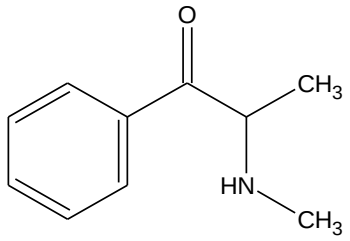
METHCATHINONE

Latest Revision: July 11, 2005



1. SYNONYMS

CFR: Methcathinone

CAS #: Base: 5650-44-2
Hydrochloride: 49656-78-2

Other Names: N-methylcathinone
Methylcathinone
2-Methylamino-1-phenyl-1-propanone
α-Methylaminopropiophenone
α-N-methylaminopropiophenone
Monomethylpropion
Ephedrone
2-(Methylamino)-1-phenylpropan-1-one

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Range (°C)
Base	$C_{10}H_{13}NO$	163.2	Liquid at room temperature
Hydrochloride	$C_{10}H_{13}NO.HCl$	199.6	176-177

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Base	SS	FS	SS	I	FS	SS
Hydrochloride	VSS	FS	I	I	FS	FS

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 TECHNIQUE

3.1. COLOR TESTS

REAGENT	COLOR PRODUCED
Marquis	No reaction
Sodium Nitroprusside	Slight ppt. of blue flecks or blue ring formed
Chen's	Slow forming yellow-orange
Janovsky	Brownish red

3.2. GAS CHROMATOGRAPHY

Method MCAT-GCS1

Instrument: Gas chromatograph operated in split mode with FID

Column: 5% phenyl/95% methyl silicone 10 m x 0.25 mm x 0.25 µm

Carrier gas: Helium at 1.0 mL/min

Temperatures: Injector: 275°C
Detector: 280°C
Oven program:
1) 100°C initial temperature
2) Ramp to 295°C at 35°C/min
3) Hold final temperature for 6.43 min

Injection Parameters: Split Ratio = 100:1, 1 µL injected

Samples are to be dissolved in appropriate solvent and filtered.

COMPOUND	RRT	COMPOUND	RRT
pseudoephedrine	0.14	guaifenesin	1.11
tridecane	0.22	acetaminophen	1.18

methamphetamine	0.55	phenacetin	1.23
MDMA	0.69	dimethyl sulfone	1.33
amphetamine	0.74	caffeine	1.73
methcathinone	**1.00 (3.07 min)**	dextromethorphan	2.67

3.3. CAPILLARY ELECTROPHORESIS

Method MCAT-CES1

Buffer Preparation:
Tris-phosphate buffer, pH 2.4 - Prepare by adding one drop H_3PO_4 per 50 mL of 25 mM Tris(hydroxymethyl)aminomethane HCl buffer

Injection Solvent:
9:1 H_20:Tris-phosphate buffer

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of l-methcathinone, d-methcathinone, l-pseudoephedrine, d-ephedrine, l-ephedrine, d-pseudoephedrine, l-methamphetamine and d-methamphetamine using approximately 1.0 mg of racemic mixture or 0.5 mg of an individual enantiomer in 10 mL of injection solvent.

Sample Preparation:
Accurately weigh an amount of sample and dilute with injection solvent to an approximate concentration of 0.05 mg/mL.

Mode:	Free zone
Column:	82 cm (60 cm effective length) x 50 µm fused silica capillary
Run Buffer:	1.2% MeOH, 98.8% 1mM β-cyclodextrin-sulfobutylether (IV) [β-CD-SBE(IV)] in 25 mM Tris-phosphate buffer, pH 2.4
Detector:	UV, 210 nm
Voltage:	30 kV
Temperature:	30°C
Injection:	0.5 s hydrodynamic
Run Time:	16 min
Rinse Time:	2 min

COMPOUND	RMT	COMPOUND	RMT
l-pseudoephedrine	0.16	**d-methcathinone**	**1.00 (10.23 min)**
l-methcathinone	**0.43**	d-pseudoephedrine	1.27
d-ephedrine	0.70	l-methamphetamine	2.59
l-ephedrine	0.93	d-methamphetamine	3.27

4. EXTRACTION TECHNIQUES

Because Methcathinone is sensitive to strong base, it is preferred that saturated sodium carbonate (pH 11) be used to basify aqueous mixtures prior to extraction.

Chloroform and methylene chloride are suitable extraction solvents. Shaking the basified aqueous solution with one of these solvents will transfer the methcathinone as the free base into the organic solvent. The solvent should then be passed through a layer of anhydrous sodium sulfate to remove any residual basic aqueous phase. Addition of 2-propanol or ethanol:hydrochloric acid mixture (4:1) to the extracted base, and subsequent solvent evaporation with a steam bath, is suitable to provide the hydrochloride salt.

5. QUANTITATIVE PROCEDURE

5.1. GAS CHROMATOGRAPHY

Method MCAT-GCQ

Internal Standard Solution (ISS):
1.00 mg/mL Tetradecane in methylene chloride.

Standard Solution Preparation:
Prepare a standard solution of Methcathinone HCl between 0.4 and 4.56 mg/mL with ISS as solvent.

Sample Preparation:
Accurately weigh an amount of sample into an appropriately sized volumetric flask so the final concentration of Methcathinone HCl is approximately equivalent to that of the standard solution. Dilute to volume with ISS.

