Department of Chemistry

University of Missouri-St. Louis

Experiment 9 Identification of Organic Unknowns

This experiment will be due at the end of the semester. You will be given one liquid and three solid unknowns to identify. The preliminary report described in the manual will be provided. This will contain the solubility properties of the unknowns as well as some physical properties, normal boiling points for the liquid and melting points for the solids. The following applies to the properties of the boiling points and melting temperatures provided:

Melting points and boiling points will be provided with an uncertainty. The uncertainty in the melting point values will be ± 4° C; The uncertainty in the boiling temperature will be ± 6° C. **This means you should consider all solid compounds melting within a temperature range of 8° C and for liquid 12° C of the value reported**

A good way of identifying the compounds is to use solubility to classify each compound. Make a list of all the compounds that have melting points or boiling points within the 8 or 12° C range within the classification and then use the spectroscopy to eliminate compounds.

All unknown compounds are included in Appendix 5 available at the bottom of my web page, www.umsl.edu/~chickosj. Also provided below are infrared, proton and carbon NMR spectra of the unknowns. Be sure to review the narrated Powerpoint presentations on spectroscopy and on the use of solubility to classify compounds. Some classification are not unique. You may have to look under different classifications in Appendix 5. Classifications are based on functional groups. Once a positive test is found that should identify the classification

In your report, in addition to identifying the structure of these materials, you should also analyze the spectra, For IR, you should identify the main functional groups and for nmr, you should identity as many protons and carbons as to the type of carbon. Examples include aromatic carbon, methyl group, … You don't have to identify each carbon if they are grouped closely together.

Each unknown is given a number from 1 to 4. In your final report, be sure to identify each unknown you are describing. The unknown spectra, the preliminary reports and the spectra are provided below. Also included are copies of the final report. Additional ones are available on my web page.

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University of Missouri-St. Louis

Liquid Unknown
Chemistry 263 Preliminary Report Form
Unknown Number __1____

Physical Properties

a) appearance: liquid

b) melting point:

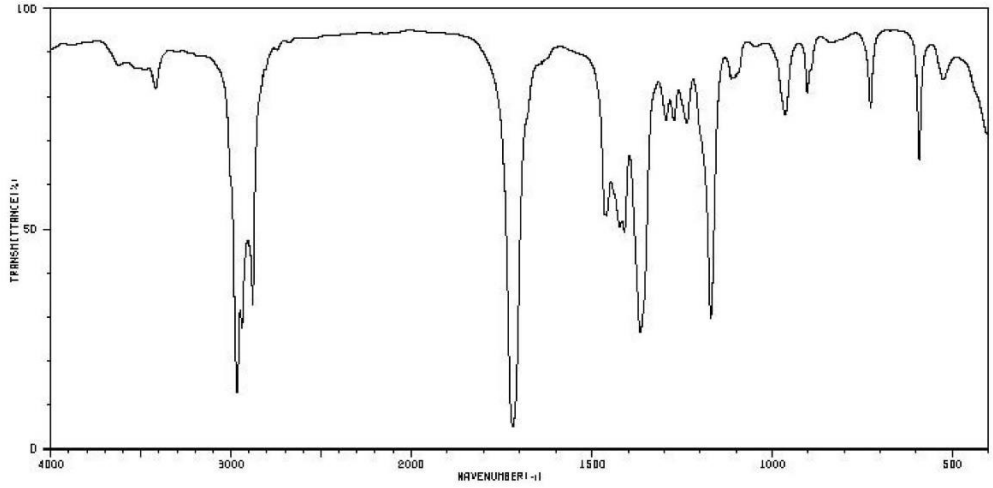
c) boiling point: 103 ° C

Solubility Tests (use +/- to indicate positive, negative tests)

H_2O: __-___; 5%NaOH, __-___; 5%$NaHCO_3$, __-___; 5%HCl, _-___ ; H_2SO_4,__+___.

