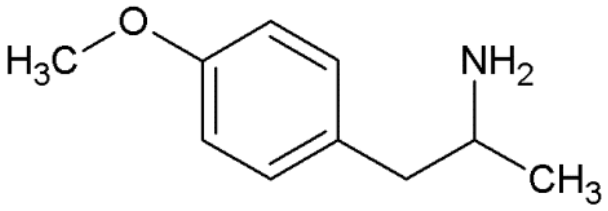
p-METHOXYAMPHETAMINE

Latest Revision: May 16, 2013



1. SYNONYMS

CFR: *p*-Methoxyamphetamine (PMA)

CAS #: Base: 23239-32-9
Hydrochloride: 3706-26-1

Other Names: Phenethylamine, *p*-methoxy-alpha-methyl-, (+/-)-
Benzeneethanamine,4-methoxy-alpha-methyl-, (+/-)
(+/-)-*p*- Methoxyamphetamine
4-Methoxyamphetamine
(+/-)-p-Methoxy-.alpha.-methyl-phenethylamine
PMA
1-(4-Methoxyphenyl)-2-propanamine

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight
Base	$C_{10}H_{15}NO$	165.2
Hydrochloride	$C_{10}H_{15}NO \cdot HCl$	201.7

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Base	*	S	*	*	VS	VSS
Hydrochloride	I	SS	I	I	VS	S

A = acetone, C = chloroform, E = ether, H = hexane, M = methanol and W = water, VS = very soluble, FS = freely soluble, S = soluble, PS = sparingly soluble, SS = slightly soluble, VSS = very slightly soluble and I = insoluble *No literature available

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
Mandelin's	Green
Froehde's	Green
Meckes	Light Olive Green

3.2. GAS CHROMATOGRAPHY

Method: PMA-GCMSQ-1

Samples are to be dissolved in an appropriate solvent such as methanol or base extracted previous to injection

Instrument: Gas Chromatograph/Mass Spectrometer

Column: HP-5MS 15.0 m x 0.25 mm x 0.25 µm film

Carrier gas: Helium at 1.0 mL/min

Temperatures: Injector: 260°C
Transfer Line: 280°C
Oven program:
1) 140°C initial temperature for 0.5 min
2) Ramp to 310°C at 38°C/min
3) Hold final temperature for 0.7 min

Injection Parameters: Split Ratio=25:1, 1 µL injected

COMPOUND	RRT	COMPOUND	RRT
dimethyl sulfone	0.46	caffeine	2.05
amphetamine	0.64	lidocaine	2.15
methamphetamine	0.71	chlorpheniramine	2.40
PMA	**1.00**	procaine	2.44
pseudo/ephedrine	1.05	cocaine	2.78
PMMA	1.11	triprolidine	2.88

MDA	1.26	O-6 monoacetylmorphine	3.34
MDMA	1.47	heroin	3.51
acetaminophen	1.70		

3.3. GAS CHROMATOGRAPHY

Method: PMA-GCMSQ-2

Samples are to be dissolved in an appropriate solvent such as methanol or base extracted before injection.

Instrument: Gas Chromatograph/Iontrap

Column: ZB-5 30.0 m x 0.25 mm dia. x 0.25µm film

Carrier gas: Helium at 1.0 mL/min

Temperatures: Injector: 250°C
Transfer Line: 280°C
Oven program:
1) 100°C initial temperature for 1.0 min
2) Ramp to 310°C at 30°C/min
3) Hold final temperature for 3.5 min

Injection Parameters: Split Ratio=20:1, 1 µL injected

COMPOUND	RRT	COMPOUND	RRT
dimethyl sulfone	0.50	caffeine	1.41
amphetamine	0.52	lidocaine	1.44
methamphetamine	0.79	chlorpheniramine	1.54
pseudo/ephedrine	0.97	procaine	1.55
PMA	**1.00**	cocaine	1.68
PMMA	1.05	triprolidine	1.71
MDA	1.09	O-6 monoacetylmorphine	1.92
MDMA	1.14	heroin	2.01
acetaminophen	1.27		

3.4. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method PMA-LCQ1

The samples are to be dissolved in an appropriate solvent such as 0.1 N HCl or water.

