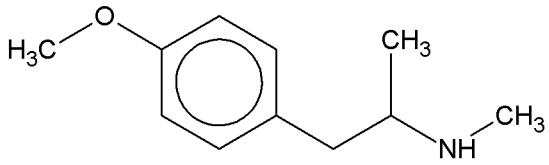
p-METHOXYMETHAMPHETAMINE

Latest Revision: August 9, 2005



1. SYNONYMS

CFR: *p*-Methoxymethamphetamine (PMMA)

CAS #: Base: 22331-70-0
Hydrochloride: unknown

Other Names: Phenethylamine, N,alpha-dimethyl-4-methoxy--alpha-(4-methoxyphenyl)-beta-methylaminopropane
(+/-)-*p*- Methoxymethamphetamine
4-Methoxymethamphetamine
(+/-)-*p*-Methoxy-.alpha.-methyl-phenethylamine
PMMA
4-MMA
1-(4-Methoxyphenyl)-N-methyl-2-propanamine

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	$C_{11}H_{17}NO$	179.3	177-178
Hydrochloride	$C_{11}H_{17}NO \cdot HCl$	215.7	

2.2. SOLUBILITY

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

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 information available.

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
Sodium Nitroprusside	Blue
Mecke's	Light Green

3.2. GAS CHROMATOGRAPHY

Method: PMMA-GCMSQ-1

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

Instrument: Gas Chromatograph / Mass Spectrometer

Column: HP-5MS 15.0 m x 0.25 mm x 0.25 µm film
Carrier gas: Hydrogen at 2.0 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	0.41	caffeine	1.84
amphetamine	0.58	lidocaine	1.93
methamphetamine	0.64	chlorpheniramine	2.16
PMA	0.89	procaine	2.19
pseudo/ephedrine	0.94	cocaine	2.50
PMMA	**1.00**	triprolidine	2.59
MDA	1.14	O6 monoacetylmorphine	3.00
MDMA	1.32	heroin	3.16
acetaminophen	1.53		

3.3. GAS CHROMATOGRAPHY

Method: PMMA-GCMSQ-2

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

Instrument: Gas Chromatograph / Ion trap

Column: ZB-5 30.0 m x 0.25 mm x 0.25 µm 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	0.48	caffeine	1.34
amphetamine	0.49	lidocaine	1.37
methamphetamine	0.75	chlorpheniramine	1.47
PMA	0.95	procaine	1.47
pseudo/ephedrine	0.95	cocaine	1.60
PMMA	**1.00**	triprolidine	1.63
MDA	1.04	O6 monoacetylmorphine	1.83
MDMA	1.09	heroin	1.91
acetaminophen	1.21		

3.4. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method PMMA-LCQ-1

Samples are to be dissolved in an appropriate solvent such as 0.1 N HCl.

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:	PMMA 6.757 min

COMPOUND	RRT
pseudoephedrine	0.48
amphetamine	0.63
MDA	0.70
methamphetamine	0.75
MDMA/PMA	0.82
PMMA	1.00
MDEA	1.10
caffeine	1.48
ketamine	1.60

*Note: MDMA/PMA coelute

4. SEPARATION TECHNIQUES

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

5. QUANTITATIVE PROCEDURES

5.1. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method PMMA-LCQ-1

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of PMMA 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:	PMMA 6.757 min
Linear Range:	0.0603-1.206 mg/mL
Repeatability:	RSD less than 3%
Correlation Coefficient:	0.99999
Accuracy:	Error less than 5%

COMPOUND	RRT
pseudoephedrine	0.48
amphetamine	0.63
MDA	0.70
methamphetamine	0.75
MDMA/PMA	0.82

PMMA	1.00
MDEA	1.10
caffeine	1.48
ketamine	1.60

*Note: MDMA/PMA coelute

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).

Del Cason, Terry, "The Identification of 4-Methoxyamphetamine (PMA) and 4-Methoxymethamphetamine (PMMA), Microgram, Volume 23, No. 8, August 2000.

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.

