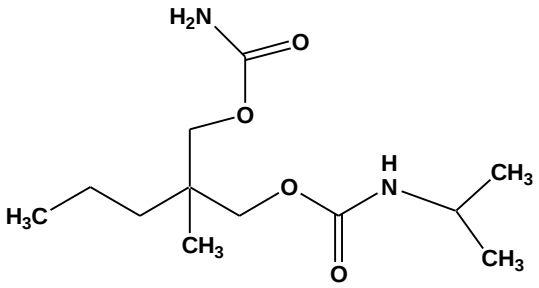
CARISOPRODOL

Latest Revision: June 23, 2005



1. SYNONYMS

CFR: Carisoprodol

CAS #: 78-44-4

Other Names:
N-Isopropylmeprobamate
2-Methyl-2-propyltrimethylene carbamate
Isopropylcarbamate
Carisoma
Flexartal
Mioxom
Rela
Sanoma
Soma
Somadril

2. CHEMICAL AND PHYSICAL DATA

2.1. CHEMICAL DATA

Chemical Formula	Molecular Weight	Melting Point (°C)
$C_{12}H_{24}N_2O_4$	260.3	92-94

2.2. SOLUBILITY

Form	A	C	M	W
Base	S	S	S	PS

A = acetone, C = chloroform, 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
Furfuraldehyde[1]	Violet to blue/black

3.2. THIN LAYER CHROMATOGRAPHY

Visualization

Furfuraldehyde reagent[1] Violet to black spots

COMPOUND	Relative R_f Values		
	System TLC7	System TLC4	System TLC12
carisoprodol	1.0	1.0	1.0
diazepam	1.6	1.05	0.90
lorazepam	0.63	0.61	0.73
oxazepam	0.61	0.60	0.69
triazolam	0.13	0.61	0.03

3. GAS CHROMATOGRAPHY

Method CAR- GCS1

Internal Standard Stock Solution (ISSS):
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 the above internal standard stock solution.

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

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

split mode equipped with a FID detector

Column: 5% Phenyl/95% Methyl silicone gum 12 m x 0.2 mm x 0.33 µm film thickness

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: Carisoprodol: 3.62 min
Tetracosane : 5.97 min

COMPOUND	RRT	COMPOUND	RRT
dimethylsulfone	0.163	propoxyphene HCl	1.488
amphetamine Sulfate	0.212	atropine Sulfate	1.498
methamphetamine	0.229	cocaine HCl	1.505
N,N-dimethylamphetamine	0.257	tetracaine HCl	1.522
phenylpropanolamine HCl	0.307	triprolidine	1.570
niacinamide	0.348	tetracosane	1.660
methylephedrine	0.378	phenylbutazone	1.702
MDA HCl	0.442	codeine phosphate	1.721
MDMA HCl	0.498	morphine sulfate	1.781
benzocaine	0.546	diazepam	1.790
MDEA	0.553	hydrocodone bitartrate	1.801
guaifenesin	0.659	acetylcodeine	1.878
acetaminophen	0.687	monoacetylmorphine	1.899
phenacetin	0.716	oxycodone Base	1.899
amphetamine Sulfate	0.212	atropine Sulfate	1.498
methamphetamine	0.229	cocaine HCl	1.505
N,N-dimethylamphetamine	0.257	tetracaine HCl	1.522
phenylpropanolamine HCl	0.307	triprolidine	1.570
niacinamide	0.348	tetracosane	1.660
methylphenidate	0.784	benzoylecgonine tartrate	1.966
caffeine	0.952	chloroquine Phosphate	1.981

carisoprodol	**1.000**	heroin HCl	2.033
ketamine HCl	1.002	quinine base	2.233
diphenhydramine HCl	1.007	quinine HCl	2.233
antipyrine	1.026	quinidine HCl	2.234
lidocaine HCl	1.032	zolpidem	2.240
doxylamine succinate	1.092	papaverine	2.275
aminopyrine	1.098	clonazepam	2.308
phenobarbital	1.161	hydroxyzine	2.319
xylazine	1.191	alprazolam	2.456
levamisole	1.193	diltiazem	2.471
dipyrone	1.231	noscapine	2.919
procaine HCl	1.275	amoxycillin	not soluble
clenbuterol HCl	1.321	creatine hydrate	not soluble
brompheniramine	1.402	creatinine HCL	not soluble
dextromethorphan	1.431	scopolamine HBr	not soluble
methadone HCl	1.441		

