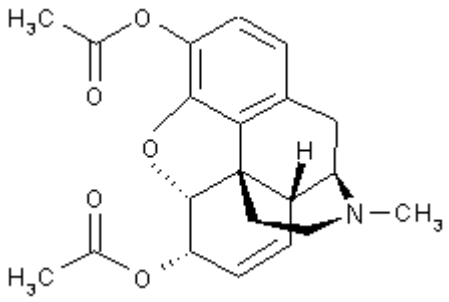
HEROIN

Latest Revision: March 18, 2005



1. SYNONYMS

CFR: Heroin

CAS #: Base: 561-27-3
Hydrochloride: 1502-95-0 (anhydrous)

Other Names: 3,6-Diacetyl-7,8-didehydro-4,5-epoxy-17-methyl-5-α,6-α-morphinan-3,6-diol 7,8-Didehydro-4,5-epoxy-17-methyl-5-α,6-α-morphinan-3,6-diol diacetate ester (5-α,6-α)-7,8-Didehydro-4,5-epoxy-17-methylmorphinan-3,6-diol diacetate ester

Diamorphine
Acetomorphine
Diacetylmorphine

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	$C_{21}H_{23}NO_5$	369.4	173
Hydrochloride monohydrate	$C_{21}H_{24}NO_5Cl{\cdot}H_2O$	423.9	243-244

2.2. SOLUBILITY

Form	A	C	E	H	M	W
Base	S	FS	SS	SS	PS	I
Hydrochloride	PS	FS	I	I	VS	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	Purple violet
Mecke	Dark green
Froehde	Purple

3.2. CRYSTAL TESTS

REAGENT	COLOR PRODUCED
Mercuric iodide	Clusters of needles and threads - also blades in clusters and dendrites
Platinic chloride	Small dark needles formed into a burr
Mercuric chloride	Fine dendrites

3.3. THIN LAYER CHROMATOGRAPHY

Visualization

UV light	Fluorescent quenching
Dragendorff reagent	Orange spots on yellow background
Acidified potassium iodoplatinate	Blue to purple spots

COMPOUND	RELATIVE R_1		
	System TLC3	System TLC4	System TLC5
morphine	0.3	0.3	0.8
codeine	0.7	0.6	0.5

heroin	**1.0**	**1.0**	**1.0**
papaverine	1.3	1.2	1.1
noscapine	1.5	1.4	1.1

3.4. GAS CHROMATOGRAPHY

Method HER- GCS1

Internal Stock Solution:
0.05 mg/mL tetracosane in chloroform:methanol (4:1)

Standard Solution Preparation:
Accurately weigh and prepare standard solutions at approximately 0.05 mg/mL using above internal standard stock solution.

Sample Preparation:
Weigh approximately 20 mg into a GC vial (~2 mL). Fill with internal standard stock solution. If necessary, filter sample through glass wool.

Instrument:	Agilent 6890 Series II (or comparable) gas chromatograph operated in split mode equipped with FID
Column:	5% phenyl/95% methyl silicone 12.0 m x 0.2 mm x 0.33 µm
Carrier gas:	Helium at 1.0 mL/min for 5 min ramped flow to 2.0 mL/min
Temperatures:	Injector: 270°C Detector: 280°C Oven program: 1) 175°C initial temperature for 1.0 min 2) Ramp to 280°C at 15°C/min 3) Hold final temperature for 4.0 min
Injection Parameters:	Split Ratio = 60:1, 1 µL injection
Typical Retention Time:	Heroin: 9.57 min Tetracosane: 6.01 min

COMPOUND	RRT	COMPOUND	RRT
dimethyl Sulfone	0.08	methadone HCl	0.71
amphetamine Sulfate	0.10	propoxyphene HCl	0.73
methamphetamine	0.11	atropine sulfate	0.74
N,N-dimethylamphetamine	0.13	cocaine HCL	0.74
phenylpropanolamine HCl	0.15	tetracaine HCl	0.75

niacinamide	0.17	triprolidine	0.77
methylephedrine	0.19	tetracosane	0.81
MDA HCl	0.22	phenylbutazone	0.84
MDMA HCl	0.24	codeine phosphate	0.85
benzocaine	0.27	morphine sulfate	0.88
MDEA	0.27	diazepam	0.88
guaifenesin	0.32	hydrocodone bitartrate	0.89
acetaminophen	0.34	acetylcodeine	0.92
phenacetin	0.35	monoacetylmorphine	0.93
caffeine	0.47	oxycodone base	0.93
ketamine HCl	0.49	benzoylecgonine tartrate	0.97
diphenhydramine HCl	0.50	chloroquine Phosphate	0.97
antipyrine	0.50	**heroin HCl**	**1.00**
lidocaine HCl	0.51	quinine base	1.10
doxylamine succinate	0.54	quinine HCl	1.10
aminopyrine	0.54	quinidine HCl	1.10
phenobarbital	0.57	zolpidem	1.10
xylazine	0.59	papaverine	1.12
levamisole	0.59	clonazepam	1.14
dipyrone	0.61	hydroxyzine	1.14
procaine HCl	0.63	alprazolam	1.21
clenbuterol HCl	0.65	diltiazem	1.22
brompheniramine	0.69	noscapine	1.44

4.0. SALT FORM DETERMINATION

Heroin is most frequently encountered either as the free base or as the hydrochloride salt. It is also not uncommon for the bulk of a heroin sample to be the hydrochloride salt while a few percent is the free base. Two other heroin salt forms, which are encountered less frequently, are the tartrate and the citrate. On rare occasions, a heroin sample will be a combination of the hydrochloride, tartrate, and citrate salts, along with a small quantity of heroin base.

To identify the salt forms, each can be separated based on their different solvent solubilities. Once separated through a series of rinses, anion testing can be done where results from selected reactions are determined by the presence or absence of a precipitate.

Base: Heroin base is soluble in anhydrous ether; whereas, salts of heroin are completely insoluble.

Hydrochloride: Heroin hydrochloride is soluble in chloroform and methylene chloride. Heroin tartrate, heroin citrate, and most inorganic chlorides are insoluble in these solvents. Therefore, dissolve the sample in chloroform or methylene chloride, filter, and then take to dryness. Redissolve the evaporated material in water and treat with silver nitrate solution. A white precipitate will form. After washing the precipitate with water, it should be insoluble in concentrated nitric acid but should be soluble in dilute ammonia solution. From the ammonia solution, it can be reprecipitated by the addition of nitric acid.

Tartrate: Heroin tartrate is insoluble in methylene chloride and chloroform, but is soluble in methanol. Therefore, dissolve the sample in methanol, filter, and then take to dryness. Redissolve the evaporated material in water and treat with silver nitrate solution. The silver nitrate will produce a white precipitate due to free tartaric acid or a tartrate salt. This precipitate is soluble in nitric acid. Congo red will produce a negative result with tartrate salts, but forms a blue color when the free acid is present.

Citrate: Heroin citrate is insoluble in methylene chloride and chloroform, but is soluble in methanol. Therefore, dissolve the sample in methanol, filter, and then take to dryness. Redissolve the evaporated material in water and treat with silver nitrate solution. The silver nitrate will produce a white precipitate due to free citric acid or a citrate salt. This precipitate is soluble in nitric acid. Acetic anhydride can also be used to test for citrates and free citric acid. The test involves adding 0.5 mL acetic anhydride to a small amount of sample in a test tube and heating the tube at 80^{o}C for 10 minutes. A purple color will develop if a citrate salt or free citric acid is present along with a tertiary amine such as heroin.

5. QUANTITATIVE PROCEDURES

5.1. GAS CHROMATOGRAPHY

Method HER-GCQ-1

Internal Standard Stock Solution:
0.5 mg/mL tetracosane in methylene chloride.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of heroin hydrochloride at approximately 0.5 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:	Agilent 6890 Series II (or comparable) gas chromatograph operated in split mode equipped with FID
Column:	100% dimethylpolysiloxane 30 m x 0.25 mm x 0.25 µm film thickness
Carrier gas:	Hydrogen at 1.2 mL/min
Temperatures:	Injector: 265°C Detector: 285°C Oven program: 1) 205°C initial temperature for 1.0 min 2) Ramp to 280°C at 12°C/min 3) Hold final temperature for 7.0 min
Injection Parameters:	Split Ratio = 60:1, 1 µL injection

Typical Retention Time: Heroin: 8.60 min
Tetracosane: 6.65 min

Linear Range: 0.02 - 4.0 mg/mL

Repeatability: RSD less than 2.0%

Correlation Coefficient: 0.999

Accuracy: Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
salicylic acid	0.18	Phenobarbital	0.46
aspirin	0.18	Tropacaine	0.47
thiamine*	0.19	Chlorpheniramine	0.48
phenylpropanolamine	0.19	Methocarbamol	0.49
nicotinamide	0.20	Methapyrilene	0.49
ephedrine	0.20	dipyrone*	0.50
thiamine*	0.20	Procaine	0.51
pseudoephedrine	0.21	Dextromethorphan	0.60
methylparaben	0.22	Methadone	0.62
salicylamide	0.22	methaqualone	0.62
dimethylphthalate	0.22	cocaine	0.65
dimethylterephthalate	0.22	amitryptyline	0.66
barbital	0.23	*c*-doxepin	0.66
diethylpropion	0.23	*t*-doxepin	0.68
benzocaine	0.26	dipyrone, acetylated*	0.75
ibuprofen	0.26	acetylprocaine	0.77
acetaminophen	0.27	phenylbutazone	0.79
phenacetin	0.29	codeine	0.81
acetylated acetaminophen	0.30	ethylmorphine	0.84

amobarbital	0.31	diazepam	0.85
butalbital	0.32	morphine	0.85
meconin	0.32	O^3-monoacetylmorphine	0.89
pentobarbital	0.32	chlorpromazine	0.89
methylphenidate	0.33	acetylcodeine	0.90
meperidine	0.34	O^6-monoacetylmorphine	0.91
secobarbital	0.35	acetylthebaine	0.94
caffeine	0.36	methandriol	0.94
glutethimide	0.40	chloroquine	1.00
carisoprodol	0.40	**heroin**	**1.00 (8.60 min)**
antipyrine	0.40	methandriol acetate	1.06
diphenhydramine	0.40	fentanyl	1.08
lidocaine	0.42	quinine	1.17
hydrocotarnine	0.42	papaverine	1.18
doxylamine	0.43	phenolphthalein	1.54
aminopyrine	0.44	strychnine	1.67
theophylline	0.45	noscapine	1.69

* thermal decomposition products

Method HER- GCQ-2

Internal Standard Stock Solution (ISSS):
4.8 mg/mL *n*-docosane in chloroform.