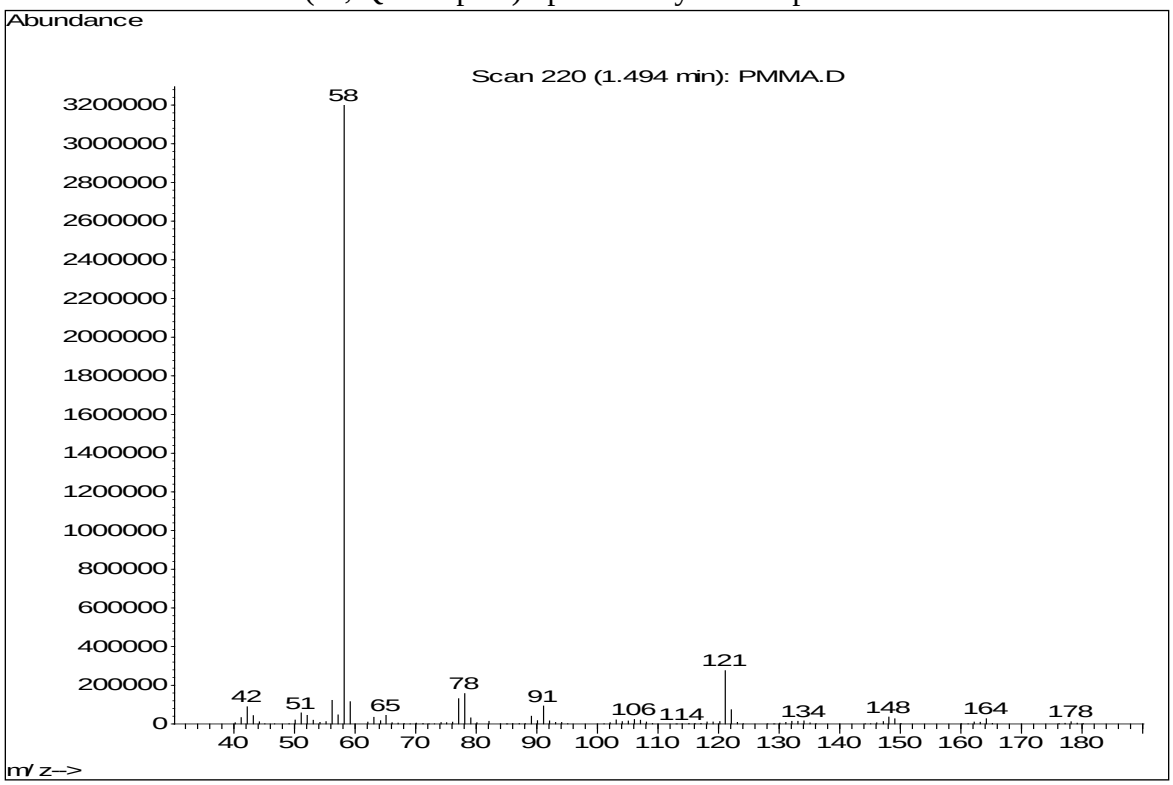
Kochana, J., Wilamowski, J., and Parczewski, A., "Profiling of Impurities in *p*-Methoxymethamphetamine (PMMA) by means of SPE/TLC method, Examination of the Influence of Experimental Conditions According to 2^4 factorial," Forensic Science International, 134, 2003.

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.