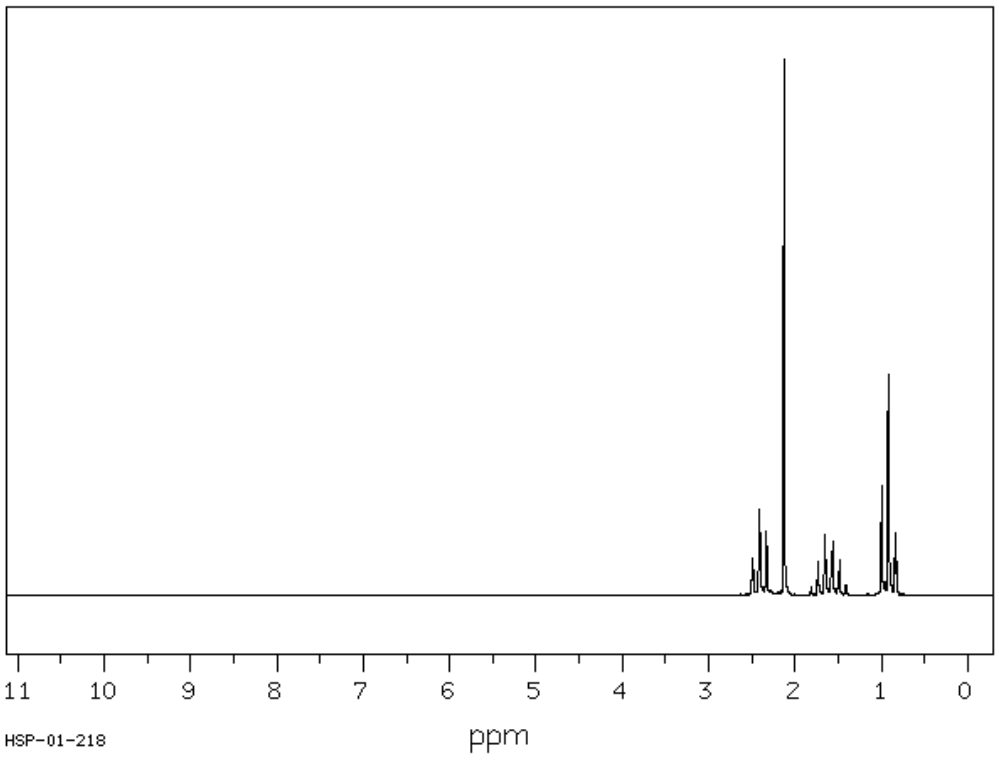
pH(aqueous solution): __~7___

Unknown. 1 IR

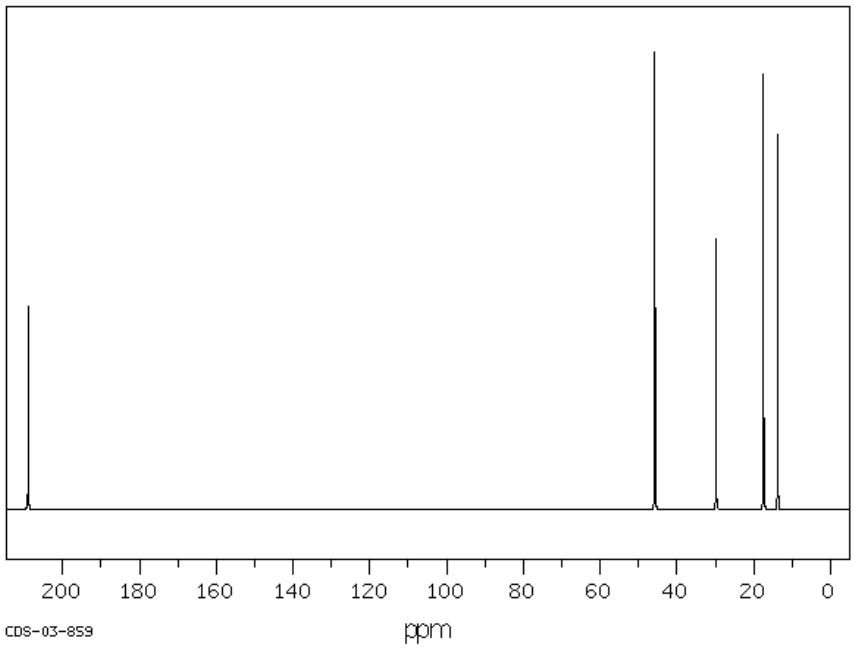


3419	79	1426	48	1114	81	692	64
2966	12	1412	47	1105	81	526	81
2939	26	1367	25	1100	81		
2879	31	1296	72	964	72		
1717	4	1274	72	903	79		
1467	50	1237	72	894	81		
1461	50	1172	28	727	74		

Unknown 1. H NMR Area left to right: 2, 3, 2, 3



Unknown 1. ^{13}C NMR: Chemical shifts from left to right 210, 45, 30, 18. 14.