Instrument: Gas chromatograph operated in split mode with FID

Column: HP-1 or equivalent, 30 m x 0.25 mm x 0.25 µm film thickness

Carrier gas: Hydrogen at 2.8 mL/min

Temperatures: Injector: 265°C
Detector: 275°C
Oven: 130°C Isothermal for 5 min

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

Typical Retention Time: 2.712 min

Linear Range: 0.40 - 4.56 mg/mL

Repeatability: %RSD less than 4%

Correlation Coefficient: 0.9997

Accuracy: %Error less than 5%

COMPOUND	RRT
methamphetamine	0.629
methcathinone	**1.00**
pseudoephedrine	1.158
tetradecane	1.421

The adulterants guaifenesin, diphenhydramine, dextromethorphan and chlorpheniramine had retention times greater than 5 min.

6. QUALITATIVE DATA

6.1. **INFRARED SPECTROSCOPY (FT-IR)**

Both sample and standard should be subjected to the same procedures, using either KBr pellet or ATR accessory.

6.2. **NUCLEAR MAGNETIC RESONANCE (NMR)**

A solution of approximately 10 to 20 mg sample to 1 mL deuterated solvent is an acceptable concentration for use on the NMR instrument.

6.3. **FT-RAMAN SPECTROPHOTOMETRY (FT-RAMAN)**

No sample preparation is needed unless two or more active substances are in a sample.

6.4. **GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)**
The same parameters from separation method MCAT-GCS1 can be utilized on a GC/MS system with the MS detector temperature at 280°C

See spectra on the following pages for FT-IR, NMR, FT-Raman, and MS.

7. REFERENCES

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

Dal Cason, T., "The Identification of Cathinone and Methcathinone,"*Microgram*, December 1992, pp. 10-29

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Lurie, I., et al., "Chiral Resolution of Cationic Drugs of Forensic Interest by Capillary Electrophoresis with Mixtures of Neutral and Anionic Cyclodextrins," *Analytical Chemistry*, Vol. 66, No. 22, Nov. 15, 1994, pp. 4019-26.

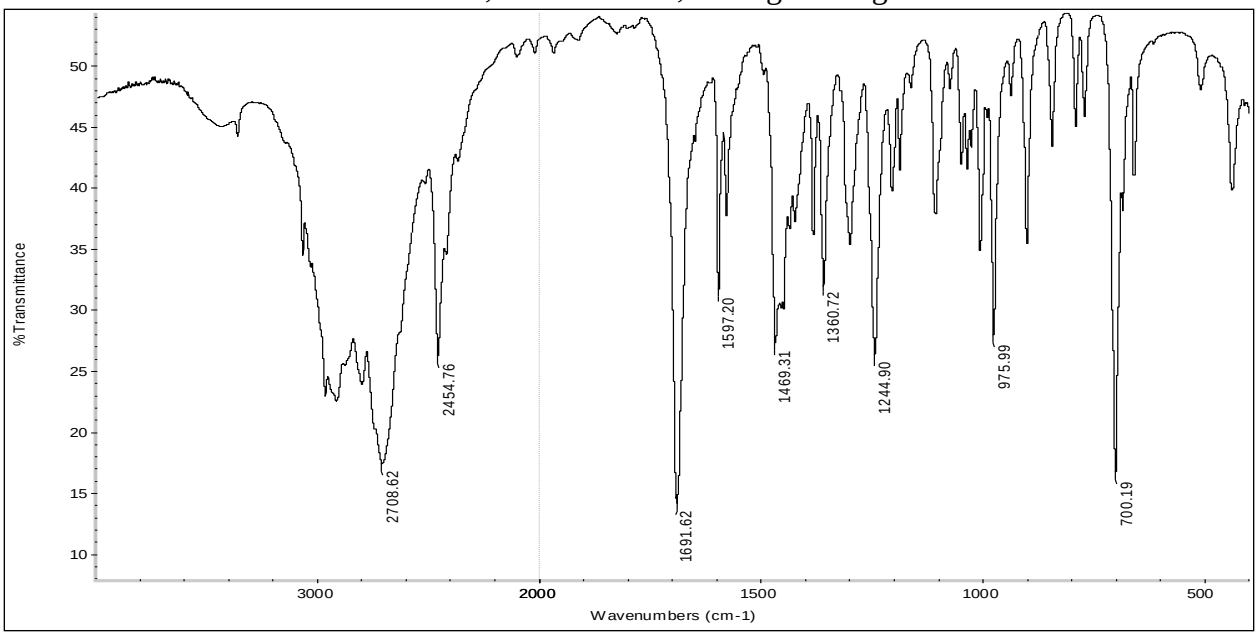
Moffat, A. C., Sr. ed., *Clarke's Isolation and Identification of Drugs*, The Pharmaceutical Press, Second Edition, 1996.

O'Neil, M. ed., *The Merck Index, 13th Edition*, Merck and Co., Inc., 2001, p. 1067.

