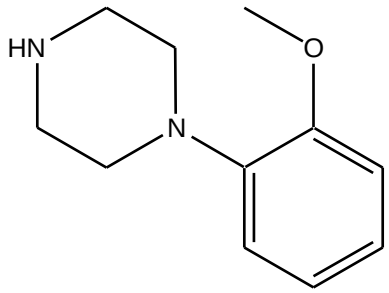
1-(2-METHOXYPHENYL)PIPERAZINE

Latest revision: June 27, 2005



1. SYNONYMS

CFR: N/A

CAS #: Base: 35386-24-4
Hydrochloride: 5464-78-8

Other Names: 2-MeOPP
2-Methoxyphenylpiperazine

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Hydrochloride	$C_{11}H_{17}ClN_2O$	228.72	213

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Hydrochloride	I	FS	VSS	I	FS	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
Cobalt thiocyanate	No reaction
Nitroprusside	Blue
Marquis	No reaction

3.2. CRYSTAL TESTS

REAGENT	COLOR PRODUCED
Platinic Bromide	clusters of rods (wide blades/rods from center core)

3.3. THIN-LAYER CHROMATOGRAPHY

Visualization

Acidified iodoplatinate solution

COMPOUND	RELATIVE R_1 System TLC5
BZP	0.9
TFMPP	1.3
2-MeOPP	**1.0**
3-MeOPP	1.1
4-MeOPP	1.0

3.4. GAS CHROMATOGRAPHY

Method PIPERAZINE-GCS1

Instrument: Gas chromatograph operated in split mode with FID

Column:	5% phenyl/95% methyl silicone 10 m x 0.32 mm x 0.52 µm
Carrier gas:	Hydrogen at 1.8 mL/min
Temperatures:	Injector: 280°C Detector: 280°C Oven program: 1) 100ºC initial temperature for 1.0 min 2) Ramp to 280°C at 25°C/min 3) Hold final temperature for 3.0 min
Injection Parameters:	Split Ratio = 50:1, 1 µL injected

Samples are to be dissolved in methanol.

COMPOUND	RRT	COMPOUND	RRT
dimethyl sulfone	0.240	3,4-methylenedioxymethamphetamine	0.903
methamphetamine	0.532	**1-(2-methoxyphenyl)piperazine**	**1.0 (4.868min)**
dimethylphthalate	0.819	1-(4-methoxylphenyl)piperazine	1.114
benzylpiperazine	0.865	1-(3-methoxyphenyl)piperazine	1.127
1-(3-trifluoromethylphenyl)piperazine	0.899	caffeine	1.178

4. SEPARATION TECHNIQUES

The solubility properties provided in the table 2.2 can be utilized to extract diluents and adulterants, including other isomeric forms of 2-MeOPP. For example, chloroform may be used to separate 3-MeOPP from 2-MeOPP. 1-(3-methoxyphenyl)piperazine is very slightly soluble in $CHCl_3$ whereas 1-(2-methoxyphenyl)piperazine is fairly soluble in $CHCl_3$.

5. QUANTITATIVE PROCEDURES

5.1. GAS CHROMATOGRAPHY

Method PIPERAZINE1-GCQ1

Internal Standard Stock Solution:
0.25 mg/mL dimethylphthalate in methanol.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of 1-(2methoxyphenyl)piperazine at approximately 1.0 mg/mL using above 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.

Instrument:	Gas chromatograph operated in split mode with FID
Column:	5% phenyl/95% methyl silicone 10 m x 0.32 mm x 0.52 µm film thickness
Carrier gas:	Hydrogen at 1.0 mL/min
Temperatures:	Injector: 280°C Detector: 280°C Oven program: 1) 130°C initial temperature for 1.0 min 2) Ramp to 200°C at 25°C/min 3) Hold final temperature for 1.0 min
Injection Parameters:	Split Ratio = 50:1, 1 µL injected
Typical Retention Time:	1-(2-Methoxyphenyl)piperazine: 2.858 min Dimethylphthalate: 2.045 min
Linear Range:	0.1364 - 2.0296 mg/mL
Repeatability:	RSD less than 0.5%
Correlation Coefficient:	0.999
Accuracy:	Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
methamphetamine	0.368	**1-(2-methoxyphenyl)piperazine**	**1.0(2.858min)**
dimethylphthalate	0.716	1-(4-methoxylphenyl)piperazine	1.176
benzylpiperazine	0.781	1-(3-methoxyphenyl)piperazine	1.209
1-(3-trifluoromethylphenyl)piperazine	0.839	caffeine	1.296

5.2. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY
Method 2MEOPP-LCQ

Sample Preparation:
Accurately weigh an amount of sample into a volumetric flask and dilute with 0.01 N HCl. If necessary, dilute the sample so the final concentration approximates the standard concentration.

Instrument:	High performance liquid chromatograph equipped with diode array
Column:	4 mm x 250 mm, 10 µm C18(2)
Detector:	UV, 210 nm
Flow:	1.00 mL/min
Injection Volume:	3.0 µL
Buffer:	4000 mL distilled water, 10 g sodium hydroxide, 30.0 mL phosphoric acid and 8.0 mL hexylamine (NaHAP buffer)
Mobile Phase:	86% NaHAP Buffer: 14% Acetonitrile
Linear Range:	0.1267 - 0.760 mg/mL
Repeatability:	Less than 3% RSD
Correlation Coefficient:	0.9999
Accuracy:	Error less than 5%

COMPOUND	**RRT**	**COMPOUND**	**RRT**
BZP	0.45	3-MeOPP	1.10
TFMPP	5.11	4-MeOPP	0.87
2-MeOPP	**1.00 (5.135 min)**		

5.3. CAPILLARY ELECTROPHORESIS

Method PIP-CEQ1

Internal Standard Stock Solution:
Thiamine hydrochloride internal standard at a concentration of 0.2 mg/mL.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution at approximately 0.4mg/mL using the internal standard stock solution.

