Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Synthesis of Chiral Sulfoximines Derived from 3-Aminoquinazolinones and Their Catalysis of Enantioselective Diethylzinc Addition to Aldehydes

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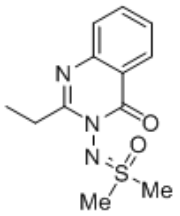

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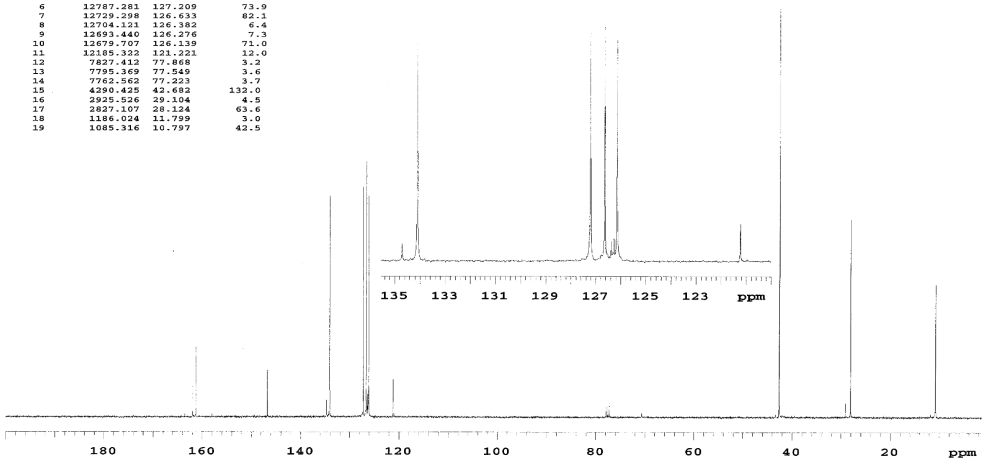
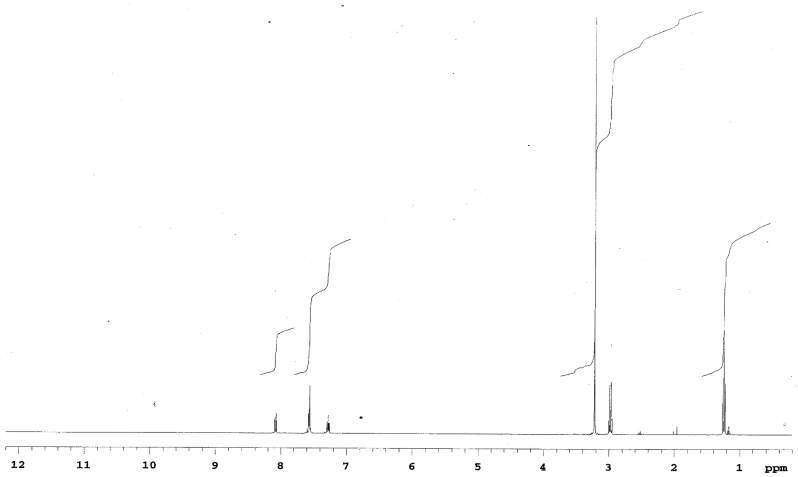
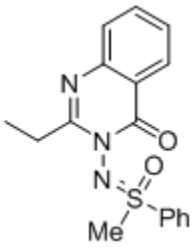
1	16283.833	161.994	11.5
2	16215.168	161.311	22.4
3	14751.088	146.746	14.9
4	13541.066	134.708	5.4
5	13480.030	134.101	71.1
6	12787.281	127.209	73.9
7	12729.298	126.633	82.1
8	12704.121	126.382	6.4
9	12693.440	126.276	7.3
10	12679.707	126.139	71.0
11	12185.322	121.221	12.0
12	7827.412	77.868	3.2
13	7795.369	77.549	3.6
14	7762.562	77.223	3.7
15	4290.425	42.682	132.0
16	2925.526	29.104	4.5
17	2827.107	28.124	63.6
18	1186.024	11.799	3.0
19	1085.316	10.797	42.5





