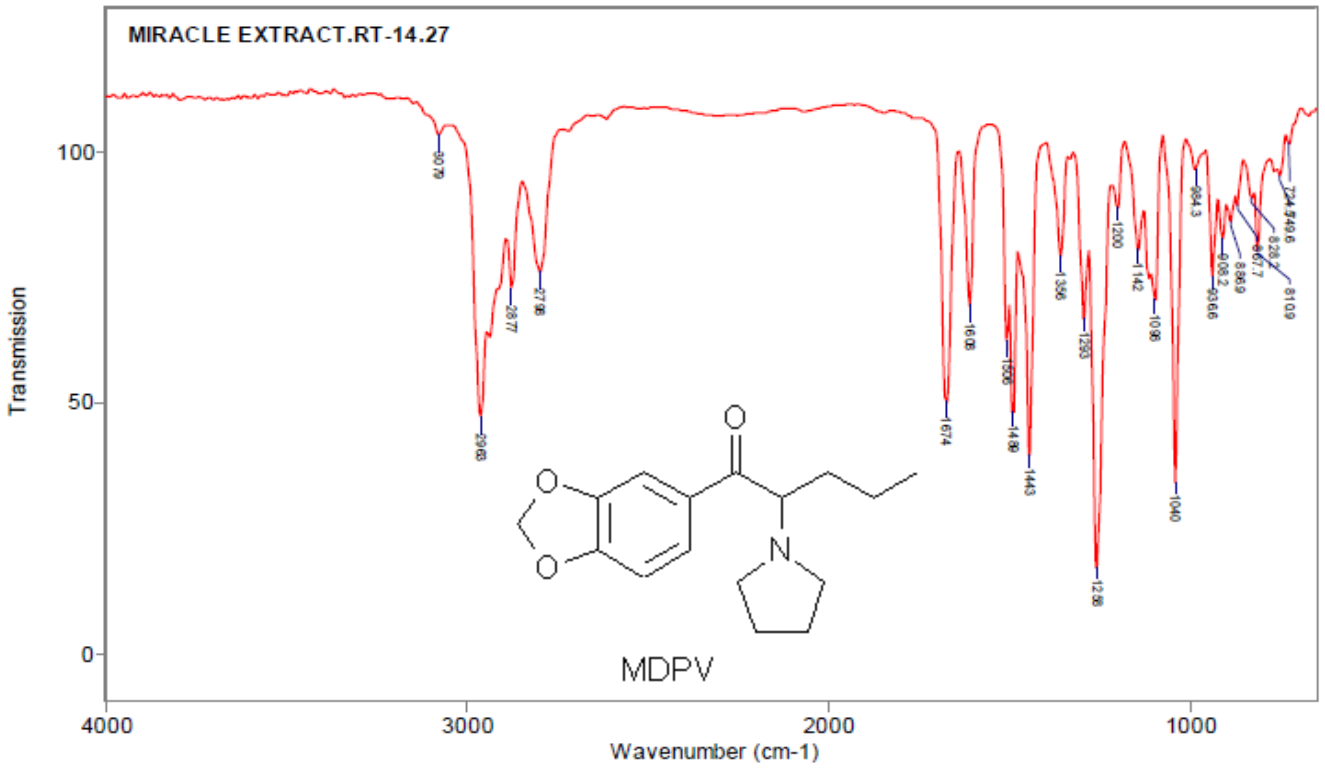
Library spectra in blue is from traditional bench top IR spectral library from Fiveash Data Management.

The overlay illustrates the excellent correlation of DiscovIR spectra with traditional solid phase spectral libraries.