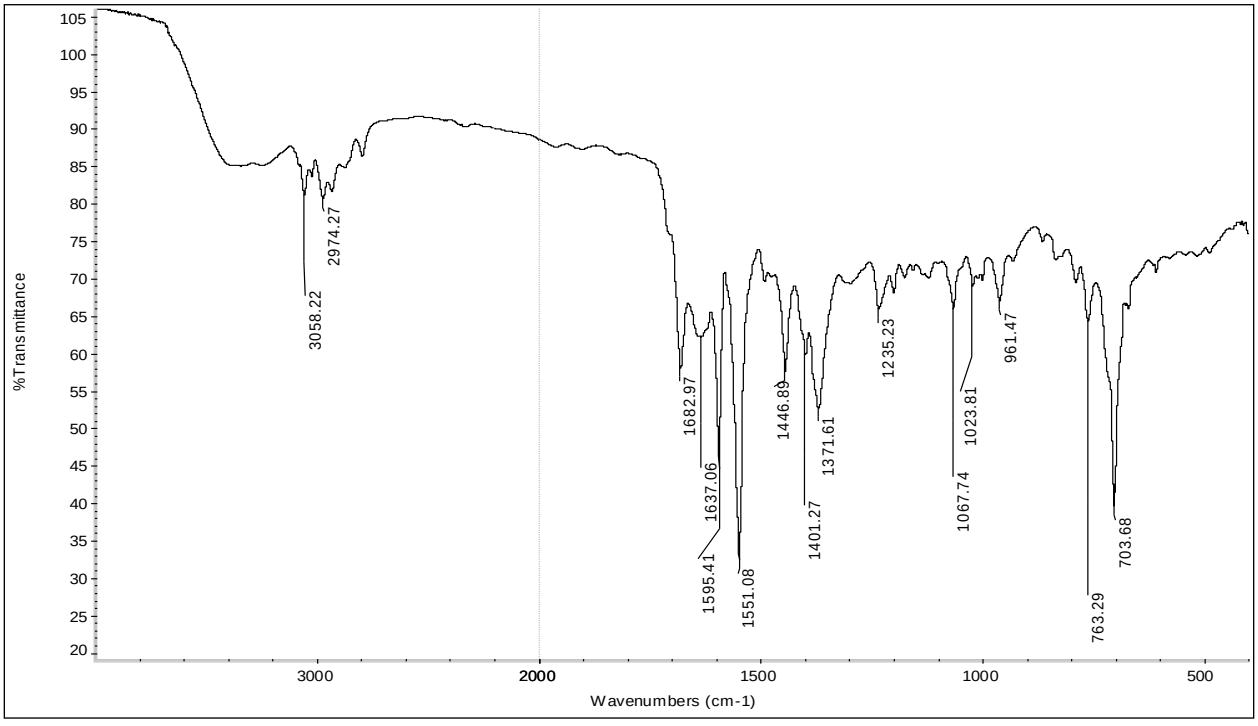
8. ADDITIONAL RESOURCES

Wikipedia

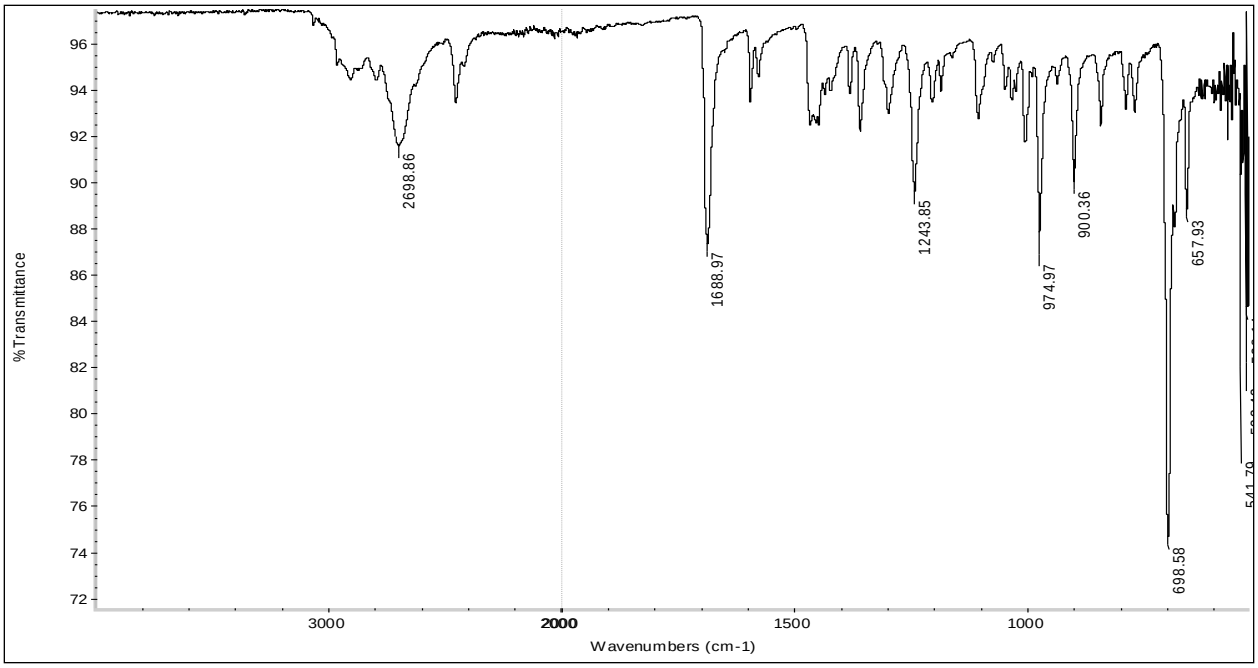
FTIR: Methcathinone hydrochloride, KBr pellet
16 Scans, resolution 4.0, 4.0 mg/100 mg



