Identification of polymorphism in ethylone hydrochloride: synthesis and characterization

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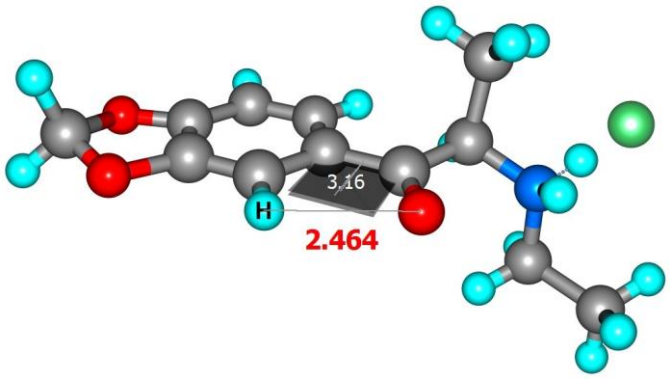
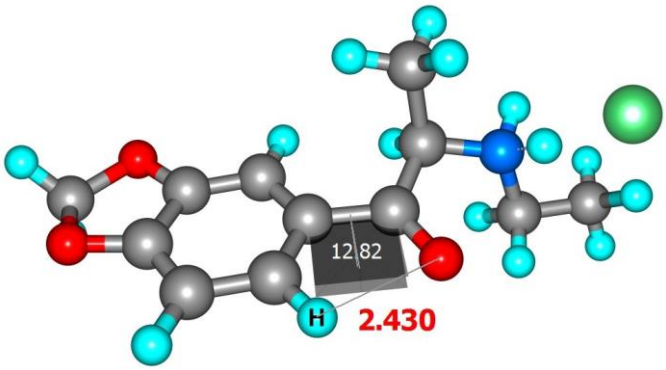
Keywords: ethylone, polymorphism, synthetic cathinones, designer drugs, new psychoactive substances

Computed, optimized structure coordinates for polymorph **1A**:



17	4.956779000	-1.451890000	0.088252000
8	-4.498044000	1.125420000	-0.331807000
8	-4.836856000	-0.892740000	0.703328000
8	0.578774000	1.663628000	-0.998763000
7	2.819337000	0.471135000	-0.215656000
6	-5.486523000	0.155746000	-0.015932000
6	-3.513686000	-0.722938000	0.479281000
6	-3.301245000	0.499122000	-0.155673000
6	-2.048962000	0.938398000	-0.479840000
6	-0.966602000	0.096285000	-0.146042000
6	-1.188232000	-1.125232000	0.492072000
6	-2.475782000	-1.558125000	0.819558000
6	0.389676000	0.572878000	-0.498274000
6	1.593562000	-0.350938000	-0.296323000
6	1.699640000	-1.329270000	-1.467937000
6	2.973709000	1.253396000	1.039555000
6	4.191159000	2.152535000	0.945856000
1	-6.250740000	0.605550000	0.615502000
1	-5.909543000	-0.253947000	-0.939956000
1	-1.874219000	1.889187000	-0.967093000
1	-0.359181000	-1.774082000	0.747414000
1	-2.650136000	-2.503017000	1.318439000
1	1.543084000	-0.901434000	0.644399000
1	3.098177000	0.515928000	1.835027000
1	2.059520000	1.823703000	1.212167000
1	5.088822000	1.561292000	0.753550000
1	4.327615000	2.685345000	1.888632000
1	4.075418000	2.899587000	0.155059000
1	1.785176000	-0.787159000	-2.413956000
1	0.808852000	-1.957091000	-1.519130000
1	2.579487000	-1.962202000	-1.340383000
1	2.850179000	1.097914000	-1.018686000
1	3.675618000	-0.216984000	-0.241544000

Computed, optimized structure coordinates for polymorph **1B**:



