

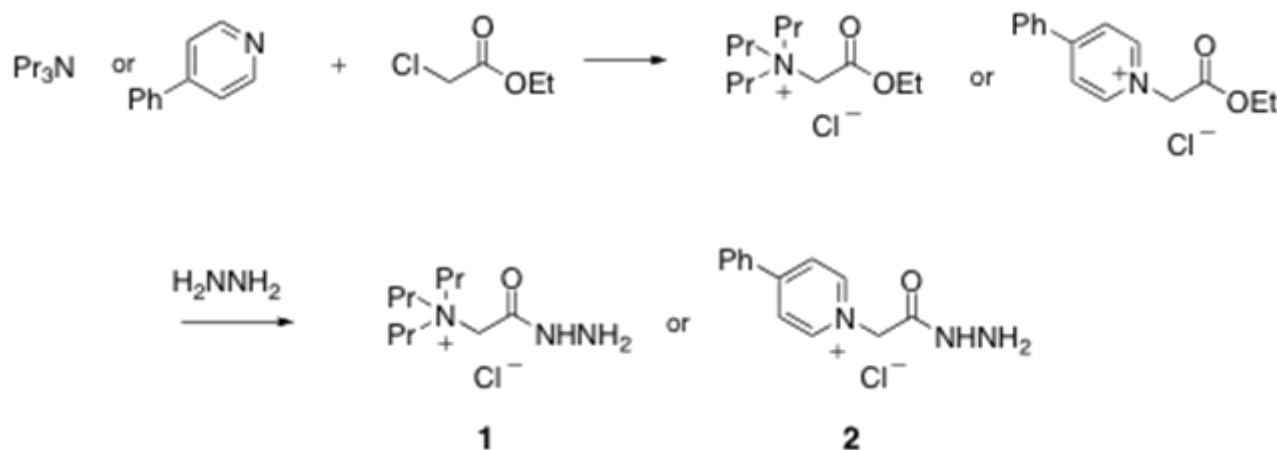
Synthesis of Reagents 1 and 2

Supplementary Material (ESI) for Chemical Communications
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General experimental for Girard Reagent derivatives 1 and 2:

To a solution of ethyl chloroacetate in 10 mL of absolute ethanol at rt was added tripropylamine (10 mmol) (or 4-phenylpyridine, 10 mmol). The mixture was refluxed for 20-24 h and then concentrated under reduced pressure. The residue was washed with hexanes (2 x 10 mL), dried under vacuum, and dissolved in absolute ethanol (10 mL). Hydrazine hydrate (100%, 1.2 equiv) was added with stirring. The mixture stirred at rt for 24 h and then concentrated under vacuum to give derivative **1** as an oil and derivative **2** as a white solid. The products (60-70%) were dried in a vacuum desiccator and used directly.

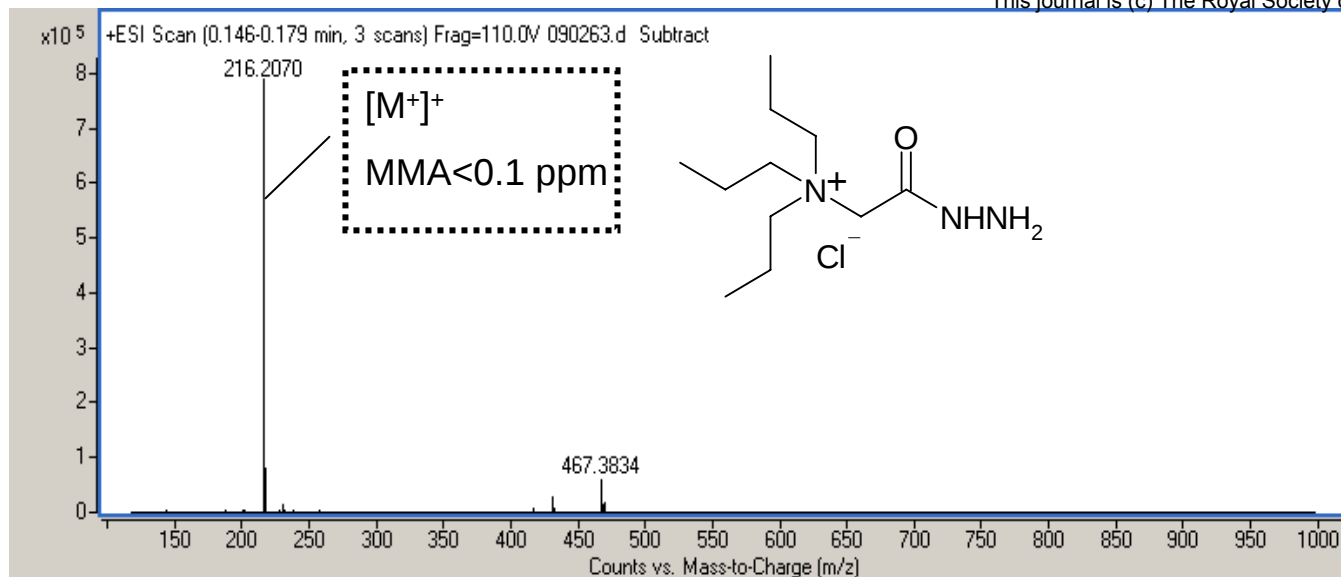
*For a large-scale synthesis of Girard's Reagent T (**3**), see: Girard, A. *Organic Syntheses*, Coll. Vol. 2, **1943**, 85.