Solid Unknown

Chemistry 263 Preliminary Report Form

Unknown Number __2____

Physical Properties

a) appearance: solid

b) melting point: 47 °C

c) boiling point: 298 °C

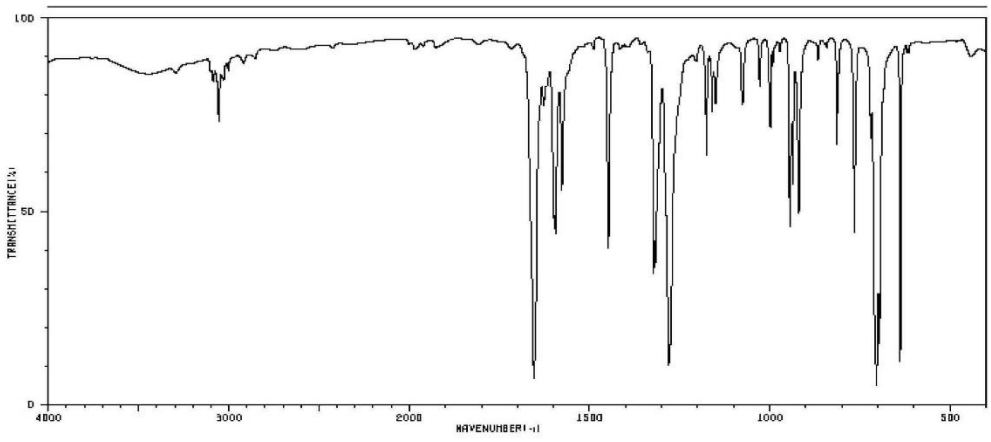
e) other properties you may have measured:

Solubility Tests (use +/- to indicate positive, negative tests)

H_2O: __-___; 5%NaOH, __-___; 5%$NaHCO_3$, __-___; 5%HCl, _-___ ; H_2SO_4,__+___.