Instrument: High performance liquid chromatograph equipped with diode array

Column: C18, 5 µm, 150 mm x 4.6 mm

Detector: UV, 210 nm, 10 BW

Flow: 1.0 mL/min

Injection Volume: 3 µL

Buffer: 4000 mL distilled water, 30 mL phosphoric acid, 10 g sodium hydroxide and 8.0 mL hexylamine at pH 2.5

Mobile Phase: Buffer: acetonitrile 93:7 for12 min

Typical Retention Time: PMA: 5.677min

COMPOUND	RRT (min)
pseudoephedrine	0.58
amphetamine	0.76
MDA	0.85
methamphetamine	0.90
MDMA/**PMA**	**1.00**
PMMA	1.20
MDEA	1.32
caffeine	1.79
ketamine	1.93

**MDMA and PMA coelute.

4. SEPARATION TECHNIQUES

PMA can be separated from matrixes by solvent extraction using the solubility.

5. QUANTITATIVE PROCEDURES

5.1. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method PMA-LCQ1

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of PMA hydrochloride at approximately 0.50 mg/mL using 0.1 N HCl.

Sample Preparation:
Accurately weigh an amount of sample into a volumetric flask and dilute with 0.1 N HCl. If necessary dilute the sample so the final concentration approximates the standard concentration or falls within the linear range. Filter sample with a 0.45-micron filter.

Instrument:	High performance liquid chromatograph equipped with diode array
Column:	C18, 5 µm, 150 mm x 4.6 mm
Detector:	UV, 210 nm, 10 BW
Flow:	1.0 mL/min
Injection Volume:	3 µL
Buffer:	4000 mL distilled water, 30 mL phosphoric acid, 10 g sodium hydroxide and 8.0 mL hexylamine at pH 2.5
Mobile Phase:	Buffer: acetonitrile 93:7 for 12min
Typical Retention Time:	PMA: 5.677min
Linear Range:	0.0612-1.224 mg/mL
Repeatability:	RSD less than 3%
Correlation Coefficient:	0.99999
Accuracy:	Error less than 5%

COMPOUND	RRT
pseudoephedrine	0.58
amphetamine	0.76

MDA	0.85
methamphetamine	0.90
MDMA/PMA	**1.00**
PMMA	1.20
MDEA	1.32
caffeine	1.79
ketamine	1.93

**Please note MDMA and PMA coelute. This method is not valid for mixtures of MDMA and PMA.

6. QUALITATIVE DATA

See spectra on the following pages for FT-IR, Mass Spectrometry, Nuclear Magnetic Resonance, UV, and Vapor Phase IR.

7. REFERENCES

Blachut, Dariusz, Wojtasiewicz, Krystyna, Czarnocki, Zbigniew, "Identification and Synthesis of Some Contaminants Present in 4-Methoxyamphetamine (PMA) Prepared by the Leukart Method," Forensic Science International, 127, 2002.

Clarke, E.G.C., *Isolation and Identification of Drugs, 2nd Edition*, The Pharmaceutical Press, 1986.

Coates, J., and Reffner, J., "Visualization of Micro-ATR Infrared Spectroscopy," *Spectroscopy*, Vol. 14, #4, April 1999.

Coumbaros, John C., Kirkbride, K. Paul, and Klass, Gunter, "Application of Solid-Phase Micrextraction to the Profiling of an Illicit Drug: Manufacturing Impurities in Illicit 4-Methoxyamphetamine," Technical Note (DEA).

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Kirkbride, K. Paul, Ward, A. David, Jenkins, Natalie F., Klass, Gunter, and Coumbaros, John C., "Synthesis of 4-Methyl-5-Arylpyrimidines and 4-Arylpyrimidines: Route Specific Markers for the Leukardt Preparation of Amphetamine, 4-Methoxyamphetamine, and 4-Methoxymethamphetamine," Forensic Science International, 115, 2001.

Kochana, J., Wilamowski, J., and Parczewski, A., "Profiling of Impurities in *p*-Methoxymethamphetamine (PMMA) by means of SPE/TLC method, Examination of the Influence of Experimental Conditions According to 2^4 factorial," Forensic Science International, 134, 2003.

Kochana, J., Wilamowski, J., Parczewski, M., Surma, M., “Synthesis of Standards of the Most Important Markers of Leuckart p-Methoxymethamphetamine (PMMA), Examination of the Influence of Experimental Conditions and a Drug Diluent on SPE/TLC Profiling,” Forensic Science International, 134, 2003.