4. SEPERATION TECHNIQUES

5. QUANTITATIVE PROCEDURE

***5.1.* GAS CHROMATOGRAPHY**

Method CAR- GCQ-1

Internal Standard Stock Solution (ISSS):
1mg/ml of eicosane into 80:20 $CHCl_3$: MEOH

Standard Solution Preparation:
Prepare a standard solution of carisoprodol at 1.0mg/ml and dilute to volume with the ISSS.

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

Instrument: Agilent 6890 Series II (or comparable) gas chromatograph operated in split mode equipped with a FID detector

Column: 5% Phenyl/95% Methyl silicone gum 12 m x 0.2 mm x 0.33 µm film thickness

Carrier gas: Helium: (constant pressure)
Flow: 1 mL/min

Temperatures: Injector: 200°C
Detector: 280°C
Oven program:
1) 165°C initial temperature for 2.0 min
2) Ramp to 250°C at 30°C/min
3) Hold final temperature for 0.5 min

Injection Parameters: Split Ratio = 60:1, 1 µL injection

Typical Retention Time: Carisoprodol: 4.053 min
Eicosane : 4.423 min

Linear Range: 0.54 - 3.25 mg/mL

Repeatability: RSD less than 2 %

Correlation Coefficient: 0.9986

Accuracy: Error less than 5%

COMPOUND	RRT
dimethylsulfone	0.136
amphetamine	0.188
methamphetamine	0.207
phenylpropanolamine	0.297
pseudoephedrine	0.338
niacinamide	0.338
ephedrine	0.344
MDA	0.462
MDMA	0.546
MDEA	0.614
methylphenidate	0.832
caffeine	0.957
ketamine	0.993
carisoprodol	**1.000**
eicosane (ISTD)	1.091
phencyclidine	1.091

6. QUALITATIVE DATA

6.1. HIGH PERFORMANCE LIQUID CHROMATOGRAPHY – MASS SPECTOMETRY

Sample Preparation:
Dissolve a small amount of sample into ammonium formate buffer and methanol. 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:	Phenomenex Hydro-RP column, 150 mm x 3.0mm, 80A, 4 µm Temperature: 40°C
Detector:	UV DAD: 210 nm, 10 nm bandwidth Reference: 450, 100 nm bandwidth MSD: Scan Mode, single quadrupole with an electrospray ionization source Polarity: Positive Fragmentor: 140 V
Ionization Mode:	API-ES Drying gas temperature: 350°C Drying gas flow: 13.0 L/min Nebulizer Pressure: 30 psi Capillary Voltage: 4000 V Scan Range: 50-300 m/z
Flow:	0.50 mL/min
Injection Volume:	2.0 µL
Buffer::	10 mM ammonium formate pH 3.7
Mobile Phase:	50% 10mM ammonium formate pH 3.7: 50% acetonitrile
Typical Retention Time:	Carisoprodol in 3.1 min.