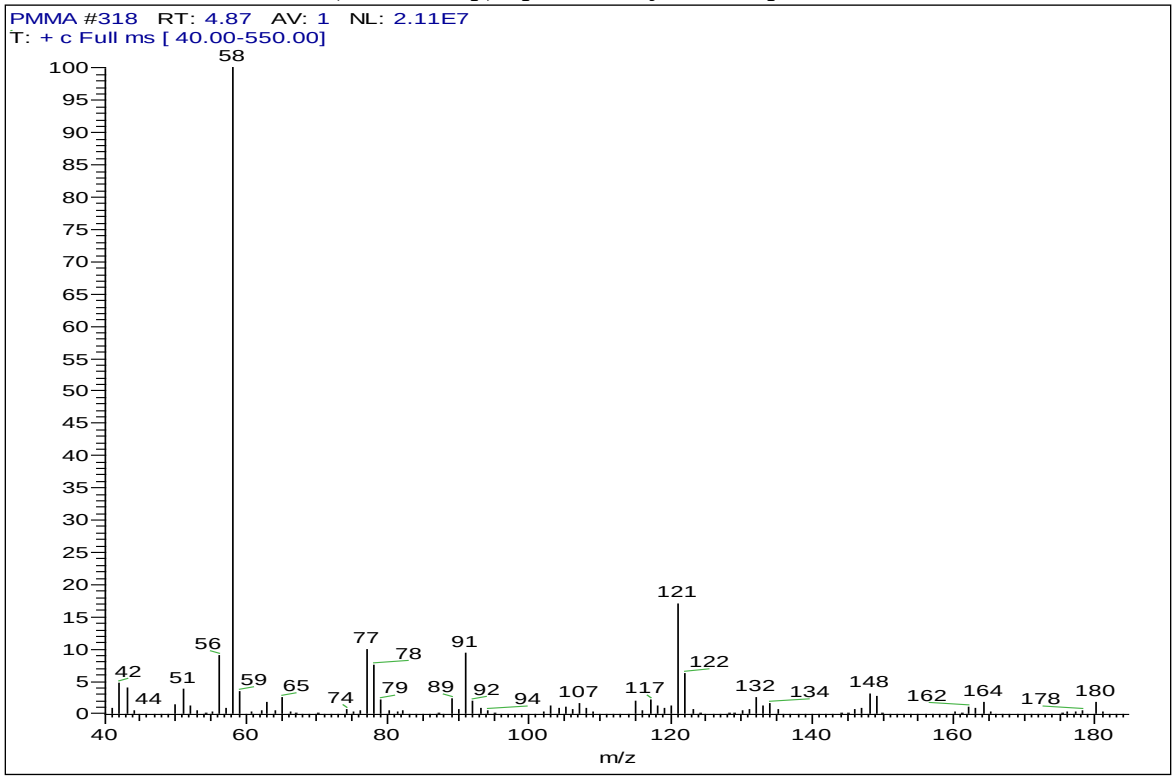
8. ADDITIONAL RESOURCES

Wikipedia

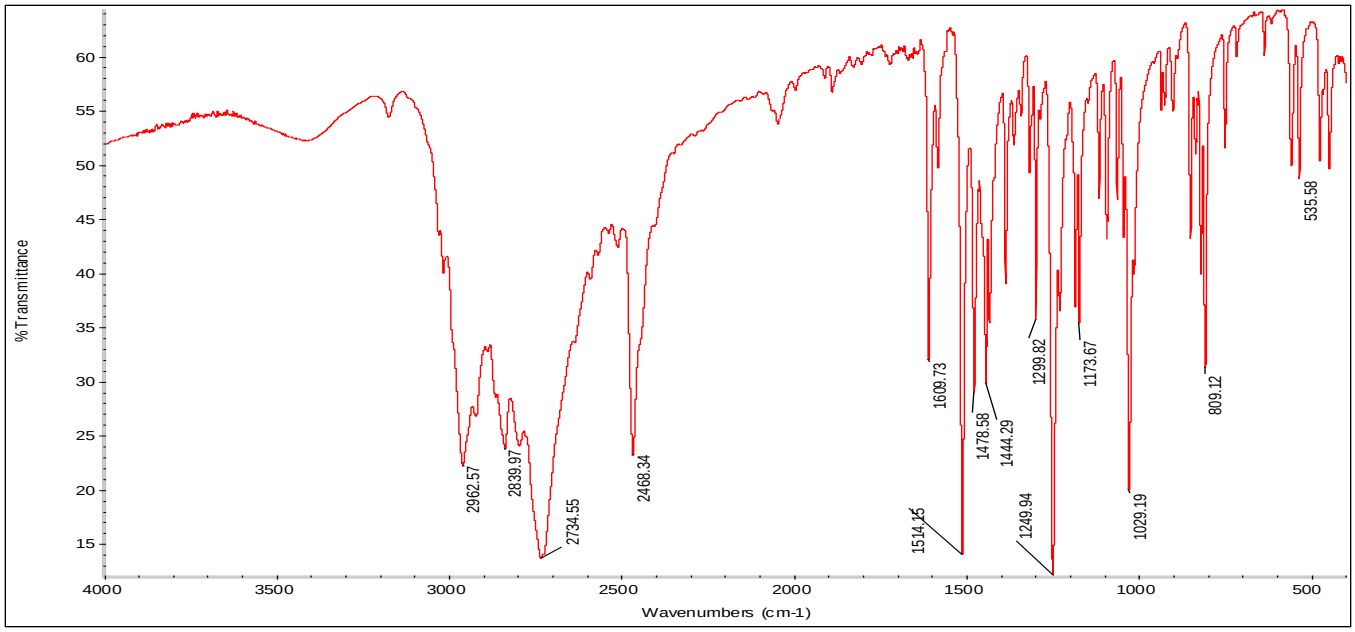
MS (EI, Quadrupole): p-Methoxymethamphetamine