^{1}H and ^{13}C NMR spectra of Sulfoximine **14**



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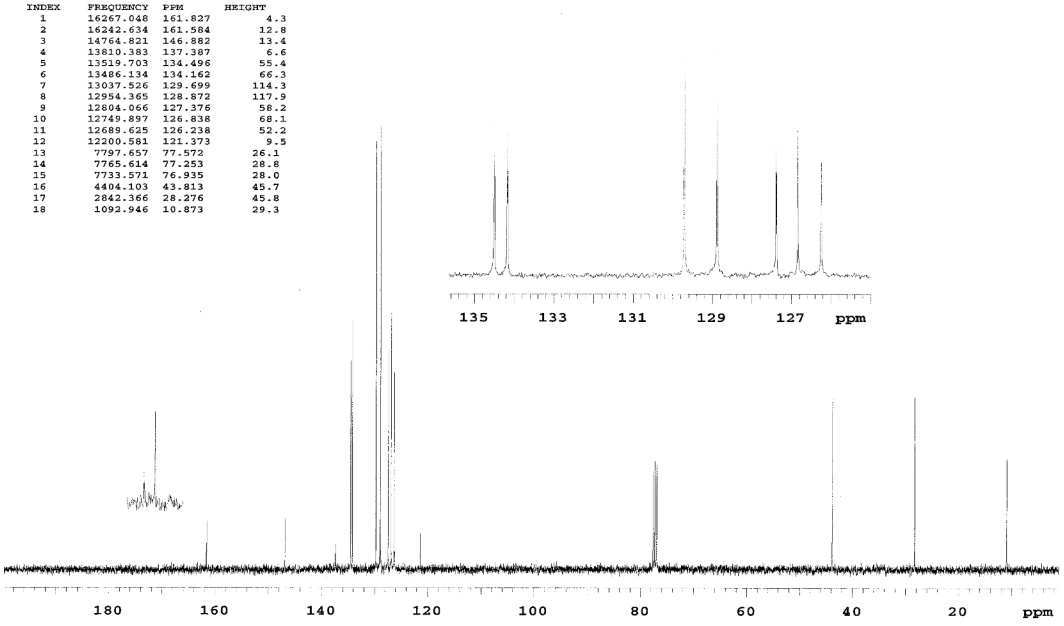
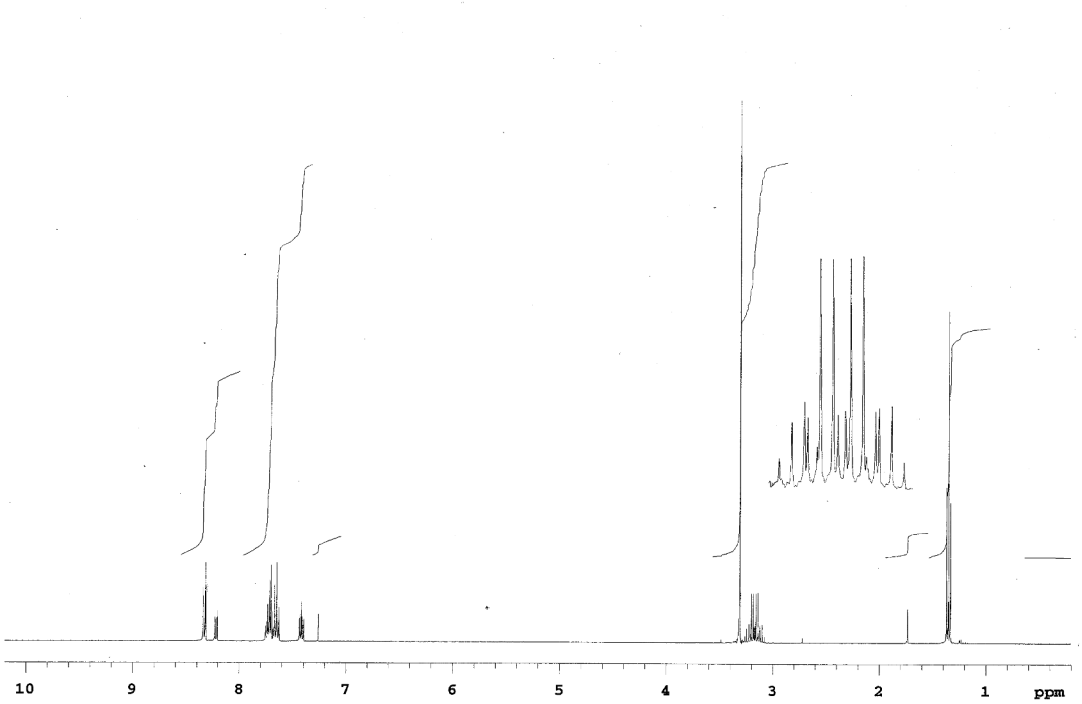
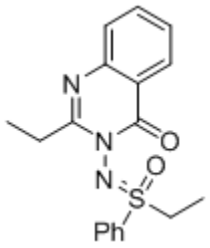
1	16267.048	161.827	4.3
2	16242.634	161.584	12.8
3	14764.821	146.882	13.4
4	13810.383	137.387	6.6
5	13519.703	134.496	55.4
6	13486.134	134.162	66.3
7	13037.526	129.699	114.3
8	12954.365	128.872	117.9
9	12804.066	127.376	58.2
10	12749.897	126.838	68.1
11	12689.625	126.238	52.2
12	12200.581	121.373	9.5
13	7797.657	77.572	26.1
14	7765.614	77.253	28.8
15	7733.571	76.935	28.0
16	4404.103	43.813	45.7
17	2842.366	28.276	45.8
18	1092.946	10.873	29.3