Sample Preparation:
Accurately weigh an amount of sample and dilute with internal standard stock solution. The sample concentration should approximate the standard.

Mode: Free zone

Column: 34 cm x 50 µm fused silica capillary

Run Buffer: 100 mM lithium phosphate buffer at pH 2.3

Detector: UV, 210 nm

Voltage: 20 kV

Temperature: 20°C air cooled

Injection: Hydrodynamic, 50 mbar for 2.5 s

Run Time: 6 min

Rinse Time: 1 min

Typical Migration Time: 1-(2-Methoxyphenyl)piperazine: 4.712
Thiamine: 3.144

Linear Range: 0.05 – 1.2 mg/mL

Repeatability: RSD less than 3%

Correlation Coefficient: 0.999

Accuracy: Error less than 5%

COMPOUND	RMT	COMPOUND	RMT
thiamine	0.677	**1-(2-methoxyphenyl)piperazine**	**1**
benzylpiperazine	0.748	1-(3-methoxyphenyl)piperazine	1.009
methamphetamine	0.942	1-(3-trifluoromethylphenyl)piperazine	1.060
1-(4-methoxylphenyl)piperazine	0.970		

6. QUALITATIVE DATA

6.1. ULTRAVIOLET SPECTROPHOTOMETRY

SOLVENT	MAXIMUM ABSORBANCE (NM)
Aqueous acid	206

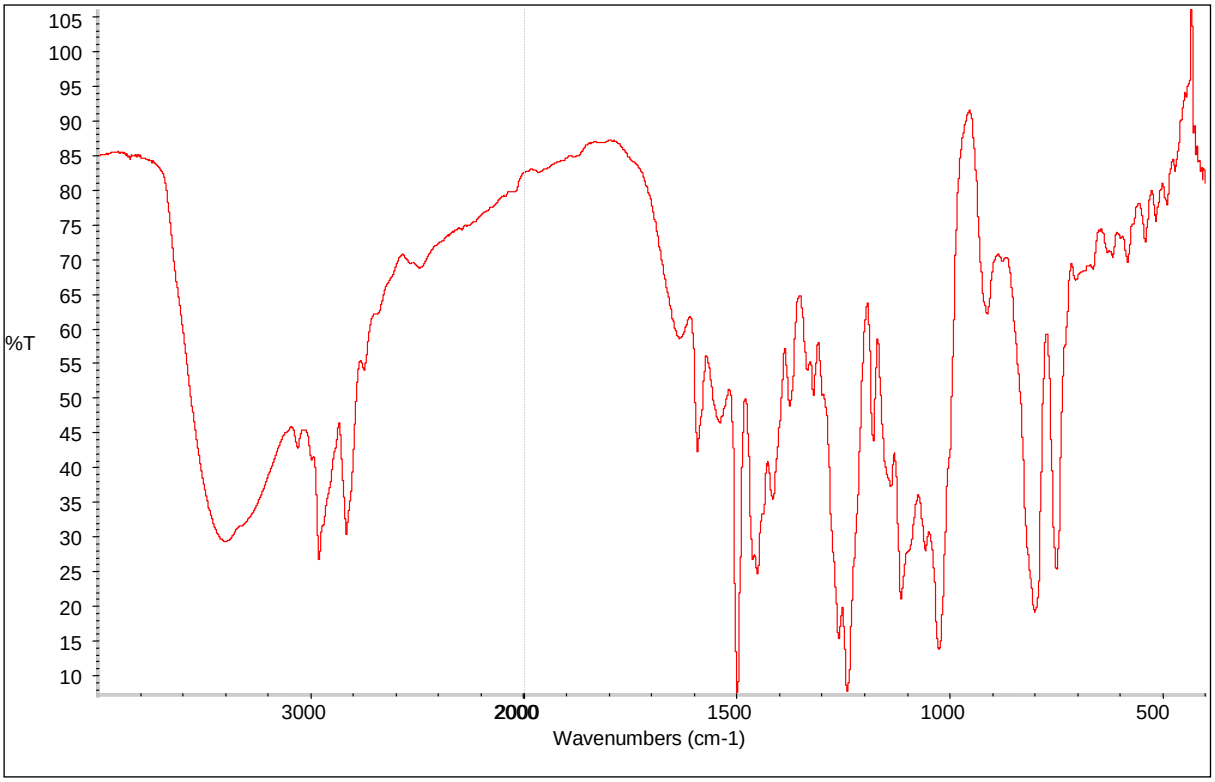
7. REFERENCES

https://fscimage.fishersci.com/msds/22615.htm

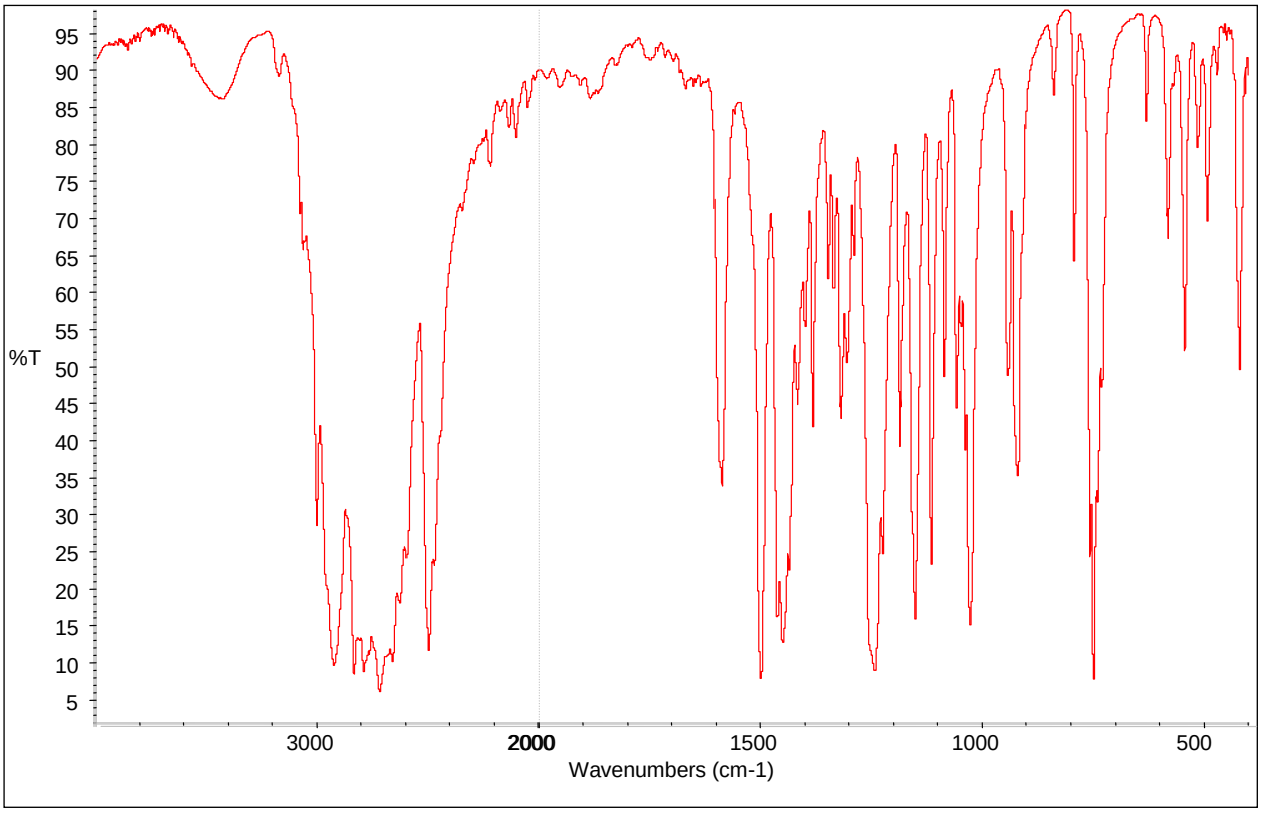
https://fscimage.fishersci.com/msds/24968.htm

Ely, Roger A., *The Forensic Examination Of Benzylpiperazine And Phenylpiperazine Homologs*, 9th Annual CLIC Technical Training Seminar, Sept. 8-11, 1999.

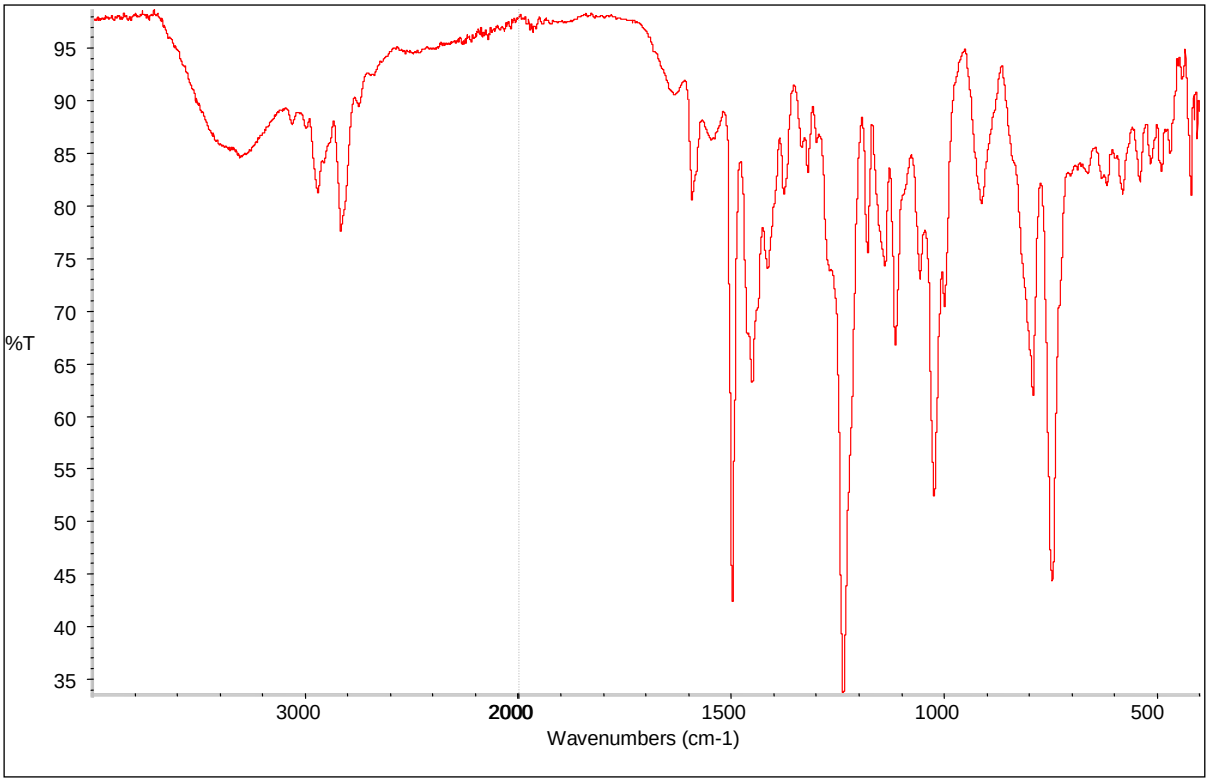
FTIR: 1-(2-Methoxyphenyl)piperazine base in KBr
16 scans; 4 cm^{-1} resolution