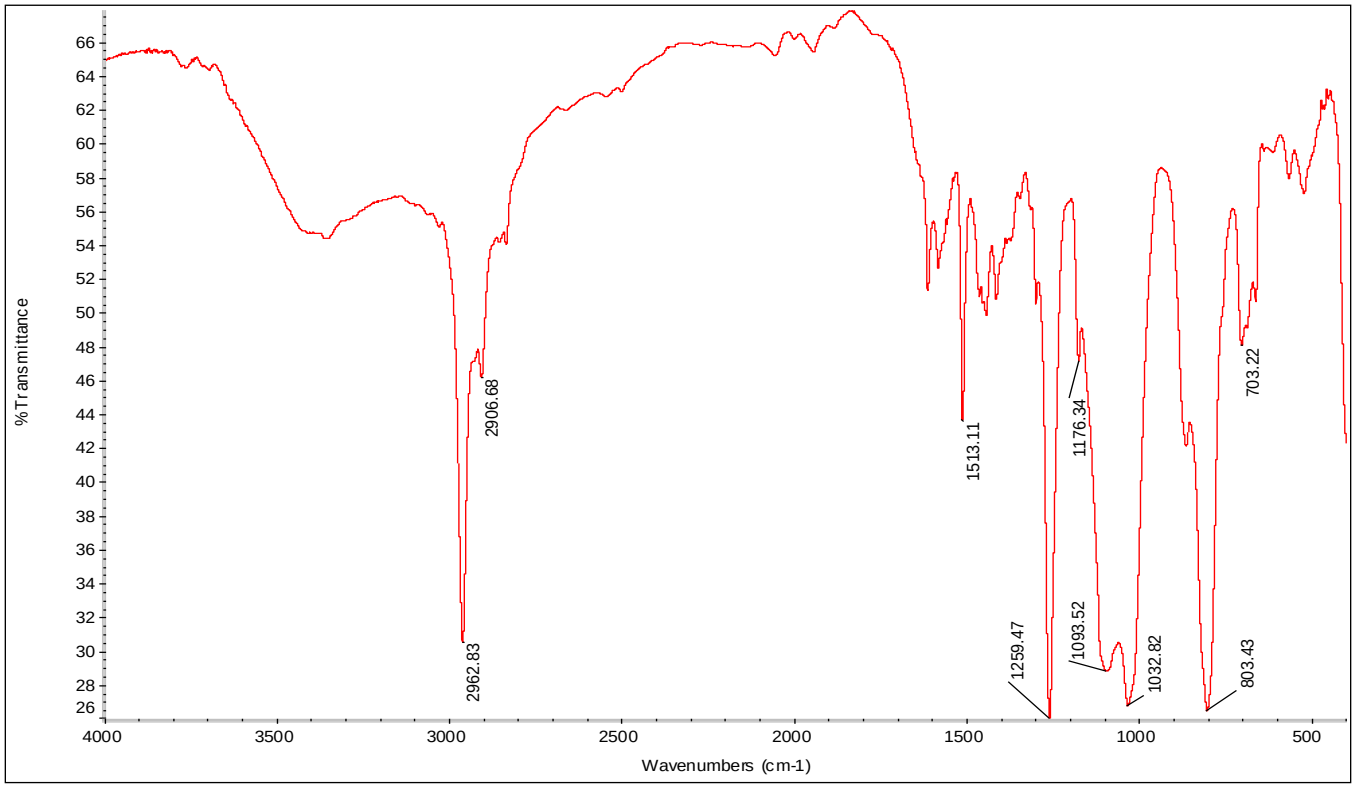
MS (EI, Ion trap): p-Methoxymethamphetamine