^{1}H and ^{13}C NMR spectra of Sulfoximine **17a**



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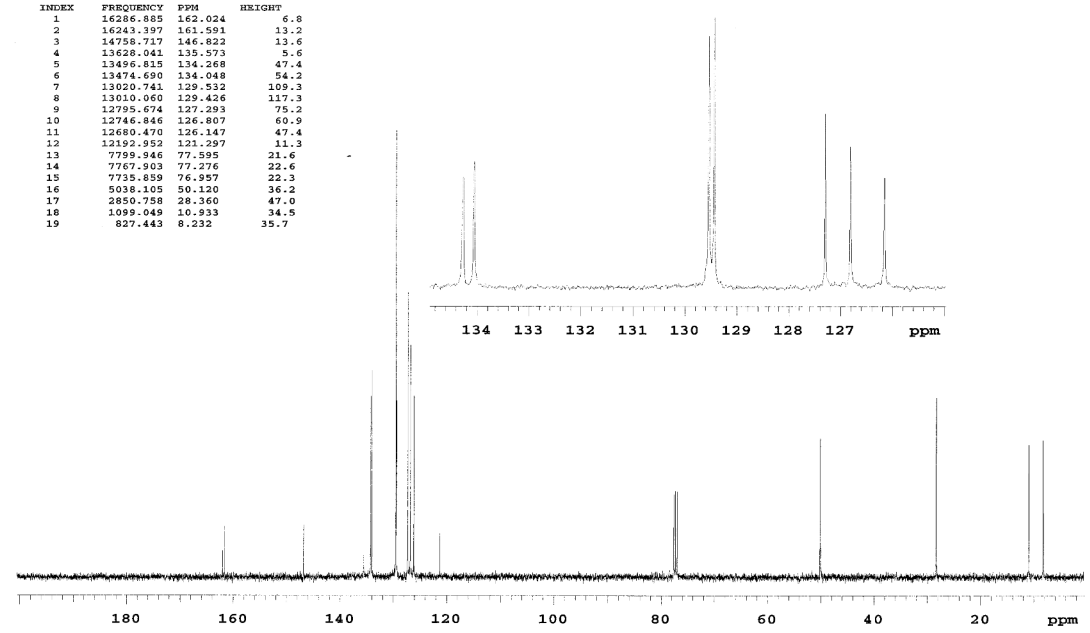
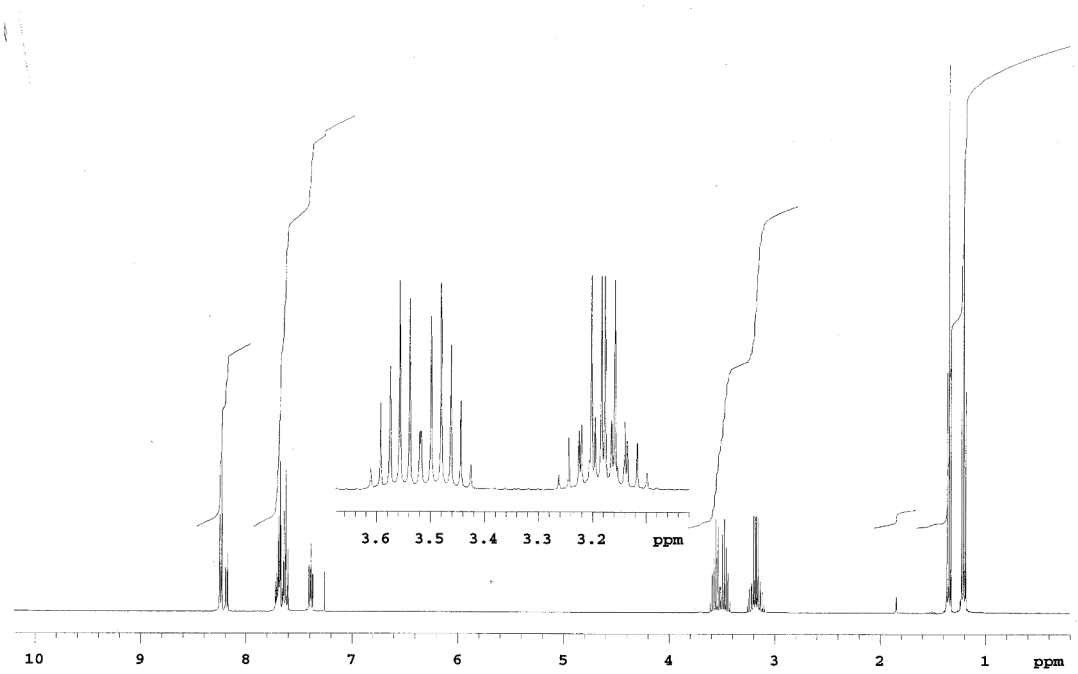
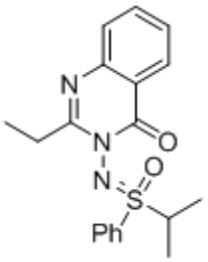
3	14758.717	146.822	13.6
4	13628.041	135.573	5.6
5	13496.815	134.268	47.4
6	13474.690	134.048	54.2
7	13020.741	129.532	109.3
8	13010.060	129.426	117.3
9	12795.674	127.293	75.2
10	12746.846	126.807	60.9
11	12680.470	126.147	47.4
12	12192.952	121.297	11.3
13	7799.946	77.595	21.6
14	7767.903	77.276	22.6
15	7735.859	76.957	22.3
16	5038.105	50.120	36.2
17	2850.758	28.360	47.0
18	1099.049	10.933	34.5
19	827.443	8.232	35.7