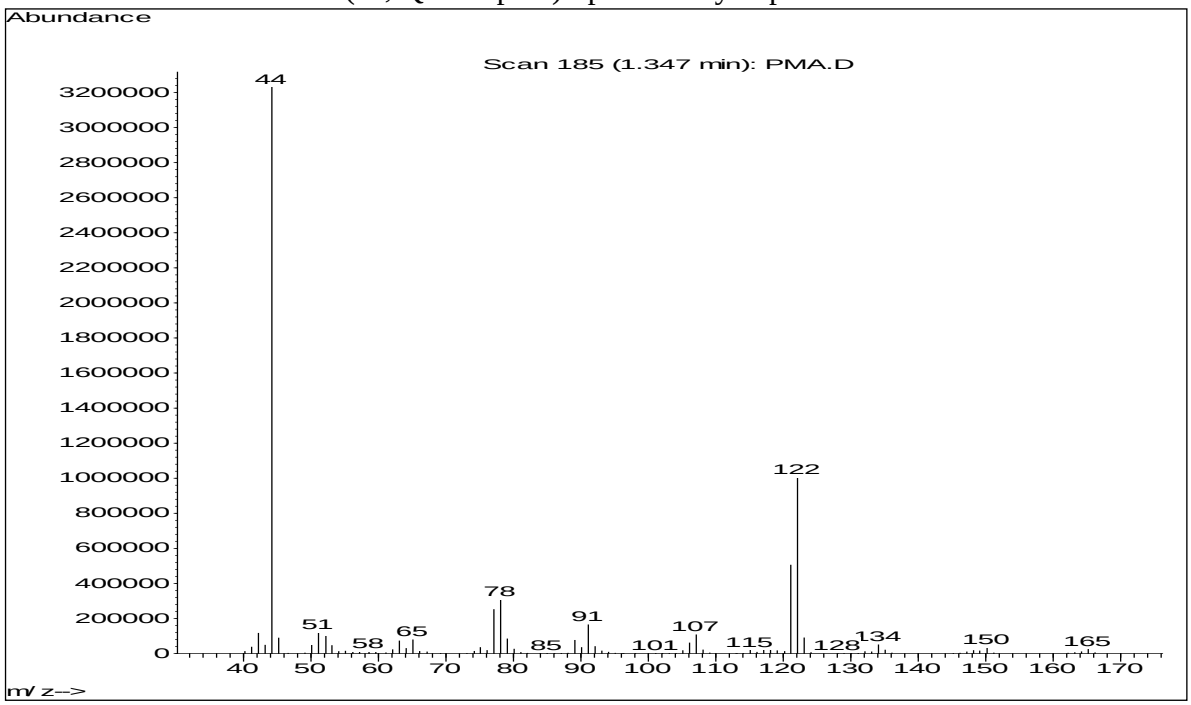
Waumans, Dieter, Bruneel, Noel, Hermans, Bas, and Tytgat, Jan, “A Rapid and Simple GC/MS Screening Method for 4-Methoxyphenol in Illicit Prepared 4-Methoxyamphetamine (PMA), Technical Note (DEA).