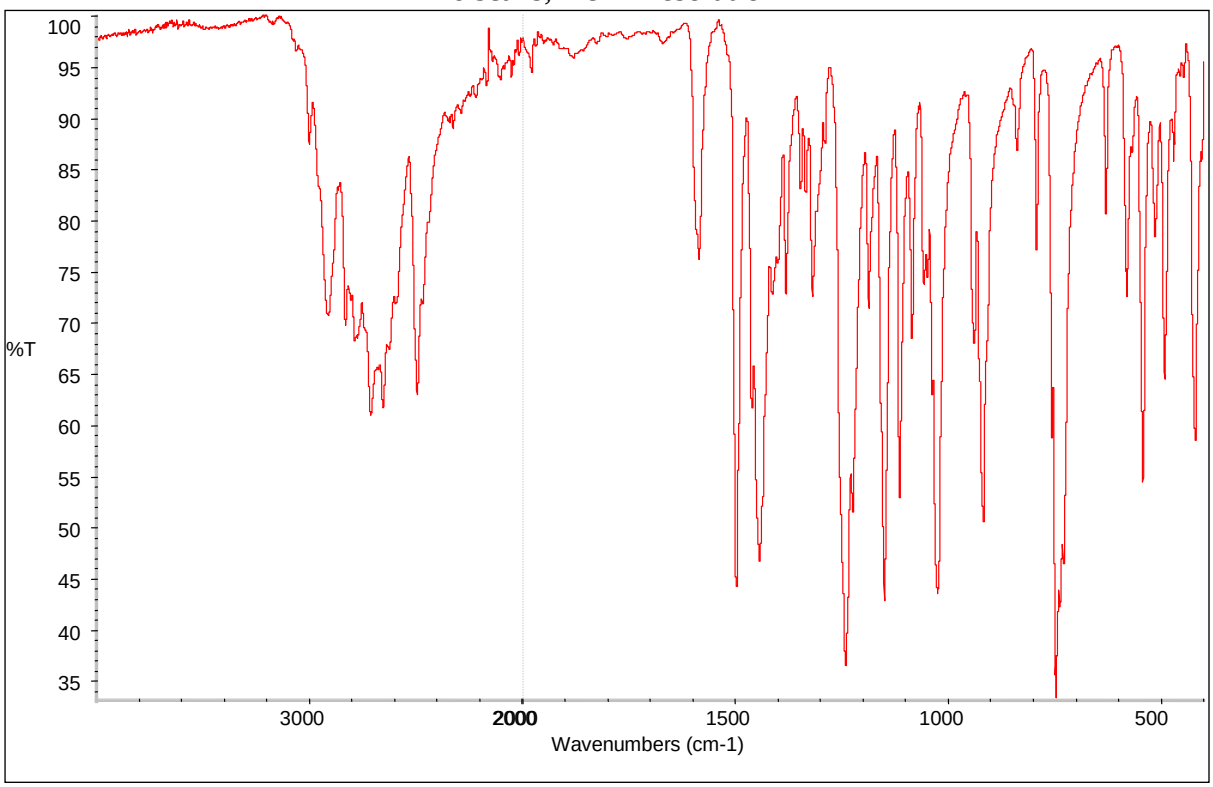
FTIR: 1-(2-Methoxyphenyl)piperazine HCl in KBr
16 scans; 4 cm^{-1} resolution