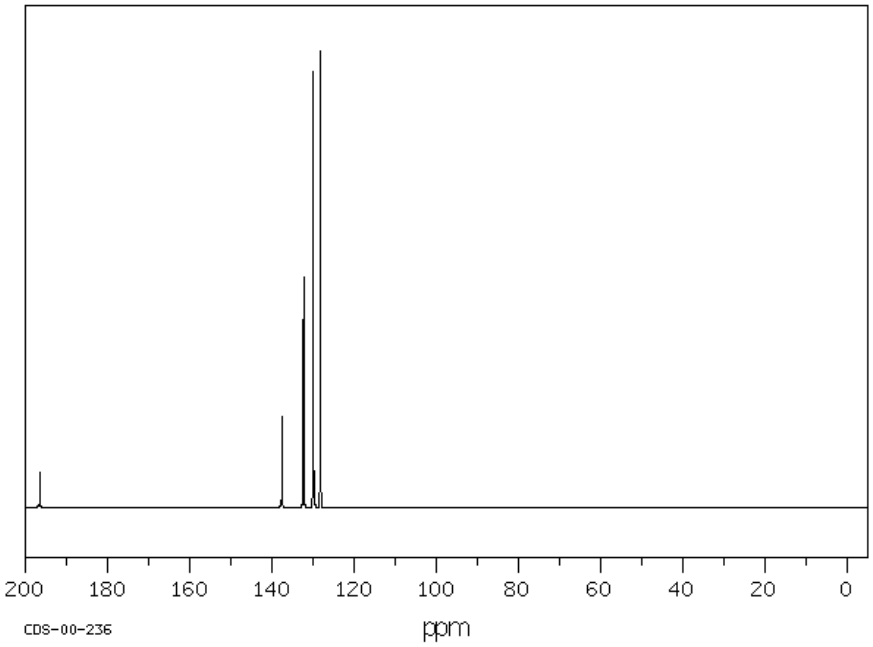
pH(aqueous solution): __~7___

Unknown 2. IR

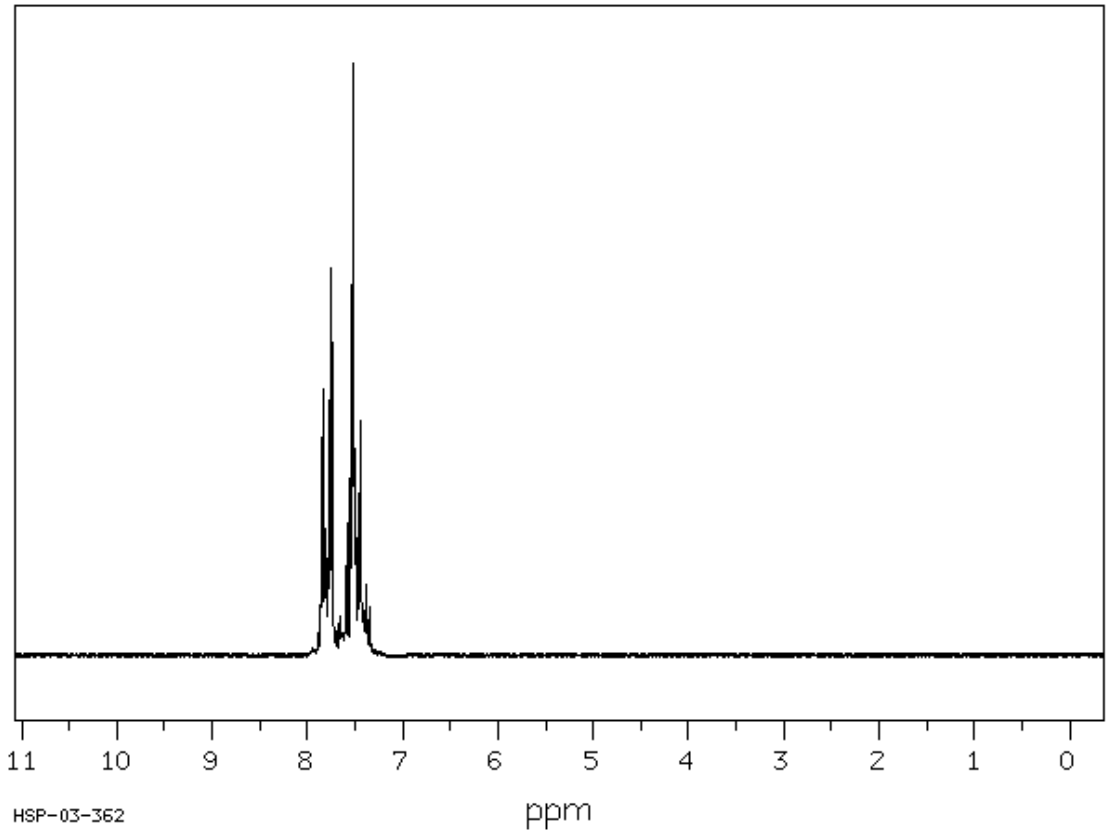


3291	81	1656	6	1205	86	992	84	720	66
3088	81	1627	74	1176	82	945	44	705	4
3067	79	1595	42	1162	72	936	55	697	15
3057	70	1577	69	1151	74	918	47	639	10
3030	81	1449	38	1076	74	867	86		
3005	84	1323	52	1028	79	814	64		
2919	84	1281	10	999	88	766	42		