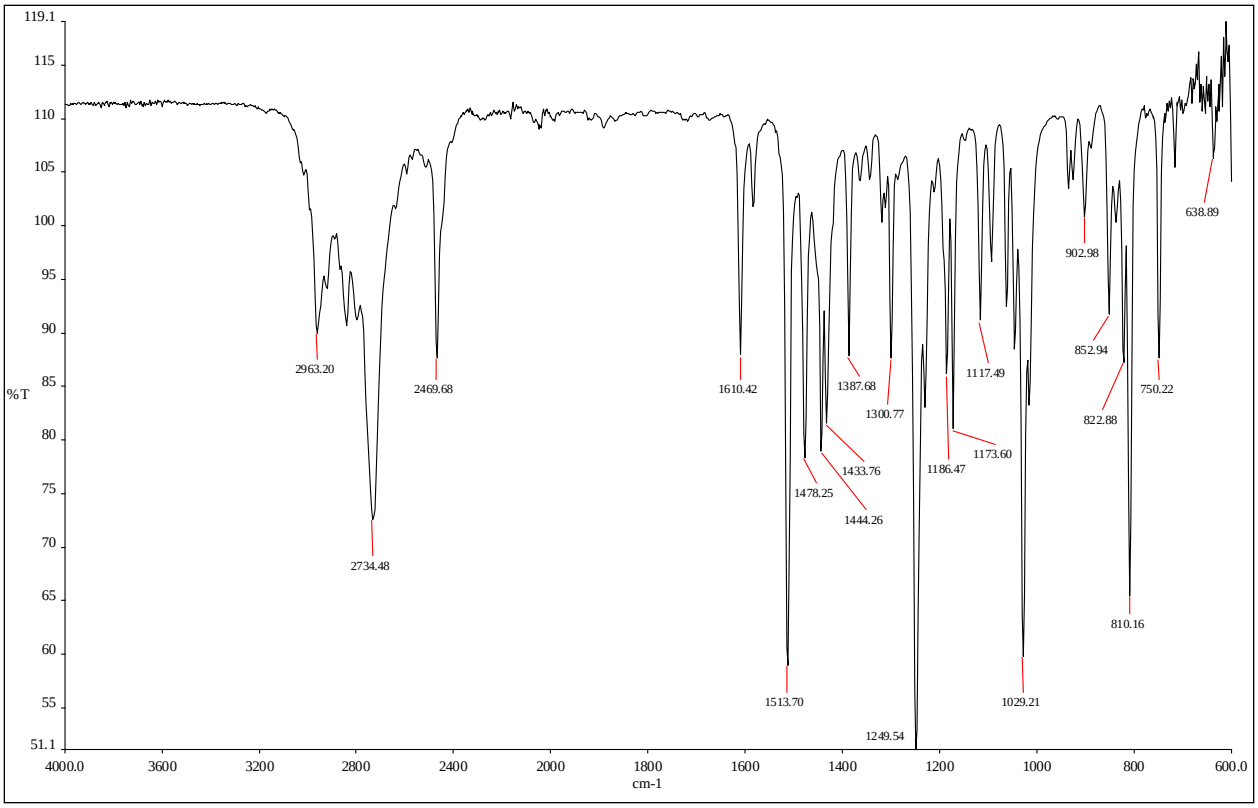
FTIR: p-Methoxymethamphetamine hydrochloride, KBr
4 Scans, 4.000 cm^{-1} resolution



