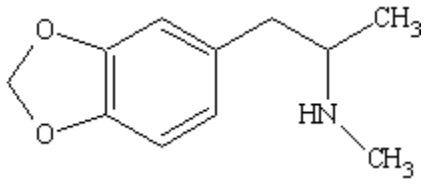
3,4-METHYLENEDIOXYMETHAMPHETAMINE

Latest Revision: August 18, 2005



1. SYNONYMS

CFR: 3,4-Methylenedioxymethamphetamine (MDMA)

CAS #:
Base: 42542-10-9
d,l-MDMA: 69610-10-2
Hydrochloride: 64057-70-1
d,l-MDMA Hydrochloride: 92279-84-0

Other Names:
N, α-dimethyl-1,3-benzodioxole-5-ethanamine
N-methyl-3,4-methylenedioxyphenylisopropylamine
Methylenedioxymethamphetamine
MDM,
MDMA
Ecstasy
E
XTC
Adam
Clarity
Elaine
Essence
Euphoria

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	$C_{11}H_{15}NO_2$	193.2	**Oil**
Hydrochloride	$C_{11}H_{15}NO_2 \cdot HCl$	229.7	147-153
Phosphate	**$C_{11}H_{15}NO_2 \cdot H_3PO_4$**	**291.24**	**184-185**

2.2. SOLUBILITY

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

3. SCREENING TECHNIQUES

3.1. COLOR TESTS

REAGENT	**COLOR PRODUCED**
Marquis	Purple to black

3.2. CRYSTAL TESTS

REAGENT	**CRYSTALS FORMED**
Gold chloride in HOAc	Rosettes of various sizes with a rusty color with high birefringence under polarized light radiating from the center outward down the arms. Feathery extensions from the arms are parallel to each other preserved on both sides of the arms.
Platinic chloride in HOAc	Burr-like rosettes of various sizes. Radii from the center resemble pine branches with fine needles. Needles have high birefringence under polarized light.

3.3. THIN LAYER CHROMATOGRAPHY

Visualization

Acidified potassium permanganate solution

COMPOUND	Relative R_f	
	System TLC 5	**System TLC 6**
3,4-methenedioxyethylamphetamine	**1.3**	**1.8**
3,4-methylenedioxyamphetamine	**1.2**	**1.5**
3,4-methylenedioxydimethylamphetamine	**1.1**	**1.8**
3,4-methylenedioxymethamphetamine	**1.0**	**1.0**

3.4. GAS CHROMATOGRAPHY

Method MDMA-GCS1

Instrument: Gas chromatograph operated in split mode with FID

Column: 5% phenyl/95% methyl silicone 30 m x 0.25 mm x 0.25 µm film thickness

Carrier gas: Hydrogen at 2.0 mL/min

Temperatures: Injector: 260°C
Detector: 270°C
Oven program:
1) 90°C initial temperature for 1.0 min
2) Ramp to 295°C at 30°C/min
3) Hold final temperature for 2.6 min

Injection Parameters: Split Ratio = 50:1, 1 µL injected

Samples are to be dissolved in 4:1 chloroform: methanol and filtered.

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.58	acetaminophen	1.18
methamphetamine	0.64	caffeine	1.27
safrole	0.76	methyl stearate	1.42
C_{14}	0.85	cocaine	1.53
3,4-MDA	0.95	methyl eicosanoate	1.54

3,4-MDMA	**1.00 (4.39 min)**	tetraphenylethylene	1.69
3,4-MDEA	1.04	heroin	1.80
3,4-MDDMA	1.06		

Method MDMA-GCS2

Instrument: Gas Chromatograph operated in split mode with FID

Column: 50% phenyl/50% methyl silicone 30 m x 0.25 mm x 0.25 µm film thickness

Carrier gas: Hydrogen at 2.0 mL/min

Temperatures: Injector: 260°C
Detector: 270°C
Oven program:
1) 90°C initial temperature for 1.0 min
2) Ramp to 295°C at 30°/min
3) Hold final temperature for 2.6 min

Injection Parameters: Split Ratio = 50:1, 1 µL injected

Samples are to be dissolved in 4:1 chloroform: methanol and filtered.

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.60	acetaminophen	1.27
methamphetamine	0.64	caffeine	1.34
safrole	0.78	methyl stearate	1.26
C_{14}	0.67	cocaine	1.53
3,4-MDA	0.97	methyl eicosanoate	1.37
3,4-MDMA	**1.00 (4.99 min)**	tetraphenylethylene	1.72
3,4-MDEA	1.02	heroin	1.97
3,4-MDDMA	1.04		

Method MDMA-GCS3

Instrument: Gas chromatograph operated in split mode with FID

Column: 5% phenyl/95% methyl silicone 5 m x 0.25 mm x 0.25 µm film thickness

Carrier gas: Hydrogen:
1) Initial pressure of 2.5 psi for 0.10 min
2) Ramp to 5.0 psi at 3.41 psi/min
3) Hold final pressure for 0.77 min

Temperature: Injector: 260°C
Detector: 270°C
Oven program:
1) 90°C initial temperature
2) Ramp to 100°C at 10°/min
3) Hold final temperature for 0.60 min

Injection Parameters: Split Ratio = 50:1, 1 µL injected
EZFlash Parameters:
1) Time 0 s = 100°C
2) Time 6 s = 110°C
3) Time 50 s = 310°C
4) Time 96 s = 310°C

Samples are to be dissolved in 4:1 chloroform: methanol and filtered.

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.70	acetaminophen	1.14
methamphetamine	0.74	caffeine	1.22
safrole	0.82	methyl stearate	1.23
C_{14}	0.88	cocaine	1.44
3,4-MDA	0.96	methyl eicosanoate	1.34
3,4-MDMA	**1.00 (4.99 min)**	tetraphenylethylene	1.62
3,4-MDEA	1.03	heroin	1.80
3,4-MDDMA	1.04		

Method MDMA-GCS4

Instrument: Gas chromatograph operated in split mode with FID

Column: 1% phenyl/99% methyl silicone 30 m x 0.25 mm x 0.25 µm film thickness

Carrier gas: Hydrogen at 2.0 mL/min

Temperature: Injector: 260°C
Detector: 270°C
Oven program:
1) 90°C initial temperature for 1.0 min
2) Ramp to 295°C at 30 degrees/min
3) Hold final temperature for 2.6 min

Injection Parameters: Split Ratio = 50:1, 1 µL injected

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.53	acetaminophen	1.19
methamphetamine	1.65	caffeine	1.28
safrole	0.72	methyl stearate	1.53
C_{14}	0.91	cocaine	1.60
3,4-MDA	0.94	methyl eicosanoate	1.67
3,4-MDMA	**1.00 (4.36 min)**	tetraphenylethylene	1.78
3,4-MDEA	1.05	heroin	1.89
3,4-MDDMA	1.08		

4. SEPARATION TECHNIQUES

MDMA **base, hydrochloride, or phosphate can be separated from the matrix** by solvent extraction using the solubility data found in Section 2.2.

5. QUANTITATIVE PROCEDURES

5.1. GAS CHROMATOGRAPHY

Method MDMA-GCQ1

Internal Standard Stock Solution:
1.0 mg/mL ***n*-butylamphetamine HCl in chloroform**.

Standard Solution Preparation:
Prepare a standard solution of **MDMA HCl at 0.4 mg/mL in chloroform**.

Sample Preparation:
Accurately weigh out 50 mg of sample into a 25 mL volumetric flask and dilute with water to the mark. Take a 2 mL aliquot of the solution, add 3 mL of I.S. and 2-3 mL of 0.5 M KOH. Shake. Separate layers and extract twice more with 2-3 mL each time of chloroform. Combine fractions, dry over sodium sulfate and dilute to total volume of 10 mL. Treat the standard in the same fashion as the sample prior to analysis.

Instrument:	Gas chromatograph operated in split mode with FID
Column:	**DB-1, 30 m x 0.25 mm x 0.25 µm film thickness**
Carrier gas:	**Hydrogen at 1.3 mL/min**
Make-Up gas:	Nitrogen at 40.0 mL/min
Temperatures:	**Injector: 230°C** **Detector: 280°C** **Oven Program:** **1) 150°C initial temperature 2 min** **2) Ramp to 190°C at 5°/min** **3) Hold final temperature for 1.0 min**
Injection Parameters:	Split Ratio = **20**:1, 1 µL injected
Typical Retention Time:	3,4-MDMA: **5.10** min ***n*-butylamphetamine HCl**: **3.92** min
Linear Range:	**0.06 mg/mL to 2.0 mg/mL**
Repeatability:	RSD less than 2.0%
Correlation Coefficient:	**0.9998**
Accuracy	Error less than 5%

COMPOUND	RRT	COMPOUND	RRT	COMPOUND	RRT
methylbenzylamine	**0.35**	**methyl paraben**	**0.77**	**caffeine**	**1.86**
P-2-P	**0.38**	**salicylamide**	**0.77**	**antipyrine**	**2.00**
amphetamine	**0.39**	**penicillin**	**0.78**	**benzphetamine**	**2.04**

nicotinic acid	**0.42**	**phenmetrazine**	**0.81**	**diphenhydramine**	**2.14**
phentermine	**0.42**	**MDP-2-P**	**0.84**	**aminopyrine**	**2.26**
methamphetamine	**0.44**	**phendimetrazine**	**0.86**	**doxylamine**	**2.31**
fenfluramine	**0.48**	**MDA**	**0.87**	**palmitic acid**	**2.45**
ethylamphetamine	**0.49**	**MDMA**	**1.00 (5.1 min)**	**phthalic acid**	**2.45**
dimethylamphetamine	**0.50**	**aminorex**	**1.05**	**dipyrone**	**2.51**
safrole	**0.55**	**methyl aminorex**	**1.05**	**procaine**	**2.66**
salicylic acid	**0.56**	**MDEA**	**1.13**	**eicosane**	**2.67**
cathine	**0.59**	**ibuprofen**	**1.24**	**dextromethorphan**	**3.12**
phenylpropanolamine	**0.59**	**MBDB**	**1.26**	**strychnine**	**3.17**
methcathinone	**0.60**	**hexadecane**	**1.28**	**phenyl-2-naphthalene**	**3.27**
nicotinamide	**0.62**	**glycerol glycolate**	**1.30**	**amitriptyline**	**3.34**
chlorpheniramine	**0.65**	**acetaminophen**	**1.35**	**scopolamine**	**3.71**
chloroephedrine	**0.66**	**MMDA**	**1.38**	**tetracosane**	**4.07**
pseudoephedrine	**0.66**	**phenacetin**	**1.41**	**chlordiazepoxide**	**4.31**
ephedrine	**0.68**	**chloro-MDMA**	**1.49**	**quinine**	**5.23**
butylamphetamine	**0.77**	**methylphenidate**	**1.63**		

5.2. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method MDMA-LCQ1

Standard Solution Preparation:
Prepare a standard solution of MDMA at approximately 0.5 mg/mL using water, buffer, or methanol.

Sample Preparation:
Accurately weigh an amount of sample into an appropriate volumetric or Erlenmeyer flask and dilute so that the final MDMA concentration is approximately that of the standard solution.