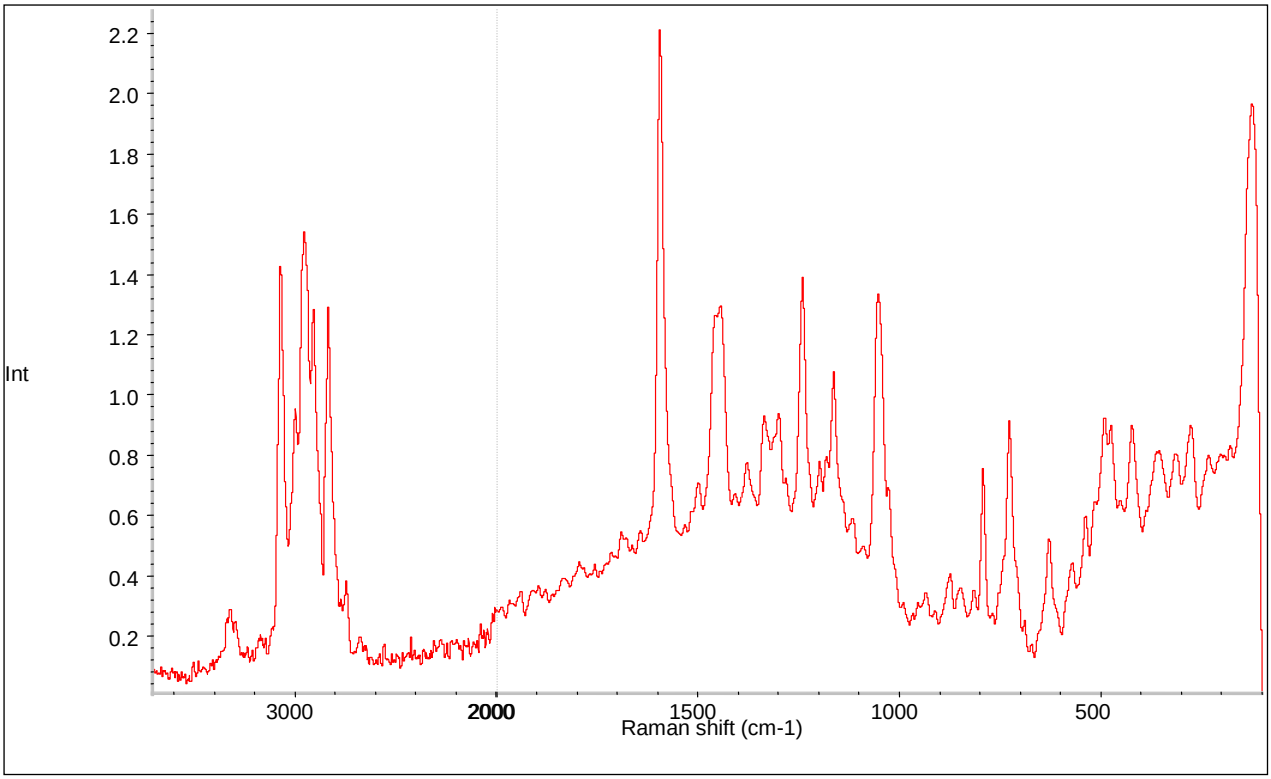
FTIR (ATR): 1-(2-Methoxyphenyl)piperazine base
16 scans; 4 cm^{-1} resolution



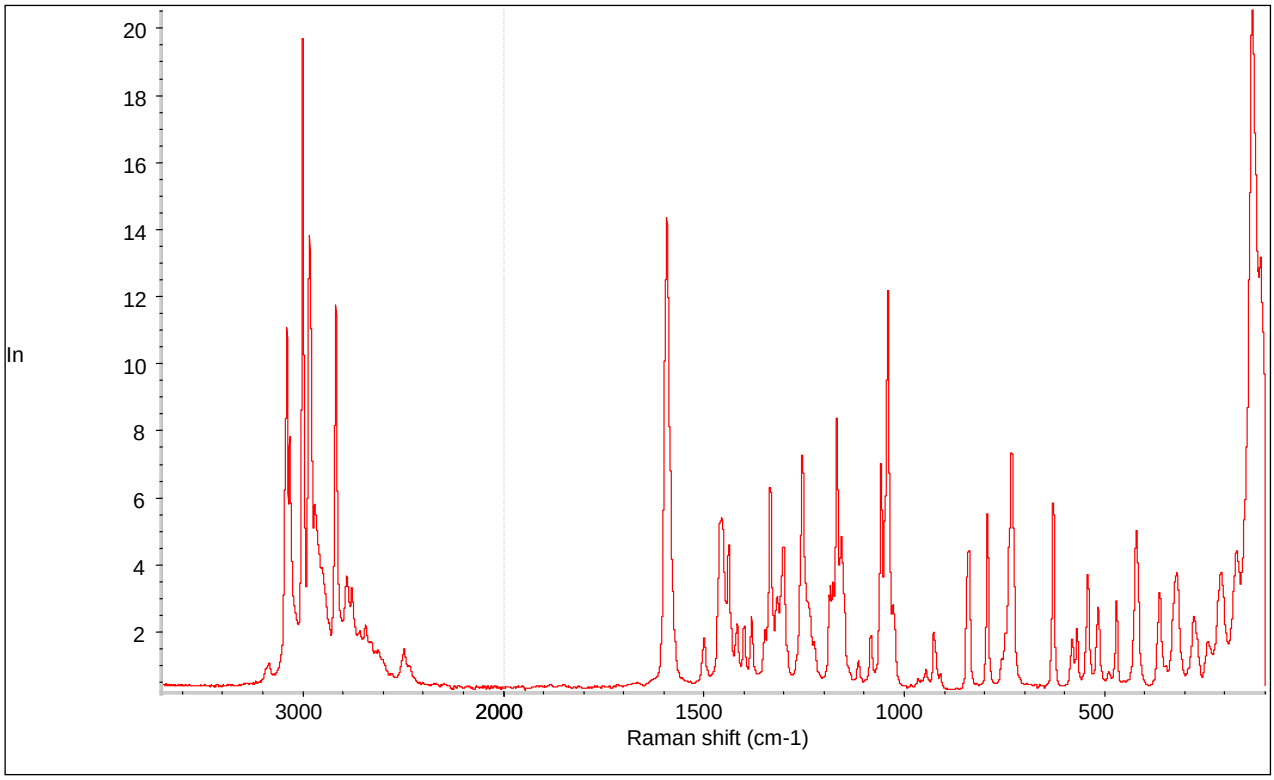
FTIR (ATR): 1-(2-Methoxyphenyl)piperazine HCl
16 scans; 4 cm^{-1} resolution