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

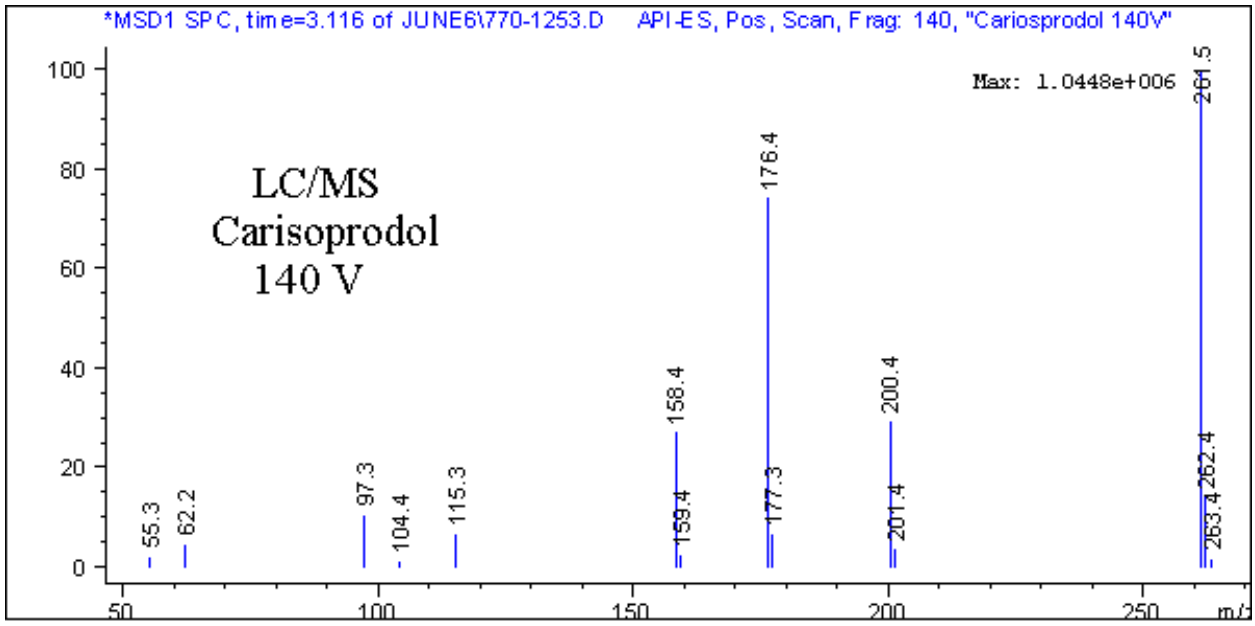
7. REFERENCES

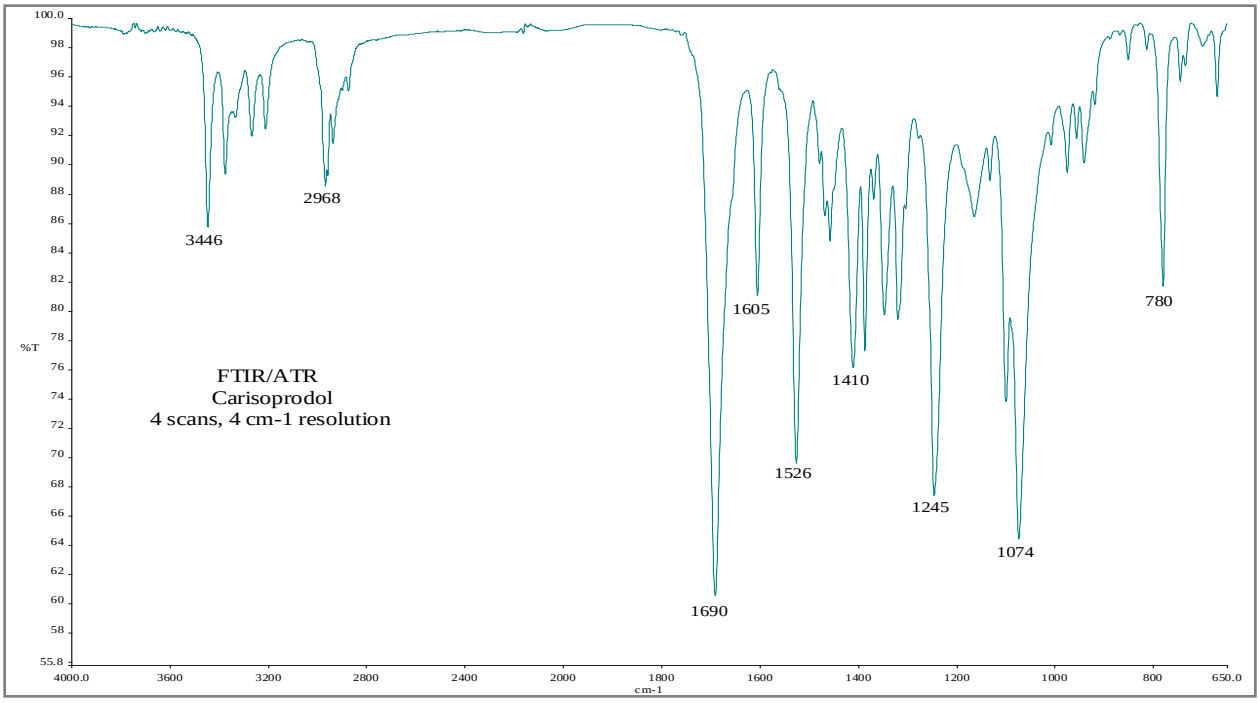
E. G. C. Clarke, *Clarke's Isolation and Identification of Drugs in pharmaceuticals, body fluids, and post-mortum material*, 2 ed., The Pharmaceutical Press, London, 1986, Part 4: Appendix of Reagents and Proprietary Test Materials.