8. ADDITIONAL RESOURCES

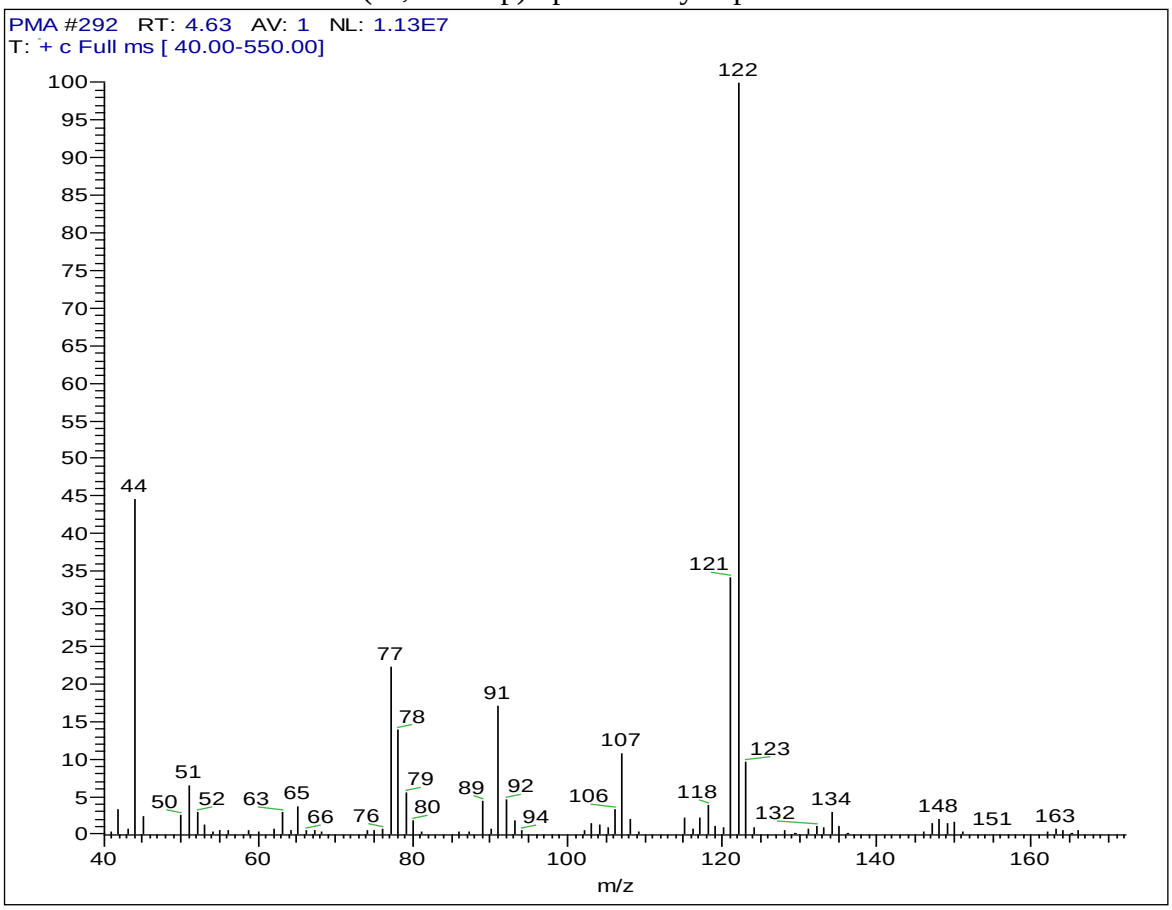
Forendex

Wikipedia

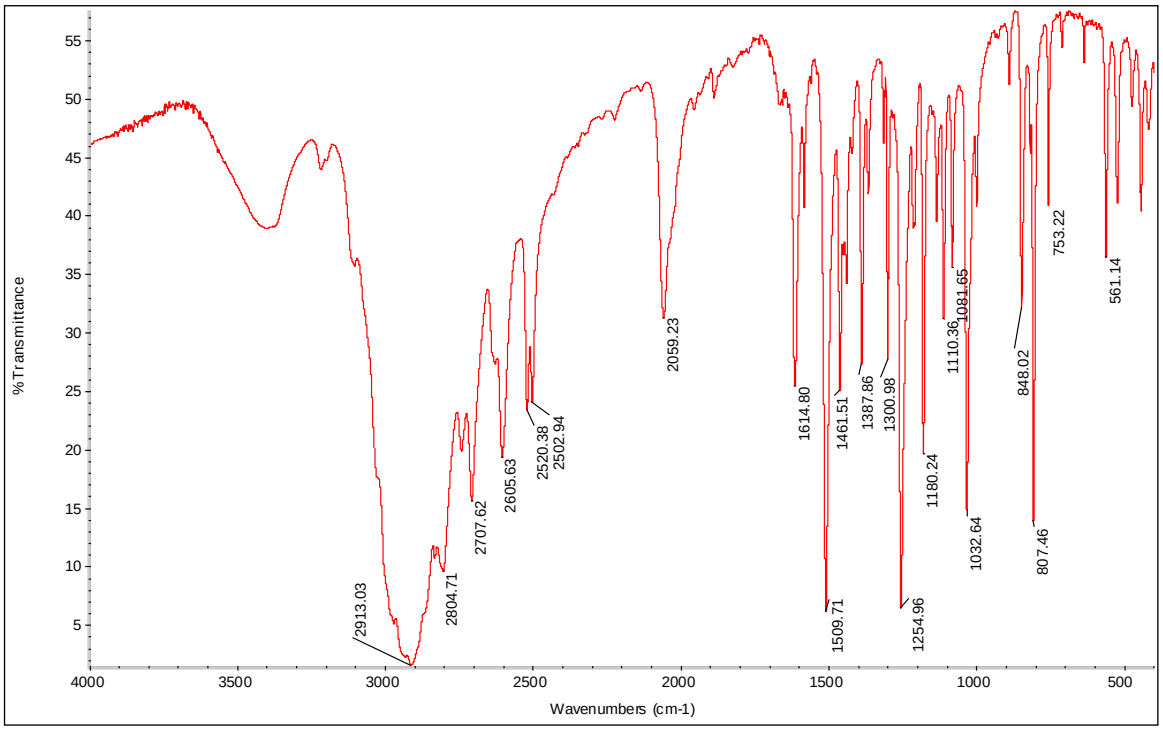
MS (EI, Quadrupole): p-Methoxyamphetamine