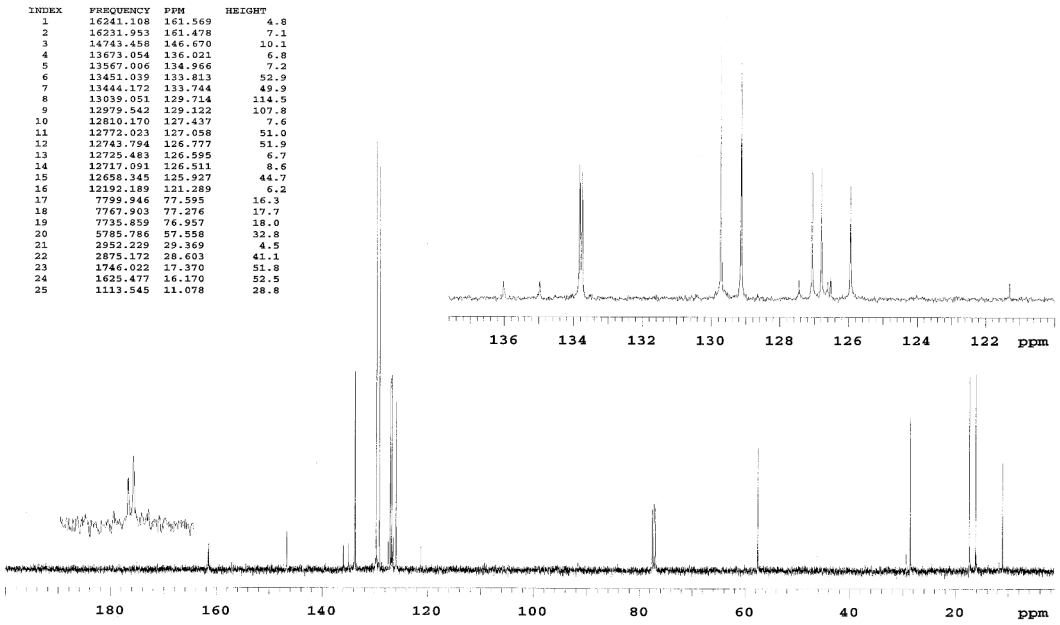


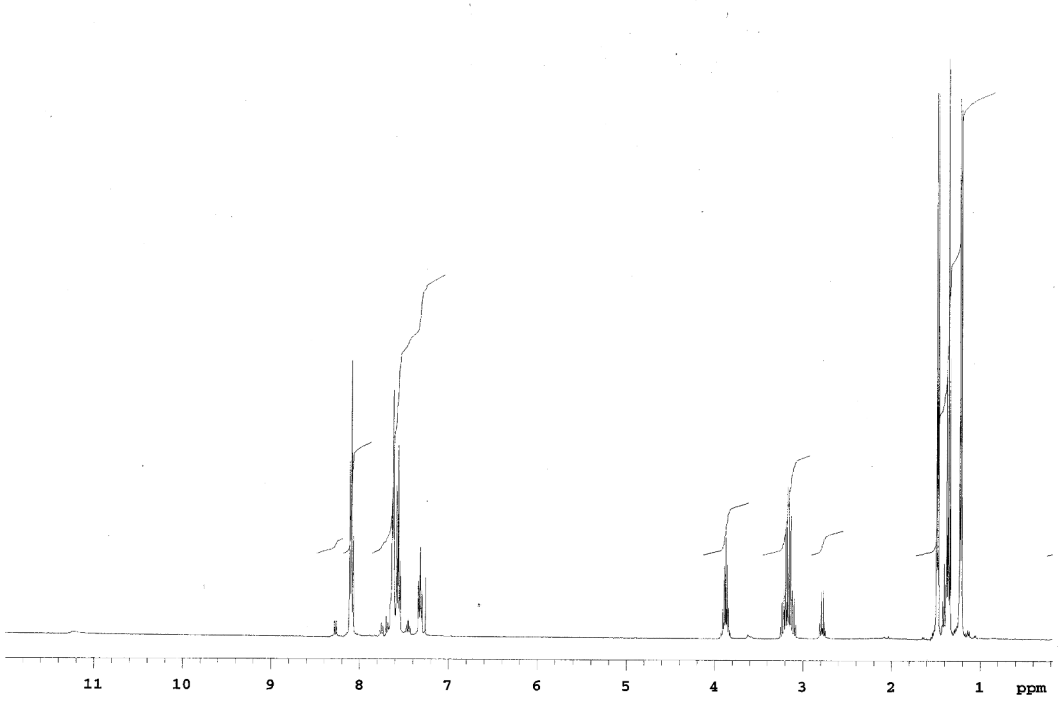


^{1}H and ^{13}C NMR spectra of Sulfoximine **17b**

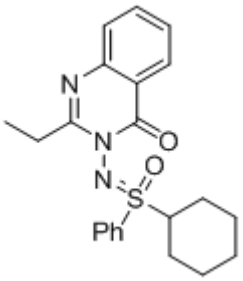


INDEX	FREQUENCY	PPM	HEIGHT
1	16241.108	161.569	4.8
2	16231.953	161.478	7.1
3	14743.458	146.670	10.1
4	13673.054	136.021	6.8
5	13567.006	134.966	7.2
6	13451.039	133.813	52.9
7	13444.172	133.744	49.9
8	13039.051	129.714	114.5
9	12979.542	129.122	107.8
10	12810.170	127.437	7.6
11	12772.023	127.058	51.0
12	12743.794	126.777	51.9
13	12725.483	126.595	6.7
14	12717.091	126.511	8.6
15	12658.345	125.927	44.7
16	12192.189	121.289	6.2
17	7799.946	77.595	16.3
18	7767.903	77.276	17.7
19	7735.859	76.957	18.0
20	5785.786	57.558	32.8
21	2952.229	29.369	4.5
22	2875.172	28.603	41.1
23	1746.022	17.370	51.8
24	1625.477	16.170	52.5
25	1113.545	11.078	28.8





