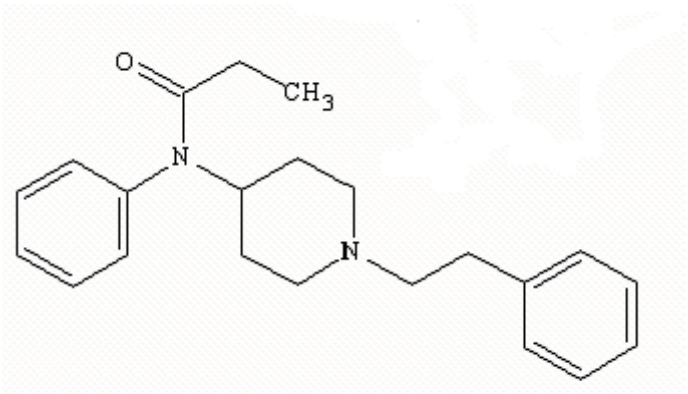
FENTANYL

Latest Revision: August 18, 2005



1. SYNONYMS

CFR:	Fentanyl
CAS #:	Base: 437-38-7 Citrate: 990-73-8
Other Names:	Fentanylum Phentanyl *N*-phenyl-*N*-[1-(2-phenylethyl)-4-piperidinyl]propanamide 1-phenethyl-4-(phenylpropionylamino)piperidine Innovar Sublimate Duragesic, China white

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Form	Chemical Formula	Molecular Weight	Melting Point (°C)
Base	$C_{22}H_{28}N_2O$	336.5	83-84
Citrate	$C_{22}H_{28}N_2O \cdot C_6H_8O_7$	528.6	149-151

2.2. SOLUBILITY

Form	A	C	E	H	M	W

Base	***	***	***	***	S	SS
Citrate	***	PS	SS	***	S	S

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	Orange

3.2. GAS CHROMATOGRAPHY

Method FEN-GCS1

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 1.1 mL/min

Make-Up gas: Nitrogen at 40.0 mL/min

Temperatures: Injector: 230°C
Detector: 280°C
Oven Program:
1) 205°C initial temperature 1 min
2) Ramp to 275°C at 11.5°C/min
3) Hold final temperature for 8.9 min

Injection Parameters: Split Ratio = 25:1, 1μL injected

Typical Retention Time: Fentanyl: 9.03 min
p-Fluorofentanyl: 8.66 min

COMPOUND	RRT	COMPOUND	RRT
acetyl codeine	0.82	methyl aminorex	0.25

COMPOUND	RRT	COMPOUND	RRT
acetyl thebol	0.84	allobarbital	0.26
acetylated dipyrone	0.68	hexadecane	0.26
acetylprocaine	0.70	ibuprofen	0.26
amitriptyline	0.60	guaifenesin	0.27
chloroquine	0.91	acetaminophen	0.28
codeine	0.73	butalbital	0.28
ethyl morphine	0.76	phenacetin	0.29
griseofulvin	0.97	amobarbital	0.30
heroin	0.91	talbutal	0.30
hydrocodone	0.77	meconin	0.31
hydromorphone	0.78	pentobarbital	0.31
morphine	0.77	acetylated acetaminophen	0.32
O3 MAM	0.81	meperidine	0.33
O6 MAM	0.83	secobarbital	0.34
oxycodone	0.83	caffeine	0.35
p-fluorofentanyl	0.96	antipyrine	0.38
phenylbutazone	0.72	diphenhydramine	0.39
promethazine	0.66	lidocaine	0.39
scopolamine	0.68	mephobarbital	0.40
tetracosane	0.73	aminopyrine	0.41
thebaine	0.82	doxylamine	0.41
strychnine	1.62	palmitic acid	0.42
noscapine	1.67	theophylline	0.42
aspirin	0.18	phenobarbital	0.43
nicotinic acid	0.18	dipyrone	0.46
salicylic acid	0.19	eicosane	0.46
nicotinamide	0.20	methapyrilene	0.46

COMPOUND	RRT	COMPOUND	RRT
methyl paraben	0.21	procaine	0.47
dimethyl phthalate	0.22	stearic acid	0.55
salicylamide	0.22	dextromethorphan	0.57
barbital	0.23	methadone	0.57
aminorex	0.25	**fentanyl**	**1.00 (9.03 min)**
		quinine	1.08

4. SEPARATION TECHNIQUES

Fentanyl Base or Citrate can be separated from the matrix by solvent extraction using the solubility.