Instrument: High performance liquid chromatograph equipped with diode array

Column: **5 µm Phenomenex Luna, 150 mm x 3.2 mm at 35°C**

Detector: UV, 210 nm

Flow: 1.0 mL/min

Injection Volume: **5 µL**

Buffer: 4000 mL distilled water, **22.5 mL phosphoric acid, adjust to pH 2.2-2.3 with triethanolamine (approx. 22 mL)**

Mobile Phase: **Buffer:acetonitrile 90:10**

Typical Retention Time: 3,4-MDMA: **10.54** min

Linear Range: **0.05 mg/mL – 2 mg/mL**

Repeatability: RSD less than 0.**5**%

Correlation Coefficient: 0.999**9**

Accuracy: Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
phenylpropanolamine	**0.60**	**methyl aminorex**	**1.01**
methyl benzylamine	**0.62**	**phentermine**	**1.02**
cathine	**0.63**	**chloroephedrine**	**1.03**
doxylamine	**0.69**	**ethylamphetamine**	**1.05**
dipyrone	**0.70**	**MDP-2-P**	**1.05**
ephedrine	**0.72**	**lidocaine**	**1.07**
methcathinone	**0.73**	**caffeine**	**q**
pseudoephedrine	**0.73**	**MDEA**	**1.13**
amphetamine	**0.84**	**phenyl-2-naphthalene**	**1.13**
theophylline	**0.87**	**phthalic acid**	**1.13**
methapyrilene	**0.89**	**strychnine**	**1.14**
phenmetrazine	**0.89**	**MMDA**	**1.17**
methamphetamine	**0.91**	**MBDB**	**1.25**
phendimetrazine	**0.91**	**salicylamide**	**1.27**

COMPOUND	RRT	COMPOUND	RRT
scopolamine	**0.92**	**acetaminophen**	**1.29**
MDA	**0.93**	**chloro-MDMA**	**1.37**
quinine	**0.96**	**methylphenidate**	**1.38**
saccharin	**0.96**	**glycerol glycolate**	**1.48**
dimethylamphetamine	**0.97**	**chlorpheniramine**	**1.55**
MDMA	**1.00 (10.54 min)**	**aspirin**	**1.81**
		chlordiazepoxide	**1.87**

5.3. CAPILLARY ELECTROPHORESIS

Method MDMA-CEQ1

Internal Standard Stock Solution:
0.50 mg/mL *n*-butylamphetamine in 50 mM sodium phosphate at pH 7.0.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of 3,4-methylenedioxymethamphetamine hydrochloride at approximately 0.8 mg/mL using the internal standard stock solution.

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

Mode:	Cyclodextrin system analysis
Column:	49 cm x 52 µm fused silica capillary
Run Buffer:	10 mM gamma-highly sulfated cyclodextrin in 50 mM sodium phosphate at pH 7.0
Detector:	UV, 210 nm
Voltage:	12 kV
Temperature:	30°C air cooled
Injection:	1 sec hydrodynamic at 50 mbar/s

Run Time: 17 min

Rinse Time: 2.0 min

Linear Range: 0.02 - 1.00 mg/mL

Repeatability: RSD of area less than 1.5%

Correlation Coefficient: 0.999

Accuracy: Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
d-methamphetamine	0.66	l-pseudoephedrine	0.98
d-ephedrine	0.74	**MDMA***	**1.00 (11.04 min)**
MDA*	0.80	l-ephedrine	1.09
cathine	0.85	MDEA*	1.12
l-methamphetamine	0.89	**MDMA***	**1.19**
d-amphetamine	0.94	l-norephedrine	1.32
d-norephedrine	0.94	l-amphetamine	1.34
d-pseudoephedrine	0.97	MDA*	1.36
		MDEA*	1.49

Method MDMA-CEQ2

Internal Standard Stock Solution:
1.0 mg/mL *n*-butylamphetamine in 3.8 mM phosphate buffer (pH 2.5)

Standard Solution Preparation:
Weigh an appropriate amount of standard 3,4-MDMA HCl into a volumetric flask to obtain a final concentration of approximately 0.08 mg/mL. Pipette appropriate amount of internal standard solution to obtain a final concentration of 0.1 mg/mL. Dilute to volume with injection solvent. Filter approximately 1.0 mL of solution with regenerated cellulose 0.45 µm 25 mm filter into a 2.0 mL glass vial (Agilent part number 5182-0567.) Make sure there are no air bubbles on bottom of glass vial. Cap vial with polypropylene cap (Agilent part number 5182-9697.)

Sample Preparation:

Weigh an appropriate amount of sample into a volumetric flask so that the final phenethylamine concentration is approximately that of the standard solution. Pipette appropriate amount of internal standard solution to obtain a final concentration of 0.1 mg/mL. Dilute to volume with injection solvent. Sonicate for 15 minutes. Filter approximately 1.0 mL of solution with regenerated cellulose 0.45 µm 25 mm filter into a 2.0 mL glass vial (Agilent part number 5182-0567.) Make sure there are no air bubbles on bottom of glass vial. Cap vial with polypropylene cap (Agilent part number 5182-9697.)

Mode:	HP 3D instrument operated in CE mode Coated Capillary Mode
Column:	50 µm i.d. x 32.2 cm (23.7 cm length to detector)
Run Buffer:	Celixir Reagent B, pH 2.5 (MicroSolv CE)
Detector:	UV, 195 nm
Voltage:	10 kV
Temperature:	15°C
Injection:	50 mbar x 2 s followed by water at 35 mbar
Run Time:	6 min
Rinse Time:	2 min
Linear Range:	0.02 - 1.00 mg/mL
Repeatability:	RSD <1.6%
Correlation Coefficient:	1.0
Accuracy:	Error less than 3.2%

COMPOUND	RRT	COMPOUND	RRT
doxylamine	0.765	ephedrine	0.932
chlorpheniramine	0.784	phenylephrine	0.951
quinine	0.804	MDEA	0.961
β-phenethylamine	0.807	ketamine	0.962

COMPOUND	RRT	COMPOUND	RRT
chlorquinine	0.812	phenyltoxolamine	0.971
nicotinamide	0.836	***n*-butylamphetamine**	**1.0(4.6 min)**
amphetamine	0.868	dextromethorphan	1.00
methamphetamine	0.883	lidocaine	1.03
procaine	0.883	benzocaine	1.25
MDA	0.900	acetaminophen	2.11
norpseudoephedrine	0.906	caffeine	2.14
MDMA	0.914	guaifenesin	2.14
norephedrine	0.917	P2P	2.24
pseudoephedrine	0.919	DMSO (neutral marker)	2.40
tetracaine	0.927	aspirin	2.71

5.4. NUCLEAR MAGNETIC RESONANCE

Method MDMA-NMR1

Solvents: Deuterium Oxide (D_2O) containing DSS or TSP for 0 PPM reference.

Internal Standard Stock Solution (ISSS): 5 mg/mL Maleic Acid (accurately weighed) in deuterium oxide (D_2O) containing DSS or TSP for 0 ppm reference.

Sample Preparation: Accurately weigh an amount of sample, usually 10-30 mg, into a capped test tube and accurately add a volume, normally 1.0 mL, of the ISSS. Vortex the sample for several seconds. If insolubles are present, add 1.0 mL D_2O (not containing maleic acid or the reference compound), vortex and sonicate 15 minutes. Vortex the sample and filter if necessary. Place in NMR sample tube.

Instrument: Varian Mercury 400 MHz with proton detection probe NMR Spectrometer

Pulse Width: 10 µs or 90°(whichever is less)

Spectral Width (SW) at least containing –3 ppm through 13 ppm

Number of Scans: multiple of 4 (greater to enhance signal to noise (S/N))
Delay between Pulses: 45 s

Shimming: automatic gradient shimming of Z1-4 shims

Total run time/sample: 6 min (NT=4) -14 min (NT=16)

Uniformity within spectral width: 0.3% RSD (-0.6 to 11.4 PPM)

Run Time: 6 min

Linear Range: 0.6 mg/mL-60 mg/mL

Repeatability: < 4%

Correlation Coefficient: 1.0

Accuracy: < 3%

Compound	**MW**	**Solvent**	**Internal Standard**	**Solubility (mg/mL)**	**Signals used for quantitation (position in ppm with number of protons). Signals in bold and underlined are preferred if other numbers present.**	**Decomposition rate (%/hour)**
MDMA HCl	229.71	D_2O	maleic acid	>30	6.7-6.9 (m)(3), 6.0 (s)(2), 3.5 (sextet)(1), 3.0dd(1), 2.8dd(1), 2.7 (s)(3), 1.3 (d)(3)	<0.1
MDMA phosphate	291.24	D_2O	maleic acid	>19	6.7-6.9 (m)(3), 6.0 (s)(2), 3.5 (sextet)(1), 3.0dd(1), 2.8dd(1), 2.7 (s)(3), 1.3 (d)(3)	<0.1

6. QUALITATIVE DATA

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

7. REFERENCES

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

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

Galichat, Laurent Y., Clarke's Analysis of Drugs and Poisons, Volume 2, p. 1256, Pharmaceutical Press, 2004.

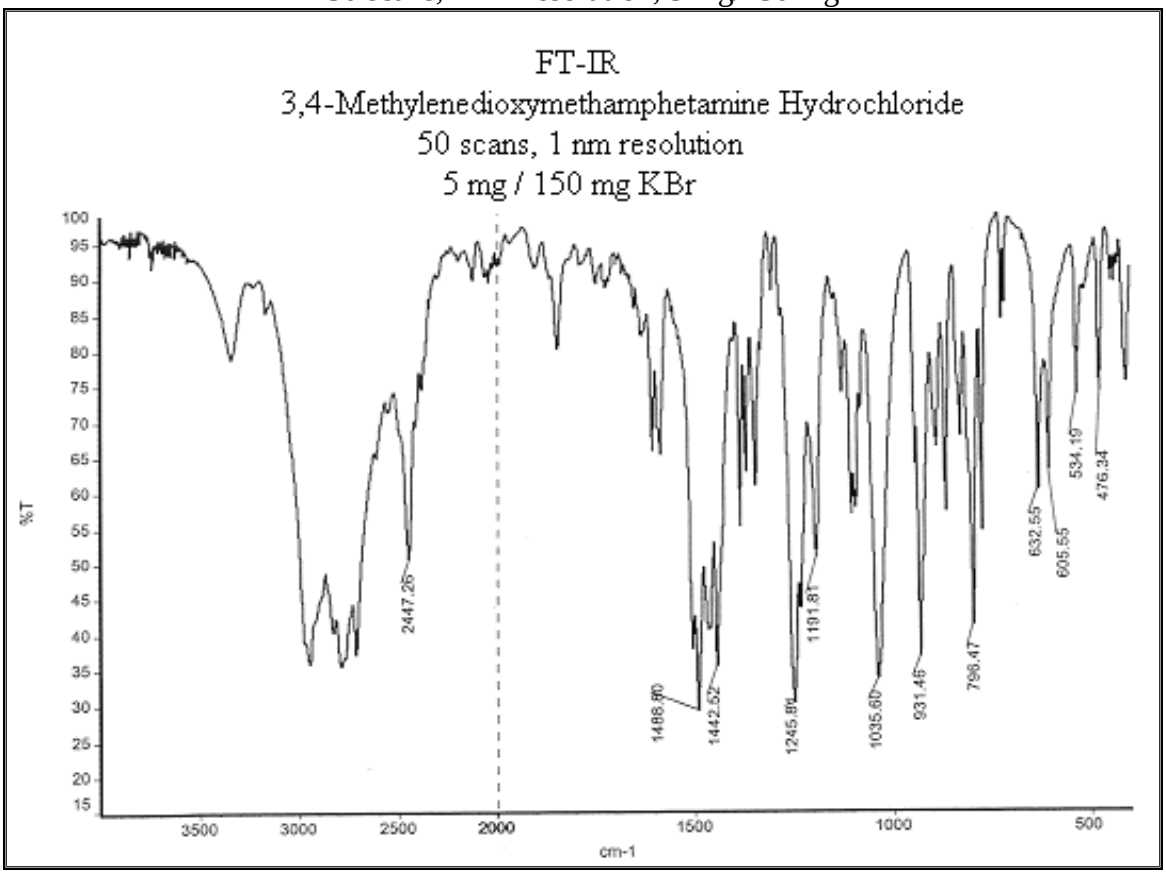
Budavari, S., The Merck Index, 13th Edition, Merck and Co., Inc., 2001.