^{1}H and ^{13}C NMR spectra of Sulfoximine **17c**



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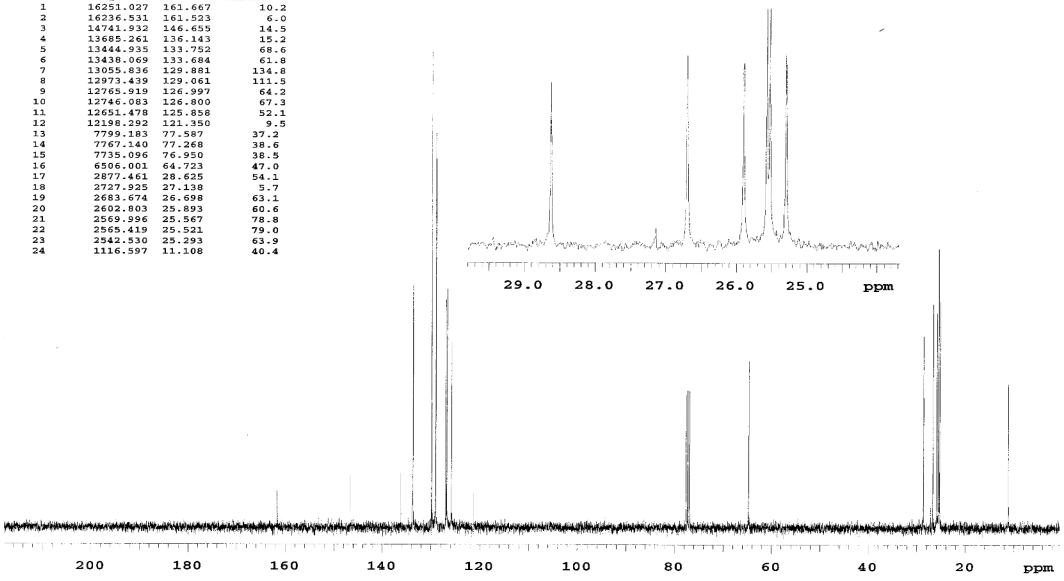
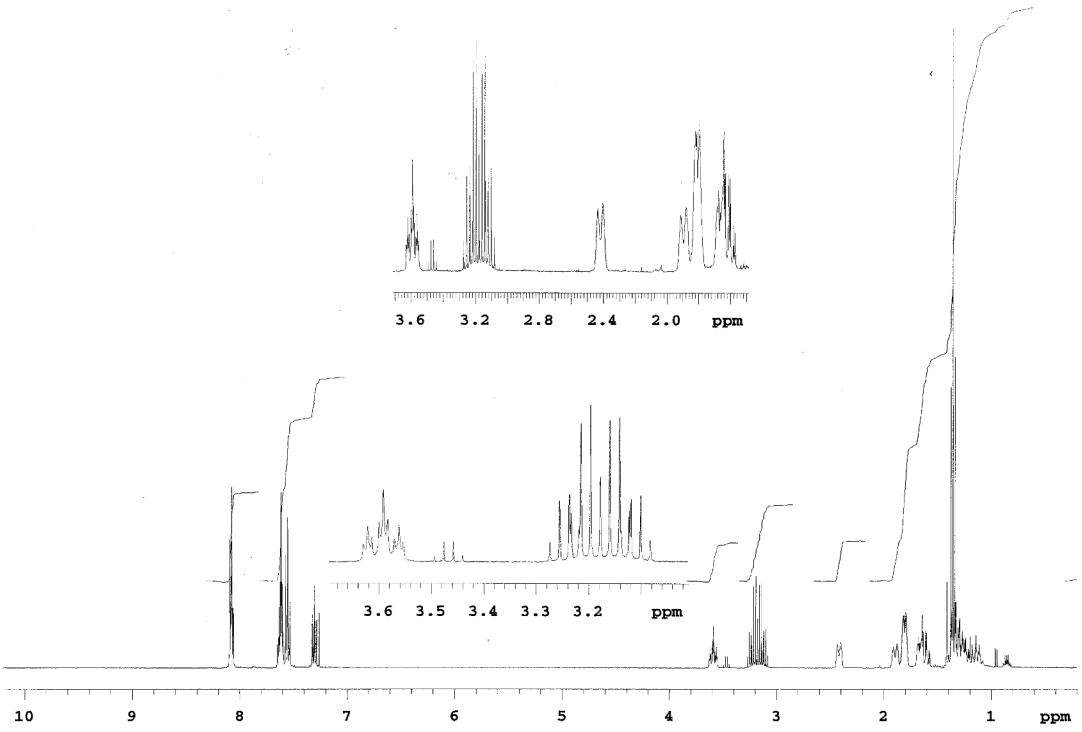
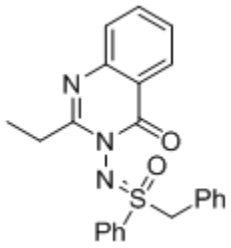
1	16251.027	161.667	10.2
2	16236.531	161.523	6.0
3	14741.932	146.655	14.5
4	13685.261	136.143	15.2
5	13444.935	133.752	68.6
6	13438.069	133.684	61.8
7	13055.836	129.881	134.8
8	12973.439	129.061	111.5
9	12765.919	126.997	64.2
10	12746.083	126.800	67.3
11	12651.478	125.858	52.1
12	12198.292	121.350	9.5
13	7799.183	77.587	37.2
14	7767.140	77.268	38.6
15	7735.096	76.950	38.5
16	6506.001	64.723	47.0
17	2877.461	28.625	54.1
18	2727.925	27.138	5.7
19	2683.674	26.698	63.1
20	2602.803	25.893	60.6
21	2569.996	25.567	78.8
22	2565.419	25.521	79.0
23	2542.530	25.293	63.9
24	1116.597	11.108	40.4