17	-4.366696000	1.399878000	1.004071000
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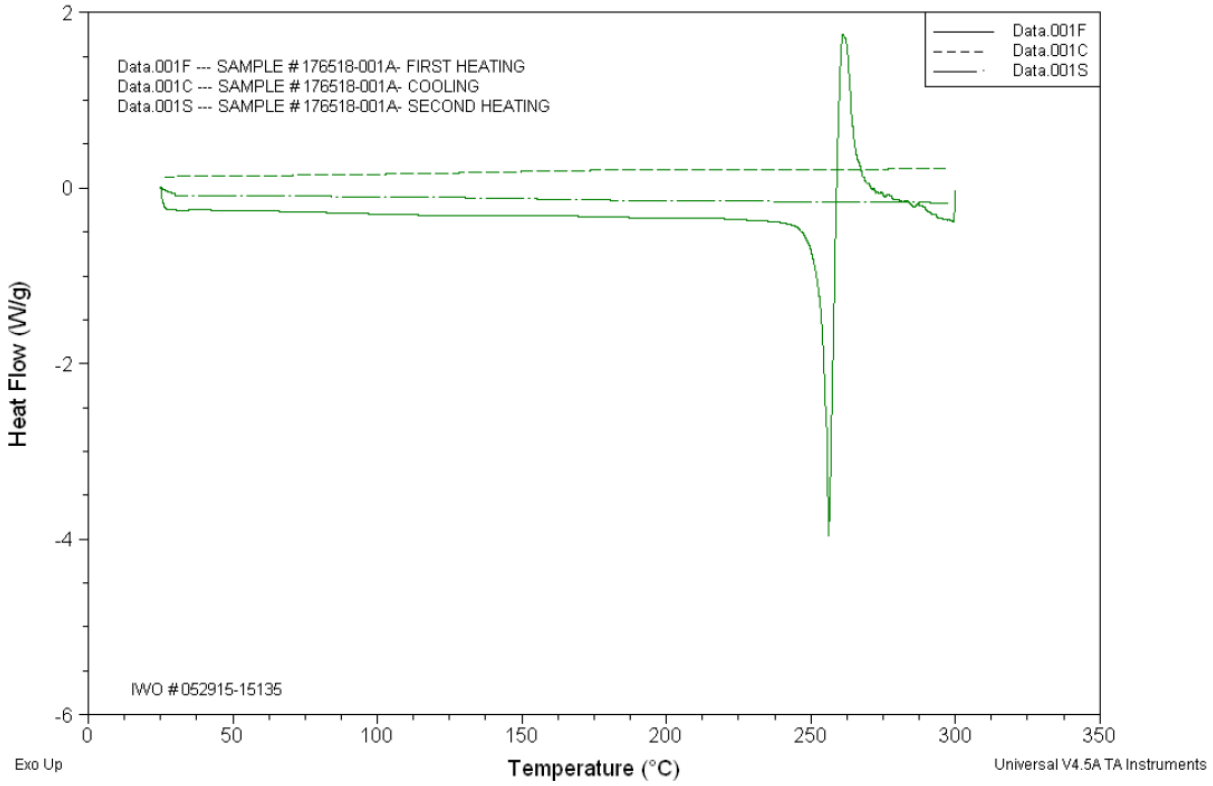
8	4.953910000	0.512165000	-0.139972000
8	-1.245438000	1.058735000	-1.089556000
7	-2.775076000	-0.819793000	0.097961000
6	0.870627000	0.349868000	-0.320895000
6	1.491385000	1.476339000	-0.860797000
6	2.878233000	1.629222000	-0.852252000
6	3.609743000	0.605155000	-0.296319000
6	3.002543000	-0.523906000	0.240877000
6	1.642276000	-0.683230000	0.251374000
6	-0.616855000	0.296962000	-0.395672000
6	-1.343695000	-0.736823000	0.472824000
6	-3.104895000	-1.233381000	-1.291573000
6	-4.582986000	-1.564807000	-1.390853000
6	5.195551000	-0.825931000	0.286947000
6	-1.249580000	-0.363198000	1.954895000
1	0.864638000	2.247102000	-1.292935000
1	3.357762000	2.510144000	-1.260382000
1	1.208466000	-1.575615000	0.685185000
1	-0.905924000	-1.726136000	0.297858000
1	-2.476320000	-2.090395000	-1.554559000
1	-2.845297000	-0.397474000	-1.936061000
1	-1.730519000	-1.124547000	2.574938000
1	-1.754553000	0.588284000	2.133994000
1	-0.209128000	-0.279419000	2.268619000
1	5.546740000	-1.417519000	-0.566584000
1	5.917960000	-0.820906000	1.101210000
1	-3.313921000	0.130337000	0.349805000
1	-3.218898000	-1.471803000	0.743271000
1	-4.847392000	-2.430476000	-0.775140000
1	-4.837491000	-1.802578000	-2.425309000
1	-5.188064000	-0.713941000	-1.071117000