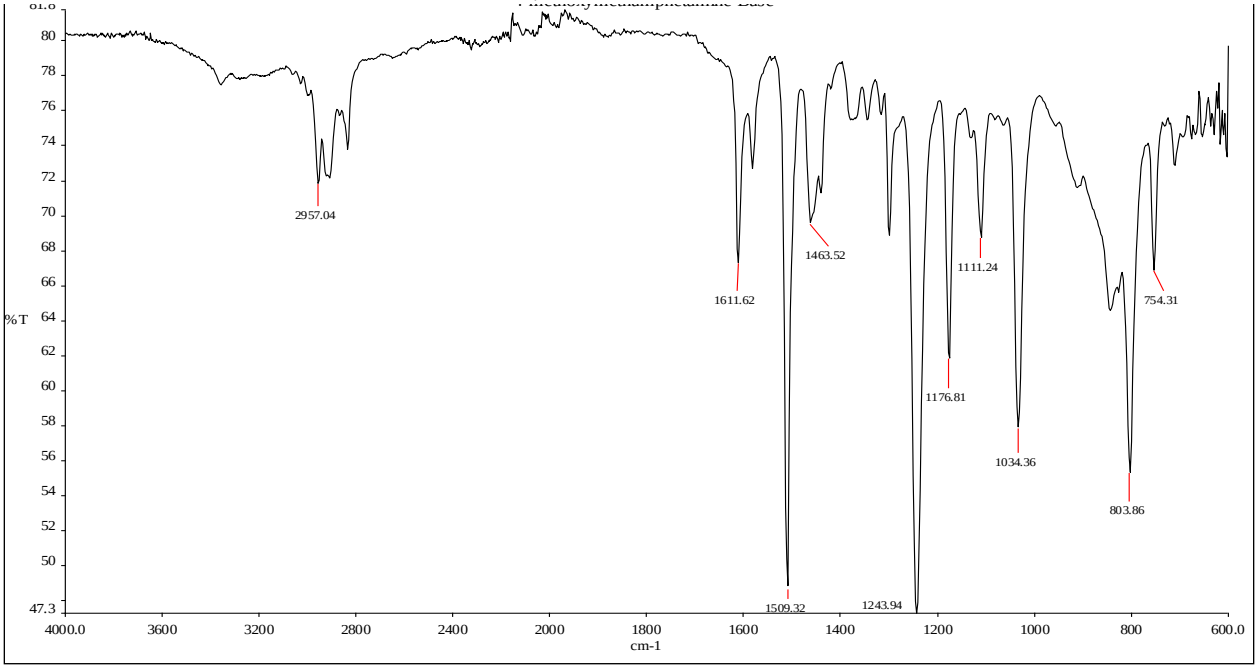
FTIR: p-Methoxymethamphetamine base KBr smear
4 scans, 4.000 cm^{-1} resolution