5. QUANTITATIVE PROCEDURES

5.1. GAS CHROMATOGRAPHY

Method FEN-GCQ1

Internal Standard Stock Solution:
1.0 mg/mL p-fluorofentanyl in chloroform or methylene chloride

Standard Solution Preparation:
Prepare a standard solution of Fentanyl at 0.2 mg/mL in chloroform with an ISSS concentration of 0.08 mg/mL (2 mL/25 mL).

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

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 1.1 mL/min
Make-Up gas:	Nitrogen at 40.0 mL/min
Temperatures:	Injector: 230°C Detector: 280°C Oven Program: 1) 205°C initial temperature 1 min 2) Ramp to 275°C at 11.5°C/min

	3) Hold final temperature for 8.9 min
Injection Parameters:	Split Ratio = 25:1, 1µL injected
Typical Retention Time:	Fentanyl: 9.03 min p-Fluorofentanyl: 8.66 min
Linear Range:	0.1 mg/mL to 2.0 mg/mL
Repeatability:	RSD less than 1.25%
Correlation Coefficient:	0.9998
Accuracy	Error less than 4.5%

6. QUALITATIVE DATA

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

7. REFERENCES

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.

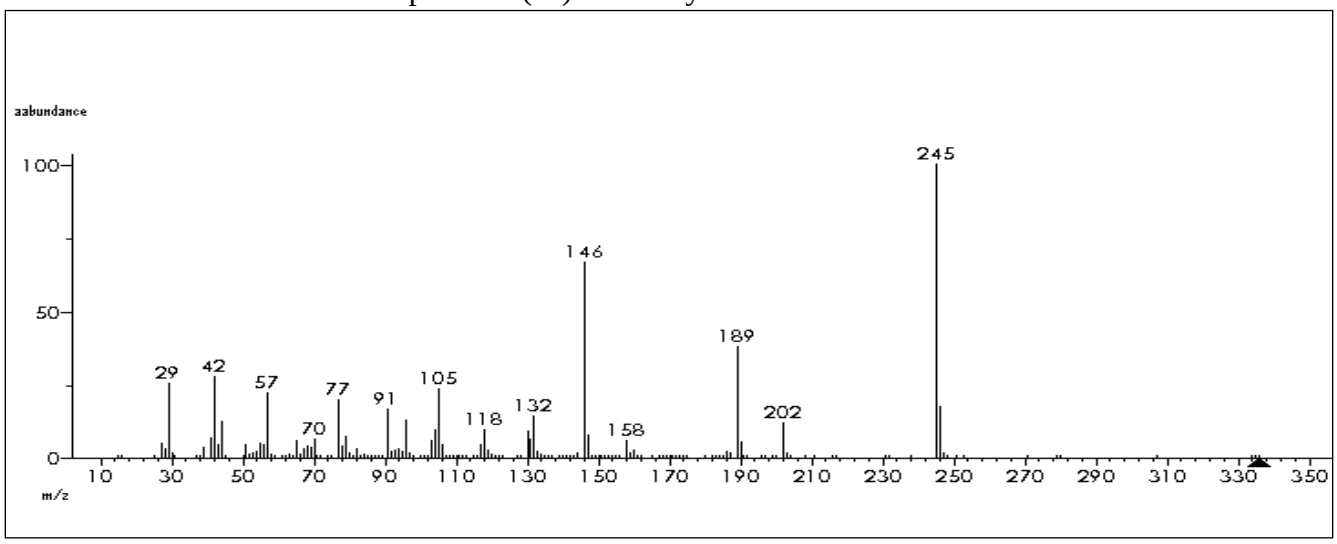
8. ADDITIONAL RESOURCES

Forendex

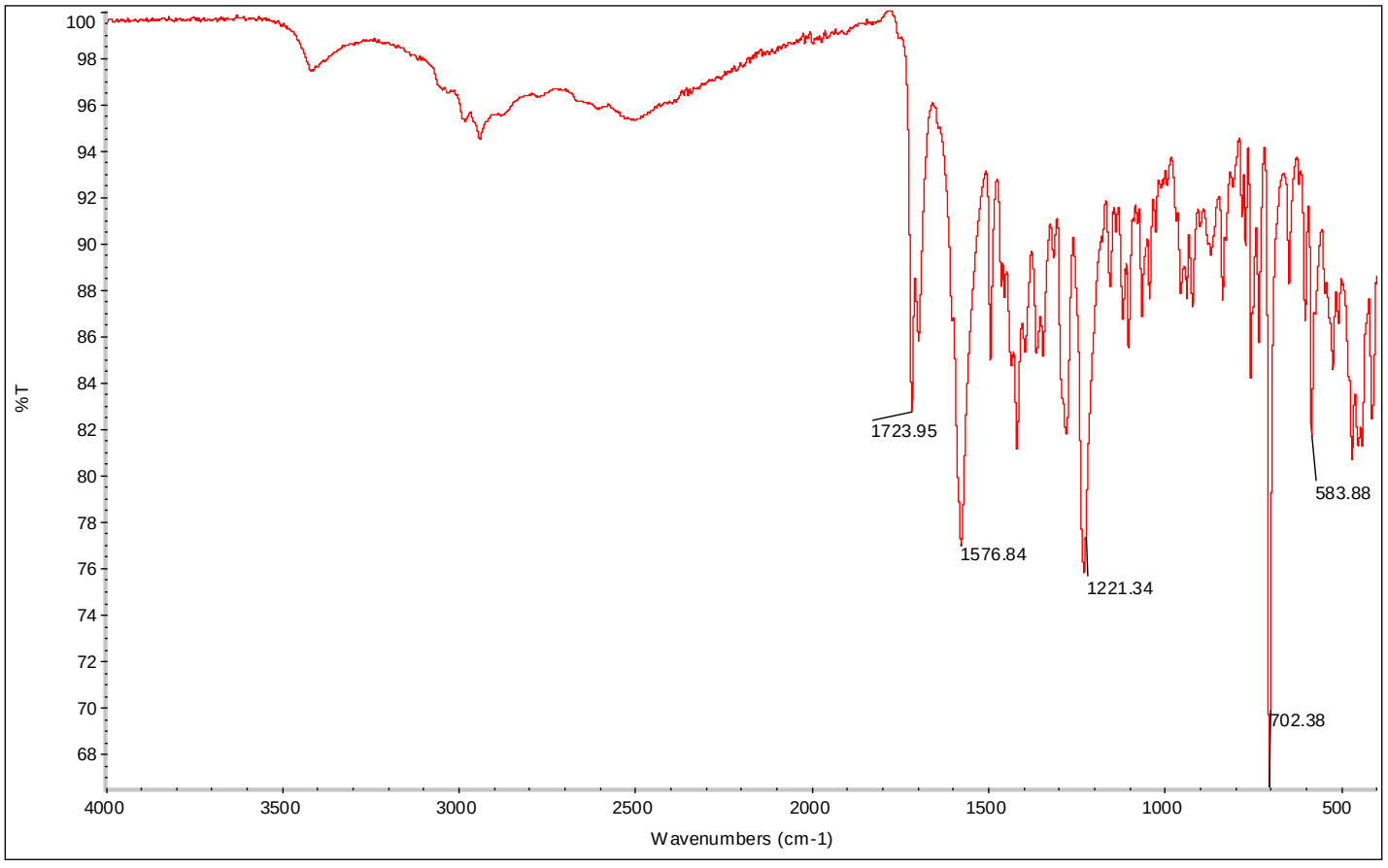
Wikipedia

***No Data Available

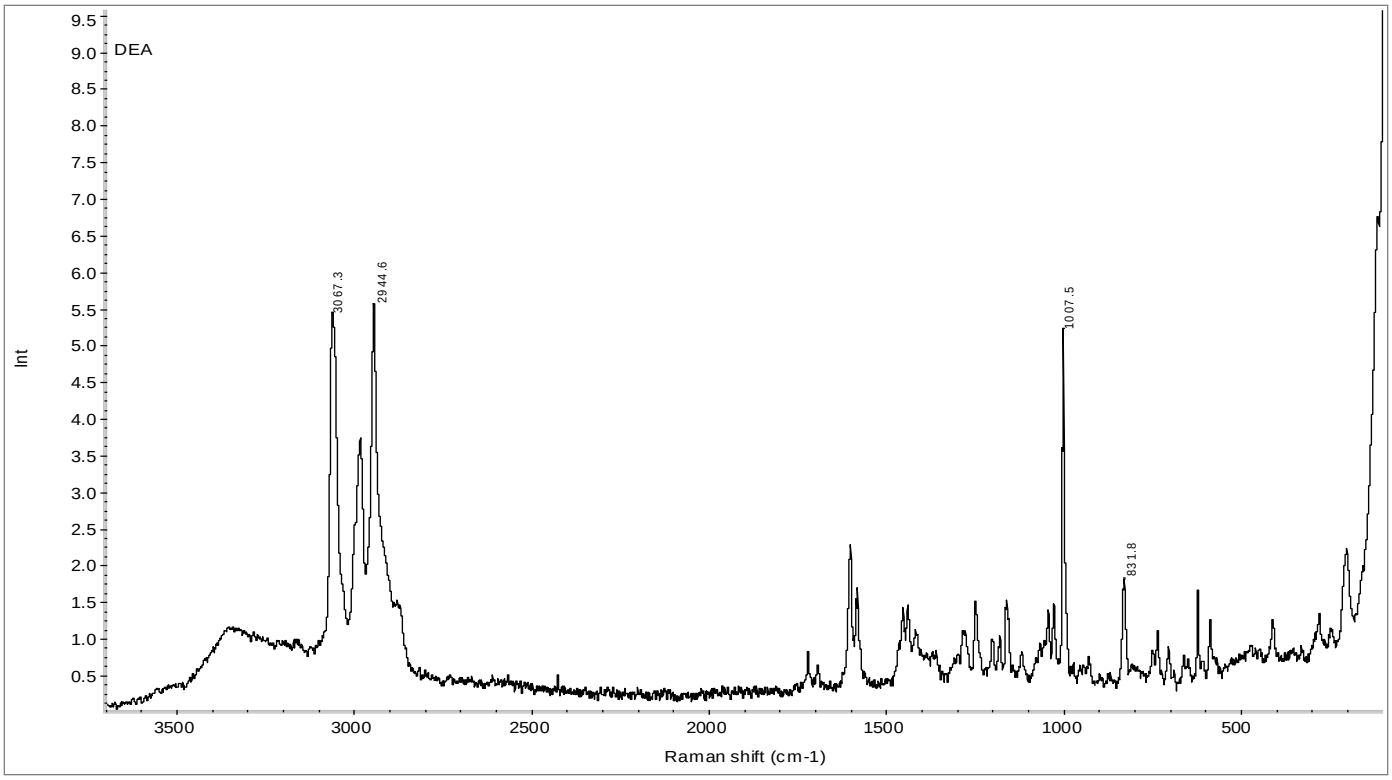
Mass Spectrum (EI): Fentanyl Citrate Lot # 121H0330



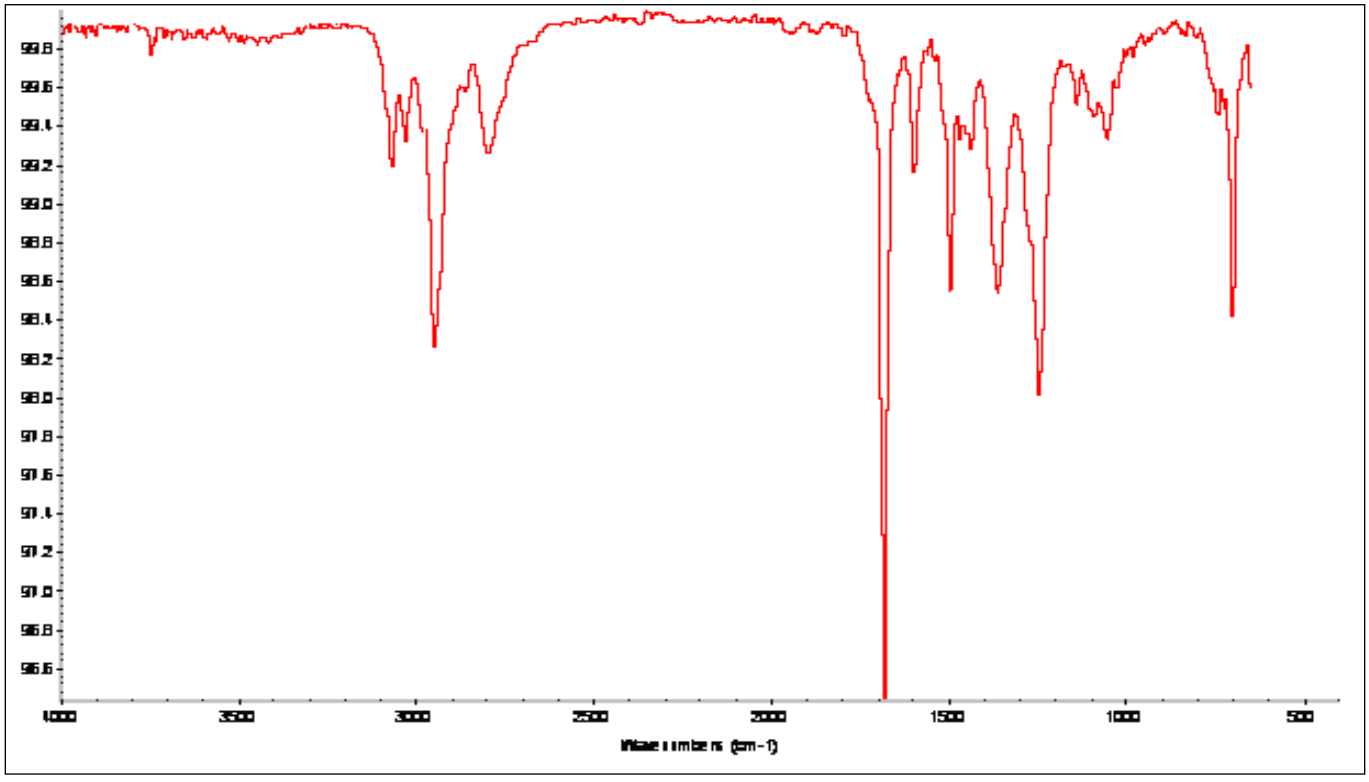
FTIR (One bounce ATR): Fentanyl citrate Lot # 121H0330
4cm^{-1} resolution, 32 scans