8. ADDITIONAL RESOURCES

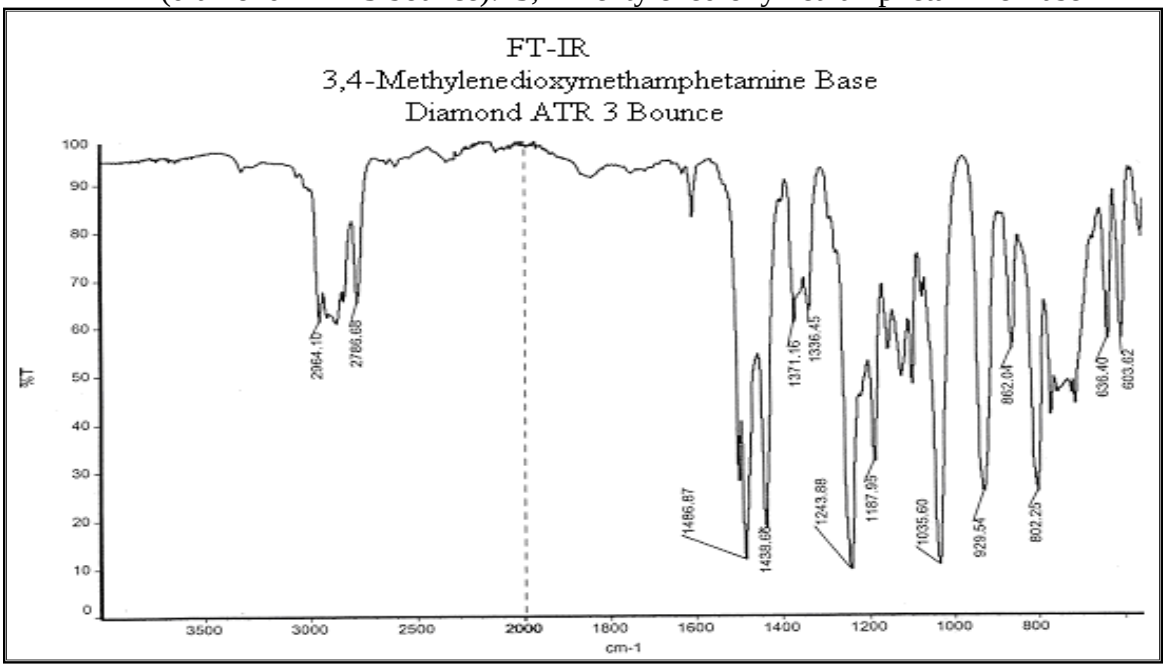
Forendex

Wikipedia

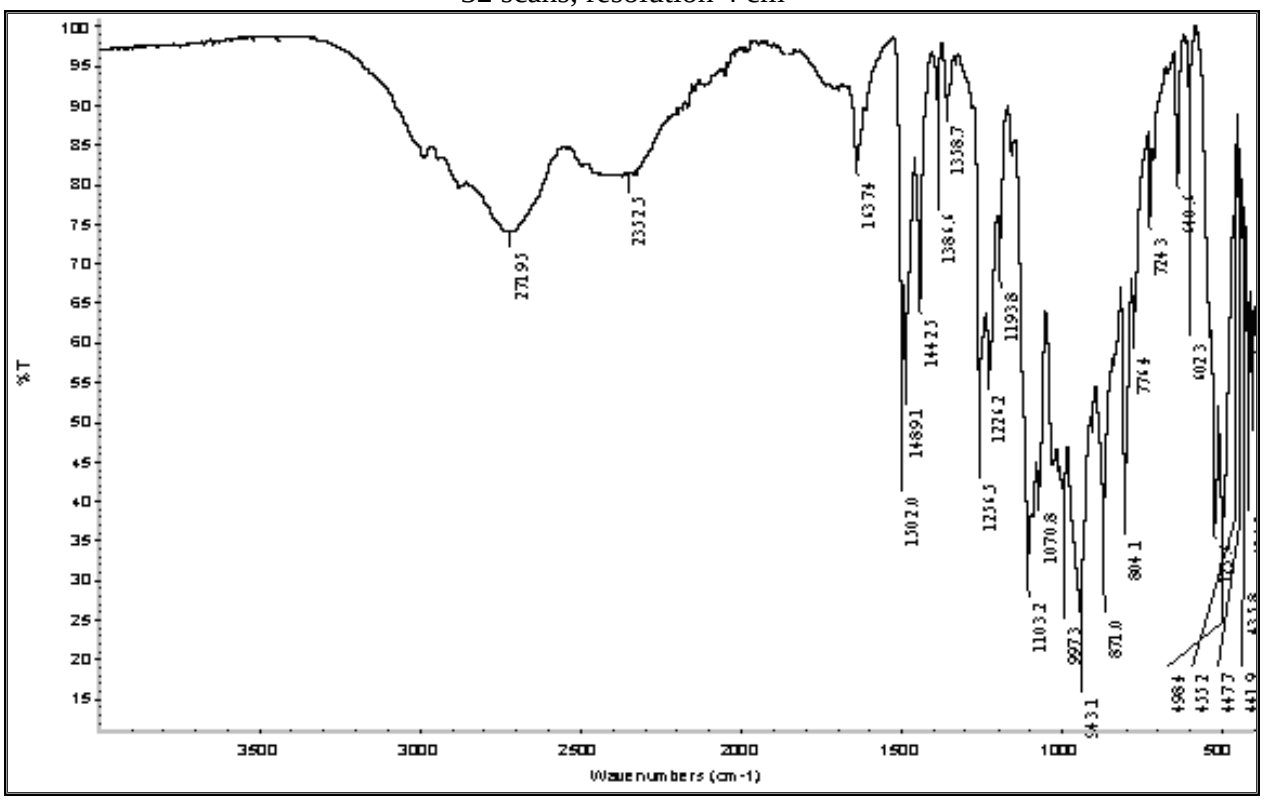
FTIR (KBr): 3,4-Methylenedioxymethamphetamine Hydrochloride
50 scans, 1 nm resolution, 5 mg/150 mg



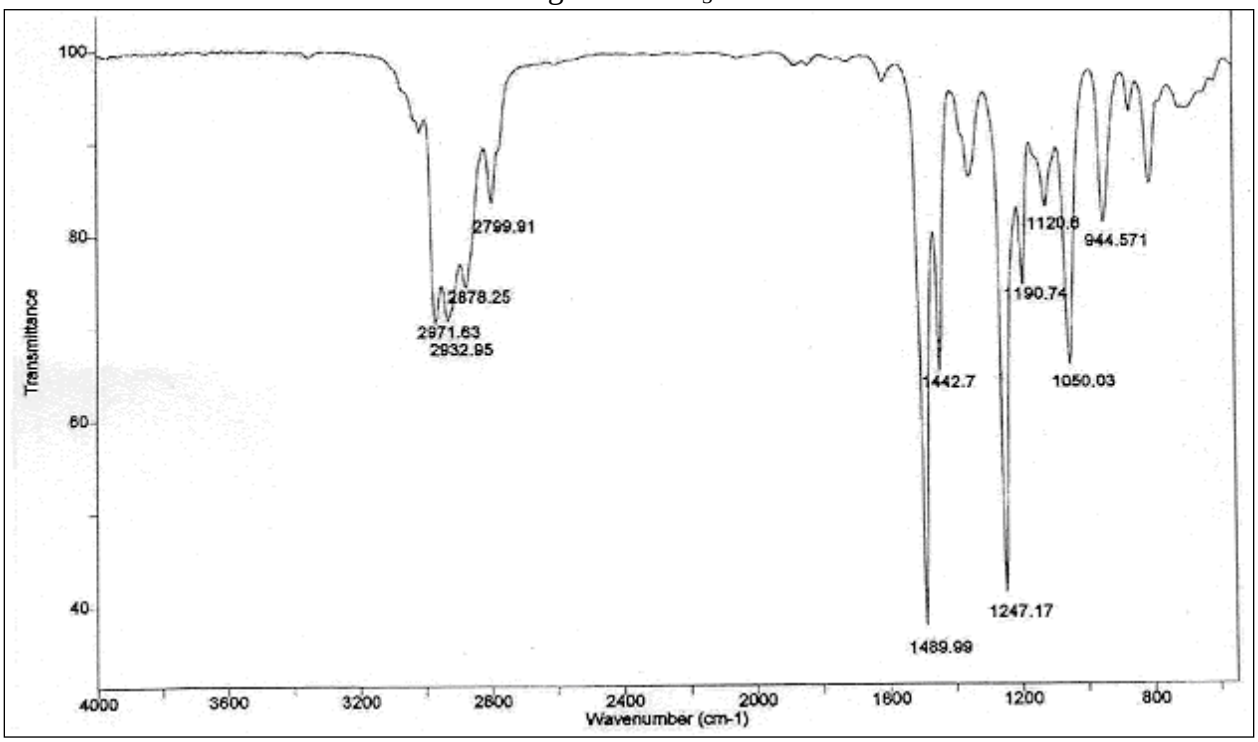
FTIR (diamond ATR 3 bounce): 3,4-Mehtylenedioxymethamphetamine Base



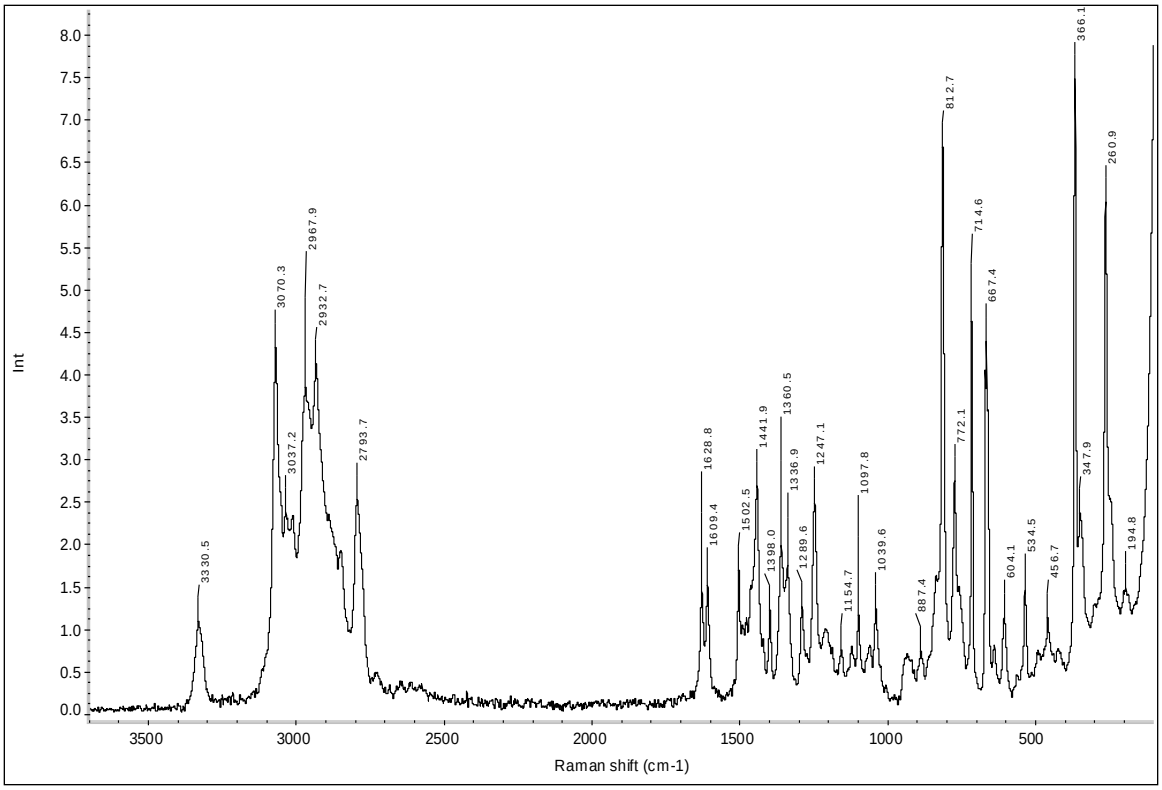
FTIR (1 bounce ATR): MDMA phosphate
32 scans, resolution 4 cm^{-1}