FTIR(Diamond ATR): p-Methoxymethamphetamine HCl
4 Scans 4.000 cm^{-1}Resolution

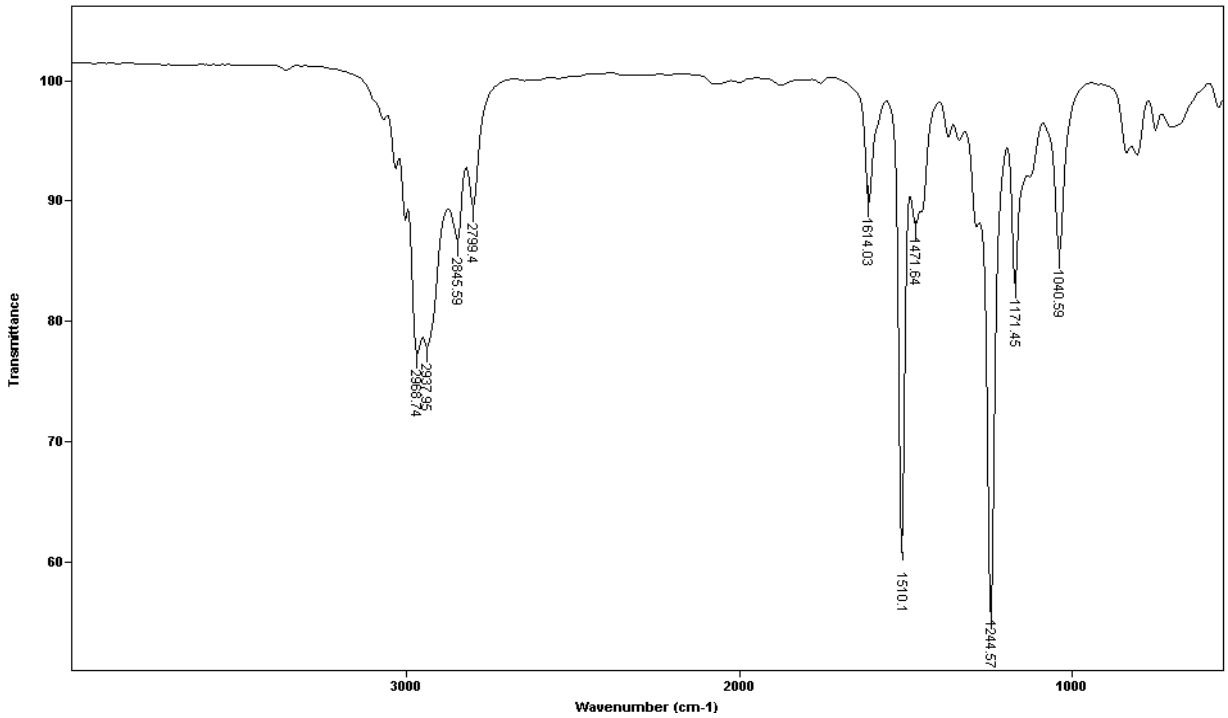


FTIR (Diamond ATR): p-Methoxymethamphetamine base
4 Scans, 4.000 cm^{-1} resolution



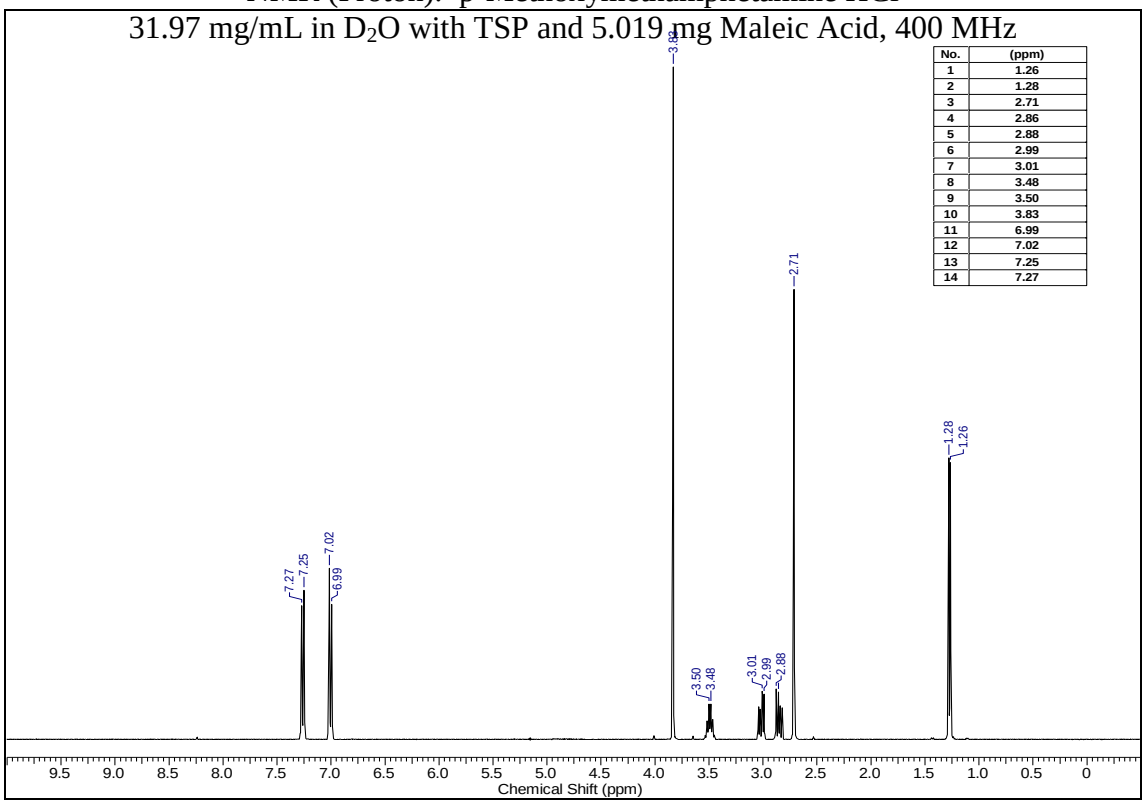
IR (Vapor Phase): p-Methoxymethamphetamine