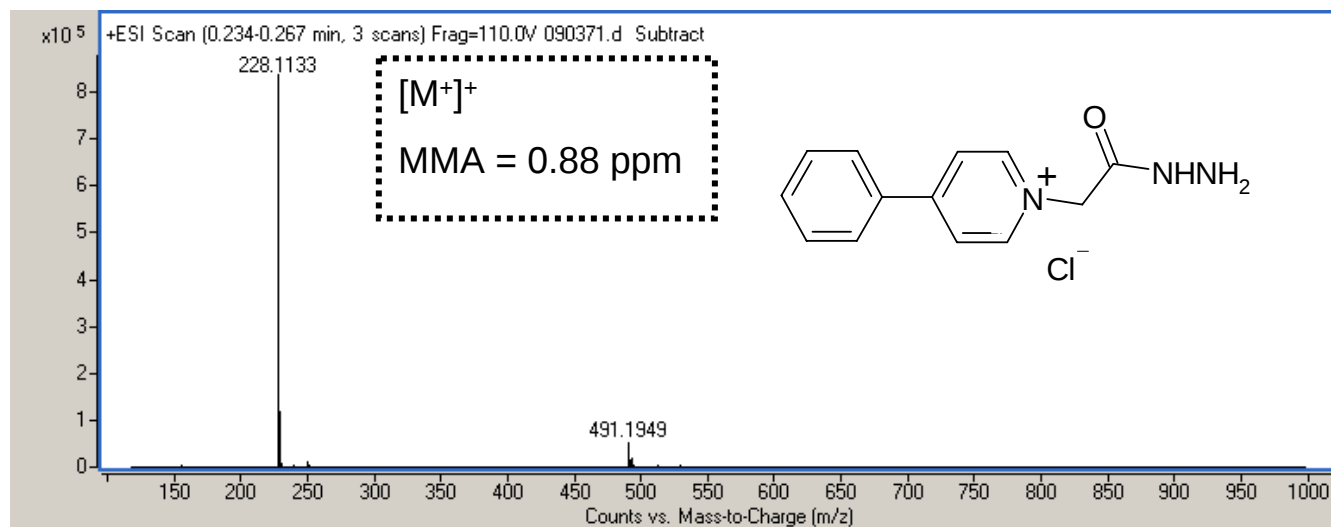
Mass Spectral Data

Reagent 1 (C₁₁H₂₆N₃OCl)

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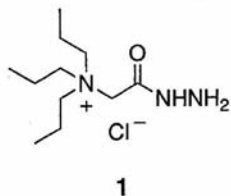


Reagent 2 (C₁₁H₂₆N₃OCl)