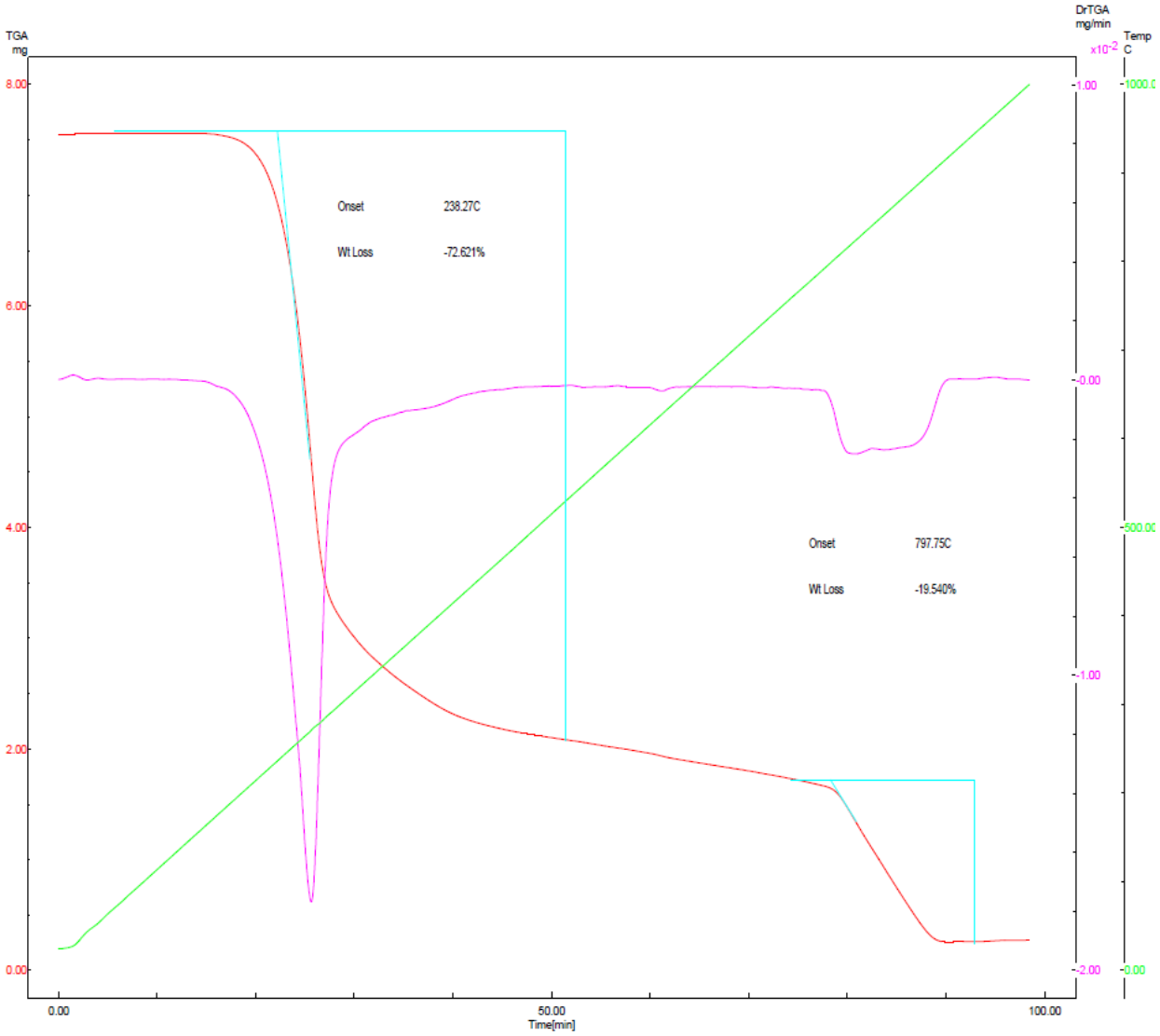
Thermal properties from the DSC and TGA

Thermal Properties from the DSC	1A	1B
First Heating		
Endothermic peak temperature [°C]	256	256
Exothermic peak temperature [°C]	261	261
Cooling		
Crystallization peak temperature [°C]	---	---
Second Heating		
Endothermic peak temperature [°C]	---	---

DSC heating curve for **1A**



TGA Thermal Analysis Report for **1A**

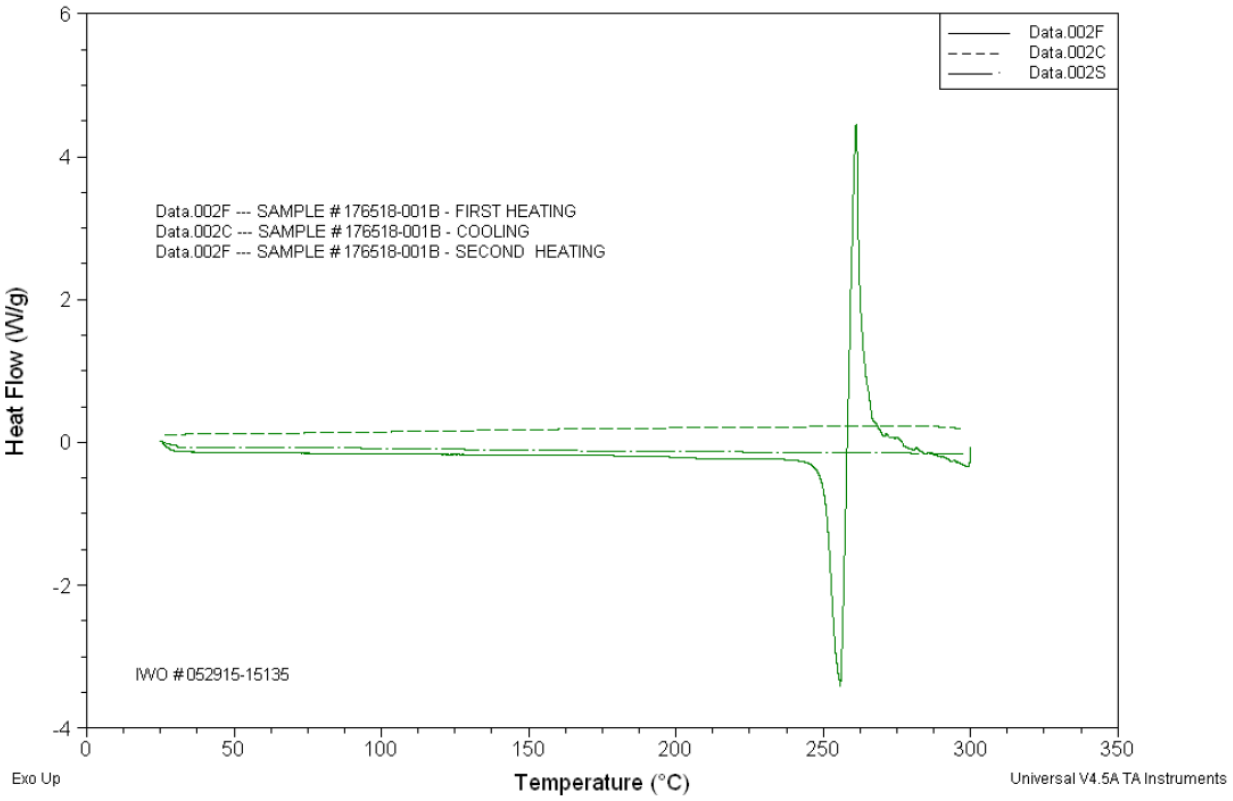


File Name:	176518-1.D20
Detector Type:	Shimadzu TGA-50
Acquisition Date:	11/06/04
Acquisition Time:	09:22:36
Sample Name:	176518-001 polymorph A
Weight:	7.542[mg]
Cell:	Platinum
Atmosphere:	Nitrogen
Rate Flow:	20.00[ml/min]
Operator:	Caroline Dulude
Comment:	Switched to 50% O2 @ 650ºC