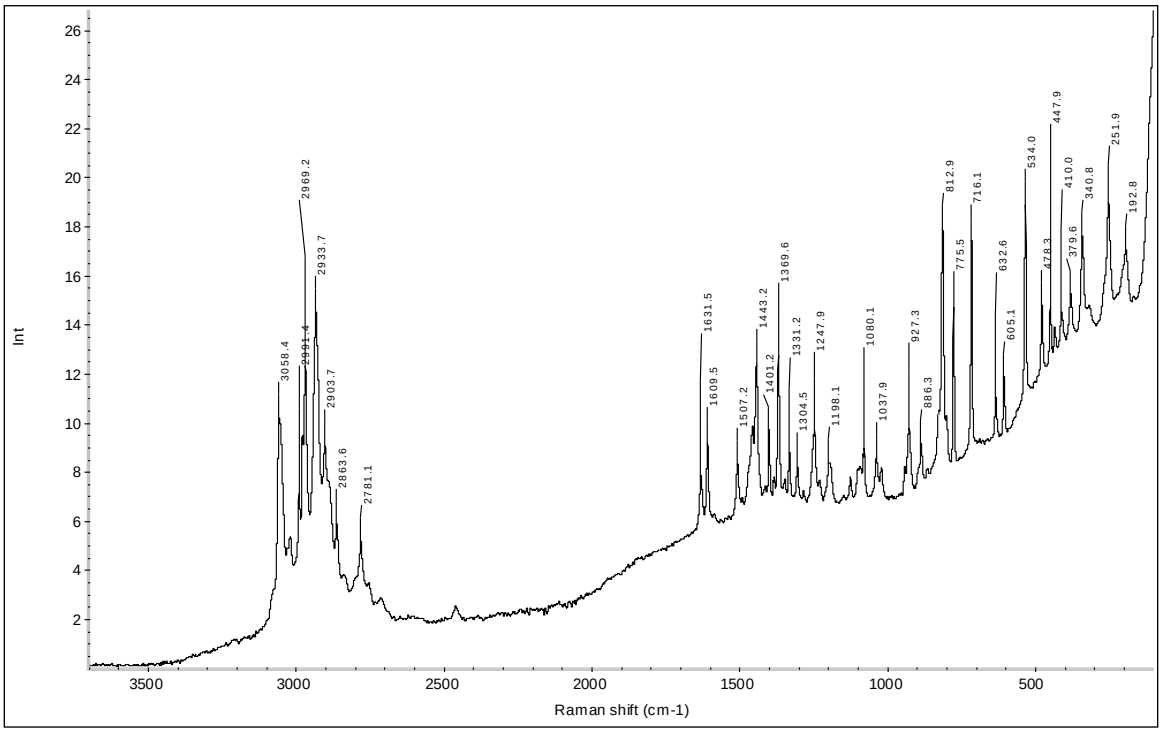
IR (Vapor Phase): 3,4-Methylenedioxymethamphetamine Lot # A150B
2 mg/mL in CH_3OH



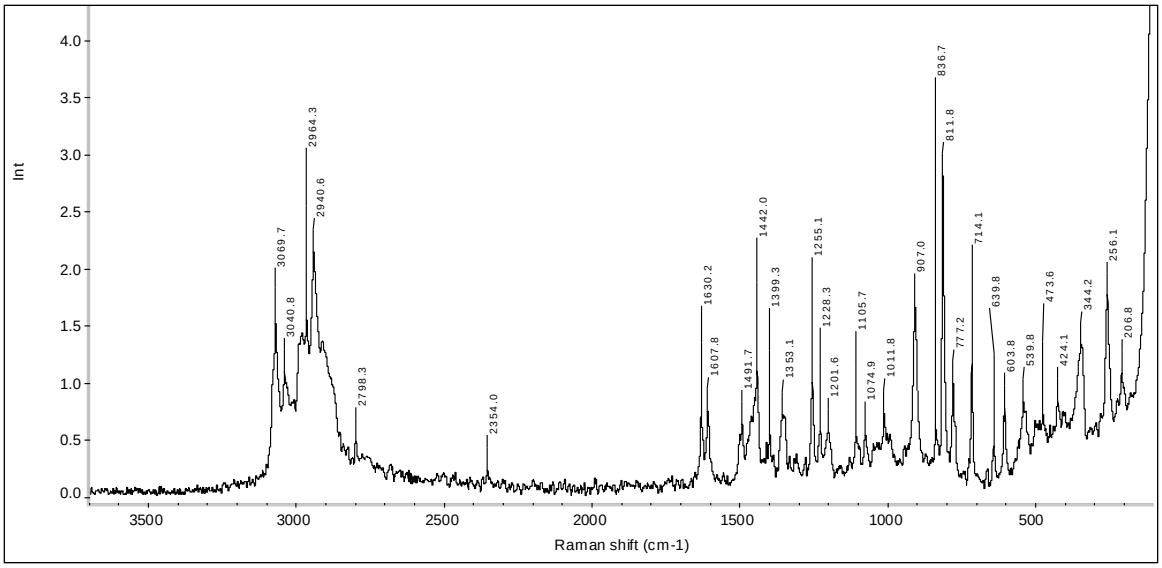
FT-RAMAN: 3,4-Methylenedioxymethamphetamine Base
Extracted from MDMA HCl, Lot #A150B, 32 scans, 4 cm^{-1} resolution



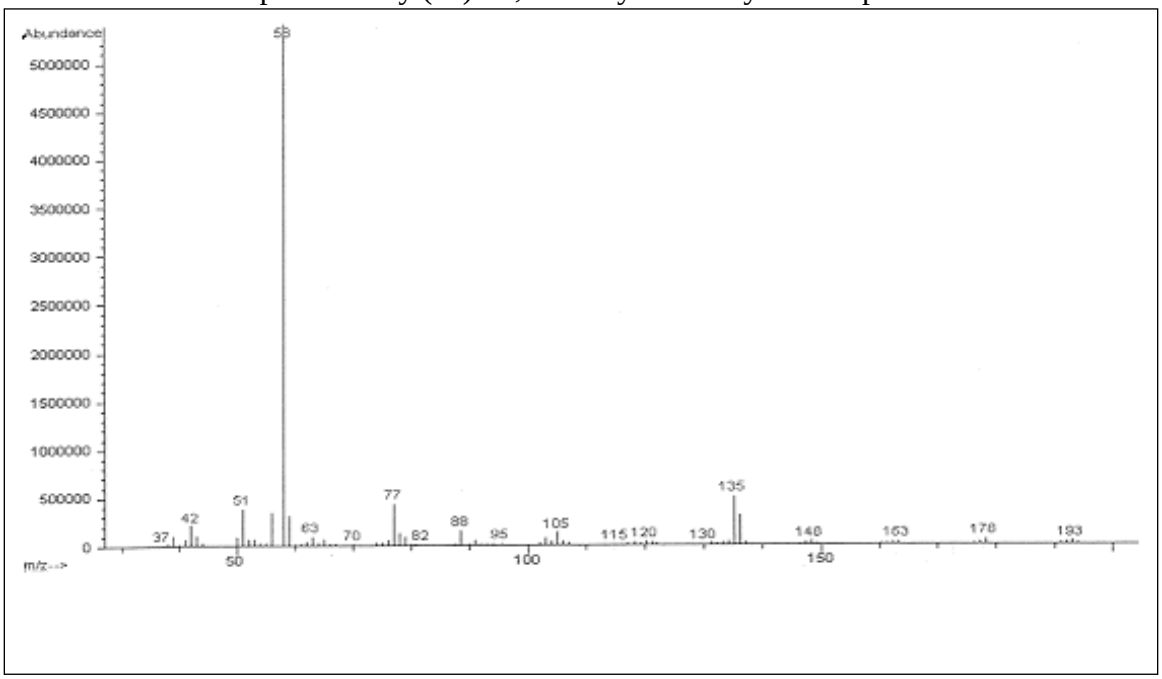
FT-RAMAN: 3,4-Methylenedioxymethamphetamine HCl
Lot # A150B, 32 scans, 4 cm^{-1} resolution



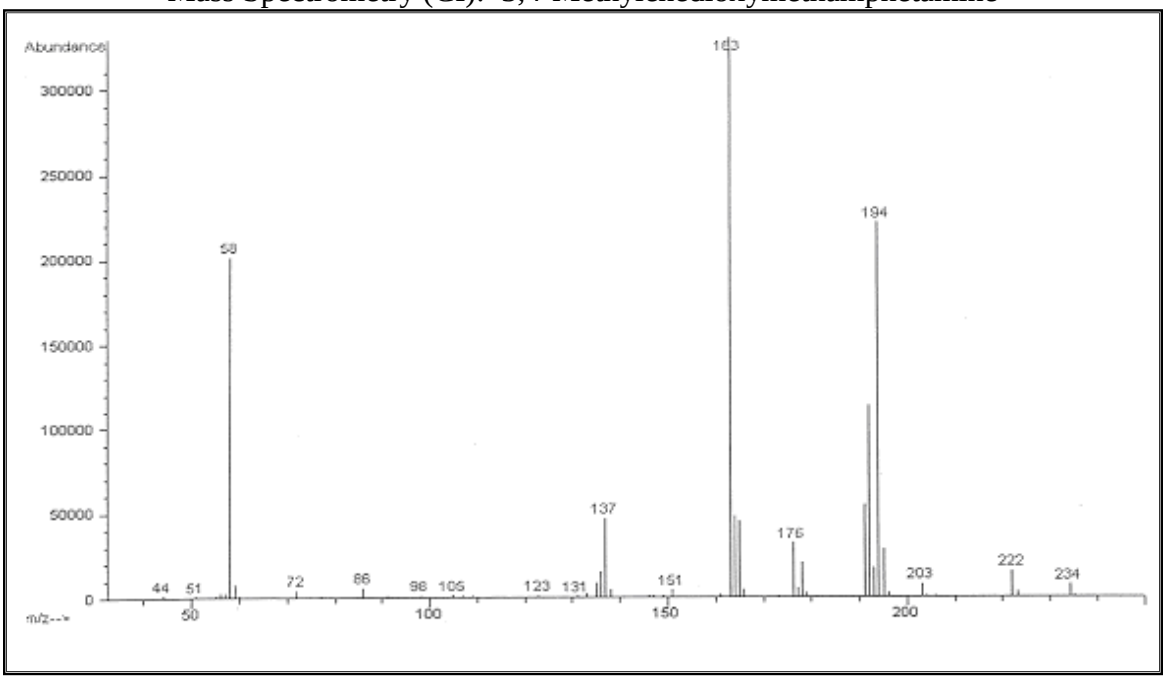
FT-RAMAN: 3,4-Methylenedioxymethamphetamine Phosphate Lot # 2 TDM-275-01
32 scans, 4 cm^{-1} resolution