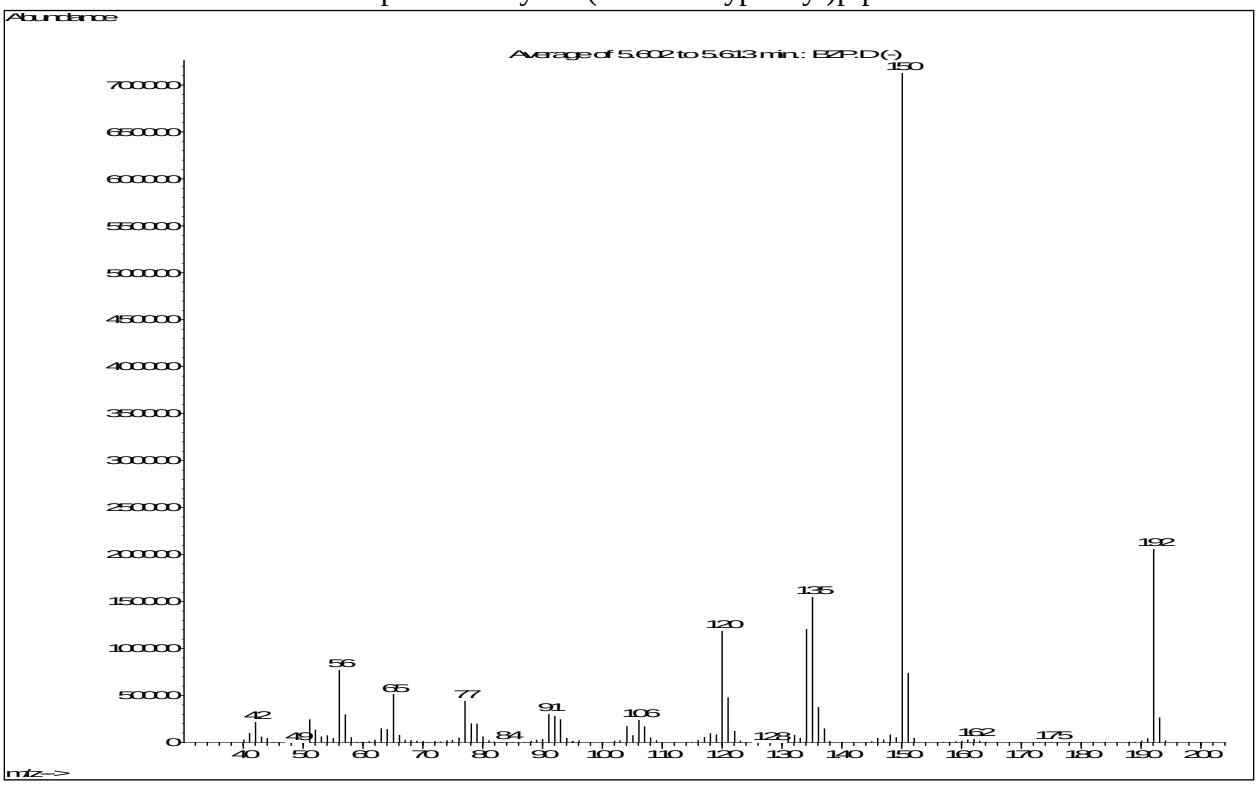
FT RAMAN: 1-(2-Methoxyphenyl)piperazine base
256 scans; 4.0 nm resolution



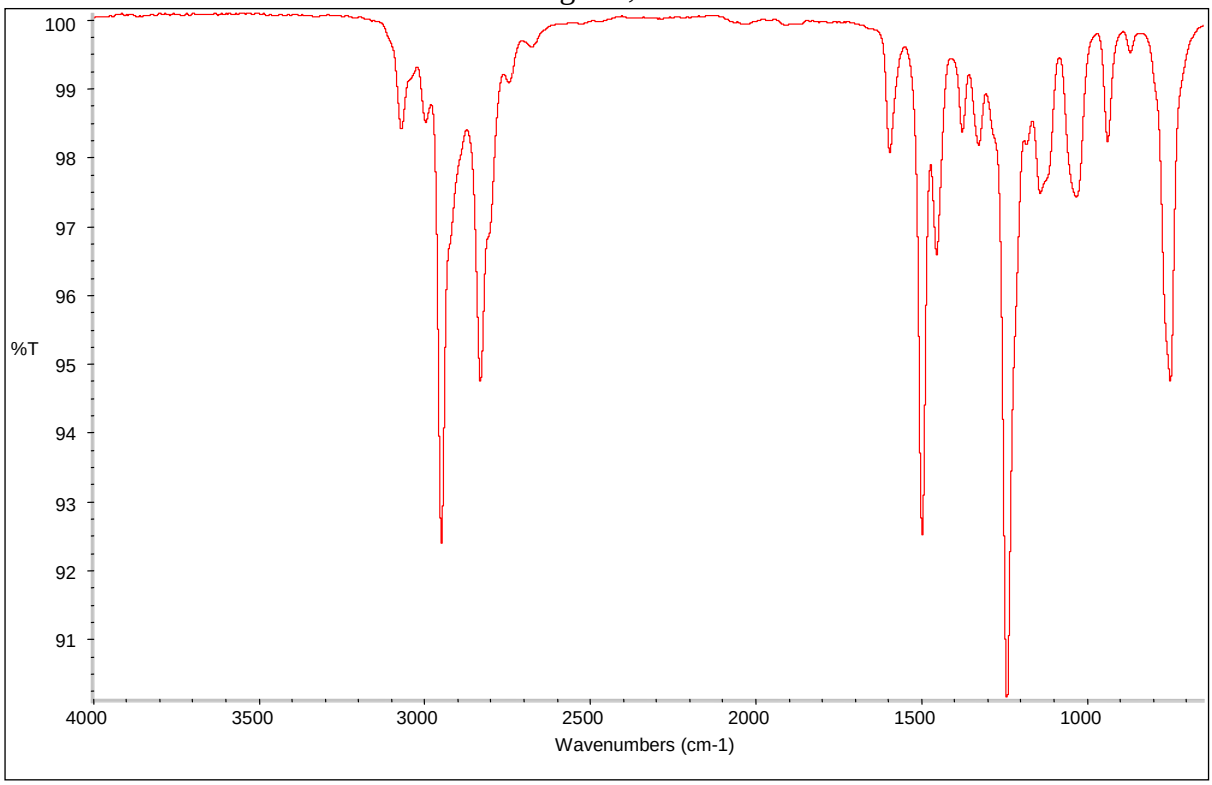
FT RAMAN: 1-(2-Methoxyphenyl)piperazine HCl
256 scans; 4.0 nm resolution