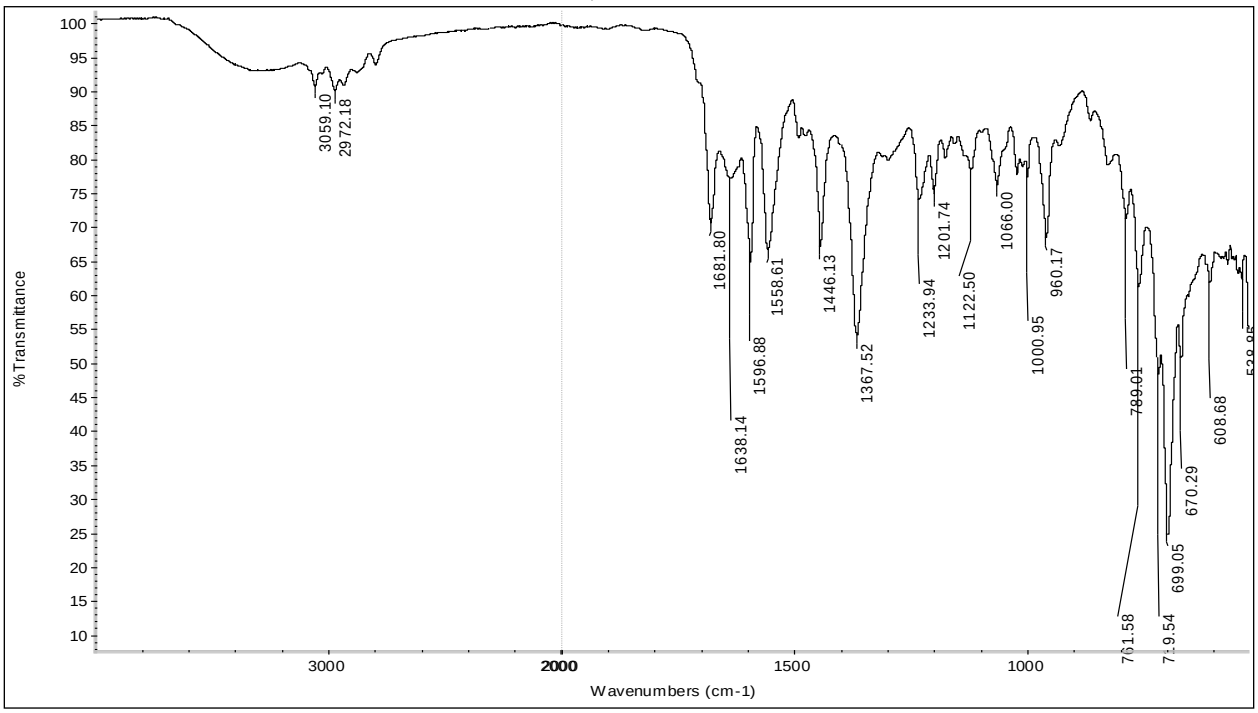
FTIR: Methcathinone base, KBr pellet
16 Scans, resolution 4.0, 4.0 mg/100 mg



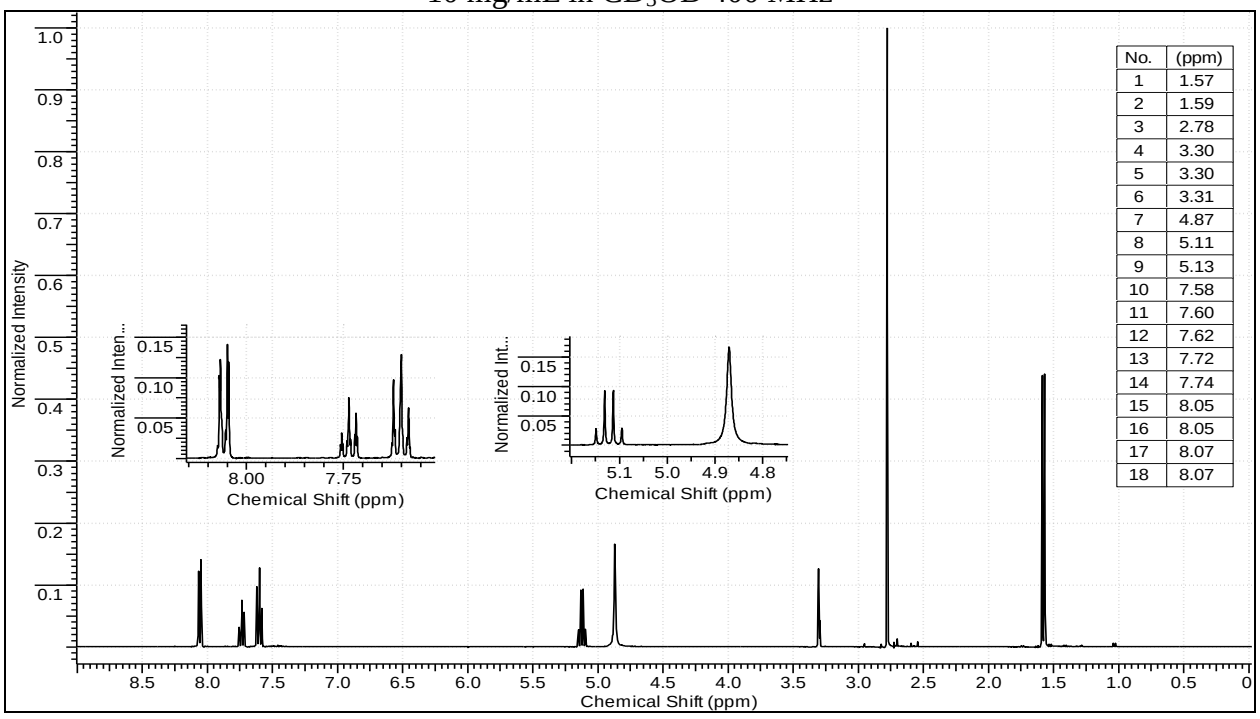
FTIR (ATR 1-bounce): Methcathinone hydrochloride
16 Scans, resolution 4.0