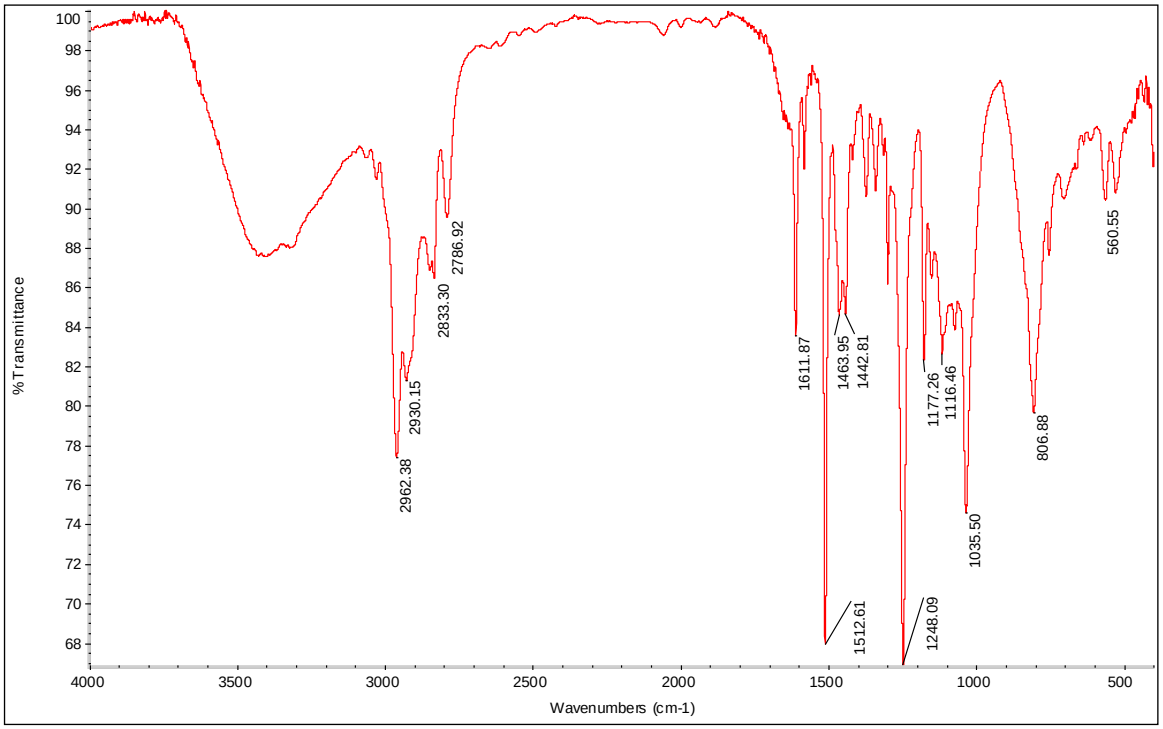
MS (EI, ion trap): p-Methoxyamphetamine



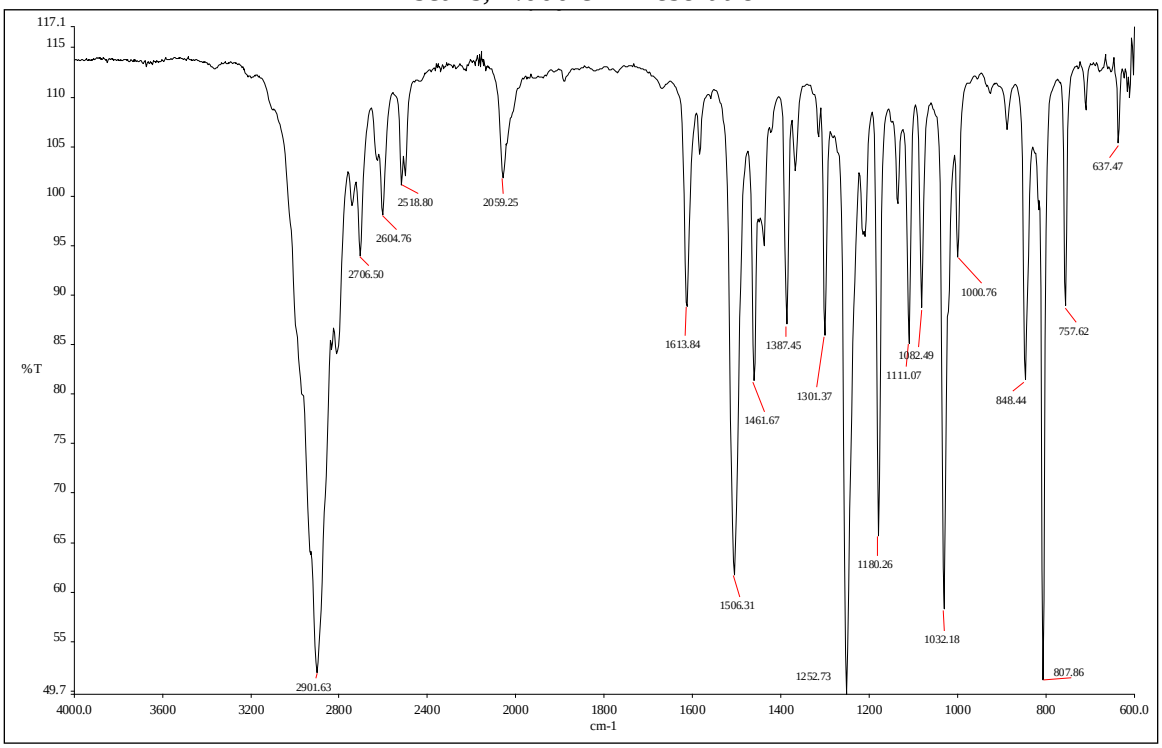
FTIR: p-Methoxyamphetamine Hydrochloride
KBr 4 scans, 4.000 cm^{-1} resolution