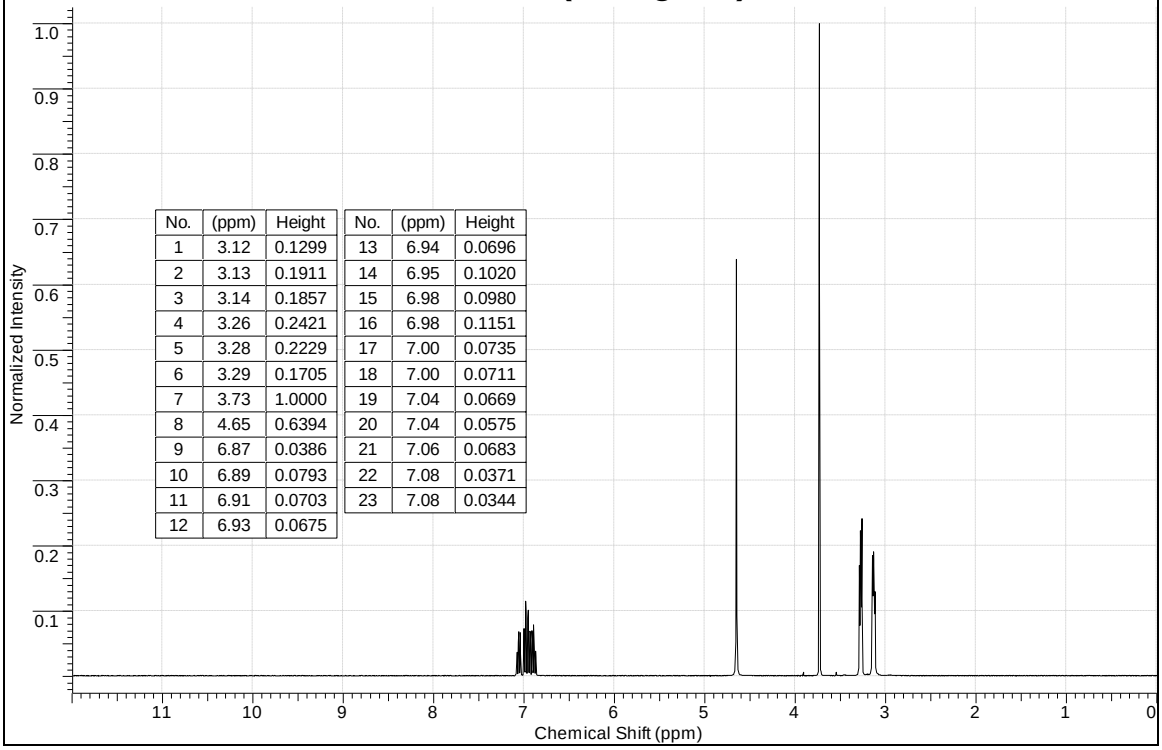
Mass Spectrometry: 1-(2-Methoxyphenyl)piperazine