Standard Solution Preparation:
Prepare a standard solution of heroin hydrochloride at 1.7 mg/mL with an ISSS concentration of 0.96 mg/mL (2 mL of ISSS per 10 mL total volume).

Sample Preparation:
Accurately weigh an amount of sample into an appropriately sized volumetric flask so that the final heroin concentration is approximately equivalent to that of the standard solution. Dissolve in a minimal amount of methanol, add appropriate amount of ISSS (2 mL into 10 mL ratio) and dilute to volume with chloroform.

Instrument: Agilent 6890 Series II (or comparable) gas chromatograph operated in

split mode equipped with FID

Column: 5% Phenyl/95% Methyl silicone gum 12.5 m x 0.2 mm x 0.33 µm film thickness

Carrier gas: Helium (ramp flow)

Temperatures: Injector: 270°C
Detector: 285°C
Oven program:
1) 260°C initial temperature for 4.75 min
2) Ramp to 290°C at 40°C/min
3) Hold final temperature for 1.0 min
4) Oven equilibration 0.5 min

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

Typical Retention Time: Heroin: 3.93 min
n-docosane: 1.32 min

Linear Range: 0.35 – 3.5 mg/mL

Repeatability: RSD less than 2.0%

Correlation Coefficient: 0.9999

Accuracy: Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
thiamine *	0.240	chlorpheniramine	0.372
salicylic Acid	0.241	procaine	0.377
pseudoephedrine	0.249	**n-docosane**	**0.413**
nicotinamide	0.250	cocaine	0.478
benzocaine	0.266	tetracaine	0.481
guaifenesin	0.277	tetracosane	0.553
acetaminophen	0.279	codeine	0.658
phenacetin	0.281	morphine	0.716
carisoprodol	0.315	acetylcodeine	0.809

caffeine	0.319	O^6-monoacetylmorphine	0.828
diphenhydramine	0.322	chloroquine	0.941
lidocaine	0.328	**heroin**	**1.00**
doxylamine	0.337	quinine	1.15
aminopyrine	0.346	papaverine	1.16
phenobarbital	0.353	noscapine	1.61
theophylline	0.354		

* thermal decomposition product

5.2. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Method HER-LCQ1

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

Sample Preparation:
Accurately weigh an amount of sample into an appropriate volumetric flask and dilute so the final heroin concentration is approximately that of the standard concentration. If necessary, filter the sample with a 0.45 µm filter prior to injection.

Instrument:	Agilent 1100 Series (or comparable) high performance liquid chromatograph equipped with diode array detector
Column:	5 µm Phenomenex Luna, 150 mm x 3.2 mm at 35°C
Detector:	UV, 210 nm
Flow:	0.76 mL/min
Injection Volume:	5 µL
Buffer:	3480 mL water 40.0 mL phosphoric acid 150 mL of 2 N sodium hydroxide pH between 2.2 to 2.3 using phosphoric acid or NaOH filter the buffer, add 12.7 mL hexylamine, cover and mix
Mobile Phase:	1) Equilibrate buffer: methanol 95:5 for 15 min 2) Gradient to buffer: methanol 70:30 over 20 min 3) Maintain buffer: methanol 70:30 for 6 min

4) Gradient to buffer: methanol 20:80 over 10 min
5) Maintain buffer: methanol 20:80 for 4 min
6) Gradient to buffer: methanol 5:95 over 5 min

Typical Retention Time: Heroin: 17.247 min

Linear Range: 0.05 mg/mL – 2.0 mg/mL

Repeatability: RSD less than 0.5%

Correlation Coefficient: 0.9999

Accuracy: Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
thiamine	0.051	theophylline	0.603
nicotinamide	0.075	caffeine	0.849
morphine	0.140	cocaine	0.887
ephedrine	0.158	acetylcodeine	0.944
pseudoephedrine	0.174	**heroin**	**1.000 (17.247min)**
acetaminophen	0.177	aspirin	1.120
procaine	0.203	noscapine	1.322
codeine	0.335	phenacetin	1.331
lidocaine	0.337	papaverine	1.479
O^6-monoacetylmorphine	0.406	diphenhydramine	1.581
chloroquine	0.507	propiophenone	1.836
quinine	0.571		

5.3. CAPILLARY ELECTROPHORESIS

Method HER-CEQ1

Internal Standard Stock Solution:
0.1 mg/mL naphazoline in 100 mM sodium phosphate at pH of 4.5.

Standard Solution Preparation:
Accurately weigh and prepare a standard solution of heroin (hydrochloride or base) at approximately 0.3 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. Dilute the sample so the final concentration approximates the standard concentration. If necessary, filter the sample with a 0.45 µm filter prior to injection if necessary.

Mode:	Free zone
Column:	37 cm x 50 µm fused silica capillary
Run Buffer:	200 mM sodium phosphate buffer, pH 4.5
Detector:	UV, 210 nm
Voltage:	20 kV
Temperature:	30°C liquid cooled
Injection:	2 s hydrodynamic
Run Time:	5 min
Rinse Time:	1 min
Typical Retention Time:	Heroin: 3.92 min Naphazoline: 2.80 min
Linear Range:	0.025 - 0.6 mg/mL
Repeatability:	RSD less than 2.0%
Correlation Coefficient:	0.999
Accuracy:	Error less than 5%

COMPOUND	RRT	COMPOUND	RRT
amphetamine	0.64	*t*-cinnamoylcocaine	0.90
methamphetamine	0.67	codeine	0.91
naphazoline	0.72	morphine	0.93

phenylpropanolamine	0.72	acetylcodeine	0.96
pseudoephedrine	0.73	O^6-monoacetylmorphine	0.97
ephedrine	0.74	papaverine	0.98
diphenhydramine	0.75	**heroin**	**1.00 (3.92 min)**
procaine	0.77	noscapine	1.02
quinine	0.79	nicotinamide	>2.5
cocaine	0.81	acetaminophen	>2.5
lidocaine	0.84	caffeine	>2.5
thebaine	0.85	benzocaineamine	>2.5
c-cinnamoylcocaine	0.86	phenacetin	>2.5
tetracaine	0.89	benzoylecgonine	>2.5
hydromorphone	0.89	aspirin	>2.5

6. QUALITATIVE DATA

6.1 INFRATED SPECTROSCOPY (FT-IR)

An additional difficulty in comparing the IR spectra of heroin hydrochloride arises from the existence of different crystalline forms or polymorphs, which generate differences in spectra. To overcome this difficulty, both sample and standard should be subjected to the same preparations.

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

6.2. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY – MASS SPECTOMETRY

Sample Preparation:
Dissolve a small amount of sample into 0.1% Formic Acid. Filter sample with 0.45-micron filter if necessary.

Instrument: High performance liquid chromatograph equipped with diode array and mass spectrometer detector (Agilent 1100 Series SL or equivalent).