Unknown 2. ^{13}C NMR From left to right: 198, 139, 132, 130 128 ppm



Unknown 2. H NMR Left to right: area ratio 2: 3



Solid Unknown Preliminary Report Form

Unknown Number __3____

Physical Properties

a) appearance: solid

b) melting point: 132 °C

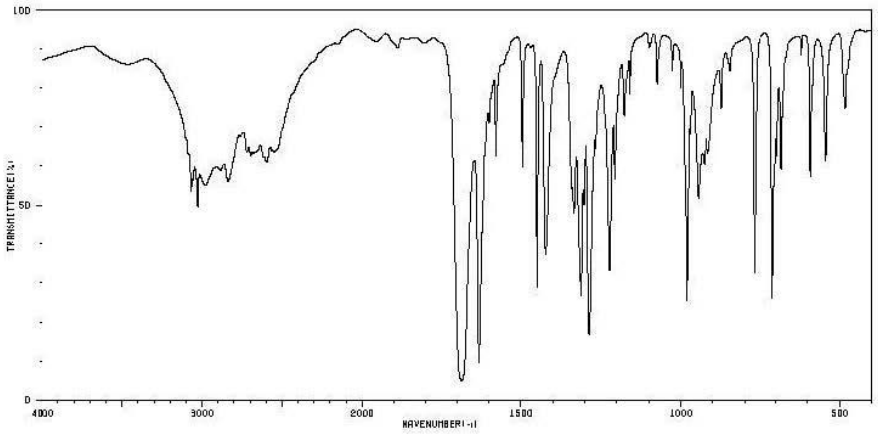
c) boiling point:

Solubility Tests (use +/- to indicate positive / negative tests)

H_2O: __-___; 5%NaOH, __+___; 5%$NaHCO_3$, __+___; 5%HCl, __-__ ; H_2SO_4,_____.