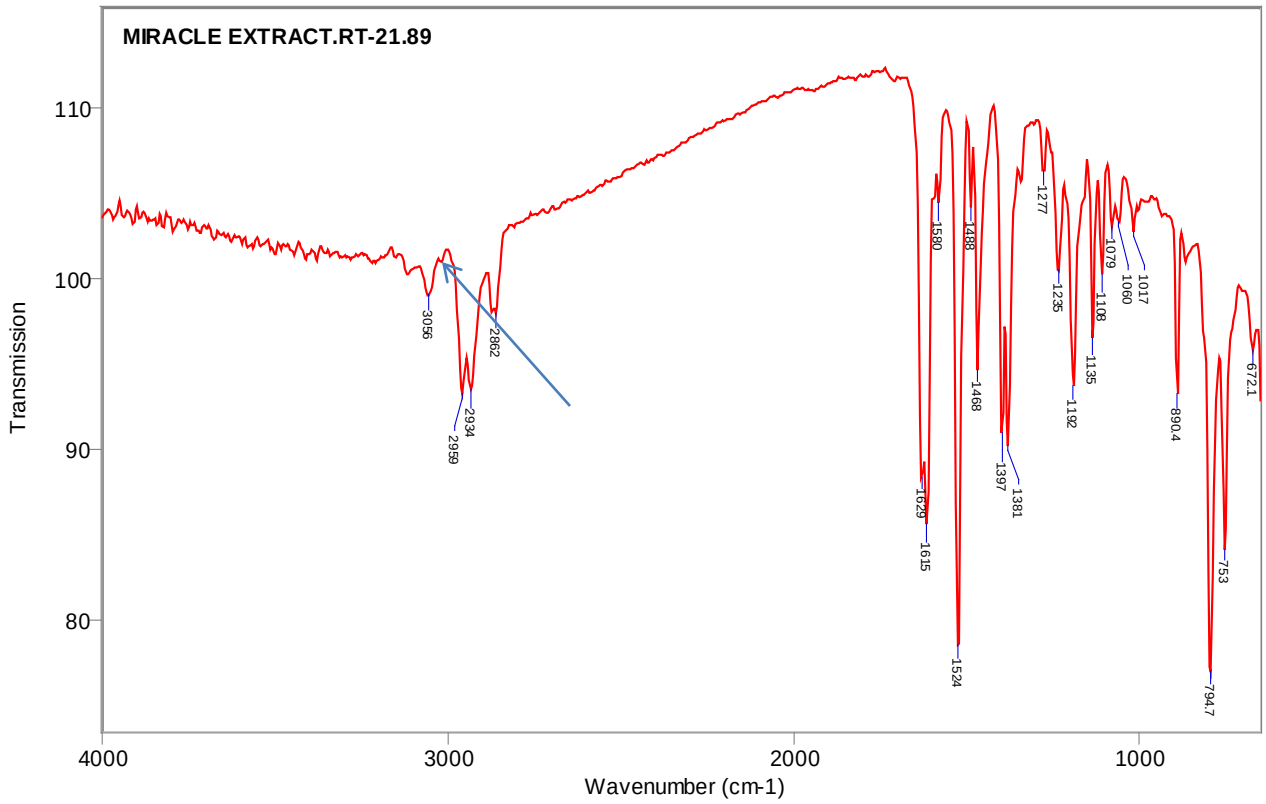
Solid Phase Mid-IR Spectrum of MDPV

❹

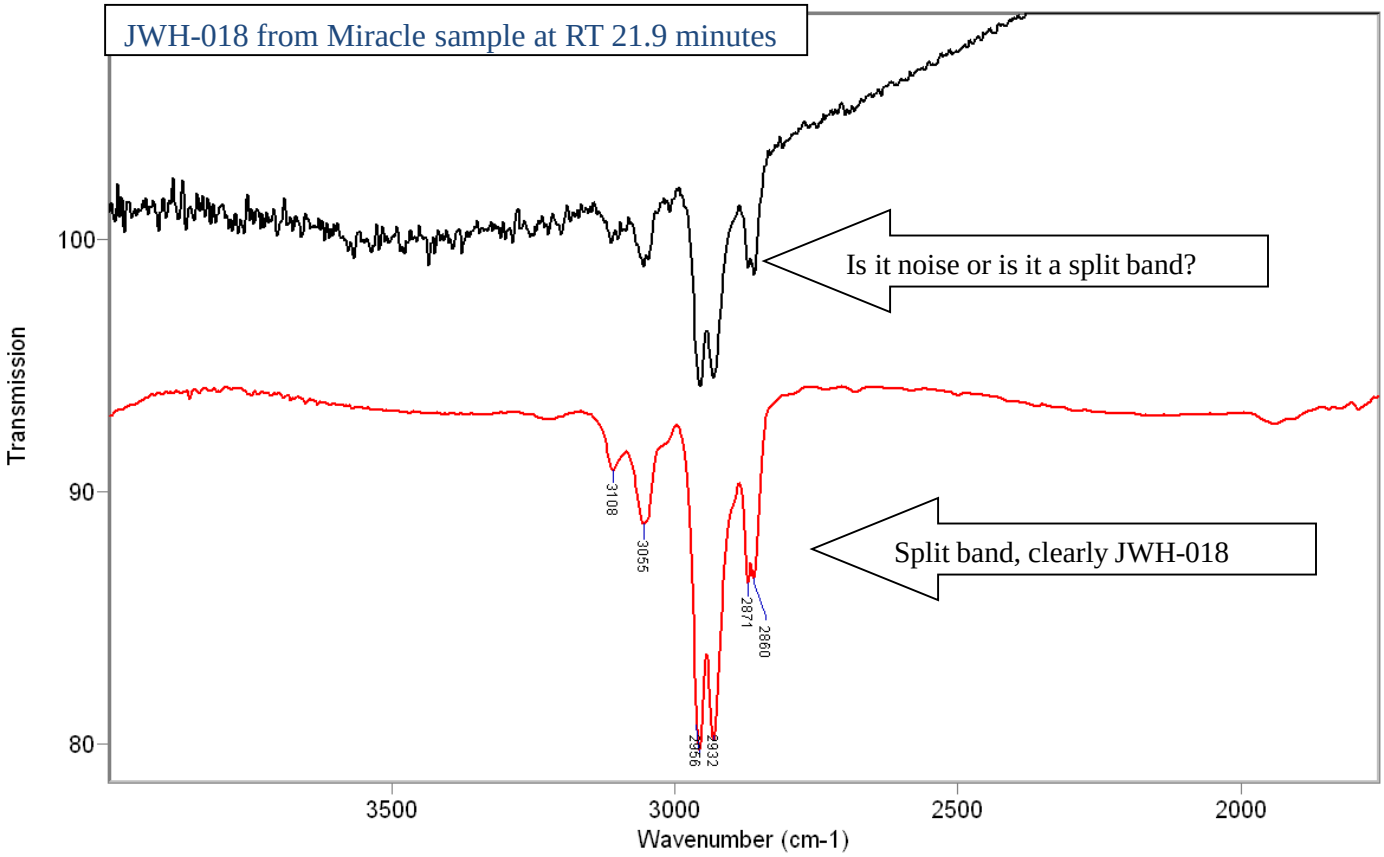


JWH-018