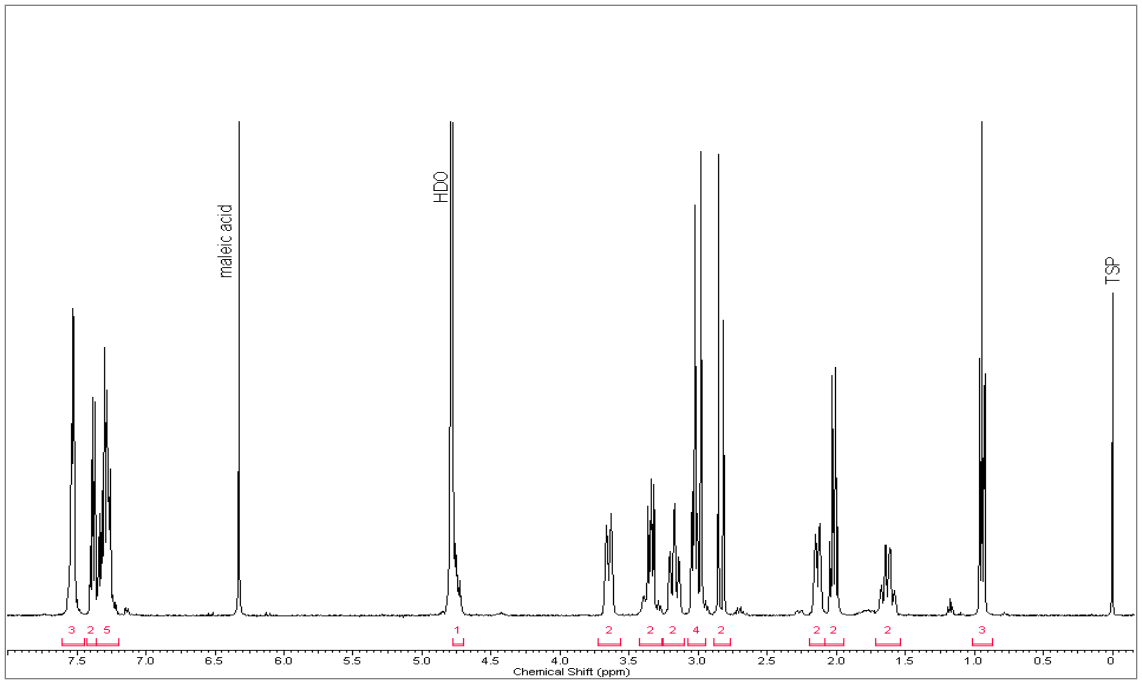
FT-RAMAN: Fentanyl citrate Lot # 121H0330
4cm^{-1} resolution, 32 scans