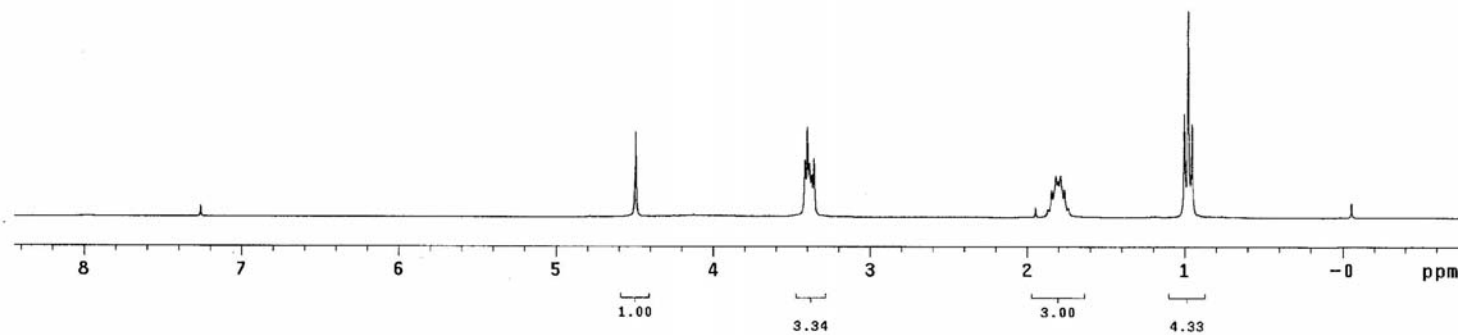


^1H NMR Data Reagent 1

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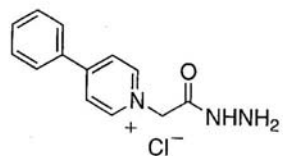


^1H NMR (CDCl_3 , 300 MHz)