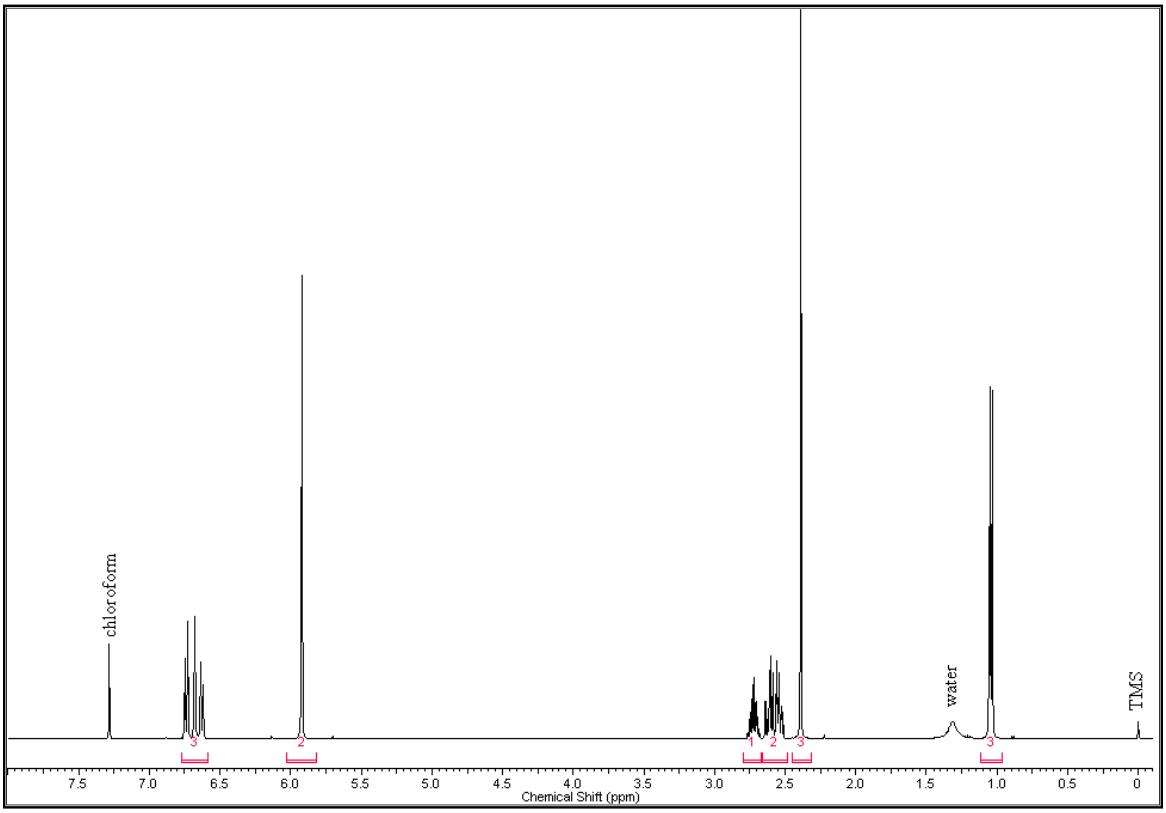
Mass Spectrometry (EI): 3,4-Methylenedioxymethamphetamine



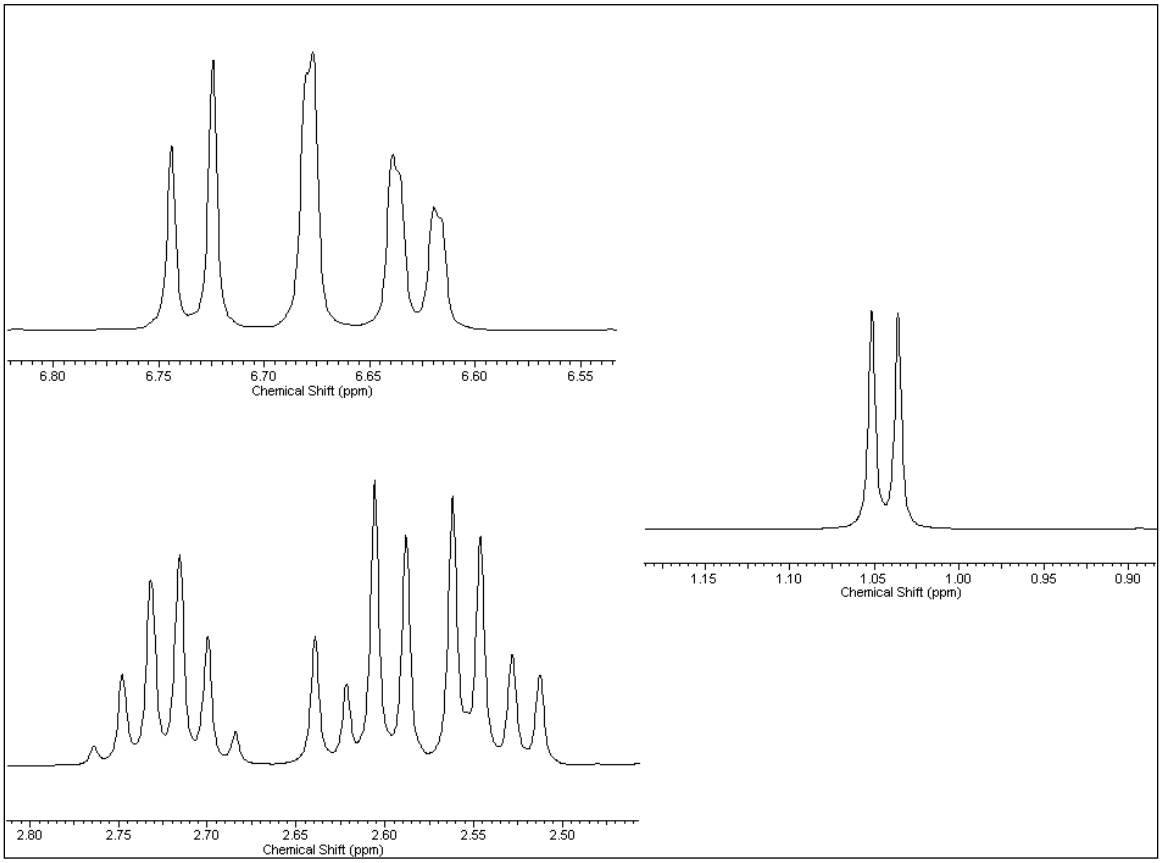
Mass Spectrometry (CI): 3,4-Methylenedioxymethamphetamine



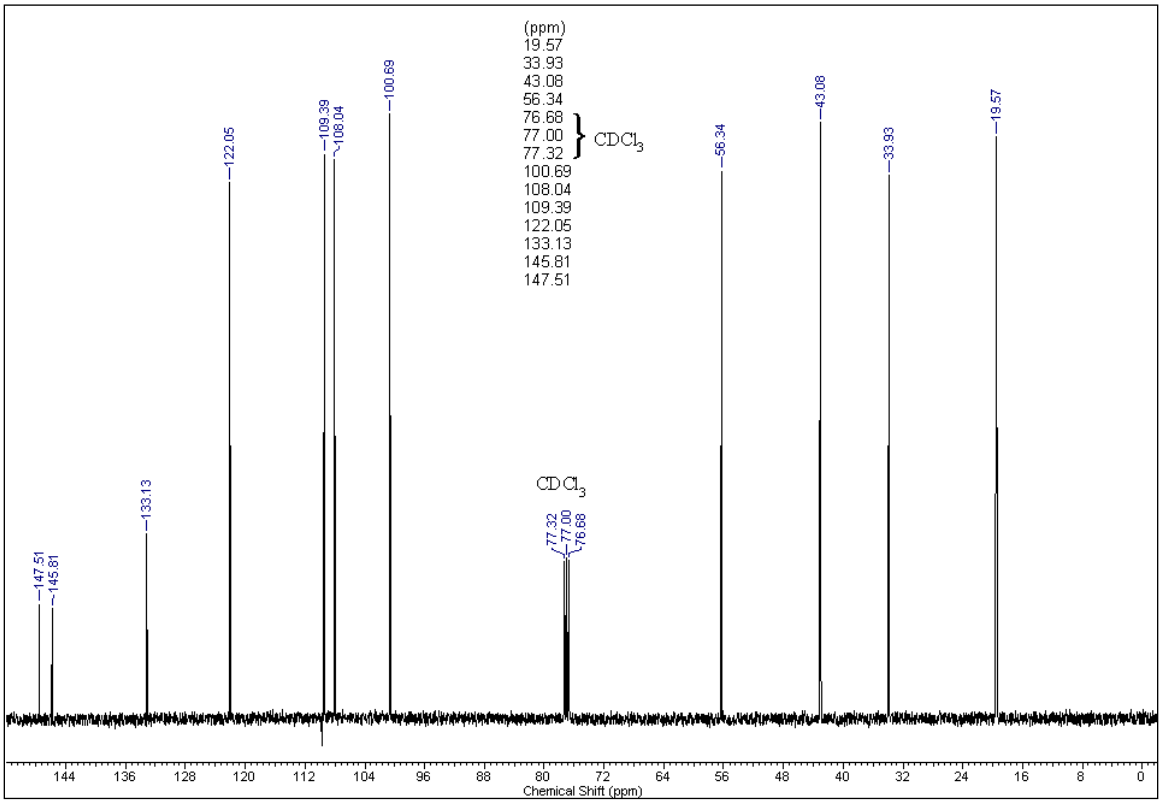
^{1}H NMR: MDMA base in CDCl3 with TMS 400 MHz
Extracted from MDMA HCl – Lot # A150B