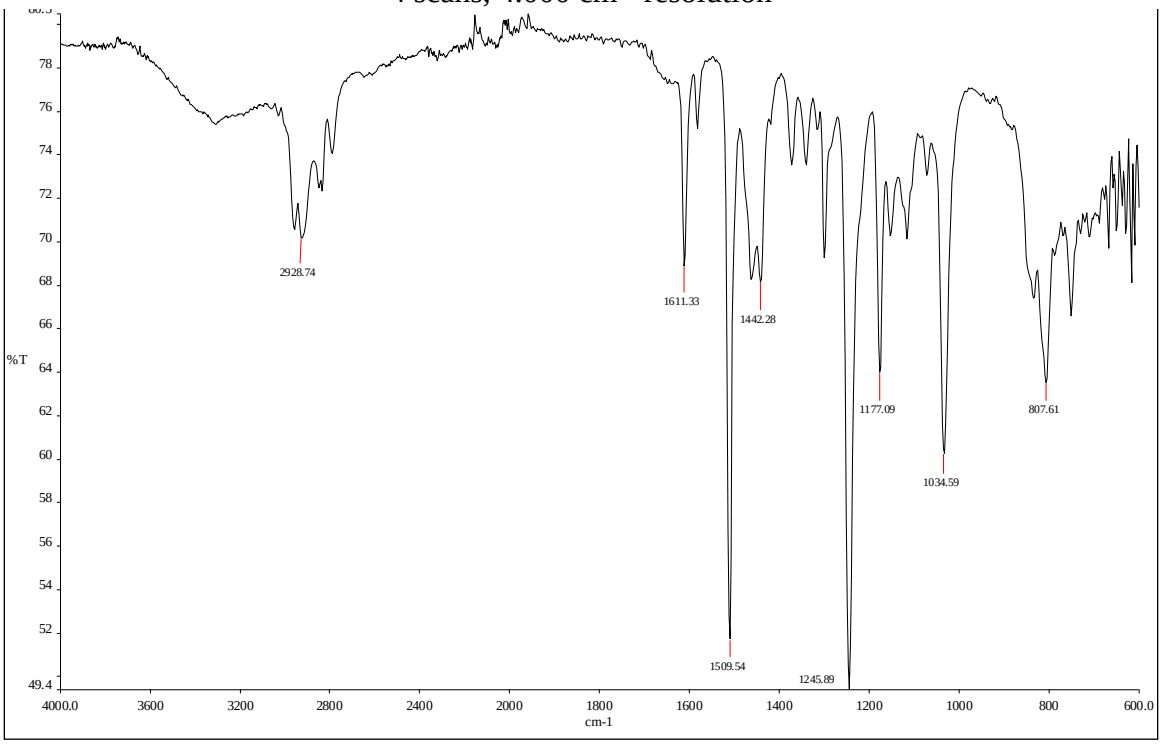
FTIR: p-Methoxyamphetamine base, KBr smear
4 scans, 4.000 cm^{-1} resolution