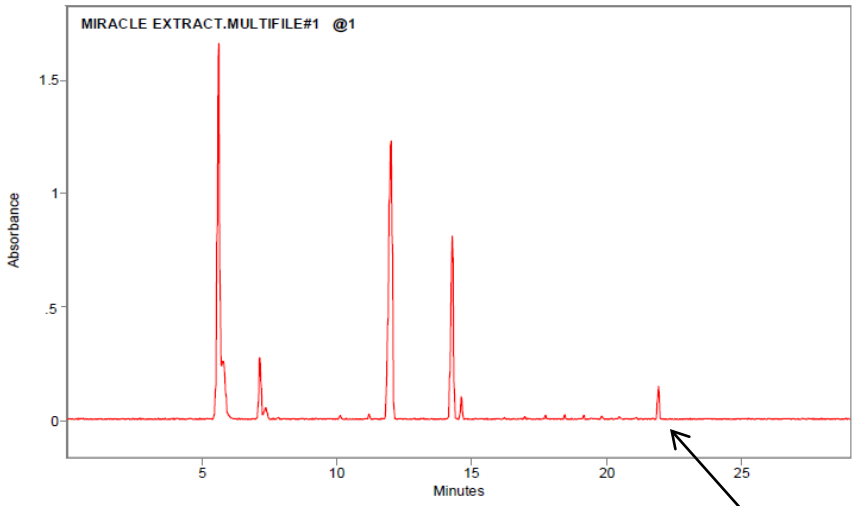
❺

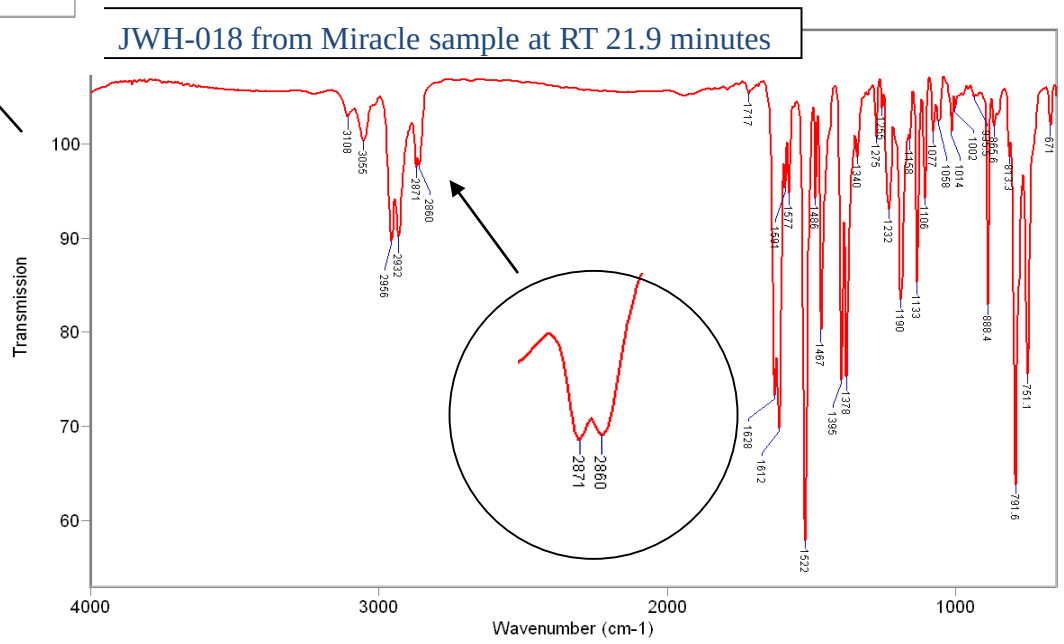

MIRACLE EXTRACT.RT-21.89
Transmission
110
100
90
80
4000
3000
2000
1000
Wavenumber (cm-1)
3056
2959
2934
2862
1629
1615
1580
1524
1488
1468
1397
1381
1277
1235
1192
1135
1108
1079
1060
1017
890.4
794.7
753
672.1