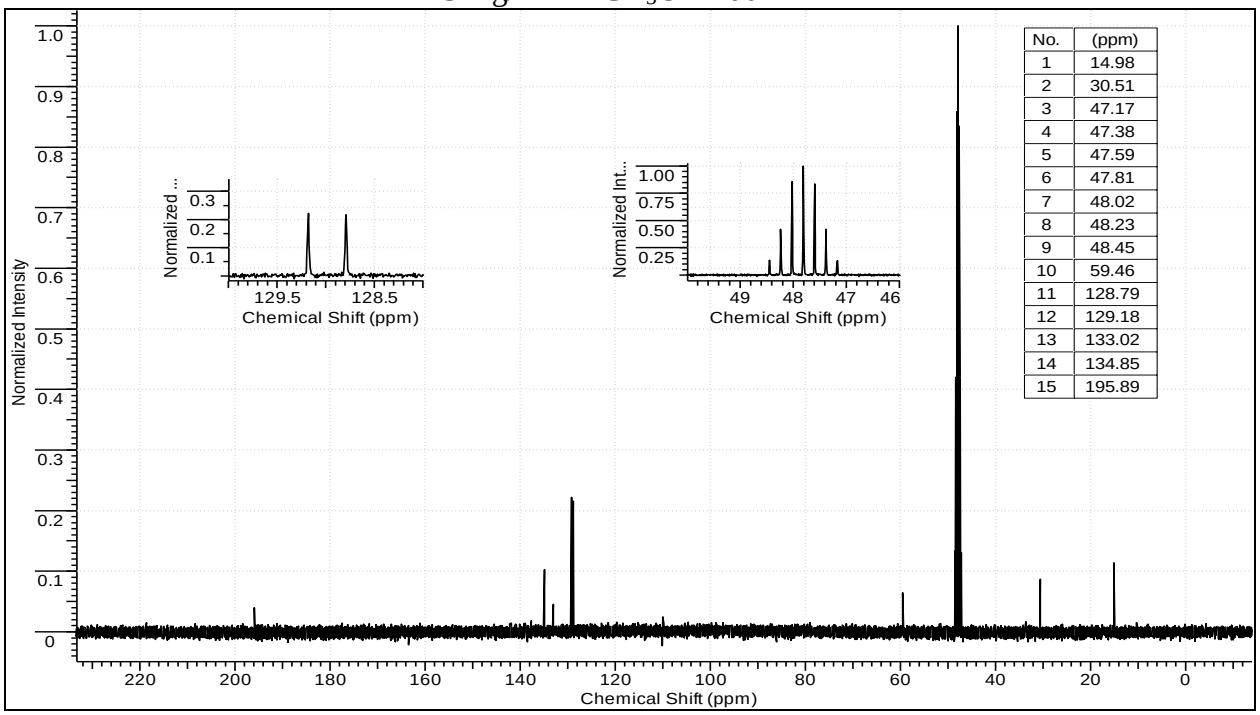
FTIR (ATR 1-bounce): Methcathinone base
16 Scans, resolution 4.0



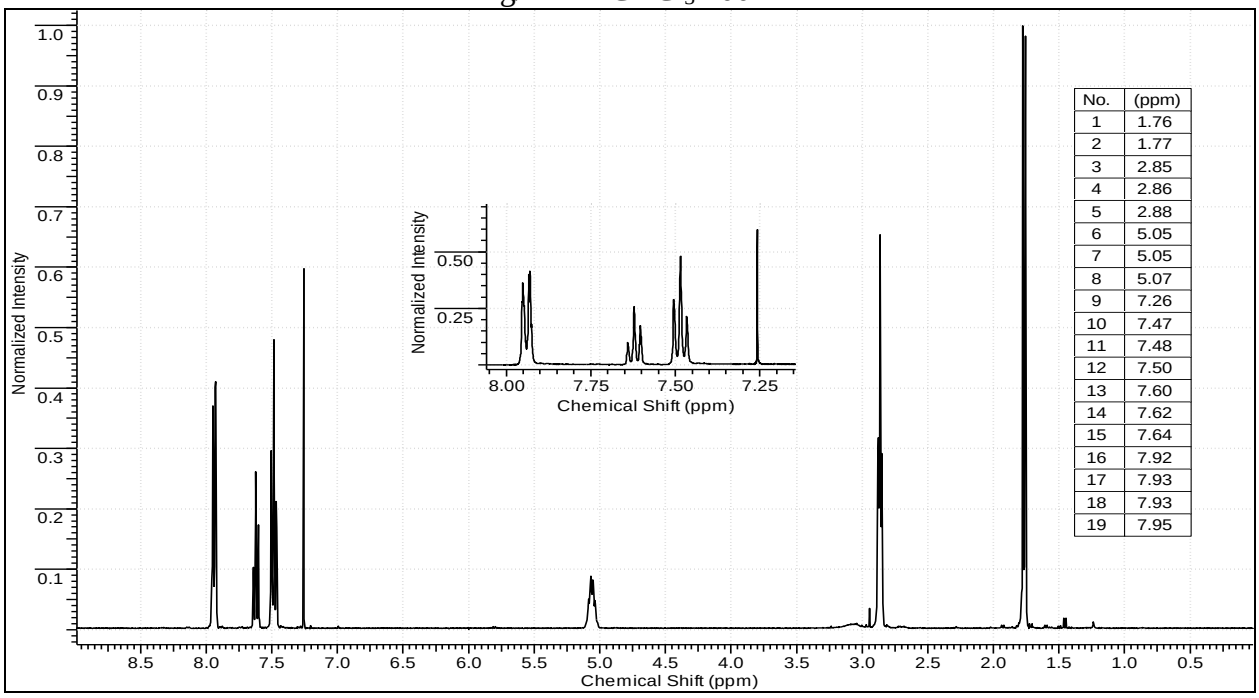
Nuclear Magnetic Resonance (proton): Methcathinone HCl
10 mg/mL in CD_3OD 400 MHz