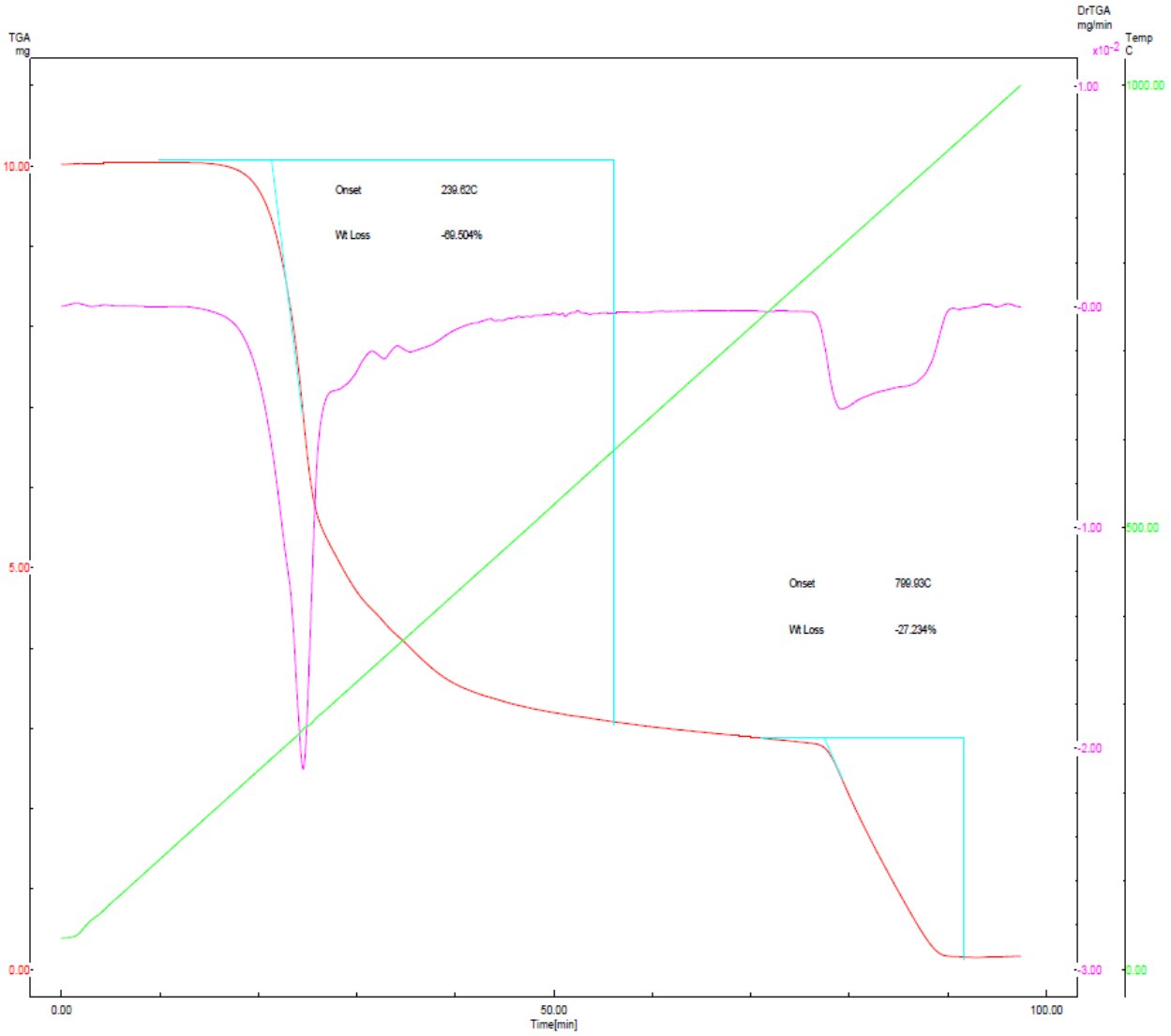
Temp Program

Rate [C/min]	Hold Temp [C]	Hold Time [min]
10.0	1000.0	0.0
10.0	600.0	0.0
5.0	800.0	0.0
10.0	1000.0	0.0