Column: Synergi MAX-RP, 150 mm x 3.2 mm at 40°C

Detector: UV, 237 nm, 280 nm, and 310 nm
MSD, Positive Mode, 110 V Fragmentor

Ionization Mode: API-ES

	Drying gas - temp 350°C, flow 13.0 L/min 40 psig Nebulizer Pressure Capillary Voltage 4000 V
Flow:	0.50 mL/min
Injection Volume:	2.0 µL
Buffer:	0.1% Formic Acid
Mobile Phase:	Linear Gradient: 95% Buffer:5% MeOH to 50% Buffer:50% MeOH in 20 minutes. Return to 95% Buffer:5% MeOH in next 2 minutes.
Typical Retention Time:	Heroin in 12.9 min

7. REFERENCES

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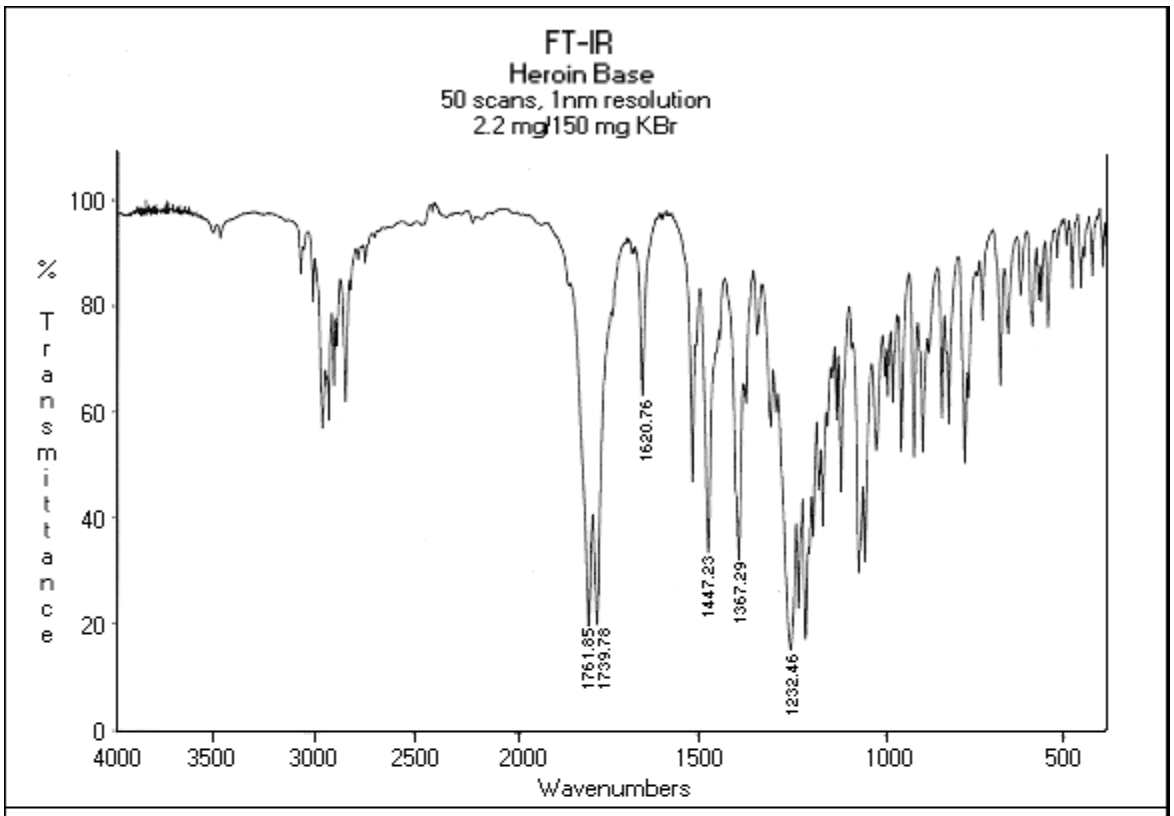
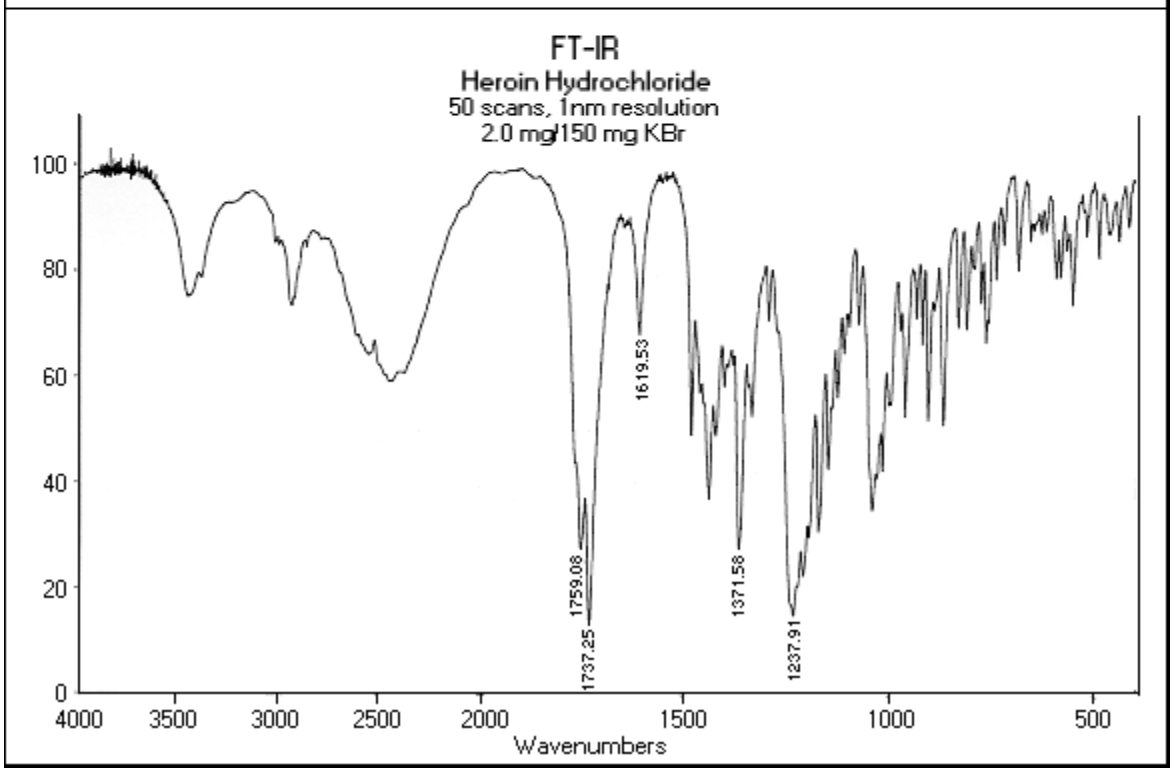
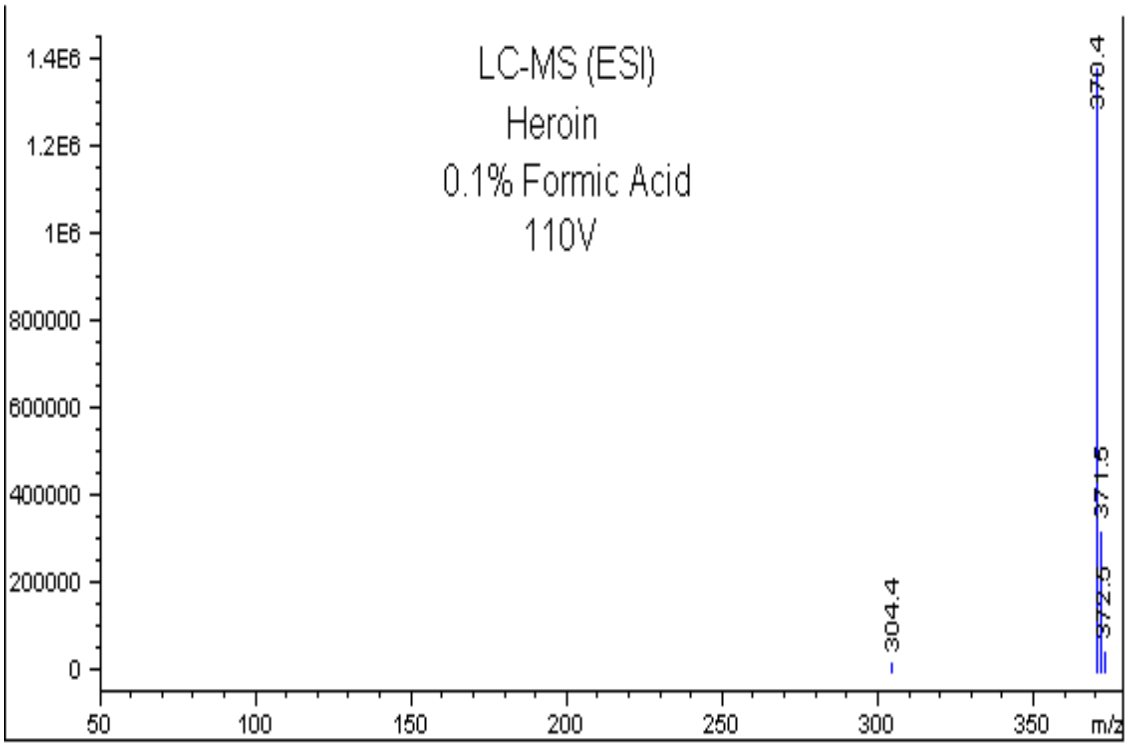
8. ADDITIONAL RESOURCES