Vapor Phase IR: 1-(2-Methoxyphenyl)piperazine
0.5mg/mL, 8cm^{-1}

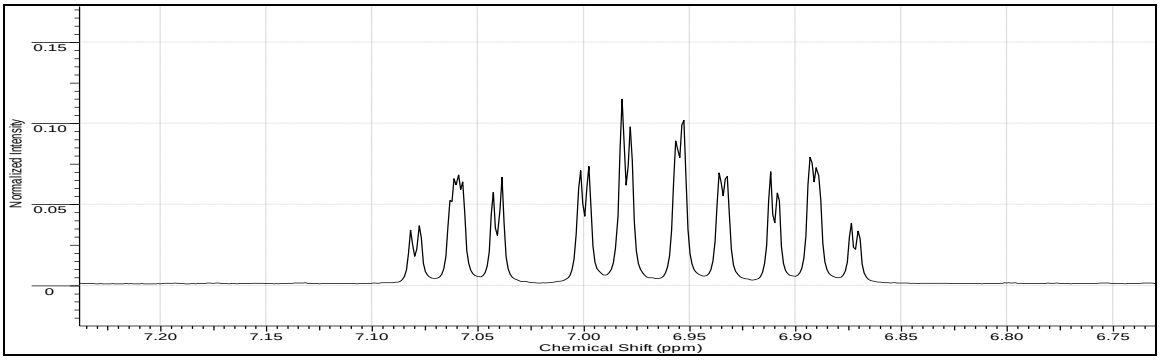


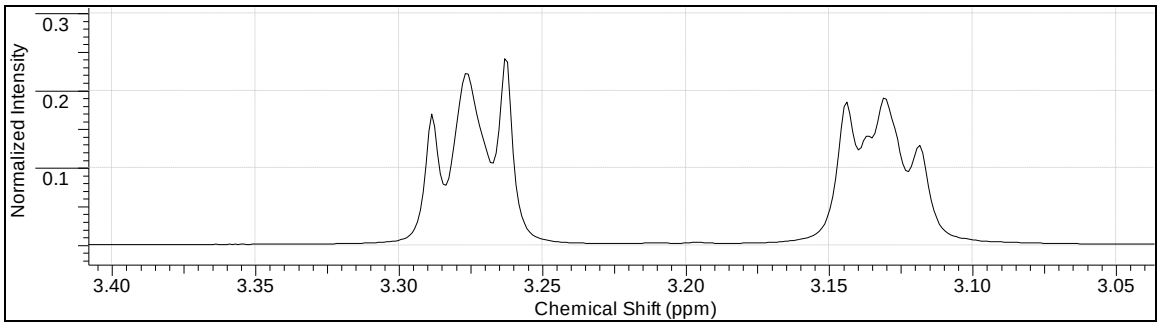
FT-NMR 400 MHz Proton
1-(2-Methoxyphenyl)-piperazine HCl
in D2O (60 mg/mL)

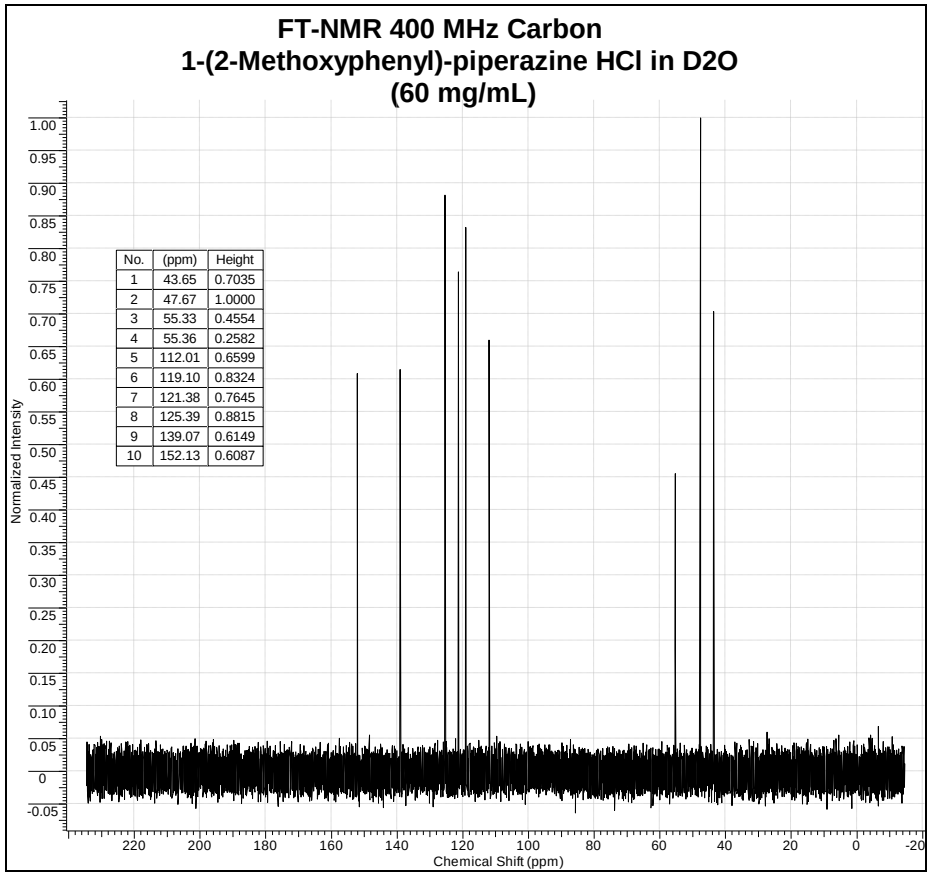


No.	(ppm)	Height
1	3.12	0.1299
2	3.13	0.1911
3	3.14	0.1857
4	3.26	0.2421
5	3.28	0.2229
6	3.29	0.1705
7	3.73	1.0000
8	4.65	0.6394
9	6.87	0.0386
10	6.89	0.0793
11	6.91	0.0703
12	6.93	0.0675

No.	(ppm)	Height
13	6.94	0.0696
14	6.95	0.1020
15	6.98	0.0980
16	6.98	0.1151
17	7.00	0.0735
18	7.00	0.0711
19	7.04	0.0669
20	7.04	0.0575
21	7.06	0.0683
22	7.08	0.0371
23	7.08	0.0344






FT-NMR 400 MHz Carbon
1-(2-Methoxyphenyl)-piperazine HCl in D2O
(60 mg/mL)
Normalized Intensity
Chemical Shift (ppm)
No. (ppm) Height
1 43.65 0.7035
2 47.67 1.0000
3 55.33 0.4554
4 55.36 0.2582
5 112.01 0.6599
6 119.10 0.8324
7 121.38 0.7645
8 125.39 0.8815
9 139.07 0.6149
10 152.13 0.6087