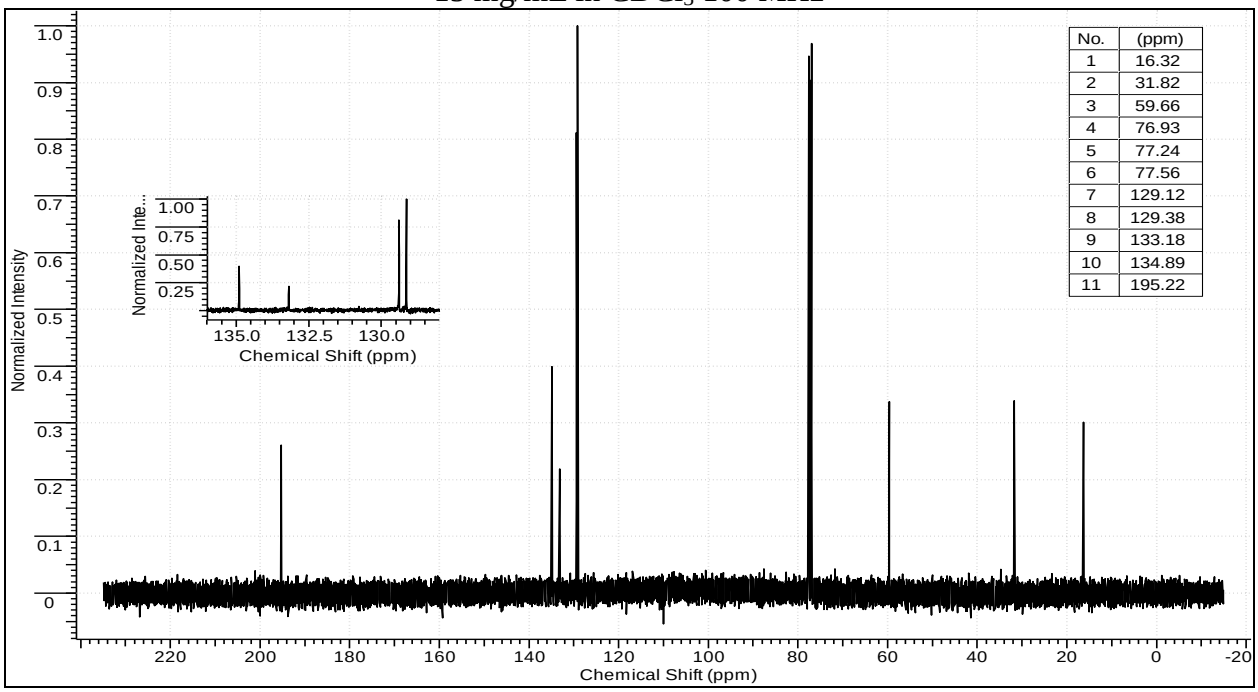
Nuclear Magnetic Resonance (carbon): Methcathinone HCl
15 mg/mL in CD_3OD 100 MHz



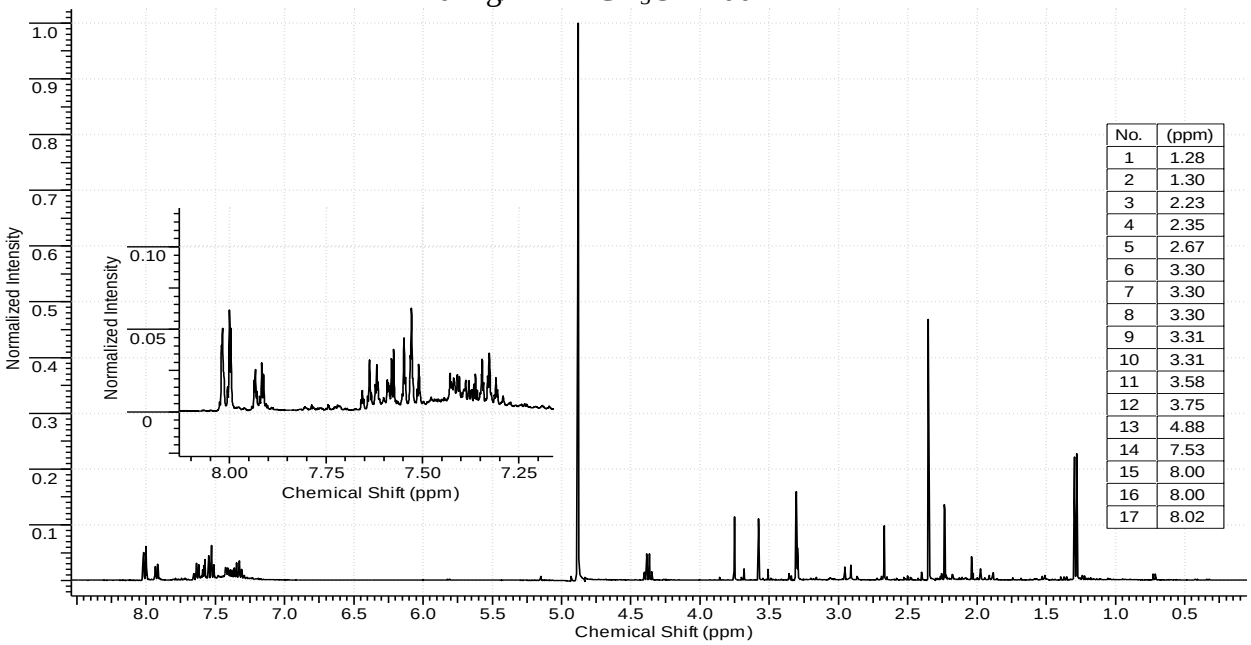
Nuclear Magnetic Resonance (proton): Methcathinone HCl
14 mg/mL in $CDCl_3$ 400 MHz