DSC heating curve for **1B**



TGA Thermal Analysis Report for **1B**

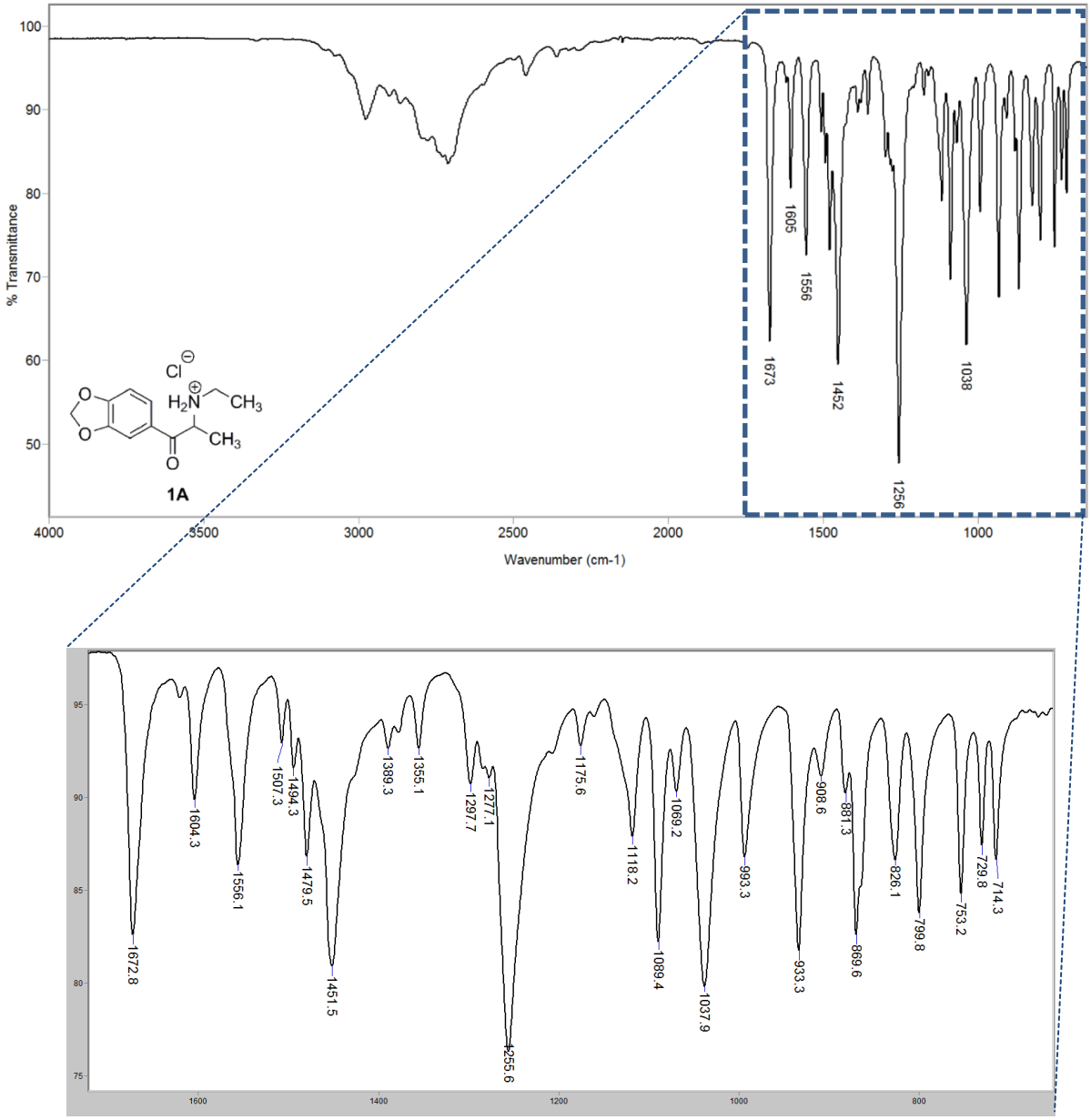


File Name:	175518_1.D20
Detector Type:	Shimadzu TGA-50
Acquisition Date:	11/06/04
Acquisition Time:	11:37:31
Sample Name:	176518-001 polymorph B
Weight:	10.02[mg]
Cell:	Platinum
Atmosphere:	Nitrogen
Rate Flow:	20.00[ml/min]
Operator:	Caroline Dulude
Comment:	Switched to 50% O2 @ 650°C