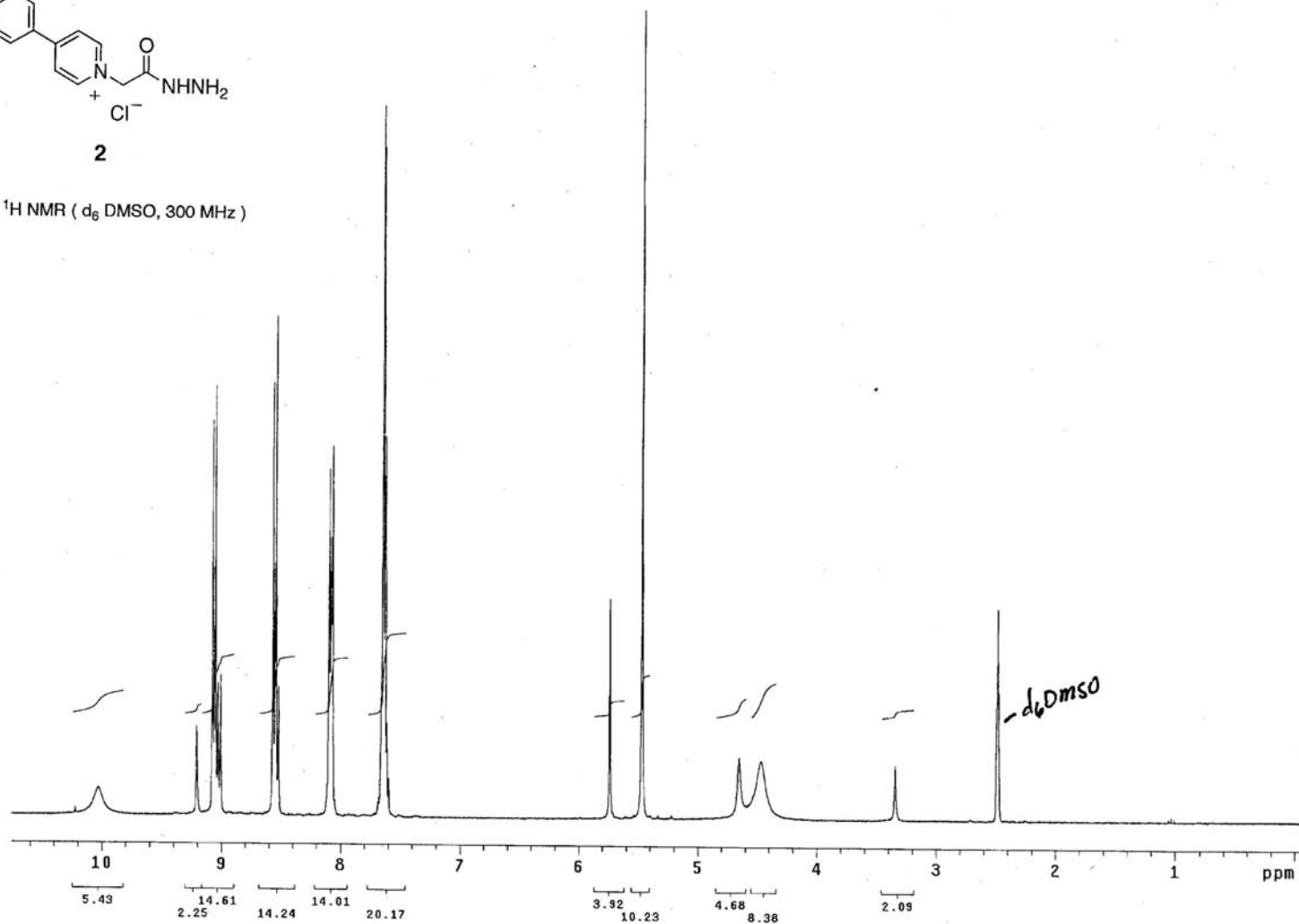
H NMR Data Reagent 2

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2

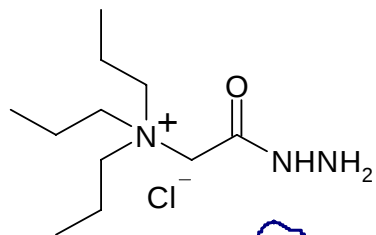
^1H NMR (d_6 DMSO, 300 MHz)