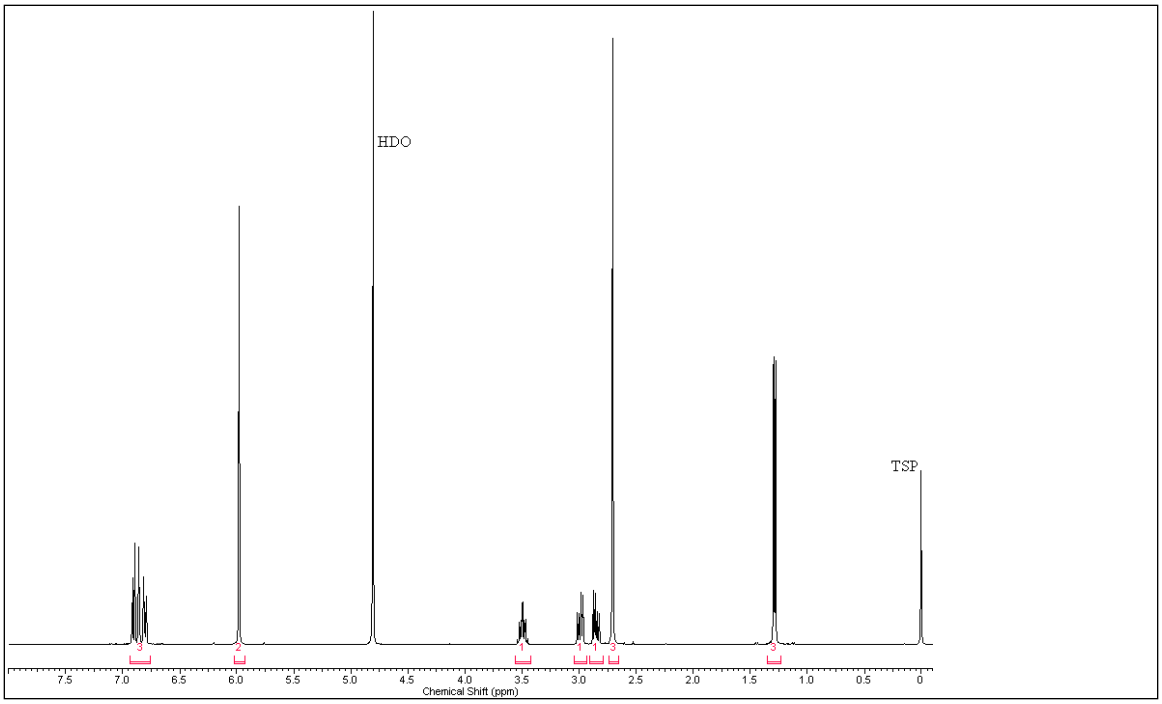
ppm 6.73 (d, J=7.83 Hz, 1 H) 6.68 (d, J=1.17 Hz, 1 H) 6.63 (dd, J=7.83, 1.17 Hz, 1 H) 5.92 (s, 2 H) 2.67 - 2.78 (ddq, J=7.20, 6.30, 6.30, 6.30, 6.30 Hz, 1 H) 2.61 (dd, J=13.50, 7.24 Hz, 1 H) 2.54 (dd, J=13.30, 6.26 Hz, 1 H) 2.39 (s, 3 H) 1.04 (d, J=6.26 Hz, 3 H)



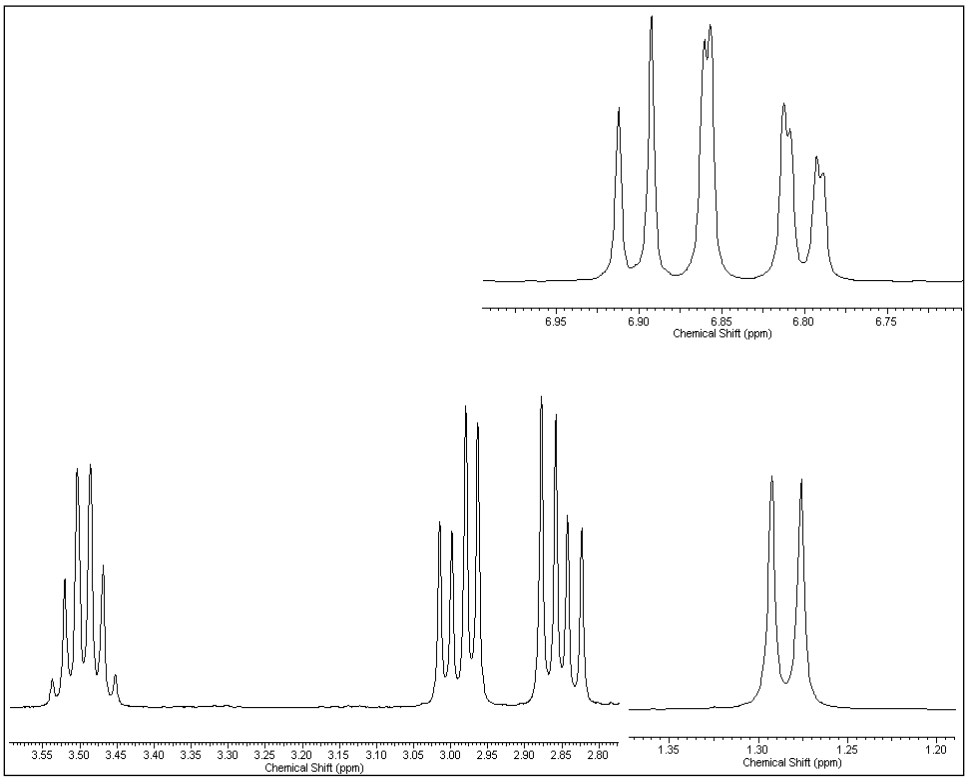
^{13}C NMR: MDMA Base
Extracted from MDMA HCl – Lot #A 150B



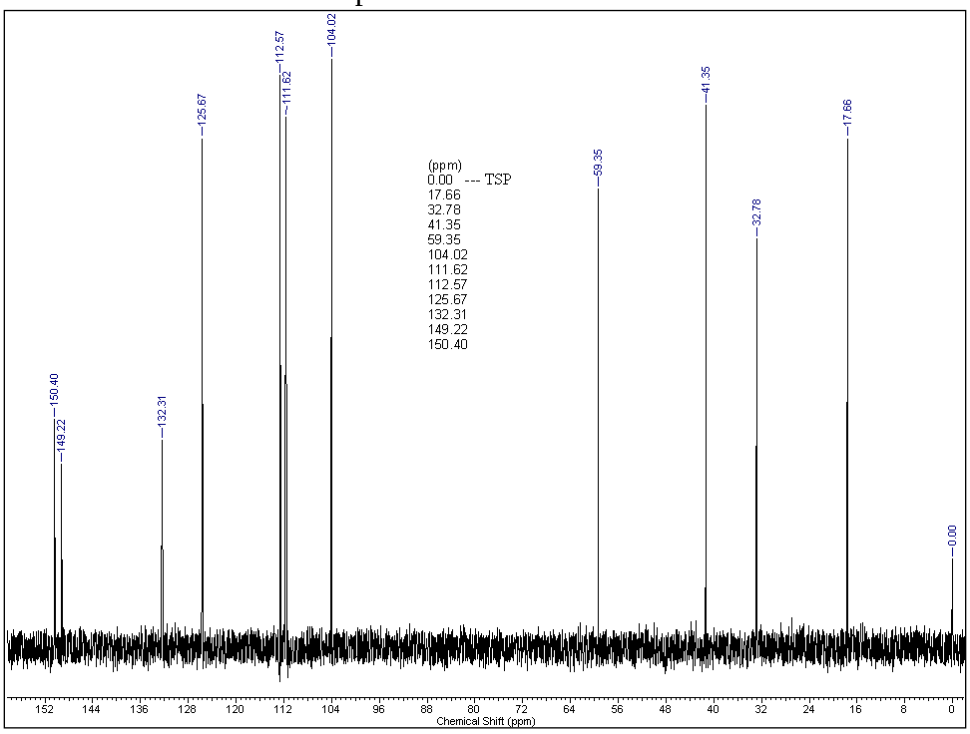
^{1}H NMR: MDMA Phosphate in D2O with TSP 400 MHz
Lot # 2 TDM-275-01



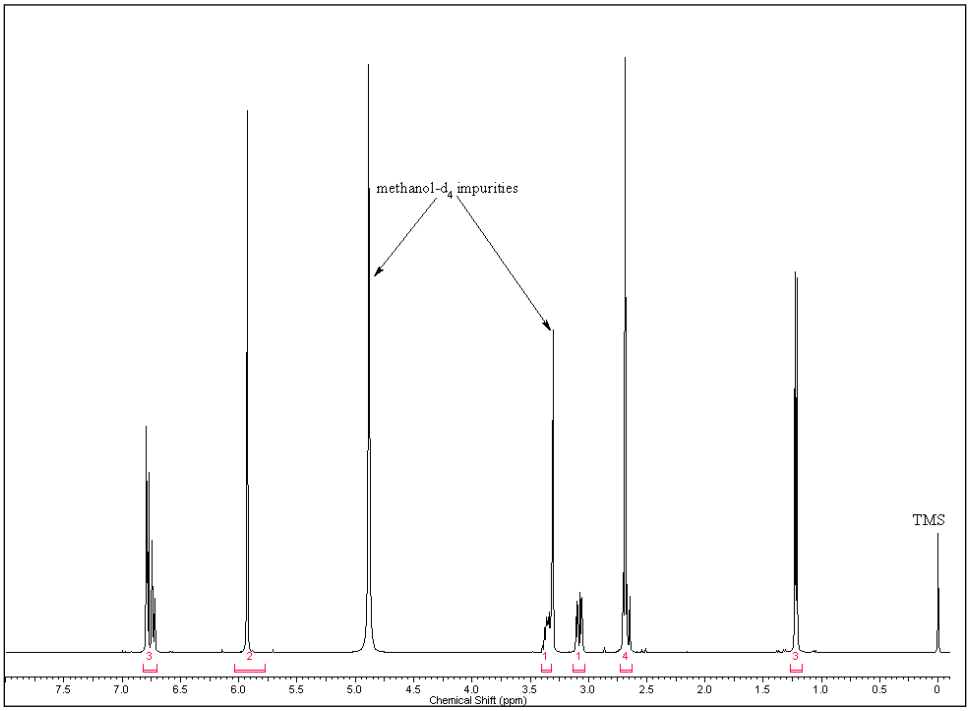
ppm 6.88 - 6.92 (d, J=8.00 Hz, 1 H) 6.86 (d, J=1.37 Hz, 1 H) 6.80 (dd, J=8.00, 1.40 Hz, 1 H) 5.98 (s, 2 H) 3.44 - 3.55 (ddq, J=7.50, 6.70, 6.70, 6.70, 6.50 Hz, 1 H) 2.99 (dd, J=13.90, 6.50 Hz, 1 H) 2.85 (dd, J=13.90, 7.60 Hz, 1 H) 2.70 (s, 3 H) 1.28 (d, J=6.65 Hz, 3 H)



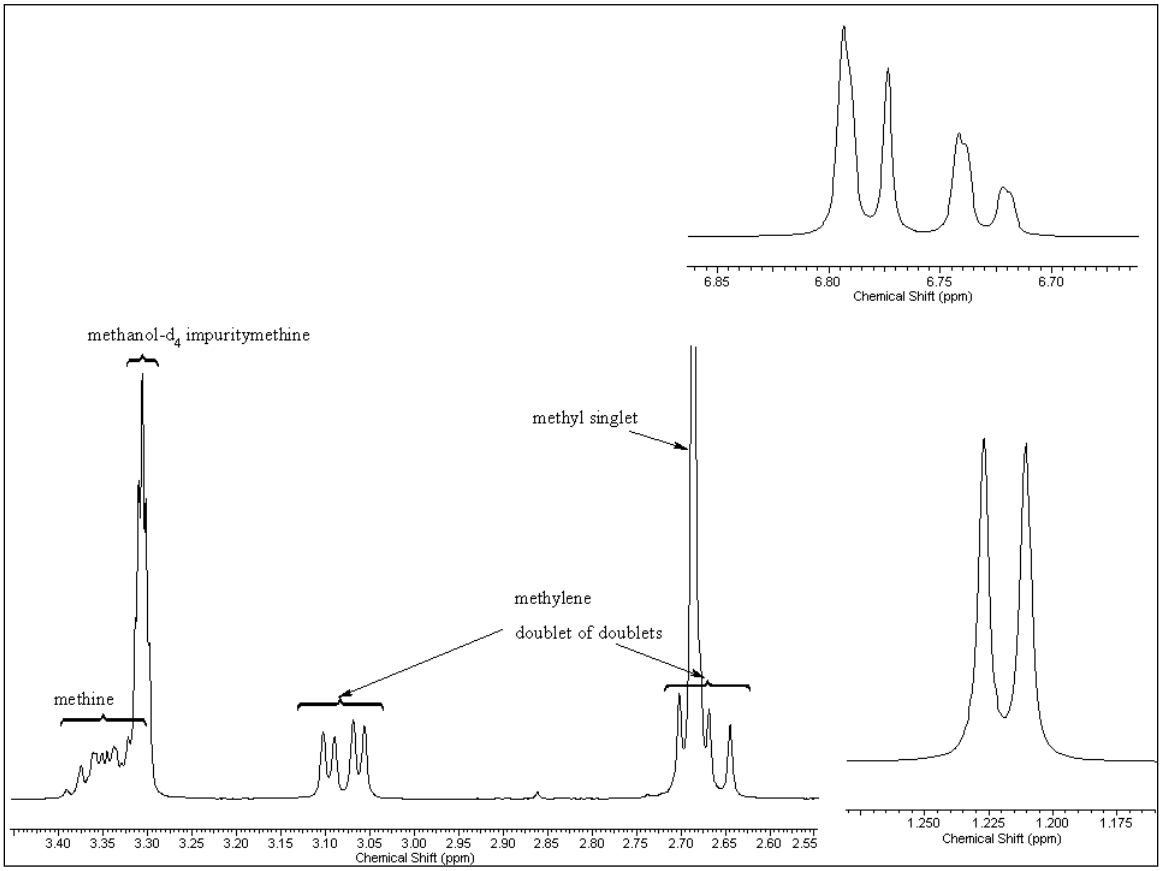
^{13}C NMR: MDMA Phosphate in D2O with TSP Lot # 2 TDM-275-01