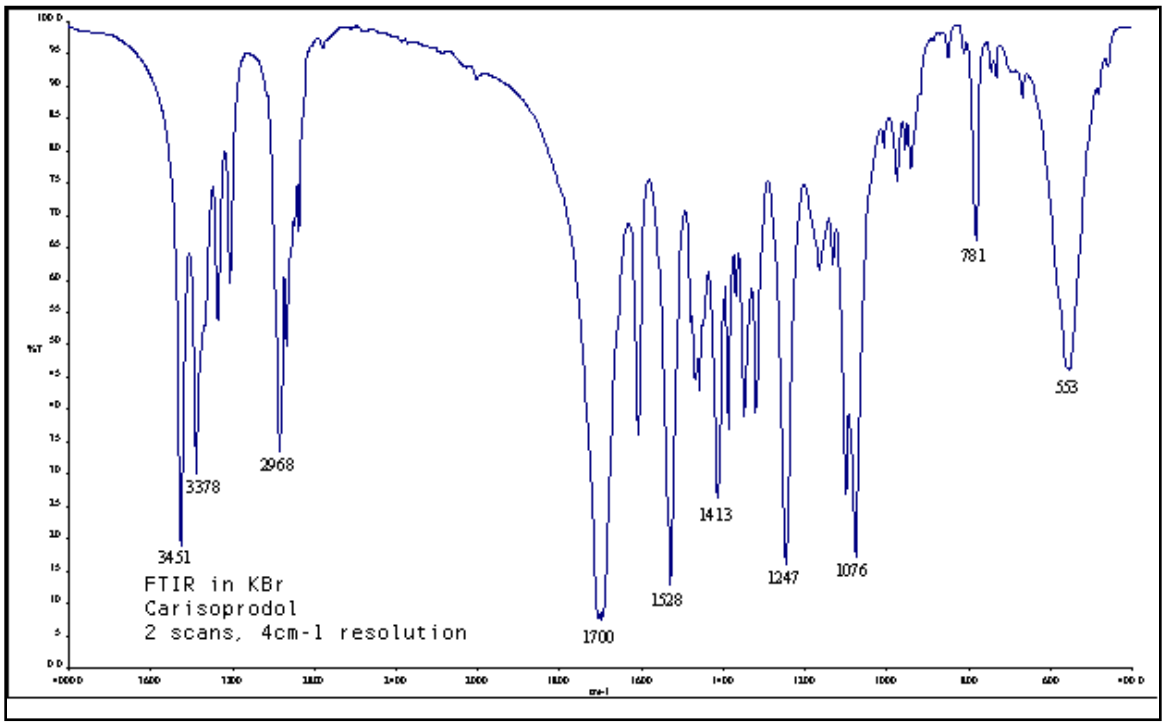
8. ADDITIONAL RESOURCES

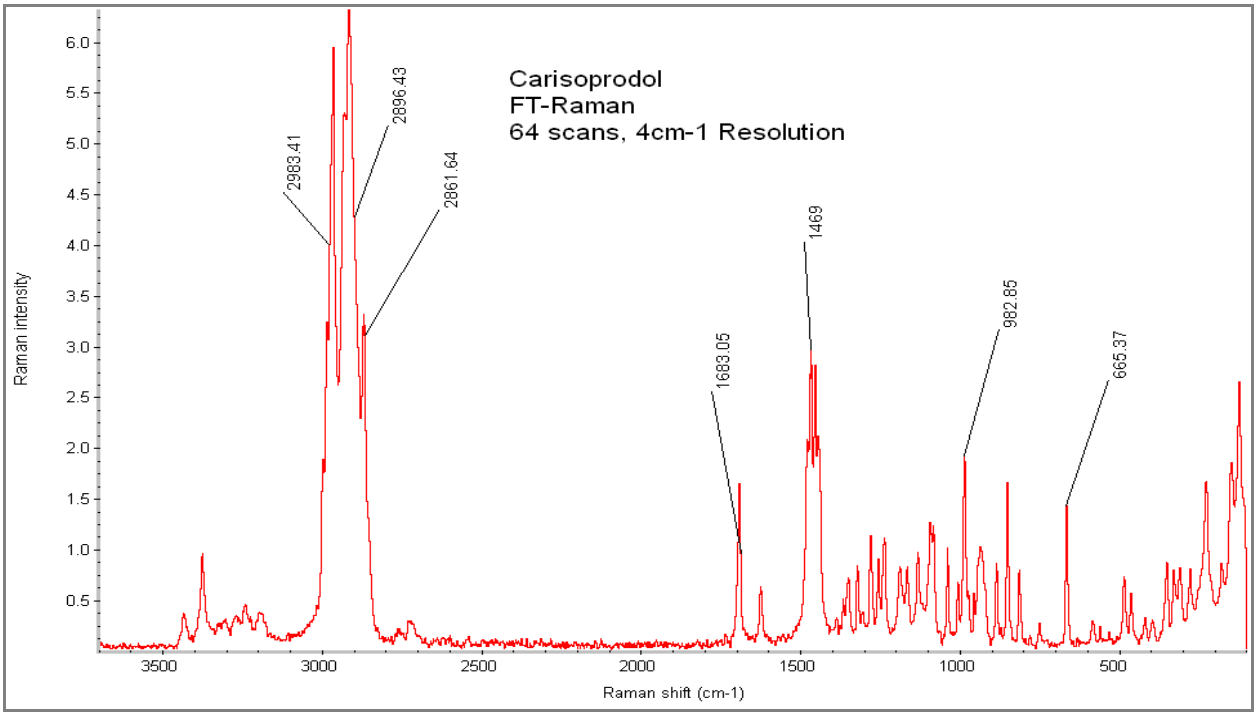
Forendex

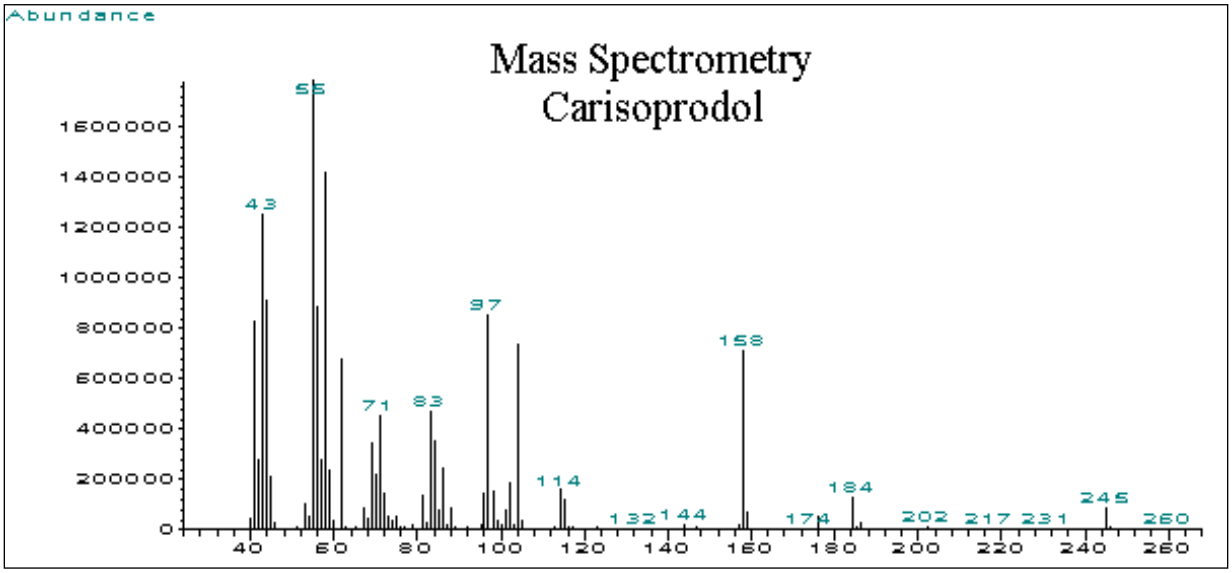