Enhancing spectral quality of minor (4%) component

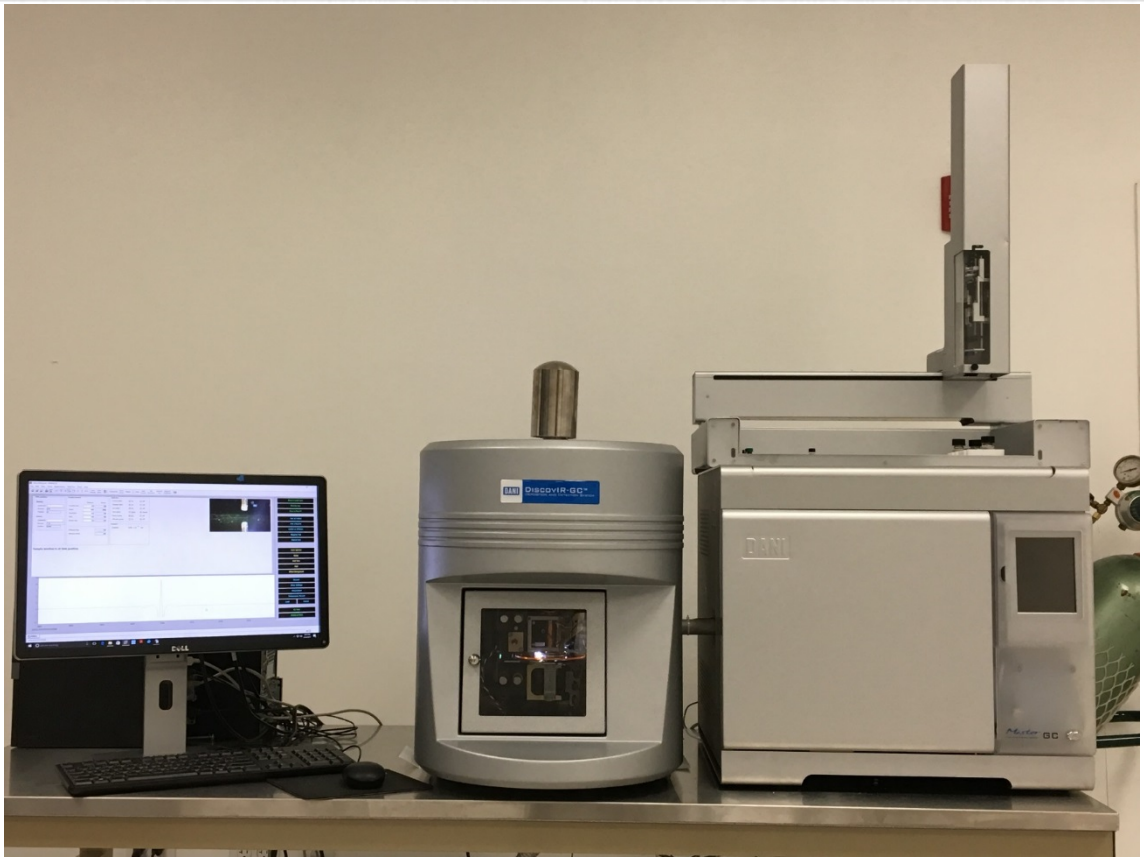


Band splitting of a weak band of the minor component.





Integrated DiscovIR-Master GC



DiscovIR Fully Automated Software Package

- Control of Master GC , Autosampler and DiscovIR Instruments
- DiscovIR detector Data Acquisition and Processing
- Library Search
- Report Generator

DiscovIR – GC Installed Customer Base

FORENSIC DRUG ANALYSIS

US DEA Southwest Laboratory, Vista, CA
US DEA Special Testing Lab, VA
US ARMY, CIL, Forest Park, GA
US Food and Drug Administration, Rockville, MD
Massachusetts State Police, Sudbury, MA
New York State Police
Alabama Dept. of Forensic Sciences, Mobile, AL
Baltimore County Police, Forensic Lab, MD
Louisiana State Police Forensic Lab. LA
Jefferson Parish Sheriff's Office, LA
Georgia Bureau of Investigation, GA
Pennsylvania State Police Forensic Lab, PA
Vermont State Police Forensic Lab, VT
Crime Lab, NV Michigan State Police
Virginia Dept. of Forensic Science – (4 systems
Miami Valley Regional Crime Lab, OH
Indiana Dept. of Forensic Science
Wausau Crime Lab, WI

Canada Border Services Agency
Canada Health Services – (2 systems) Washoe County
Forensic Sciences of South Australia, AU
Victoria Forensic Science Centre, AU
Forensic Science Institute of Zurich
Saint Gallen Cantonal Police
Health Canada DAS – (3 systems)
Singapore Health Sciences Authority
Japan National Police (9 systems)
Japan Custom Laboratory
Slovenia State Police
Landeskriminalamt Germany (3 systems)
Finnish Customs Laboratory

CHEMICAL WEAPONS ANALYSIS

Oak Ridge National Laboratory, TN
Lawrence Livermore National Lab, CA
Bundeswehr Institute, Germany
US ARMY, ECBC, Aberdeen
US Naval Research Laboratory , DC
OPCW, The Hague
ARMSCOR, Dept. Defense, So. Africa
Dstl Porton Down, United Kingdom