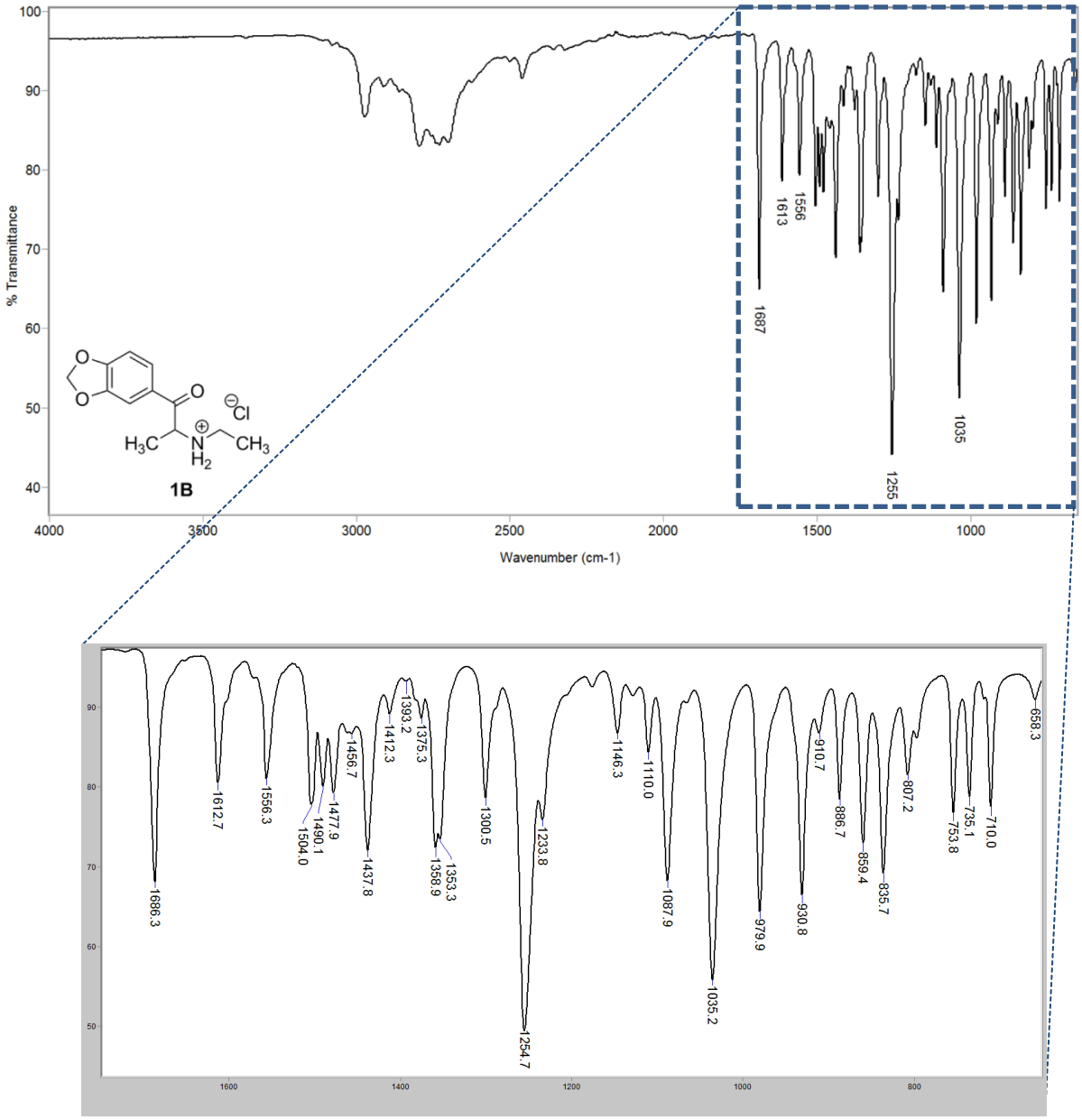
Temp Program

Rate [C/min]	Hold Temp [C]	Hold Time [min]
10.0	1000.0	0.0
10.0	600.0	0.0
5.0	800.0	0.0
10.0	1000.0	0.0