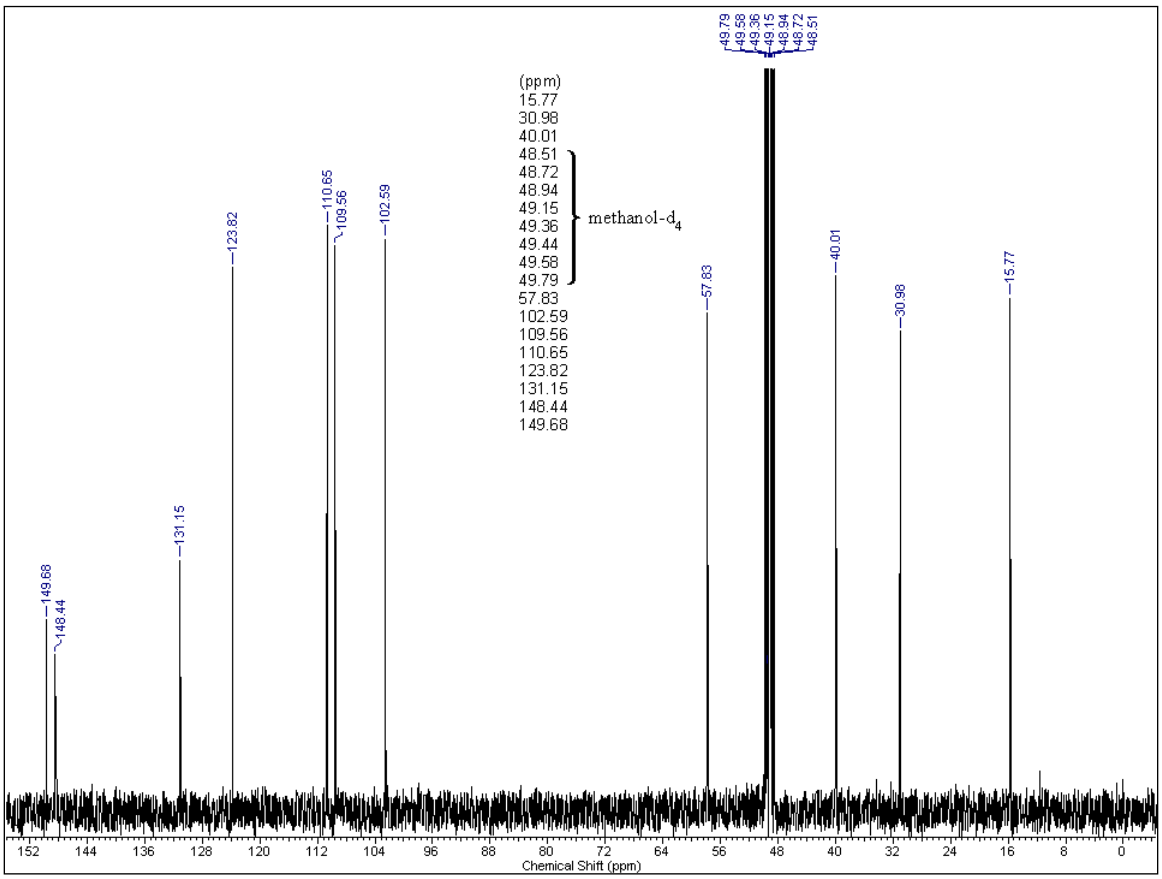


^{1}H NMR: MDMA Phosphate in CD_3OD with TMS Lot #2 TDM-275-01, 400 MHz

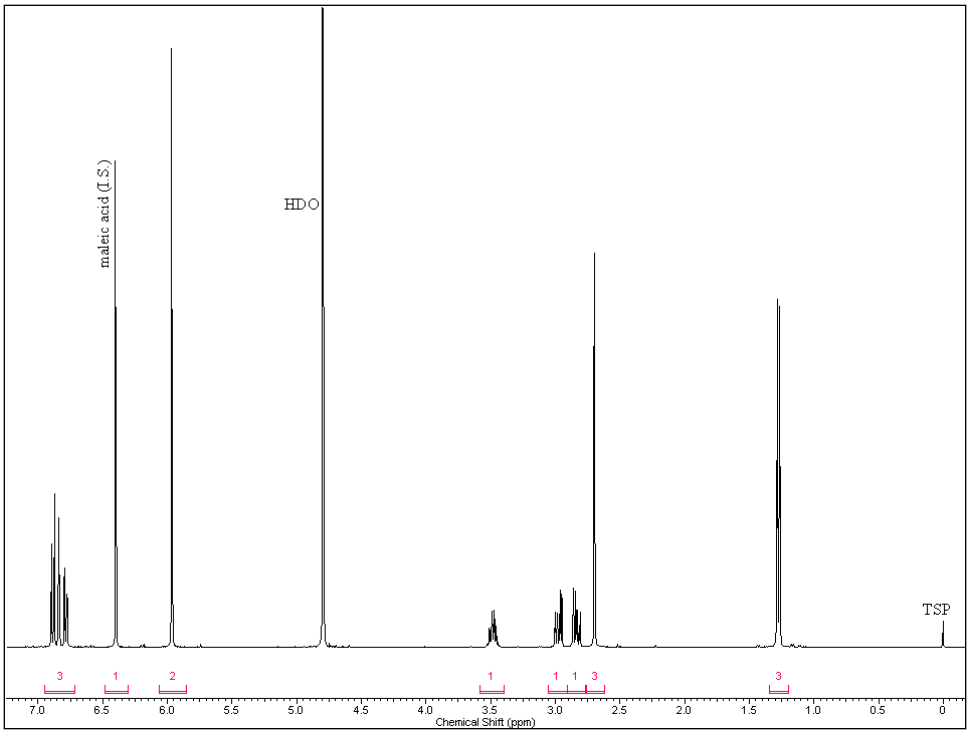


ppm 6.79 (d, J=0.98 Hz, 1 H) 6.78 (d, J=8.02 Hz, 1 H) 6.73 (dd, J=8.00, 1.20 Hz, 1 H) 5.93 (s, 2 H) 3.29 - 3.40 (m, 1 H) 3.08 (dd, J=13.50, 5.09 Hz, 1 H) 2.69 (s, 3 H) 2.67 (dd, J=13.30, 9.60 Hz, 1 H) 1.22 (d, J=6.46 Hz, 3 H)


methanol-d4 impuritymethine
methyl singlet
methylene
doublet of doublets
methine
Chemical Shift (ppm)
Chemical Shift (ppm)
Chemical Shift (ppm)

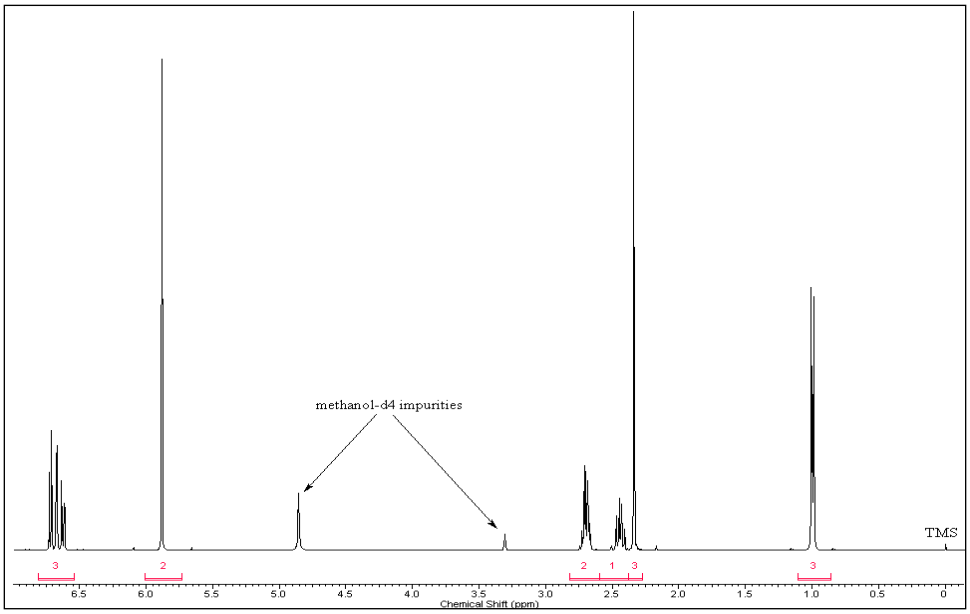

(ppm)
15.77
30.98
40.01
48.51
48.72
48.94
49.15
49.36
49.44
49.58
49.79
methanol-d4
57.83
102.59
109.56
110.65
123.82
131.15
148.44
149.68
Chemical Shift (ppm)

1H NMR: MDMA HCl Lot # A150B
with maleic acid as internal standard in D_2O with TSP, 400 MHz