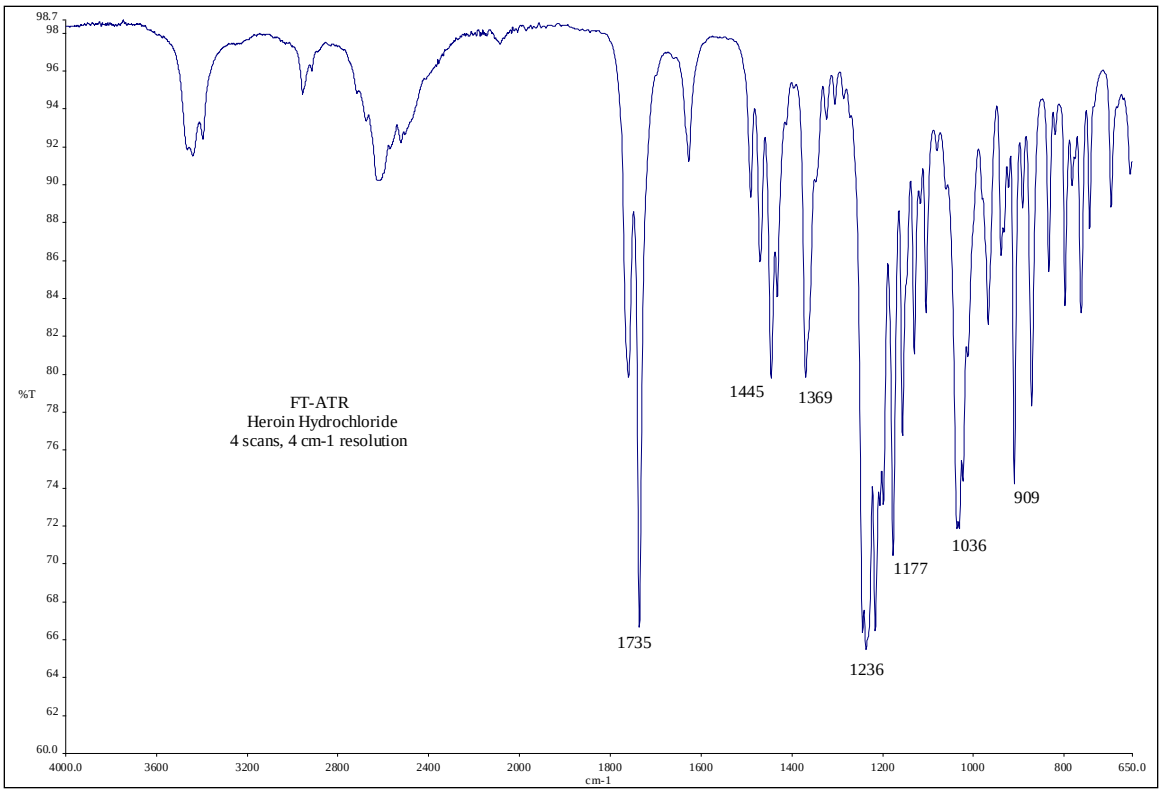
Forendex

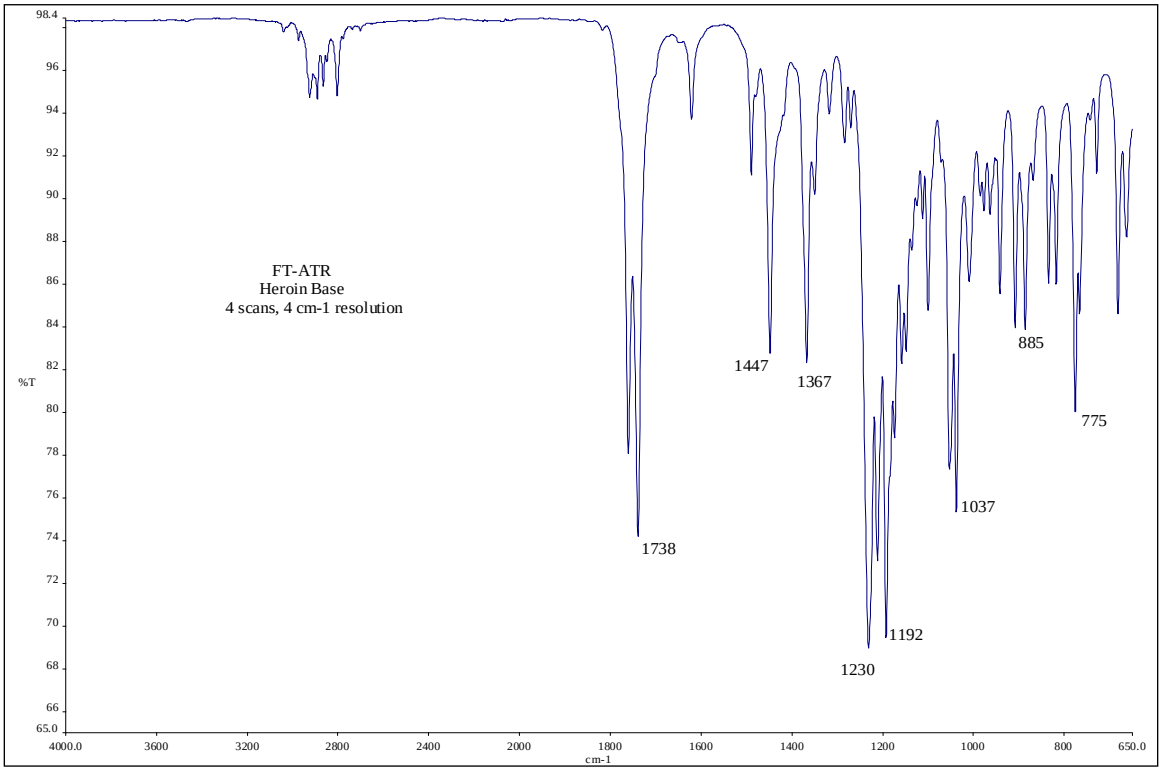
Wikipedia


FT-IR
Heroin Base
50 scans, 1nm resolution
2.2 mg/150 mg KBr
% Transmittance
100
80
60
40
20
0
1761.85
1739.78
1620.76
1447.23
1367.29
1232.46
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers


FT-IR
Heroin Hydrochloride
50 scans, 1nm resolution
2.0 mg/150 mg KBr
100
80
60
40
20
0
1759.08
1737.25
1619.53
1371.58
1237.91
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers


LC-MS (ESI)
Heroin
0.1% Formic Acid
110V
1.4E6
1.2E6
1E6
800000
600000
400000
200000
0
50
100
150
200
250
300
350
m/z
304.4
372.5
371.5
370.4