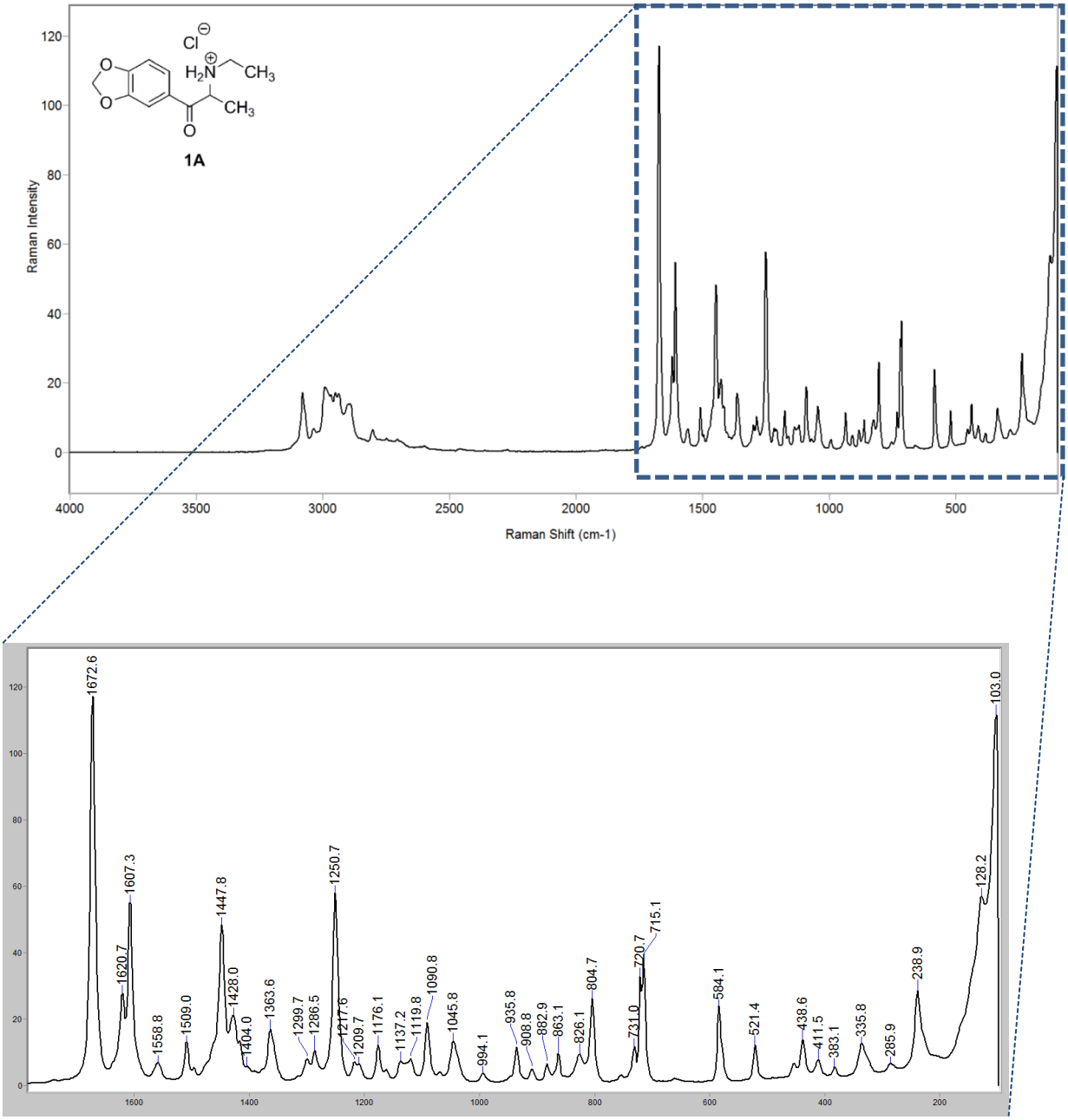
ATR-FTIR of **1A**



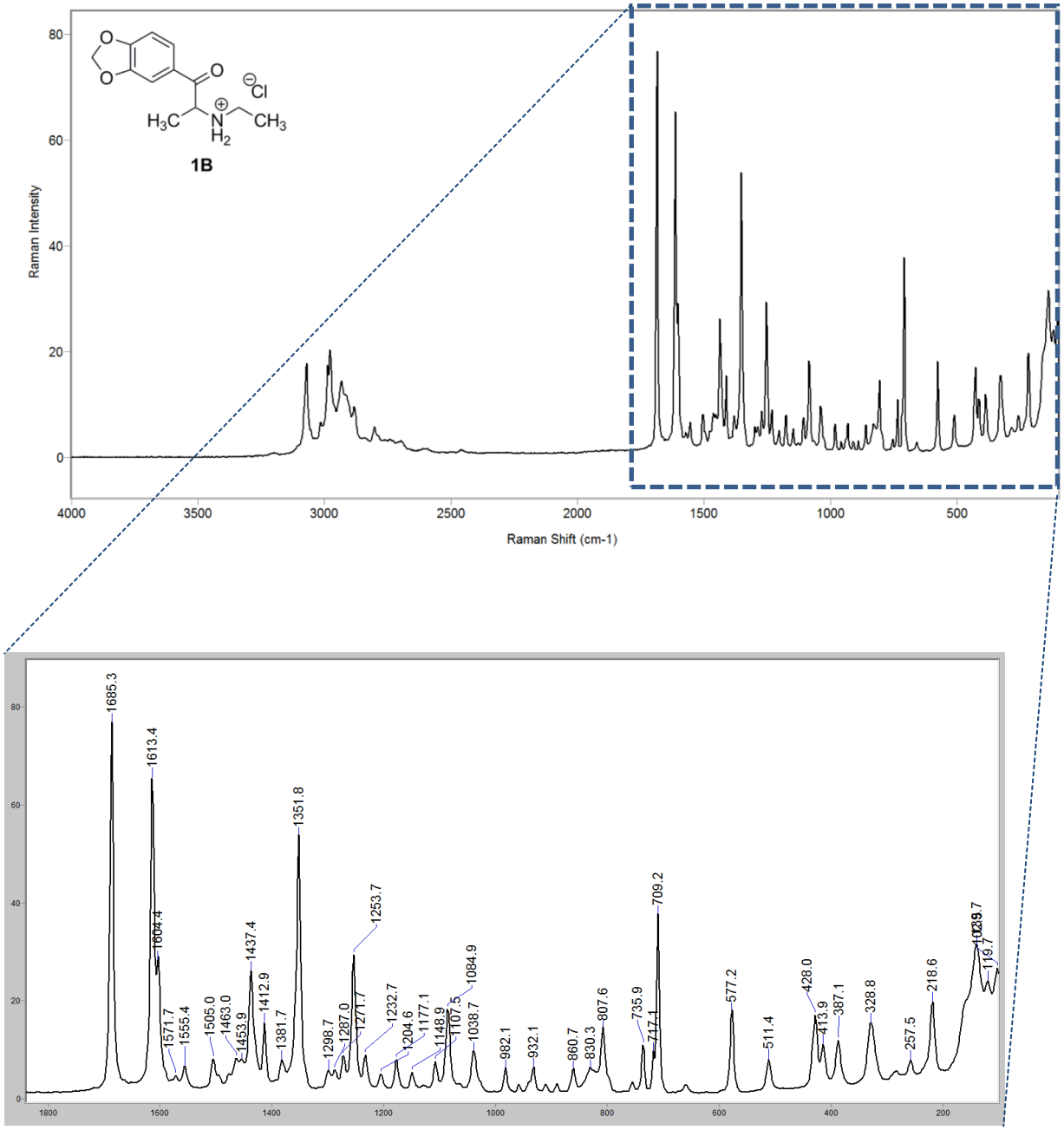
ATR-FTIR of **1B**



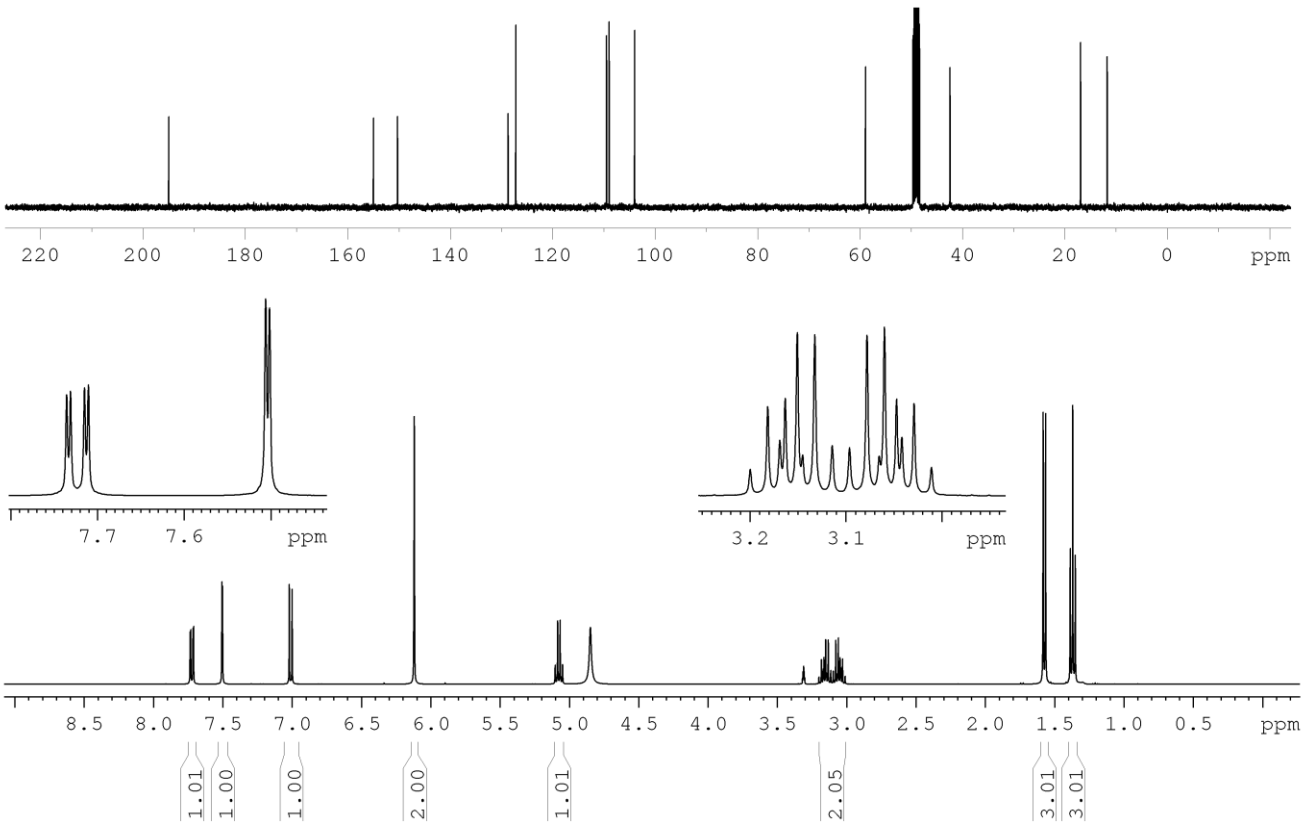
FT-Raman of **1A**



FT-Raman of **1B**



ATR-FTIR and FT-Raman peak list for **1A** and **1B**

ATR-FTIR		FT-Raman			
1A (cm^{-1})	**1B** (cm^{-1})	**1A** (cm^{-1})		**1B** (cm^{-1})	
2974.3	2973.1	3079.0	1119.8	3070.8	1107.5
2709.1	2910.8	3036.4	1090.8	2987.8	1084.9
2692.9	2793.2	2991.6	1045.8	2977.7	1038.7
1672.8	2728.7	2950.2	994.1	2932.7	982.1
1604.3	2700.1	2937.3	935.8	2882.3	932.1
1556.1	2459.2	2900.1	908.8	2802.8	860.7
1507.3	1686.3	2891.7	882.9	1685.3	830.3
1494.3	1612.7	2803.5	863.1	1613.4	807.6
1479.5	1556.3	2748.8	826.1	1604.4	735.9
1451.5	1504.0	2705.7	804.7	1571.7	717.1