Nuclear Magnetic Resonance (carbon): Methcathinone HCl
15 mg/mL in $CDCl_3$ 100 MHz

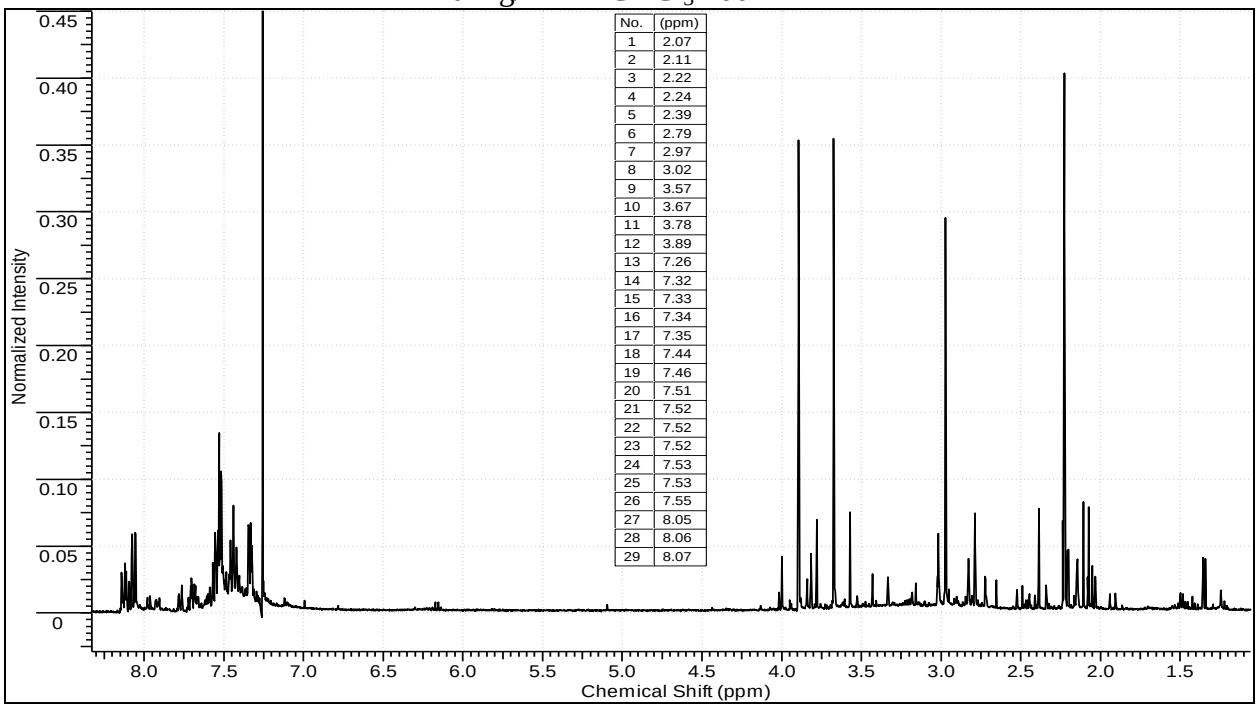


Nuclear Magnetic Resonance (proton): Methcathinone base
10 mg/mL in CD_3OD 400 MHz