FT-ATR
Heroin Hydrochloride
4 scans, 4 cm-1 resolution
%T
cm-1
1735
1445
1369
1236
1177
1036
909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Heroin Base
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%T
cm-1
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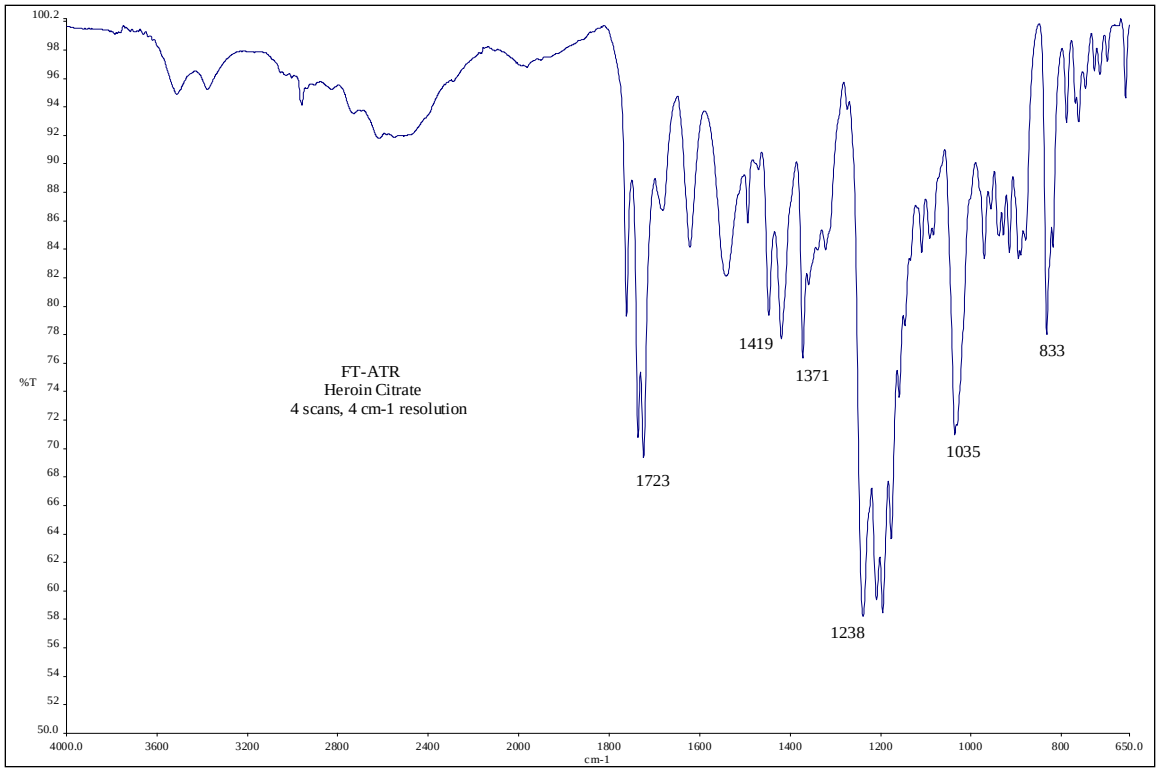
1447
1367
1230
1192
1037
885
775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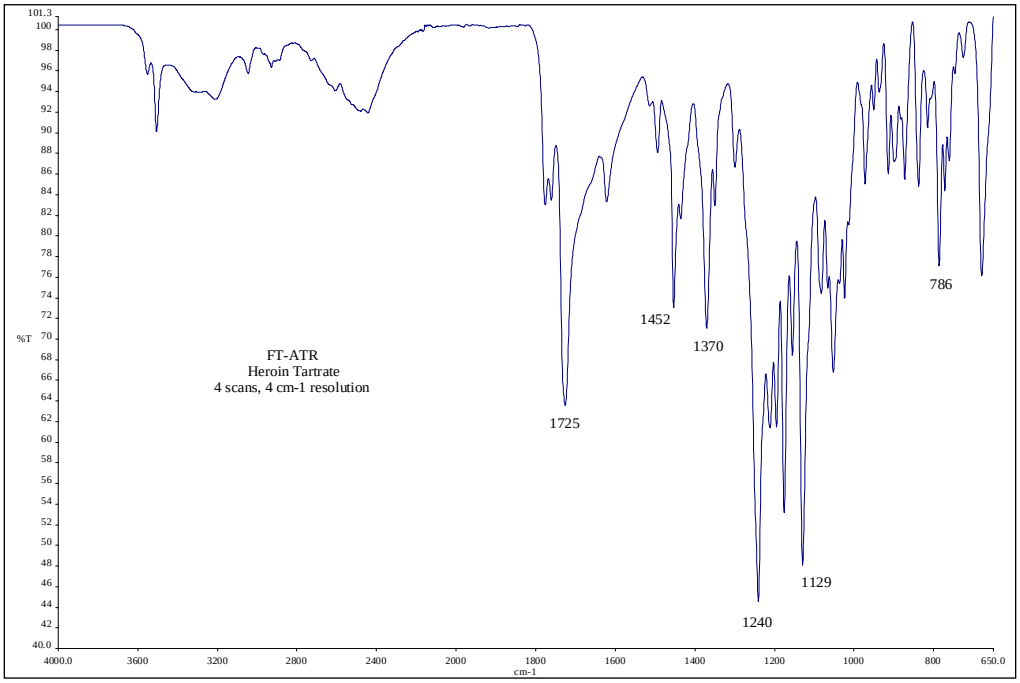
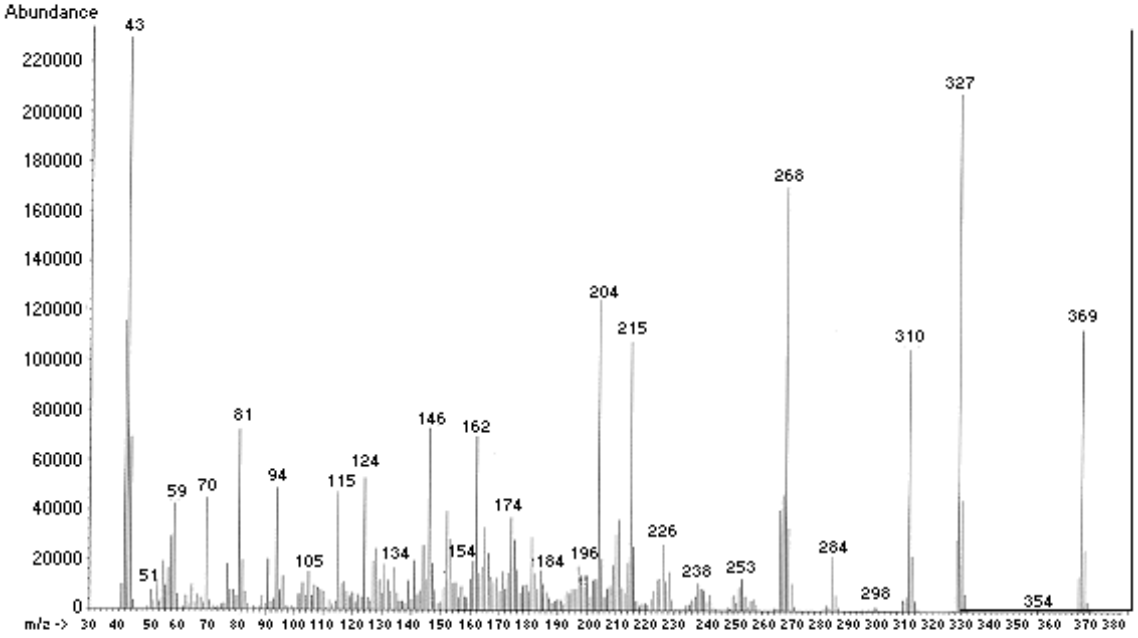
Heroin Citrate
4 scans, 4 cm-1 resolution
%T
cm-1
1723
1419
1371
1238
1035
833

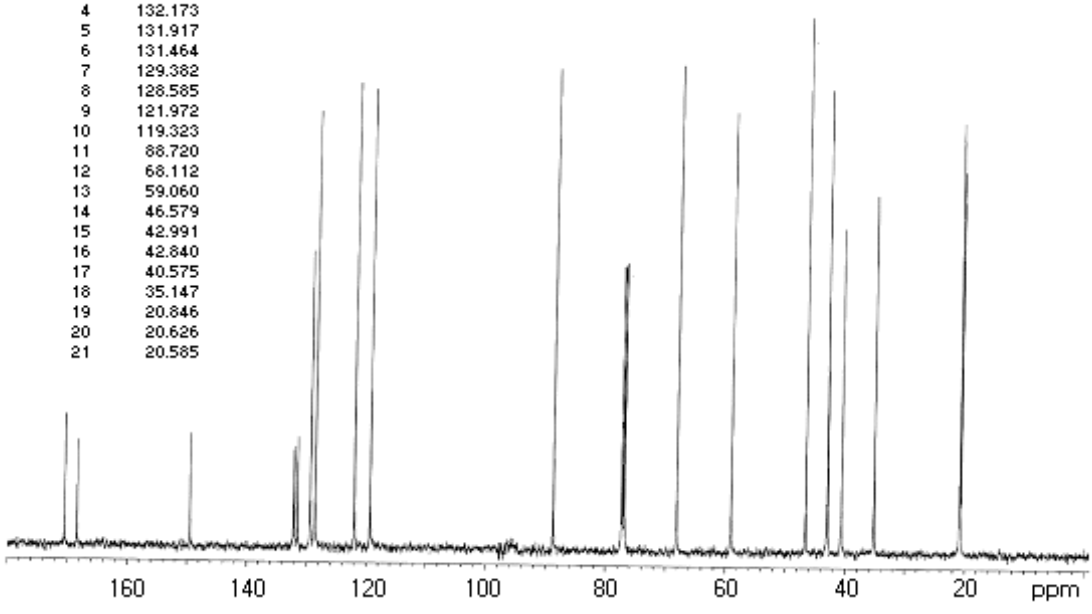

%T
cm-1
FT-ATR
Heroin Tartrate
4 scans, 4 cm-1 resolution
1725
1452
1370
1240
1129
786

Mass Spectrometry
Heroin



Nuclear Magnetic Resonance (carbon)
Heroin Base
50 mg/mL in $CDCL_3$ with TMS
125 MHz

PEAK	PPM
1	170.405
2	168.319
3	149.468
4	132.173
5	131.917
6	131.464
7	129.382
8	128.585
9	121.972
10	119.323
11	88.720
12	68.112
13	59.060
14	46.579
15	42.991
16	42.840
17	40.575
18	35.147
19	20.846
20	20.626
21	20.585

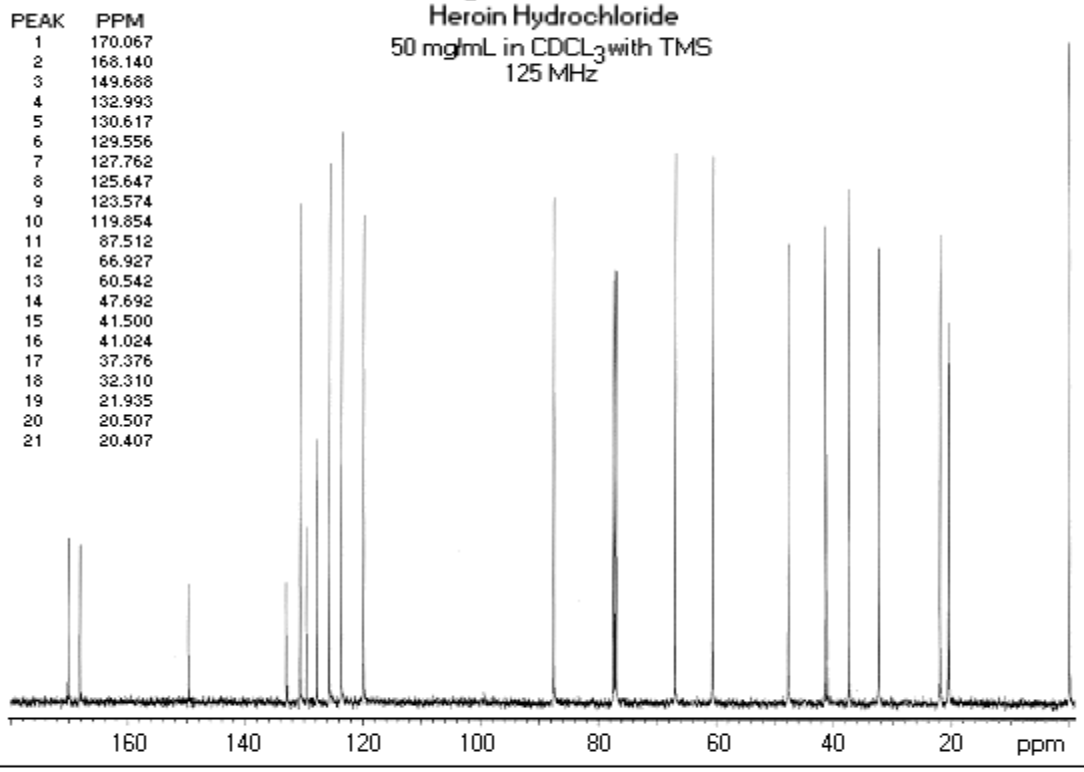


Nuclear Magnetic Resonance (carbon)