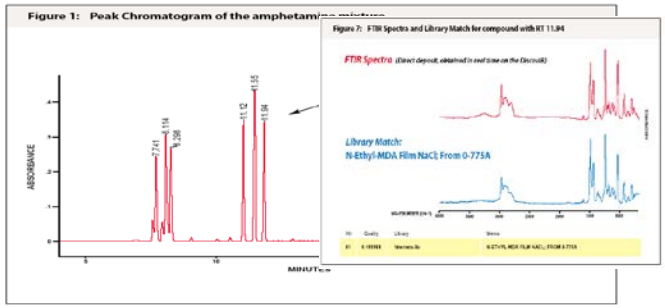
CROP SCIENCE

Universidade do Parana, Sao Paulo, Brazil
University of Tokyo, Japan
DuPont, Newark, DE
El Colegio de la Frontera Sur, Mexico
Technical University Braunschweig, Germany

DiscovIR-GC™ The Solution for Emerging Drugs

Step #1 Separation by Gas Chromatography

Step #2 Controlled Deposition - unique solid phase deposition.



Step #3 Chemical Identification - using FTIR specificity (positional and stereoisomers)

DiscovIR-GC Benefits

- **Solid-Phase transmission IR Spectra**
  - **4000-700 cm^{-1} Range with 4 cm^{-1} Resolution**
  - **High Sensitivity: 25 nanogram**
  - **Isomer Determination**
- **Analysis of Mixtures with Infrared Spectroscopy**
  - **Chromatography followed by Infrared identification of purified components**
- **Fits into existing lab workflow**
  - **1mg/ml concentrations**
  - **1 ul injections**
  - **IR spectra compatible with labs existing ATR and Transmission libraries**
- **Can be coupled with Mass Spectrometer**

Types of Mass Spectrometers

• SQ	Most common	
• ITD		
• TOF	$$	More Sensitive, Higher resolution
• QQQ	$$$	High sensitivity, target compounds only
• Orbitrap	$$$$	High mass accuracy, very high price, research tool

TOF
Typically higher resolution
Long flight tube = higher mass accuracy = $$$

New trend emerging into market: Shorter flight tube = lower mass accuracy = Much lower cost = $

Single Quadrupole MS

Well proven mass analyser

Relatively inexpensive

Strength: high efficiency of target ions (SIM)
Filters out non-target ions

Weakness: scanning instrument
- Faster scanning = poorer sensitivity
- Fewer scans per peak
- Poor resolution for fast GC
- Sens reduction at higher masses
  - Mass discrimination during scanning
  - Low resolution, unit mass

New Trend TOF MS

Well proven mass analyser

More expensive historically with long flight tube

No scanning, all masses all the time
- Sens ~ SQ SIM but with all masses
- More scans per peak, 100's per sec
- Great for fast GC, GC X GC
- High sens over entire mass range
- Higher resolution, 0.01 – 0.0001mass
- Long flight tube = high res high mass accuracy

Single Quadrupole MS

Scanning based analyzer

Acquisition rate: 1000amu/sec, 3-5 spectra/sec
Dependent on mass range
Scanning takes time, high mass discrimination

Spectral data points/peak: 7-20
Somewhat compatible with Fast GC
Incompatible with GC X GX
Poor smoothing of data: too few data points

Mass accuracy: 1.0amu Unit Mass

Cost: 60-80K, 50K with diffusion pump

Sensitivity: 100pg full spectrum, 1pg SIM
Satisfactory- it is what everyone is used to

New Trend TOF MS

Pulsed based Analyzer: all ions each pulse – no scanning

Acquisition rate: 1000 spectra/sec
Independent of mass range
No high mass discrimination

Spectral data points/peak: 1000
Compatible with FAST GC (200 peaks/chromatogram)
Compatible with GC X GC (1000 peaks/chromatogram)
Good peak smoothing: plenty of data points

Mass accuracy: 0.01 – 0.0001amu flight tube length
Short flight tube = 1.0amu Unit Mass

Cost: Long flight tube-higher mass accuracy = 130-240K
Short flight tube: 70K ~ single quadrupole

Sensitivity: 1pg full spectrum – more than satisfactory

Master TOF-MS with Master GC by DANI Instruments

Please Contact for Questions

Jeff Kearney

Jeff.Kearney@dani-instruments.com