Wikipedia

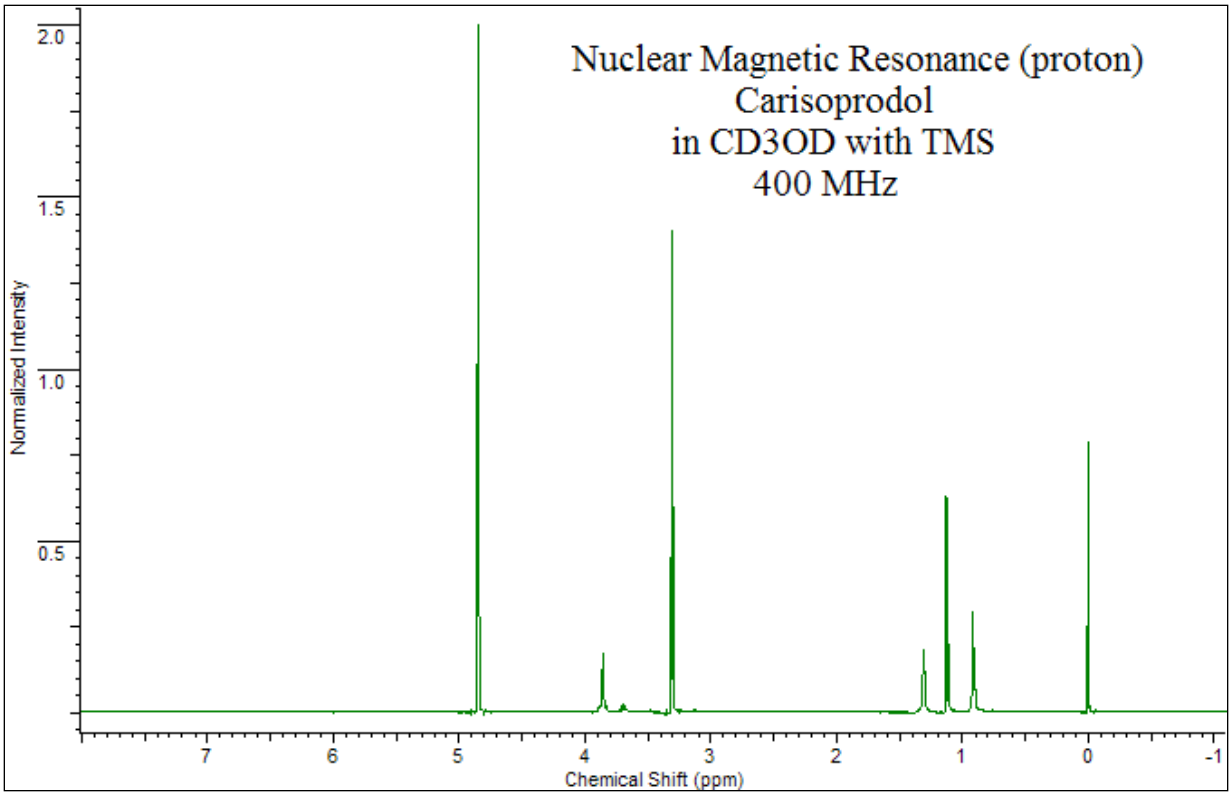

*MSD1 SPC, time=3.116 of JUNE6\770-1253.D API-ES, Pos, Scan, Frag: 140, "Cariosprodol 140V"
Max: 1.0448e+006
LC/MS
Carisoprodol
140 V
261.5
176.4
158.4
200.4
97.3
115.3
55.3
62.2
104.4
159.4
177.3
201.4
262.4
263.4
100
80
60
40
20
0
50
100
150
200
250
m/z