Heroin Hydrochloride

50 mg/mL in $CDCL_3$ with TMS
125 MHz

PEAK	PPM
1	170.067
2	168.140
3	149.688
4	132.993
5	130.617
6	129.556
7	127.762
8	125.647
9	123.574
10	119.854
11	87.512
12	66.927
13	60.542
14	47.692
15	41.500
16	41.024
17	37.376
18	32.310
19	21.935
20	20.507
21	20.407

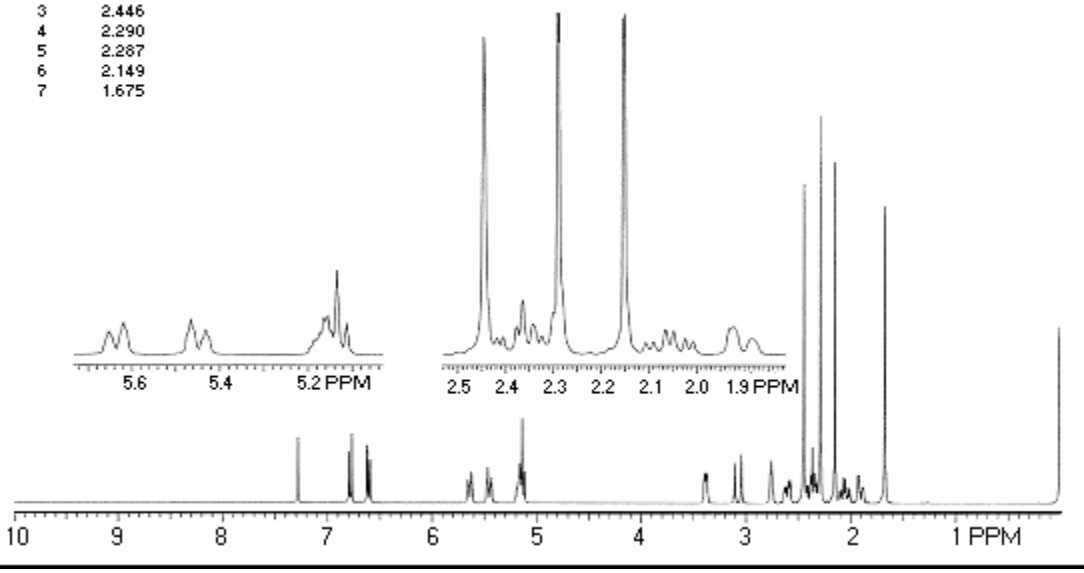


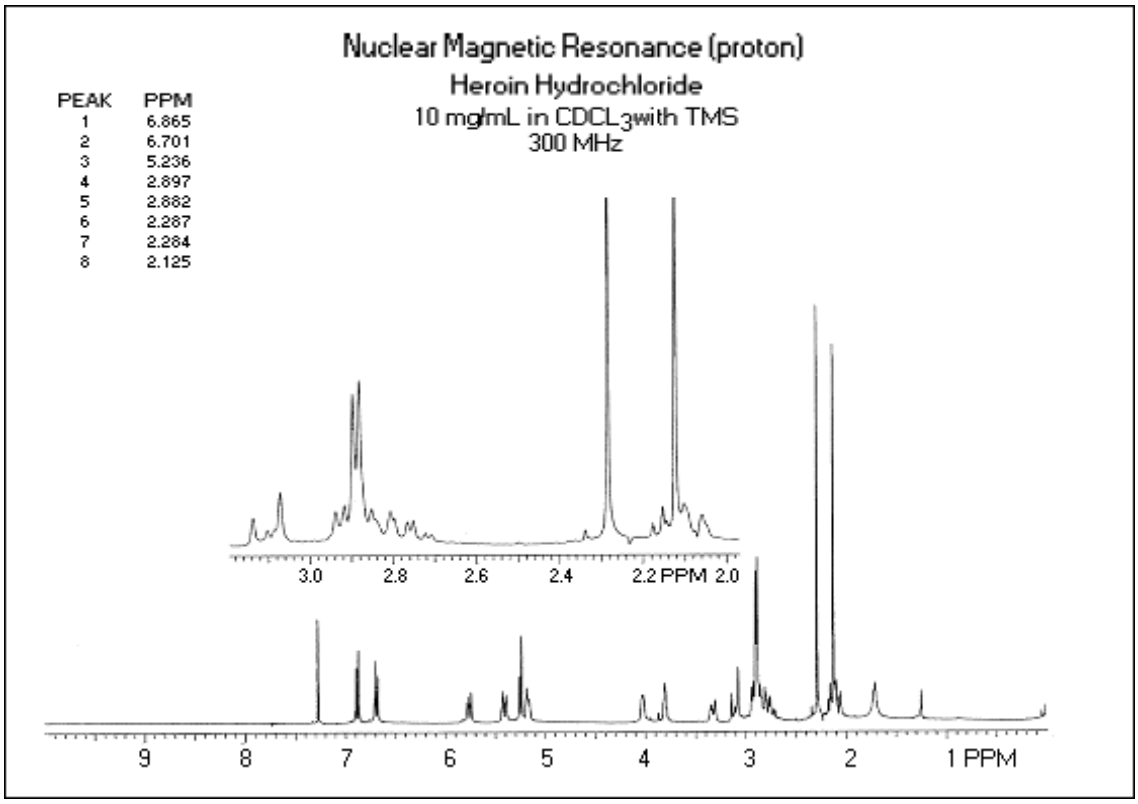
Nuclear Magnetic Resonance (proton)