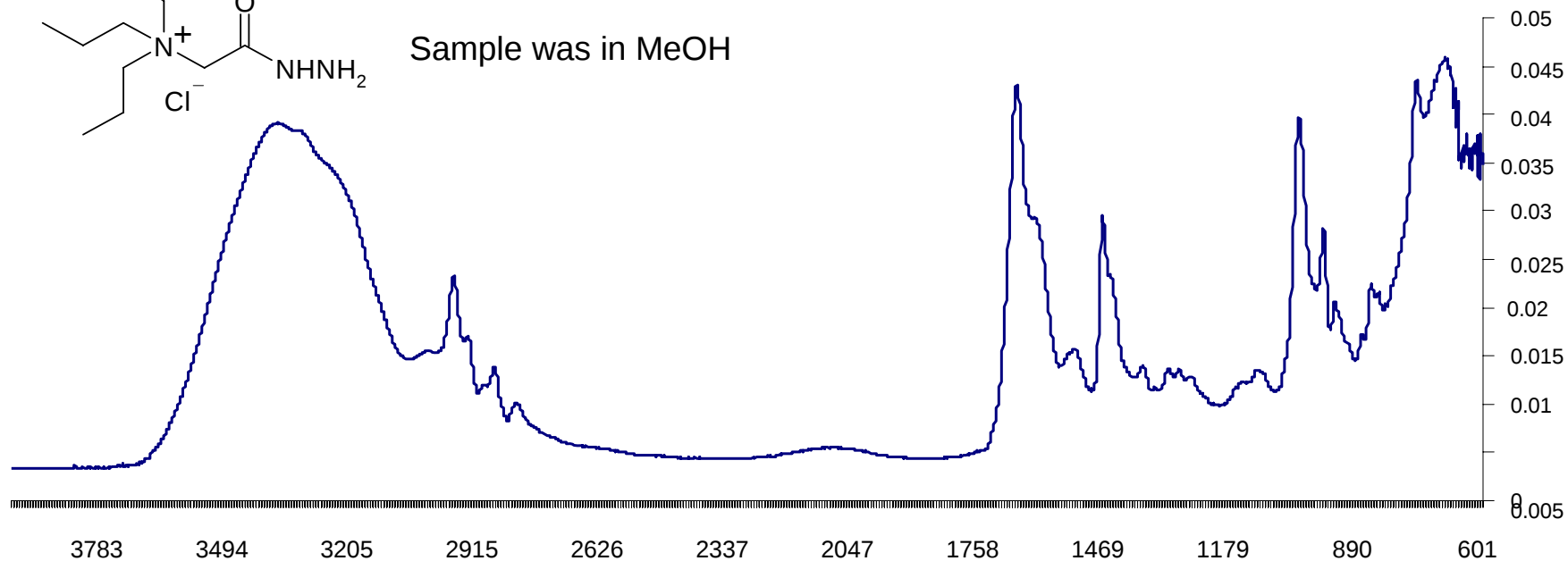
Reagent 1

ATR-IR Spectroscopy

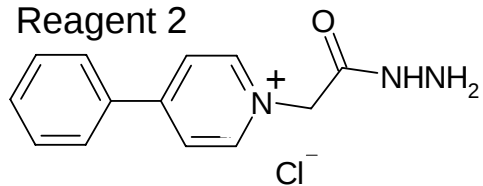
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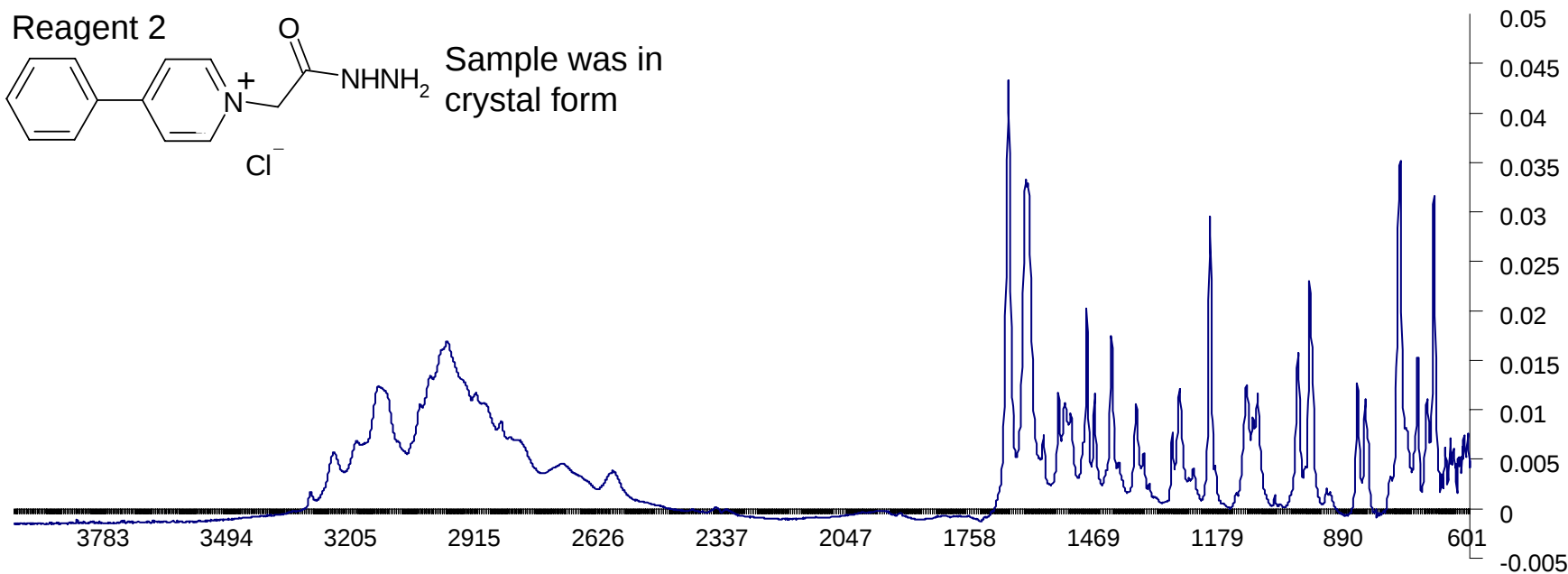
Sample was in MeOH