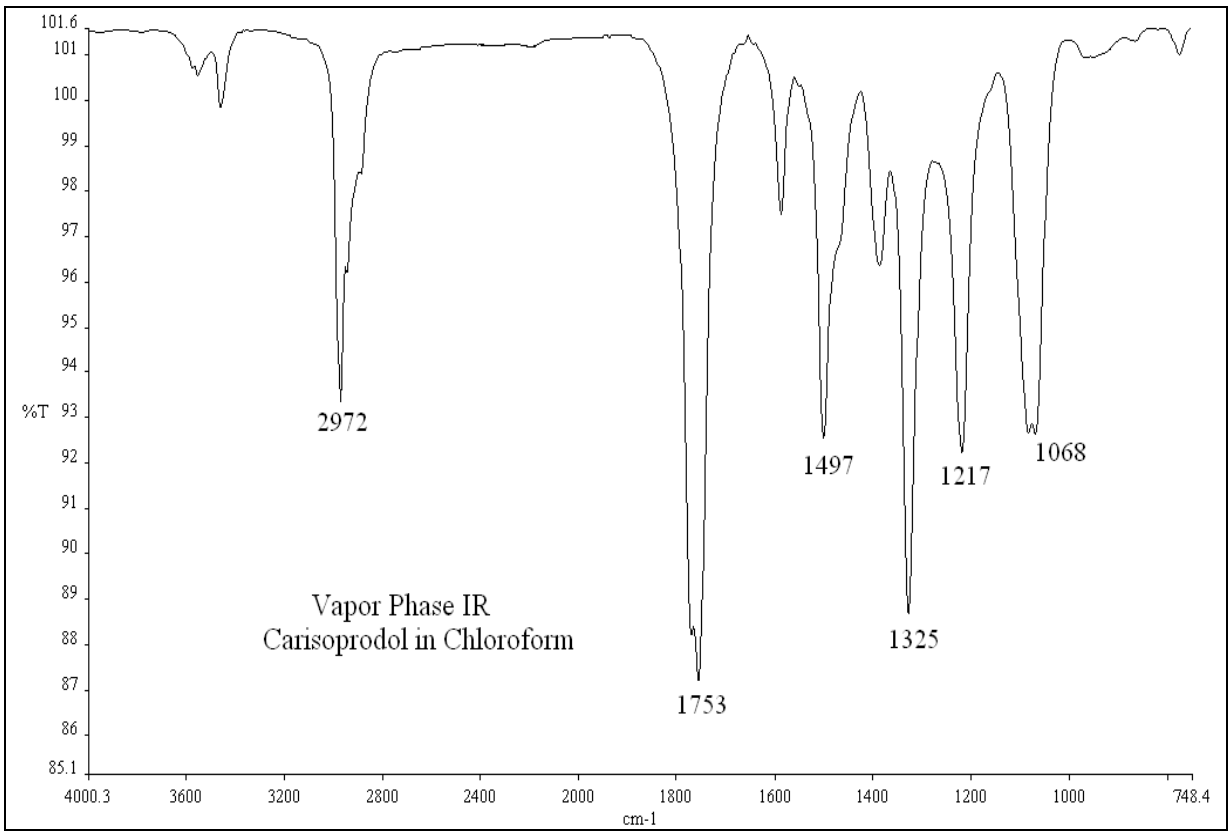

FTIR/ATR
Carisoprodol
4 scans, 4 cm-1 resolution
%T
cm-1
3446
2968
1690
1605
1526
1410
1245
1074
780
4000.0
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650.0


FTIR in KBr
Carisoprodol
2 scans, 4cm-1 resolution
3451
3378
2968
1700
1528
1413
1247
1076
781
553


Carisoprodol
FT-Raman
64 scans, 4cm-1 Resolution
2983.41
2896.43
2861.64
1683.05
1469
982.85
665.37
Raman intensity
Raman shift (cm-1)


Abundance
Mass Spectrometry
Carisoprodol
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
55
43
97
158
71
83
114
184
245
132
144
174
202
217
231
260
40
60
80
100
120
140
160
180
200
220
240
260


Nuclear Magnetic Resonance (proton)
Carisoprodol
in CD3OD with TMS
400 MHz
Normalized Intensity
2.0
1.5
1.0
0.5
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)


101.6
101
100
99
98
97
96
95
94
%T 93
92
91
90
89
88
87
86
85.1
2972
1753
1497
1325
1217
1068
Vapor Phase IR
Carisoprodol in Chloroform
4000.3
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
748.4
cm-1