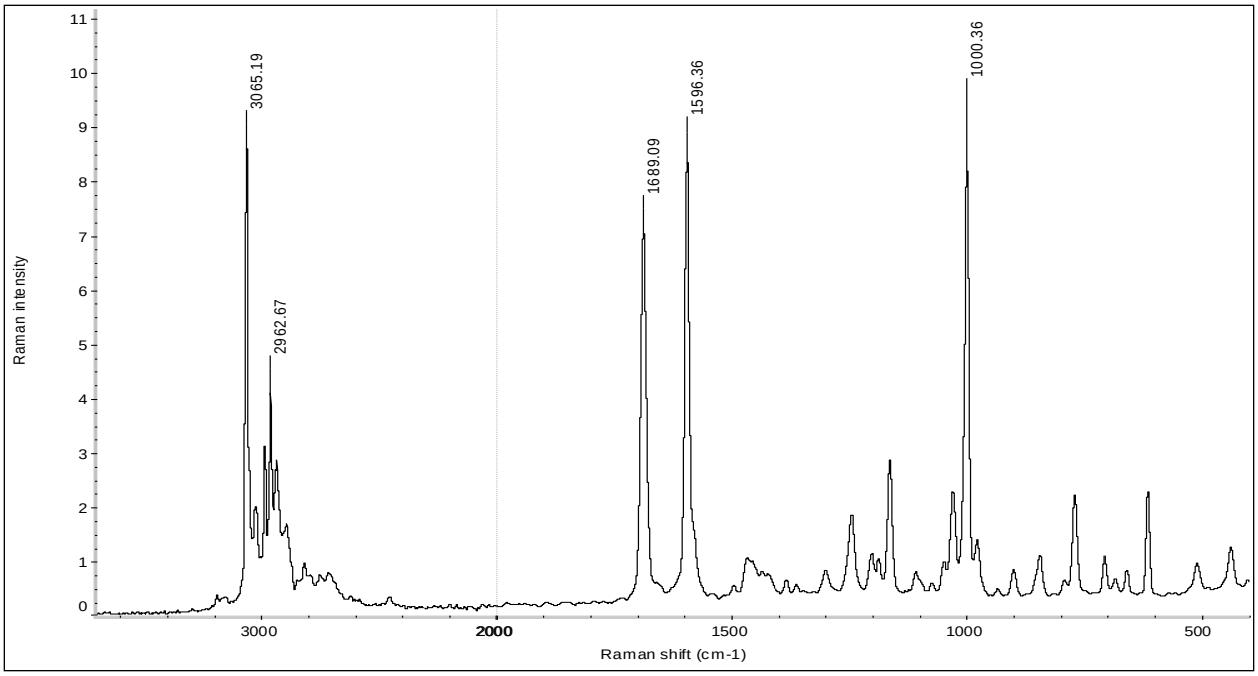
No.	(ppm)
1	1.28
2	1.30
3	2.23
4	2.35
5	2.67
6	3.30
7	3.30
8	3.30
9	3.31
10	3.31
11	3.58
12	3.75
13	4.88
14	7.53
15	8.00
16	8.00
17	8.02

Nuclear Magnetic Resonance (proton): Methcathinone base
20 mg/mL in $CDCl_3$ 400 MHz

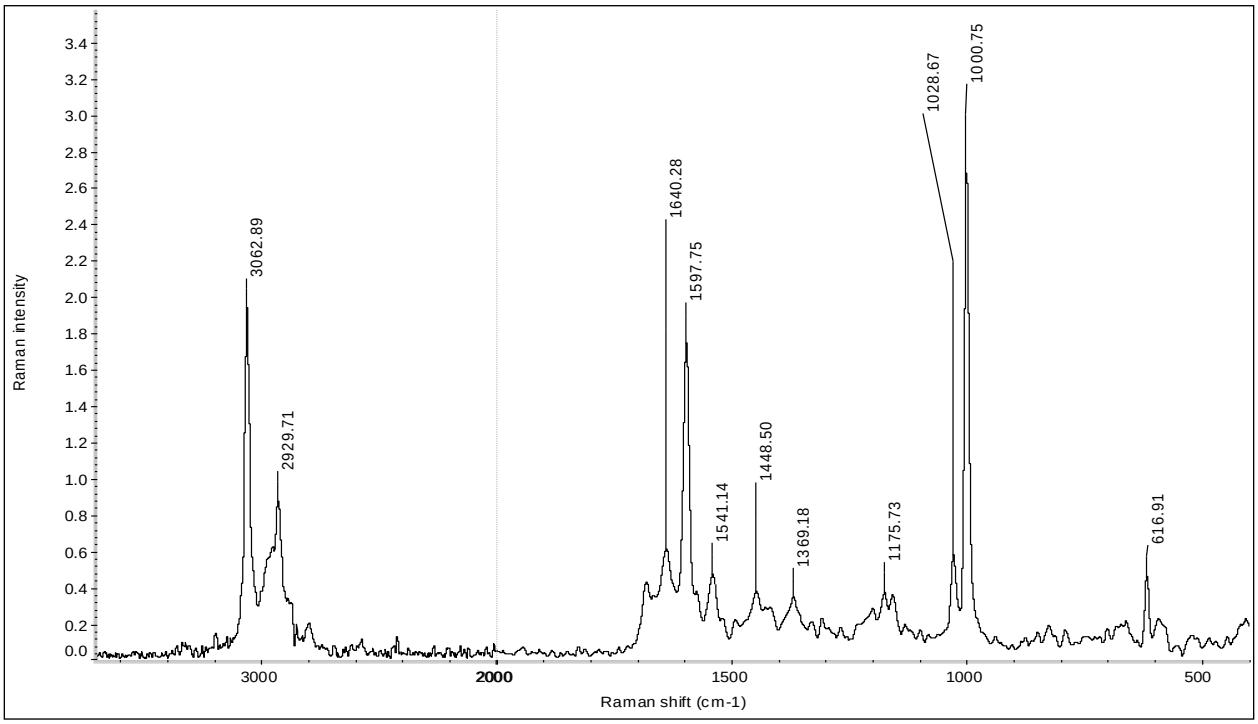


No.	(ppm)
1	2.07
2	2.11
3	2.22
4	2.24
5	2.39
6	2.79
7	2.97
8	3.02
9	3.57
10	3.67
11	3.78
12	3.89
13	7.26
14	7.32
15	7.33
16	7.34
17	7.35
18	7.44
19	7.46
20	7.51
21	7.52
22	7.52
23	7.52
24	7.53
25	7.53
26	7.55
27	8.05
28	8.06
29	8.07

FT RAMAN: Methcathinone HCl
64 Scans, resolution 8.0



FT-RAMAN: Methcathinone Base
64 Scans, resolution 8.0



MS: Methcathinone HCl in Chloroform