Reagent 2



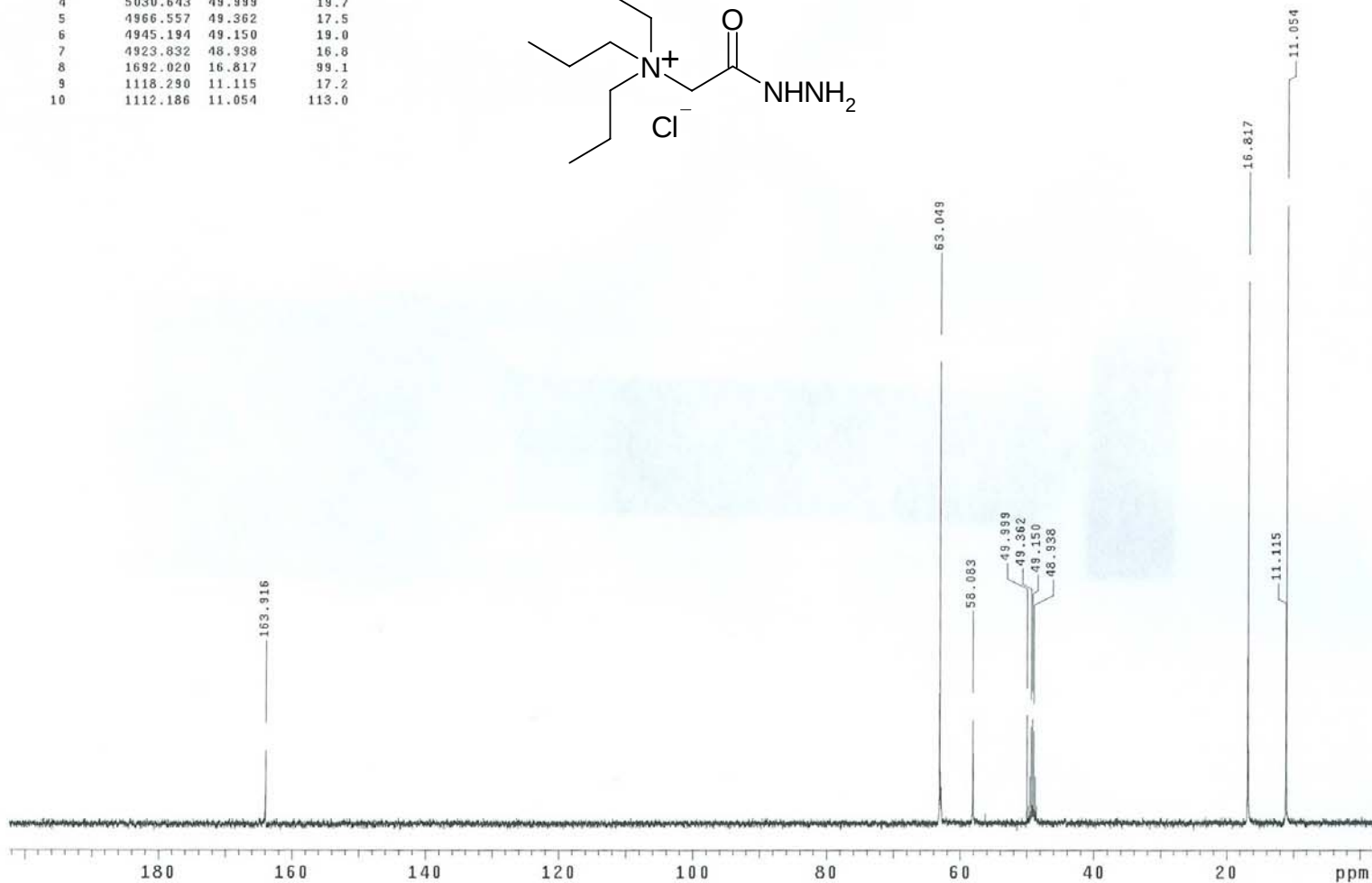
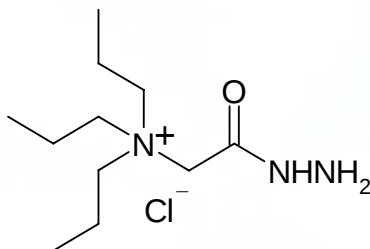
Sample was in crystal form



¹³C NMR Reagent 1

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INDEX	FREQUENCY	PPM	HEIGHT
1	16492.283	163.916	13.2
2	6343.662	63.049	84.5
3	5843.937	58.083	18.8
4	5030.643	49.999	19.7
5	4966.557	49.362	17.5
6	4945.194	49.150	19.0
7	4923.832	48.938	16.8
8	1692.020	16.817	99.1
9	1118.290	11.115	17.2
10	1112.186	11.054	113.0



¹³C NMR Reagent 2

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INDEX	FREQUENCY	PPM	HEIGHT
1	16707.155	166.051	11.0
2	15961.000	158.635	9.0
3	14854.738	147.640	10.9
4	14831.086	147.405	37.4
5	13608.095	135.250	12.0
6	13460.084	133.779	24.3
7	13447.877	133.658	8.7
8	13194.581	131.140	66.7
9	13021.394	129.419	56.1
10	13016.816	129.373	20.4
11	12647.554	125.703	52.1
12	12631.532	125.544	19.4
13	6152.650	61.151	17.0
14	5011.293	49.807	9.6
15	4989.930	49.595	28.0
16	4968.568	49.382	57.8
17	4947.206	49.170	67.7
18	4925.844	48.958	56.8
19	4904.481	48.745	28.7
20	4883.119	48.533	9.3