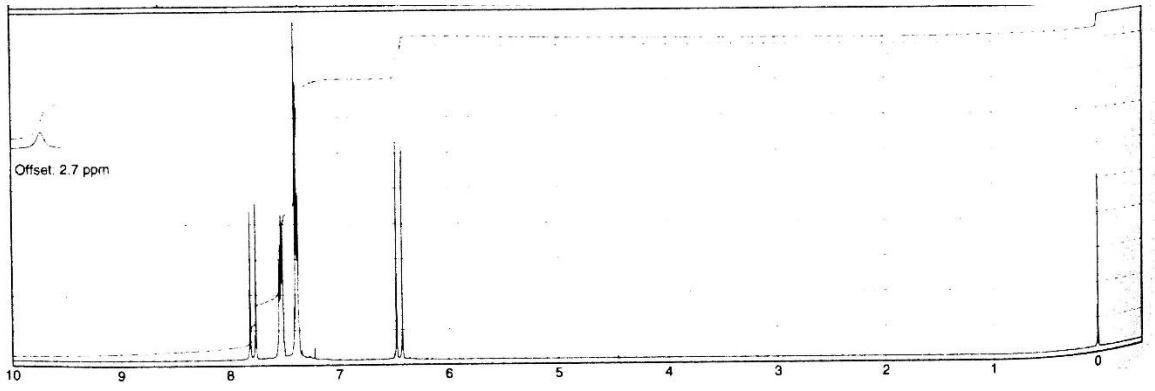
pH(aqueous solution): __~2

Unknown 3. IR

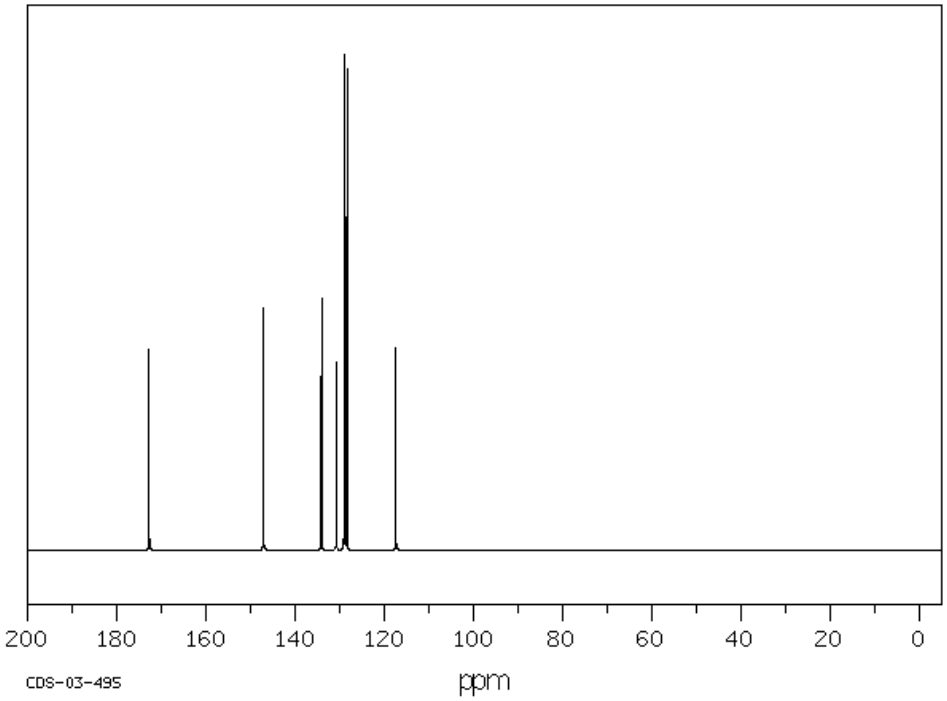


3067	52	2671	60	1496	57	1288	16	917	50
3027	47	2614	60	1450	27	1224	31	769	31
2977	53	2598	58	1423	35	1207	55	712	25
2837	53	2560	60	1341	52	987	66	698	60
2720	60	1686	4	1335	46	982	24	684	57
2696	60	1631	9	1312	26	945	50	592	55
2683	60	1578	60	1303	47	926	58	544	58

Unknown 3. H NMR: The peak at 0 ppm is tetramethylsilane (standard); the chemical shift that is offset 2.7 ppm is 12.7 ppm. The area ratio left to right starting with 12.7 ppm is: 1, 1, 2, 3, 1.



Unknown 3, ^{13}C NMR: Chemical shifts: 172.7, 147.0, 133.9, 130.7, 128.9, 117.3



Solid Unknown

Chemistry 263 Preliminary Report Form

Unknown Number __4____

Physical Properties

a) appearance: solid

b) melting point: 40 °C

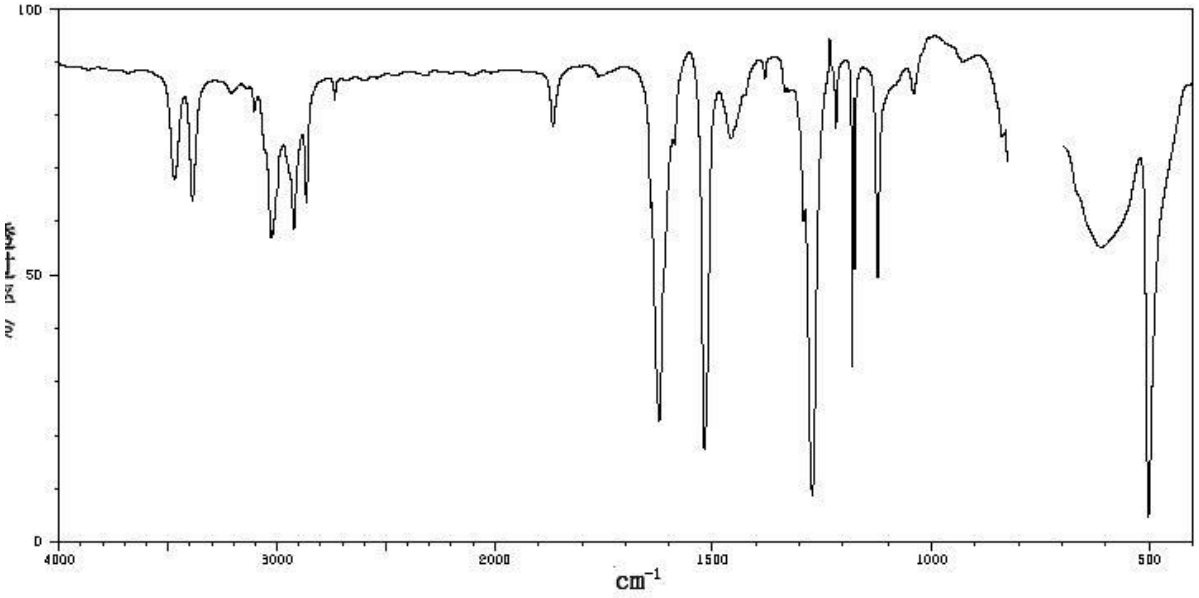
c) boiling point: 195 °C

Solubility Tests (use +/- to indicate positive, negative tests)