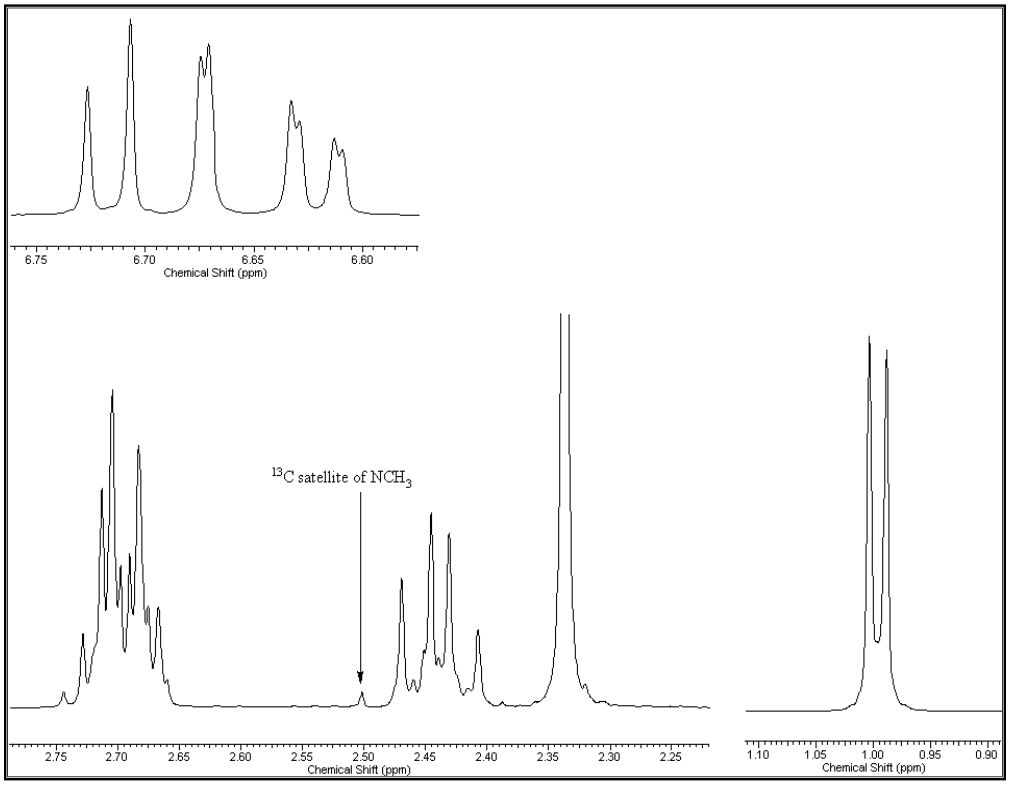


ppm 6.88 (d, J=7.92 Hz, 1 H) 6.84 (d, J=1.66 Hz, 1 H) 6.76 - 6.81 (dd, J=7.90, 1.80 Hz, 1 H) 5.97 (s, 2 H) 3.41 - 3.54 (ddq, J=7.70, 6.70, 6.70, 6.70, 6.50 Hz, 1 H) 2.93 - 3.02 (dd, J=14.00, 6.50 Hz, 1 H) 2.83 (dd, J=14.00, 7.70 Hz, 1 H) 2.70 (s, 3 H) 1.27 (d, J=6.65 Hz, 3 H). Maleic acid 6.40 (s, 2H).

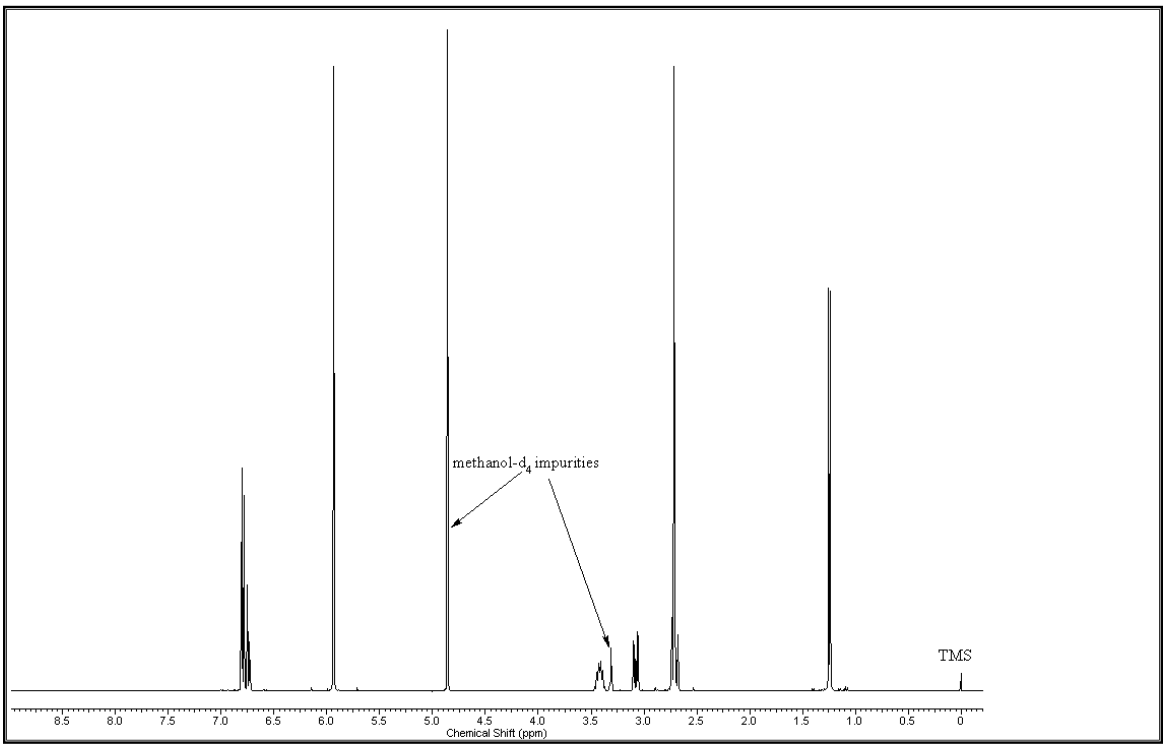
^{1}H NMR: MDMA base in CD_3OD with TMS
Extracted from MDMA HCl – Lot #A150B, 400 MHz



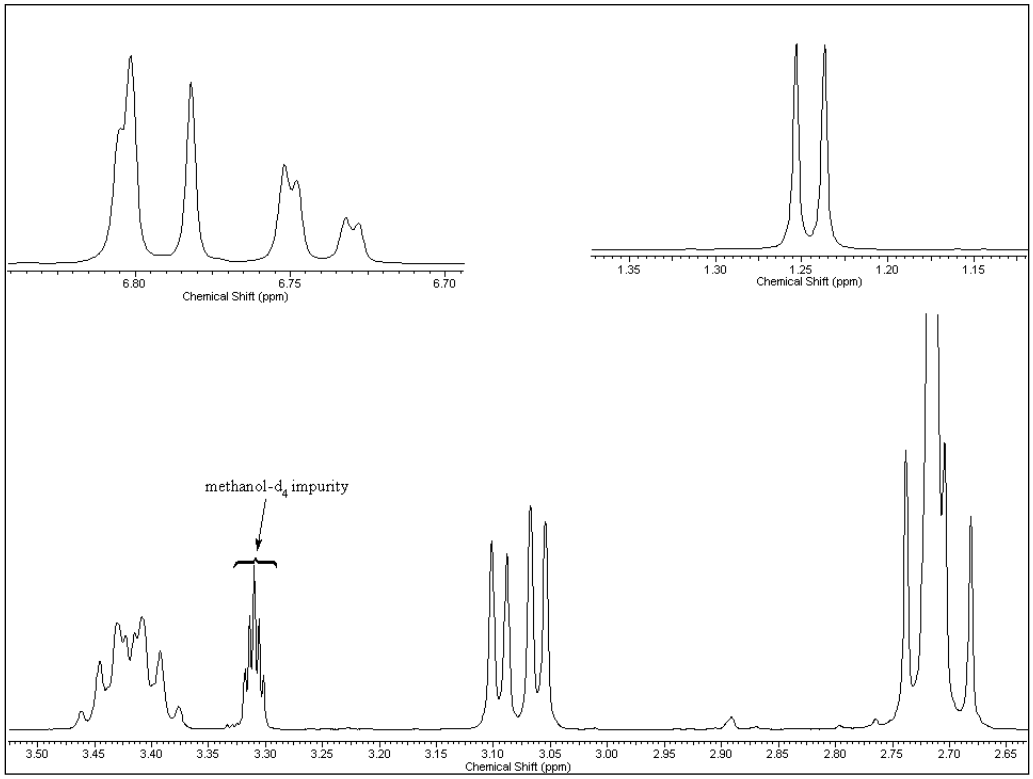
ppm 6.69 - 6.74 (d, J=7.83 Hz, 1 H) 6.67 (d, J=1.37 Hz, 1 H) 6.62 (dd, J=7.80, 1.40 Hz, 1 H) 5.88 (s, 2 H) 2.65 - 2.76 (m, 2 H) 2.44 (dd, J=15.30, 9.40 Hz, 1 H) 2.34 (s, 3 H) 1.00 (d, J=6.06 Hz, 3 H)



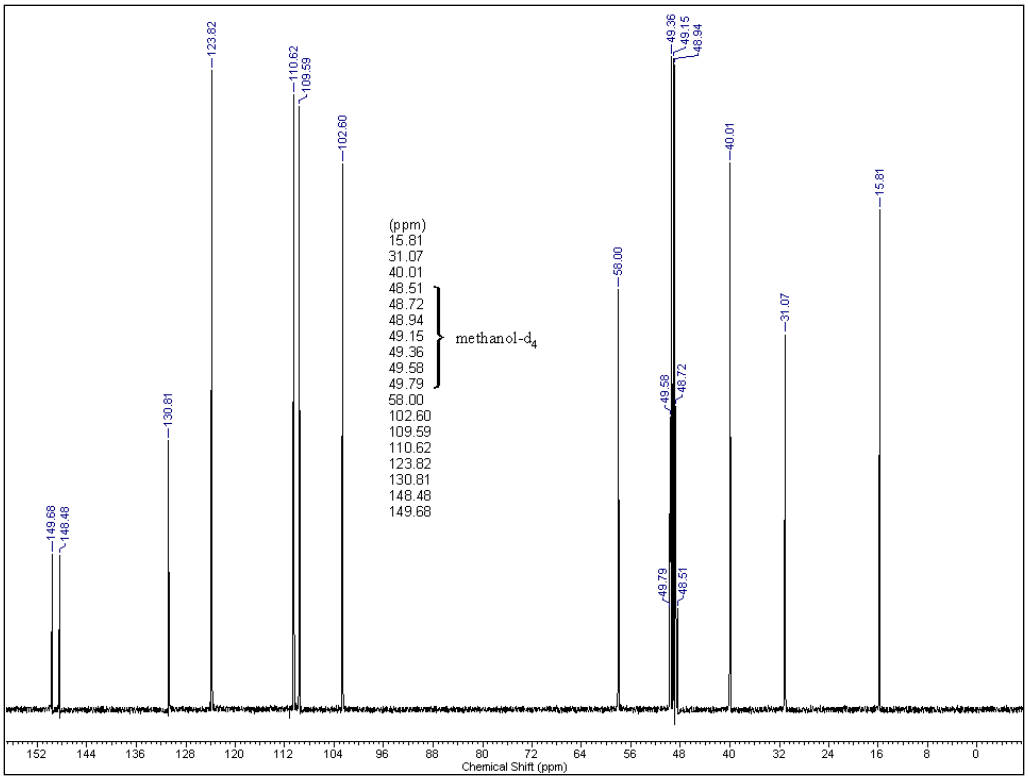
^{1}H NMR: MDMA HCl in CD_3OD Lot # A150B
400 MHz



ppm 6.80 (d, J=1.60 Hz, 1 H) 6.79 (d, J=8.02 Hz, 1 H) 6.74 (dd, J=8.02, 1.56 Hz, 1 H) 5.93 (s, 2 H) 3.36 - 3.48 (ddq, J=9.40, 6.50, 6.50, 6.50, 5.30 Hz, 1 H) 3.08 (dd, J=13.50, 5.28 Hz, 1 H) 2.71 (dd, J=13.30, 9.40 Hz, 1 H) 1.25 (d, J=6.46 Hz, 3 H)



^{13}C NMR: MDMA HCl in CD_3OD Lot # A150B



*Optical isomers standards for these compounds were unavailable.
***No Data Available