1389.3	1490.1	1672.6	731.0	1555.4	709.2
1355.1	1477.9	1620.7	720.7	1505.0	577.2
1297.7	1456.7	1607.3	715.1	1463.0	511.4
1277.1	1437.8	1558.8	584.1	1453.9	428.0
1255.6	1412.3	1509.0	521.4	1437.4	413.9
1175.6	1393.2	1447.8	438.6	1412.9	387.1
1118.2	1375.3	1428.0	411.5	1381.7	328.8
1089.4	1358.9	1404.0	383.1	1351.8	257.5
1069.2	1353.3	1363.6	335.8	1298.7	218.6
1037.9	1300.5	1299.7	285.9	1287.0	139.7
993.3	1254.7	1286.5	238.9	1271.7	119.7
933.3	1233.8	1250.7	128.2	1253.7	102.5
908.6	1146.3	1217.6		1232.7	
881.3	1110.0	1209.7		1204.6	
869.6	1087.9	1176.1		1177.1	
826.1	1035.2	1137.2		1148.9	
799.8	979.9				
753.2	930.8				
729.8	910.7				
714.3	886.7				
	859.4				
	835.7				
	807.2				
	753.8				
	735.1				
	710.0				
	658.3				

^{1}H and ^{13}C NMR data for ethylone hydrochloride in CD_3OD.