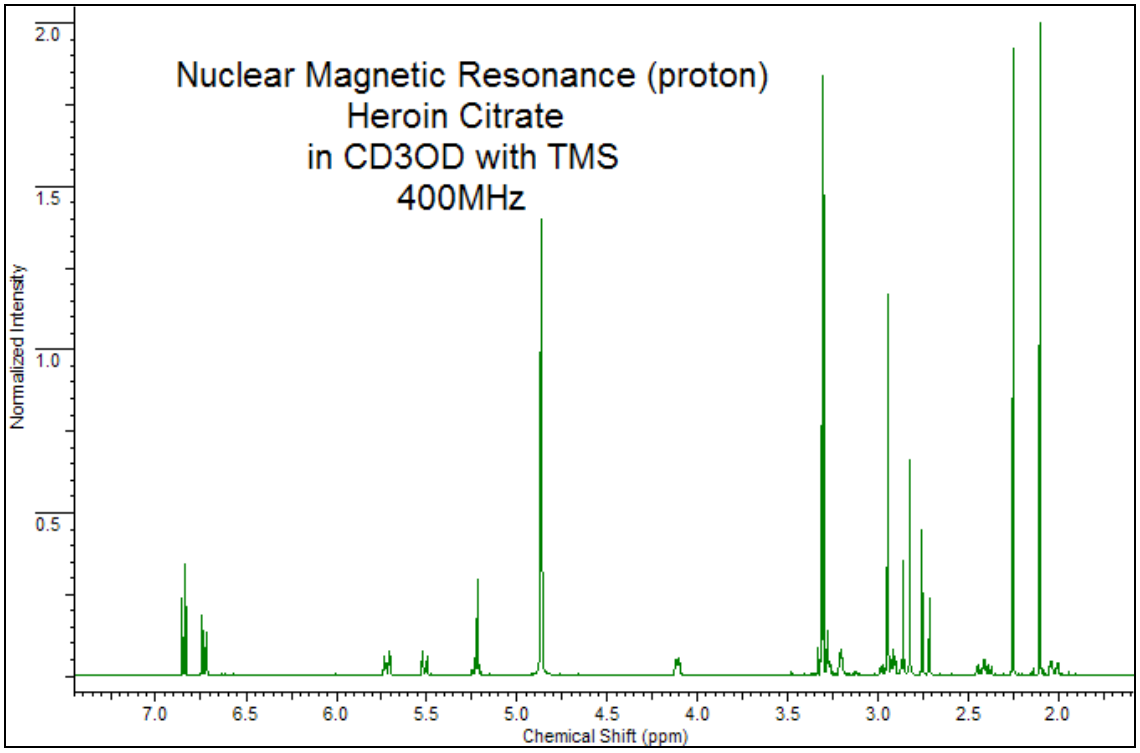
Heroin Base

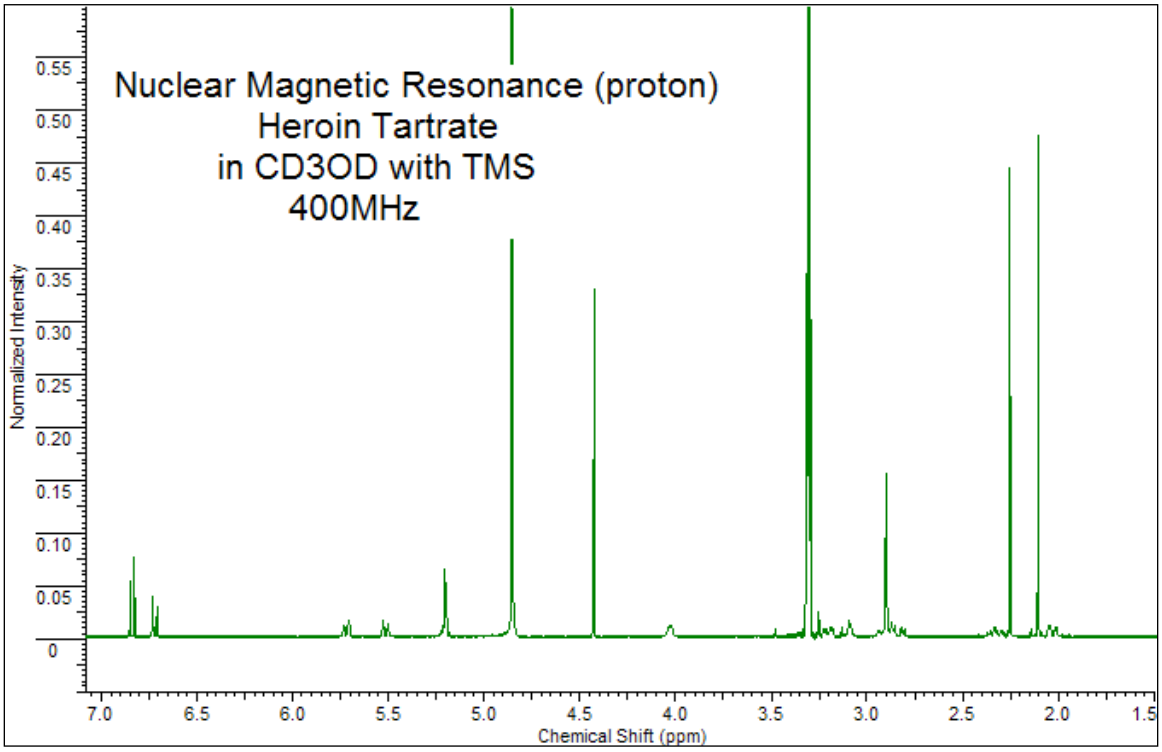
10 mg/mL in $CDCL_3$ with TMS
300 MHz

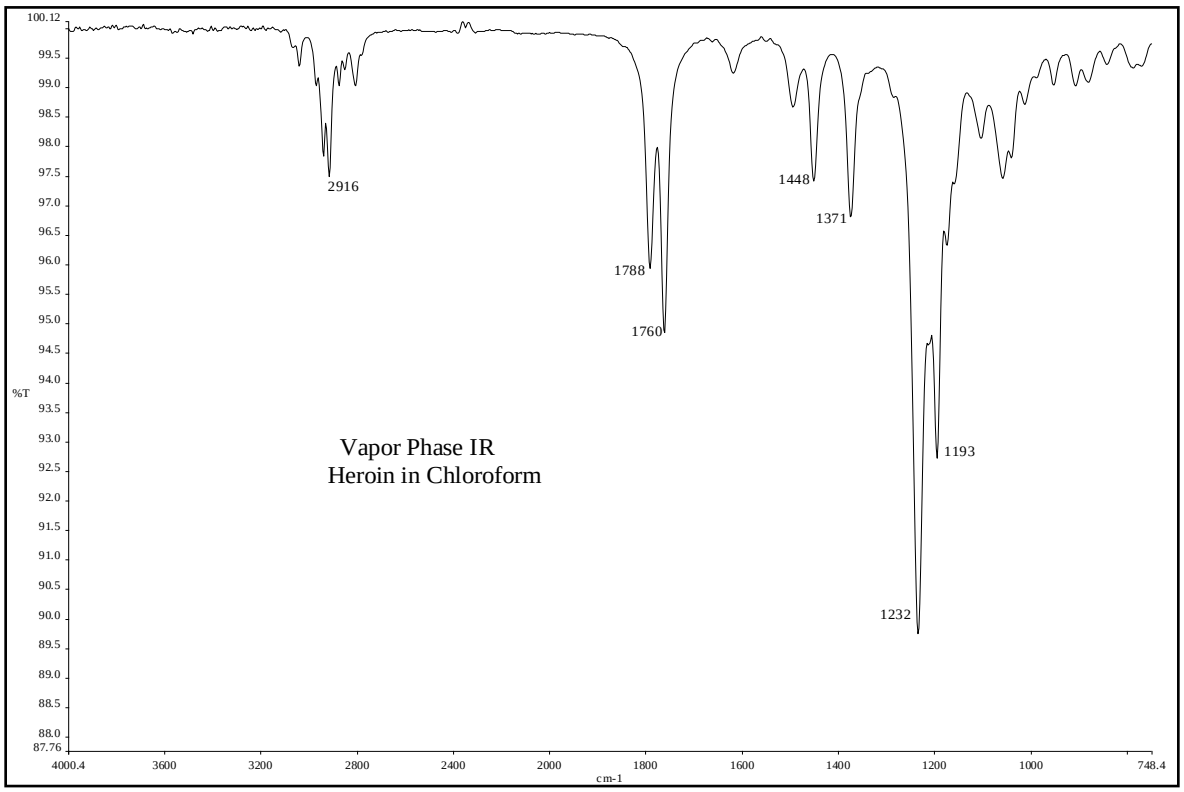
PEAK	PPM
1	6.767
2	5.136
3	2.446
4	2.290
5	2.287
6	2.149
7	1.675