H_2O: __-___; 5%NaOH, __-___; 5%$NaHCO_3$, __-___; 5%HCl, _+___ ; H_2SO_4,__+___.

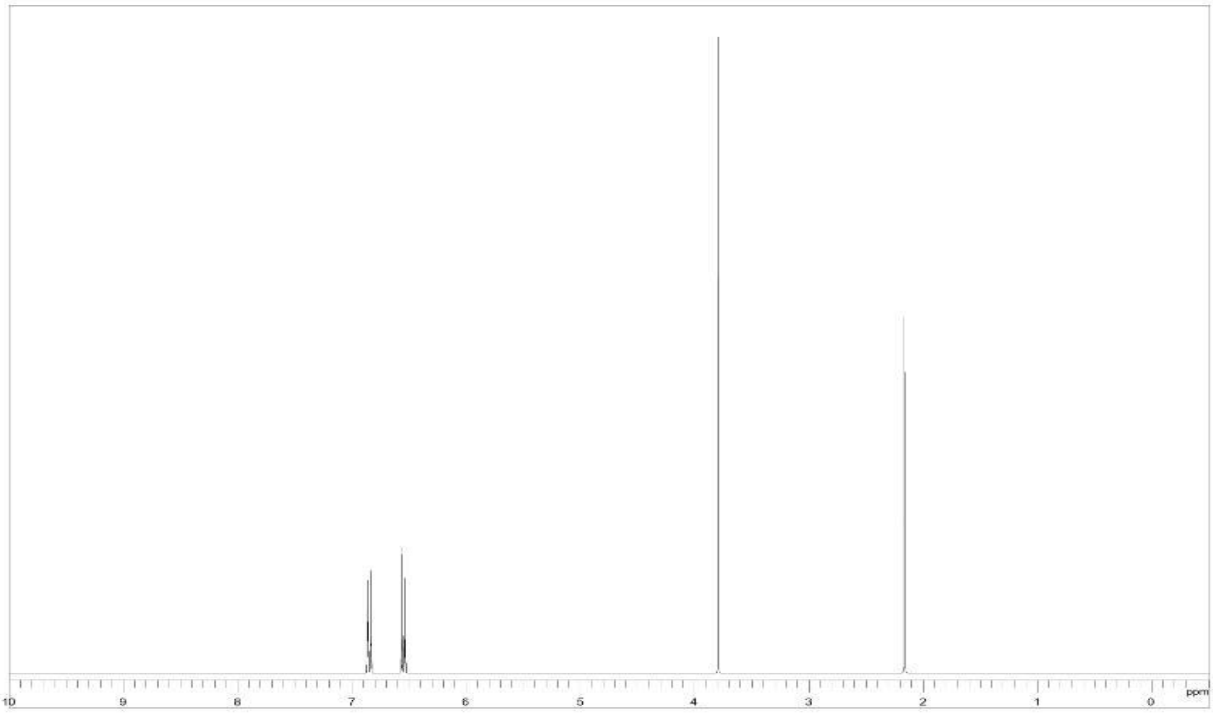
pH (aqueous solution): __~9___ .

Unknown 4. IR



3470	66	2866	62	1291	68	831	72
3388	62	2735	79	1271	8	611	53
3208	81	1868	74	1216	74	502	4
3105	79	1622	22	1179	32		
3029	55	1519	17	1125	49		
3017	55	1457	72	1041	81		
2923	57	1380	84	839	74		

Unknown 4. H NMR: Area ratio from left to right: 2, 2, 3, 2



Unknown 4. ^{13}C NMR: Chemical shifts from left to right 148, 135, 130, 115, 20 ppm