Na_2CO_3 extracted CH_2Cl_2

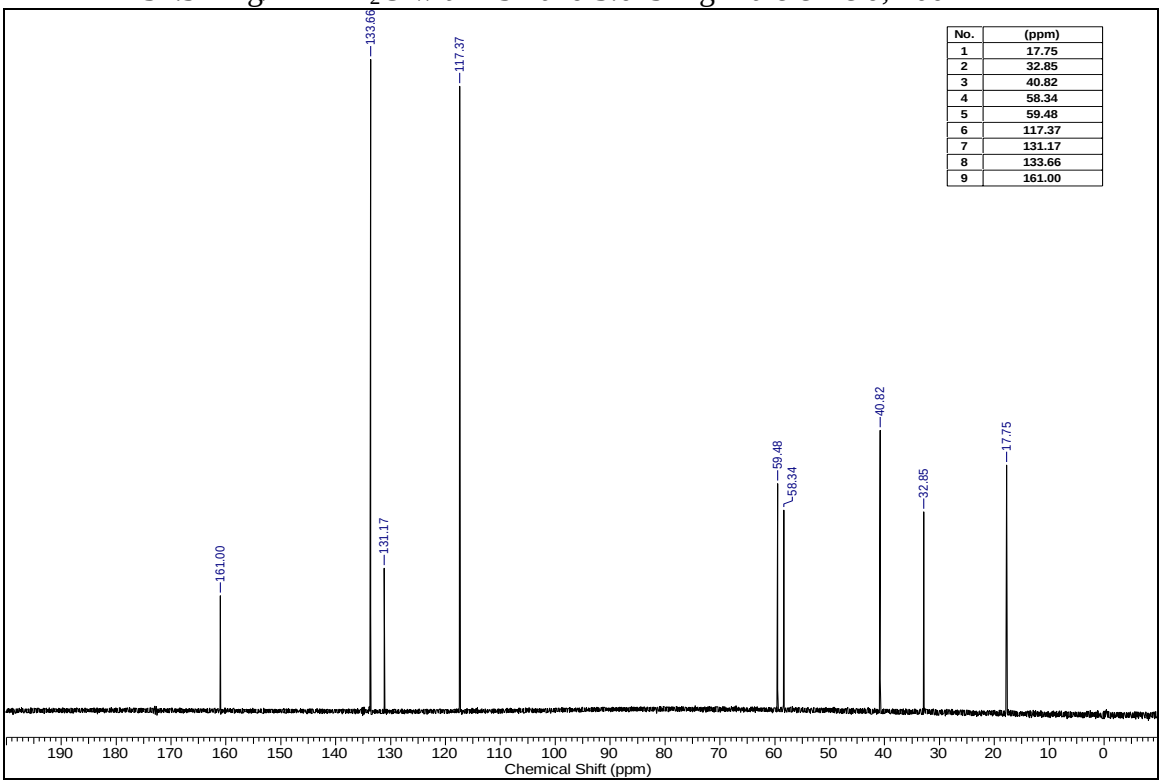


NMR (Proton): p-Methoxymethamphetamine HCl

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



NMR (Carbon): p-Methoxymethamphetamine HCl
31.97 mg/ml in D_2O with TSP and 5.019 mg Maleic Acid, 400 MHz



No.	(ppm)
1	17.75
2	32.85
3	40.82
4	58.34
5	59.48
6	117.37
7	131.17
8	133.66
9	161.00

UV: p-Methoxymethamphetamine HCl
in 0.1 N HCl