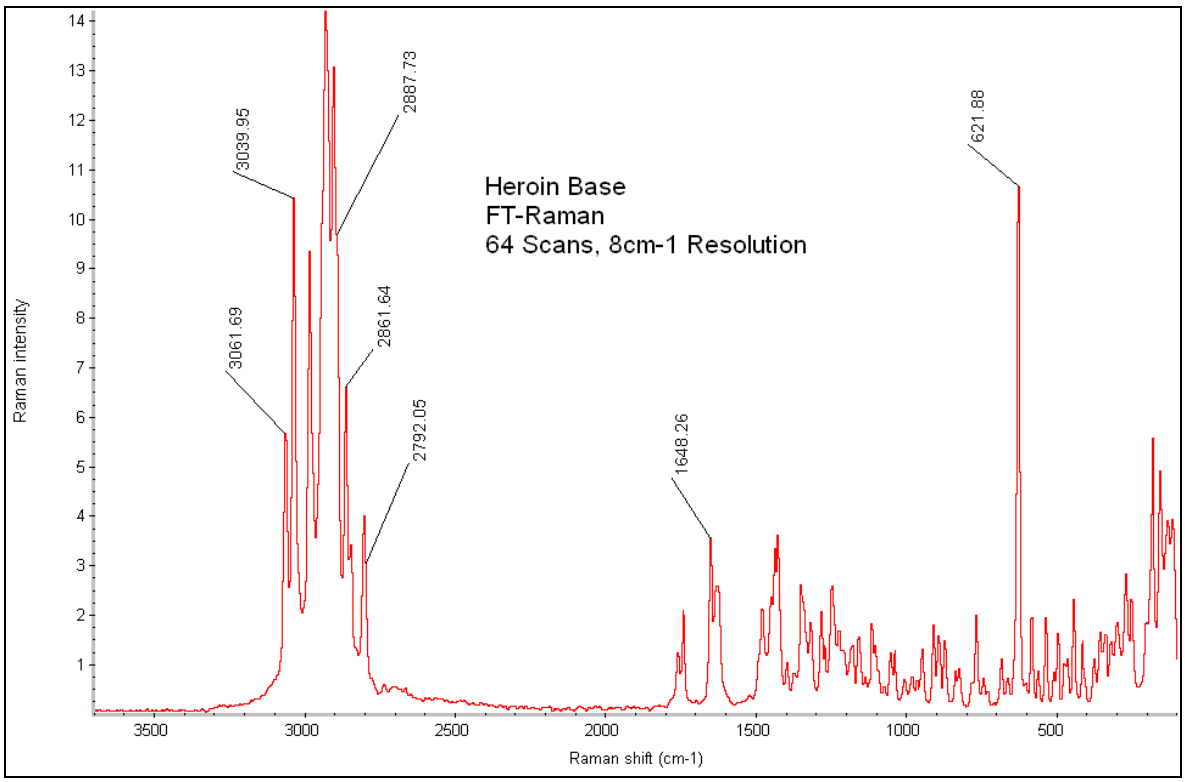


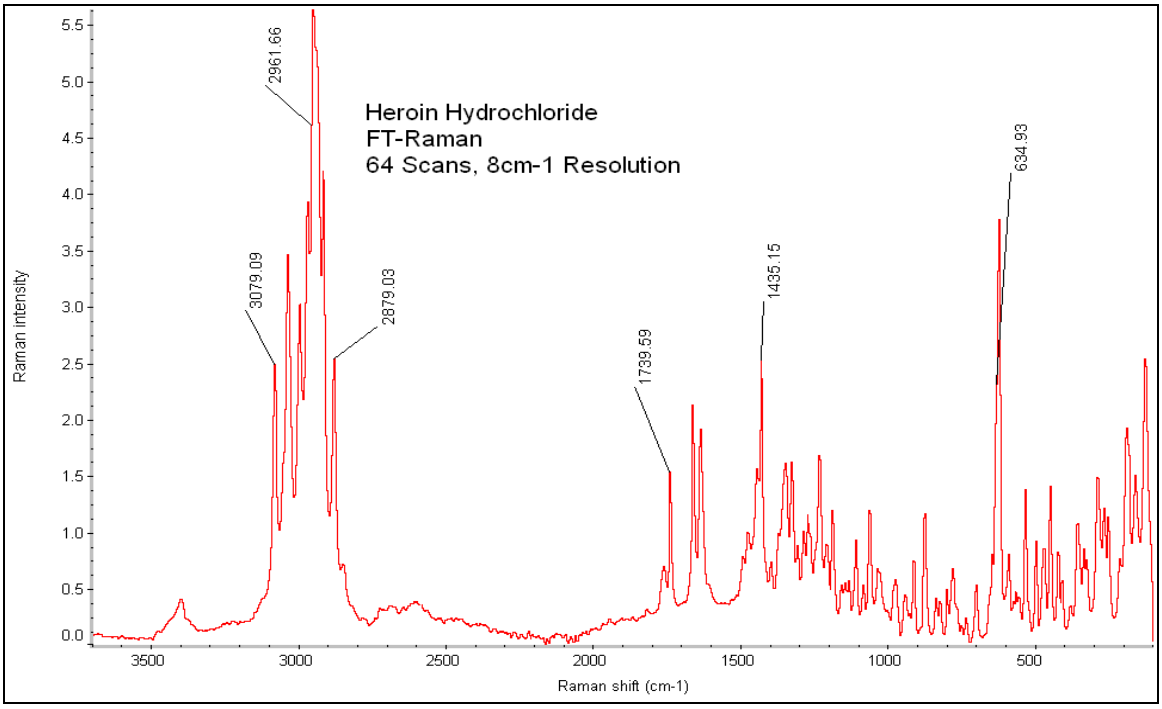

Nuclear Magnetic Resonance (proton)
Heroin Hydrochloride
10 mg/mL in CDCL3with TMS
300 MHz
PEAK PPM
1 6.865
2 6.701
3 5.236
4 2.897
5 2.882
6 2.287
7 2.284
8 2.125
3.0 2.8 2.6 2.4 2.2 PPM 2.0
9 8 7 6 5 4 3 2 1 PPM

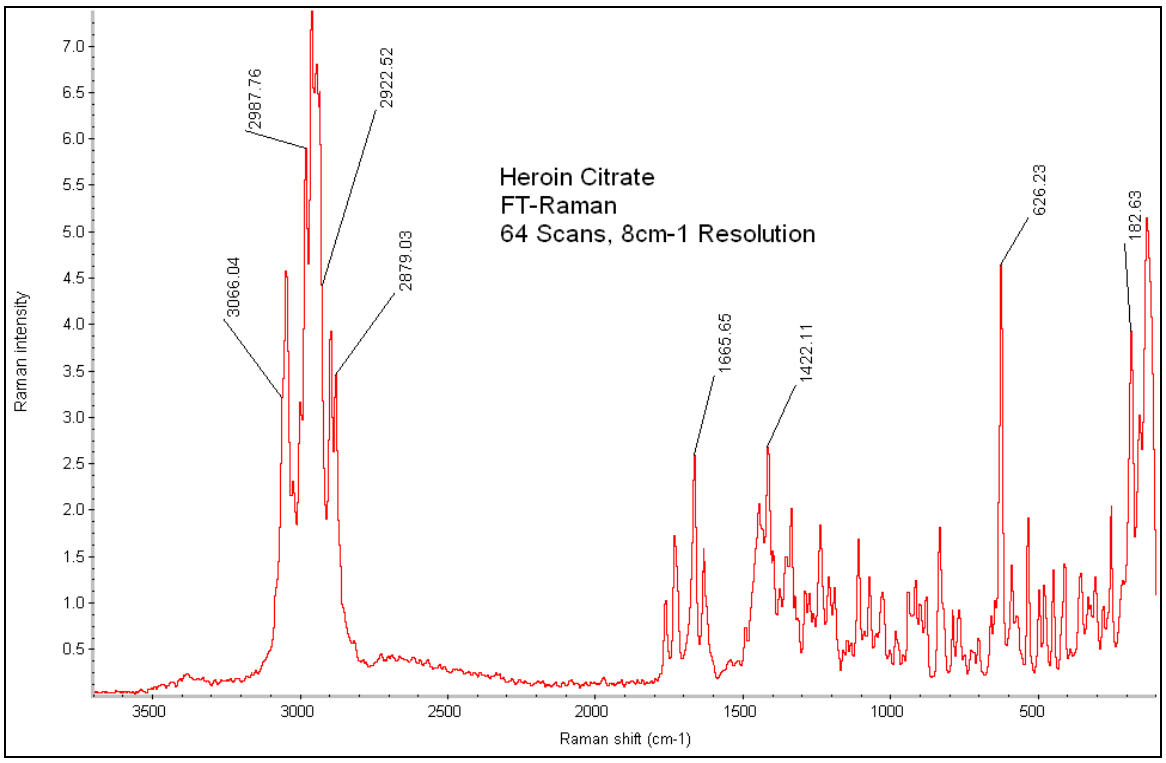

Nuclear Magnetic Resonance (proton)
Heroin Citrate
in CD3OD with TMS
400MHz
Normalized Intensity
2.0
1.5
1.0
0.5
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
Chemical Shift (ppm)

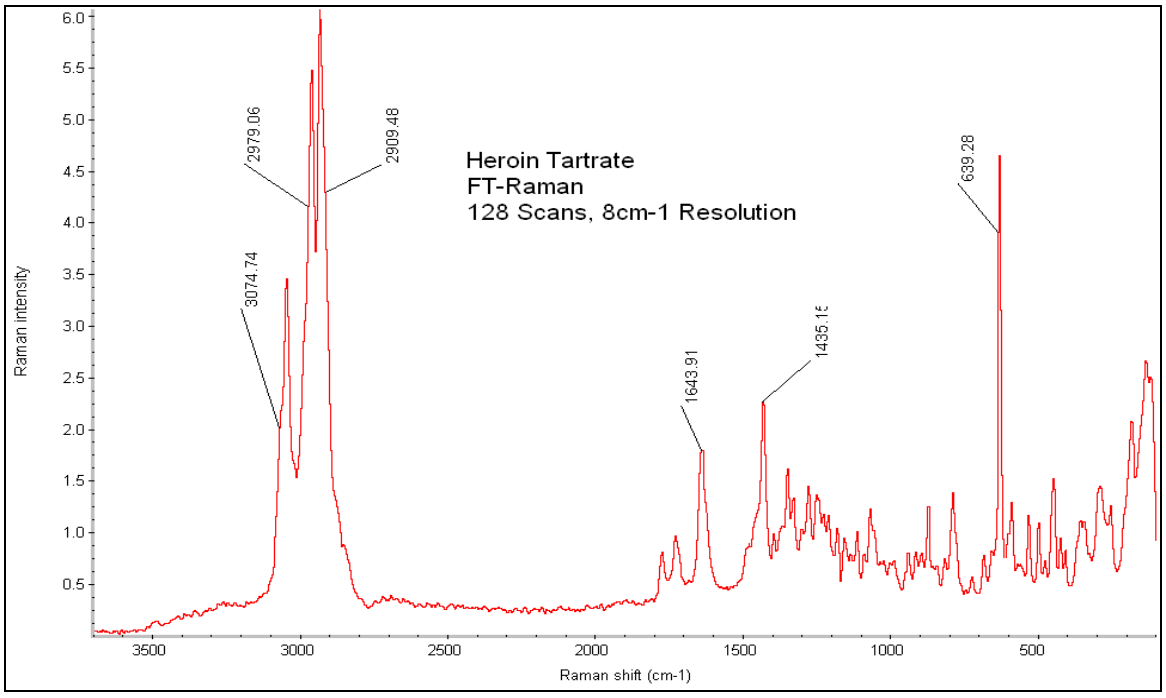

Nuclear Magnetic Resonance (proton)
Heroin Tartrate
in CD3OD with TMS
400MHz
Normalized Intensity
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
Chemical Shift (ppm)


100.12
99.5
99.0
98.5
98.0
97.5
97.0
96.5
96.0
95.5
95.0
94.5
94.0
93.5
93.0
92.5
92.0
91.5
91.0
90.5
90.0
89.5
89.0
88.5
88.0
87.76
%T
2916
1788
1760
1448
1371
1232
1193
Vapor Phase IR
Heroin in Chloroform
4000.4
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
748.4
cm-1


Heroin Base
FT-Raman
64 Scans, 8cm-1 Resolution
3039.95
2887.73
621.88
3061.69
2861.64
2792.05
1648.26
Raman intensity
Raman shift (cm-1)


Heroin Hydrochloride
FT-Raman
64 Scans, 8cm-1 Resolution
2961.66
634.93
3079.09
2879.03
1435.15
1739.59
Raman intensity
Raman shift (cm-1)


Heroin Citrate
FT-Raman
64 Scans, 8cm-1 Resolution
2987.76
2922.52
3066.04
2879.03
1665.65
1422.11
626.23
182.63
Raman intensity
Raman shift (cm-1)


Heroin Tartrate
FT-Raman
128 Scans, 8cm-1 Resolution
2979.06
2909.48
3074.74
1643.91
639.28
Raman intensity
Raman shift (cm-1)