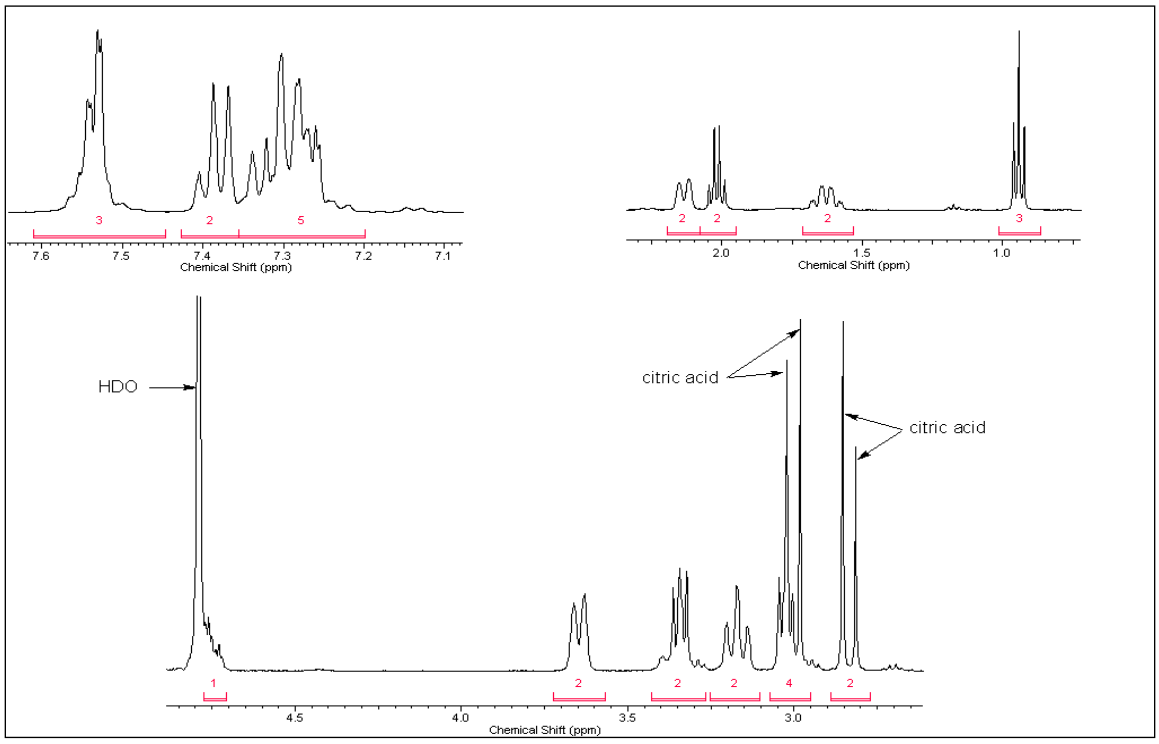
IR (Vapor Phase): Fentanyl citrate Lot # 121H0330
4cm^{-1} resolution, 64 scans



^{1}H NMR: Fentanyl Citrate Lot # 121H0330, 400 MHz
Deuterium oxide with TSP, maleic acid as IS



ppm 7.49 - 7.58 (m, 3 H) 7.39 (t, *J*=7.19 Hz, 2 H) 7.24 - 7.35 (m, 5 H) 6.33 (s,2 H, maleic acid) 4.74 (td, *J*=12.10, 3.60 Hz, 1 H) 3.65 (d, *J*=12.62 Hz, 2 H) 3.30 - 3.42 (m, 2 H) 3.17 (td, *J*=12.84, 1.61 Hz, 2 H) 3.03 (dd, *J*=10.60, 5.60 Hz, 2 H) 3.00 (d, *J*=15.75 Hz, 2 H, citric acid) 2.83 (d, *J*=15.75 Hz, 2 H, citric acid) 2.14 (d, *J*=13.89 Hz, 2 H) 2.02 (q, *J*=7.53 Hz, 2 H) 1.52 - 1.72 (m, *J*=13.35, 13.16, 13.16, 3.57 Hz, 2 H) 0.94 (t, *J*=7.53 Hz, 3 H)



^{13}C NMR: Fentanyl Citrate Lot # 121H0330
Deuterium oxide with TSP, maleic acid as IS