FTIR (Diamond ATR): p-Methoxyamphetamine HCl
4 scans, 4.000 cm^{-1} resolution

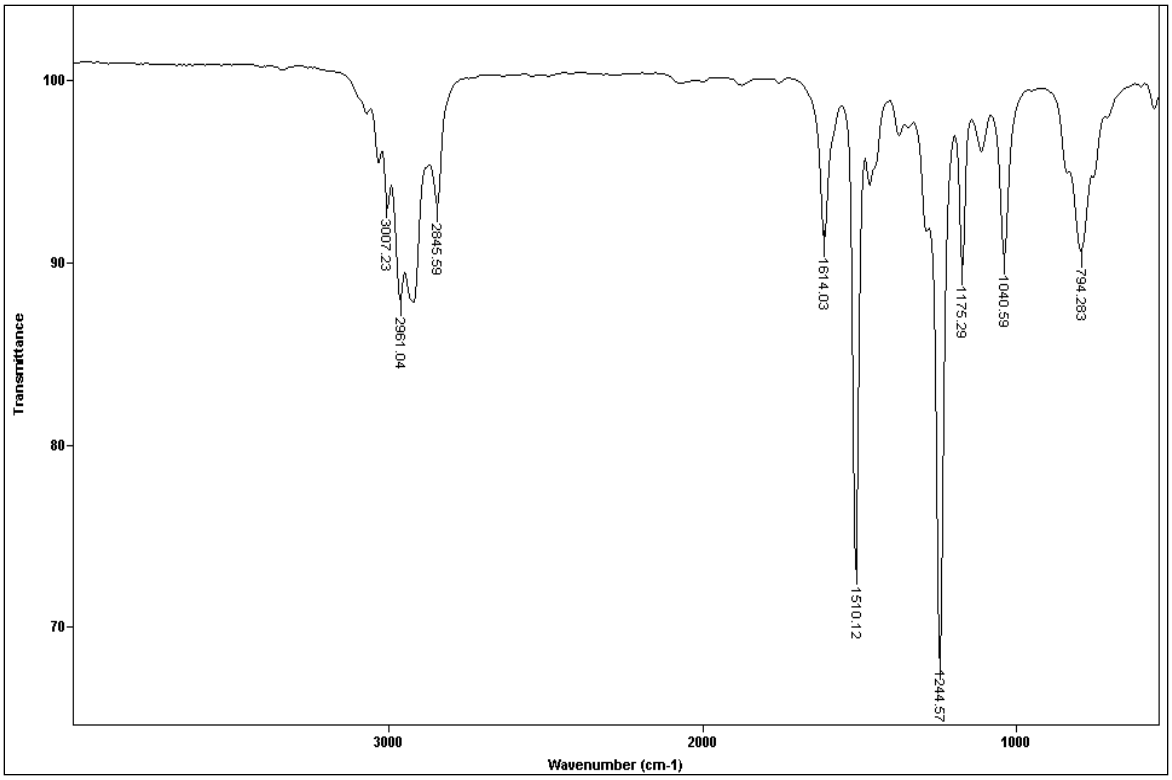


FTIR (Diamond ATR): p-Methoxyamphetamine base
4 scans, 4.000 cm^{-1} resolution



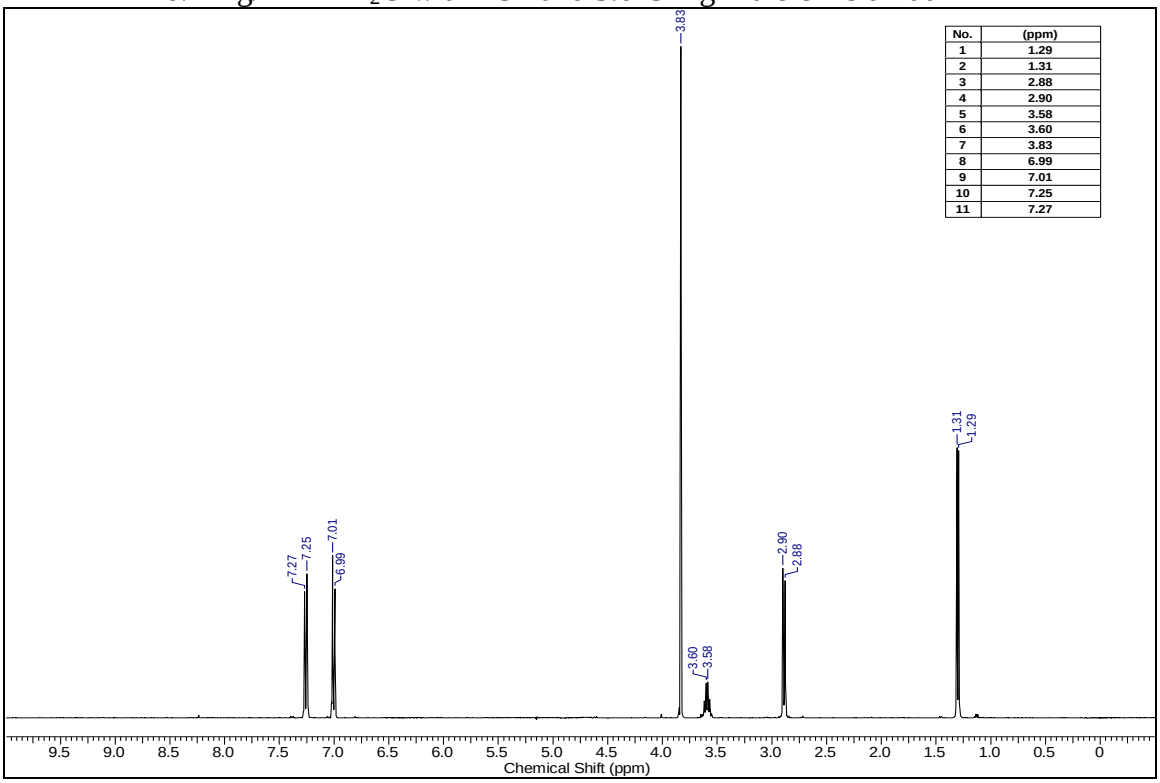
IR (VAPOR PHASE): p-Methoxyamphetamine

Na_2CO_3 extracted CH_2Cl_2

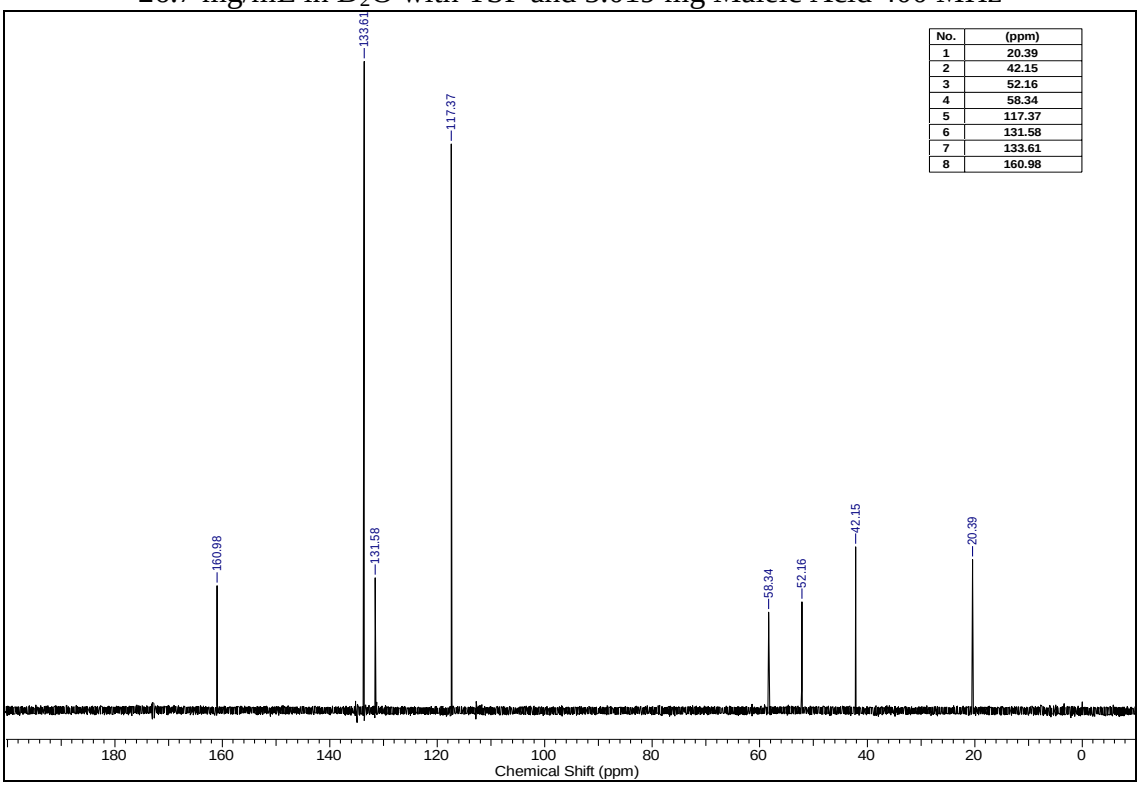


NMR (Proton): p-Methoxyamphetamine

26.7 mg/mL in D_2O with TSP and 5.019 mg Maleic Acid 400 MHz



NMR (Carbon): p-Methoxyamphetamine
26.7 mg/mL in D_2O with TSP and 5.019 mg Maleic Acid 400 MHz



No.	(ppm)
1	20.39
2	42.15
3	52.16
4	58.34
5	117.37
6	131.58
7	133.61
8	160.98

UV: p-Methoxyamphetamine HCl
in 0.1 N HCl