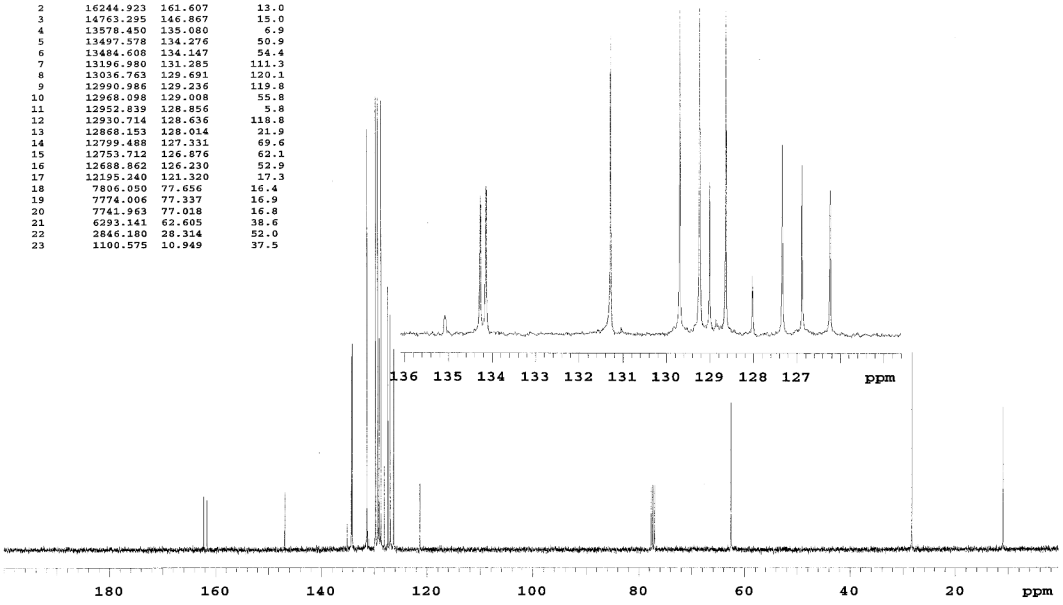


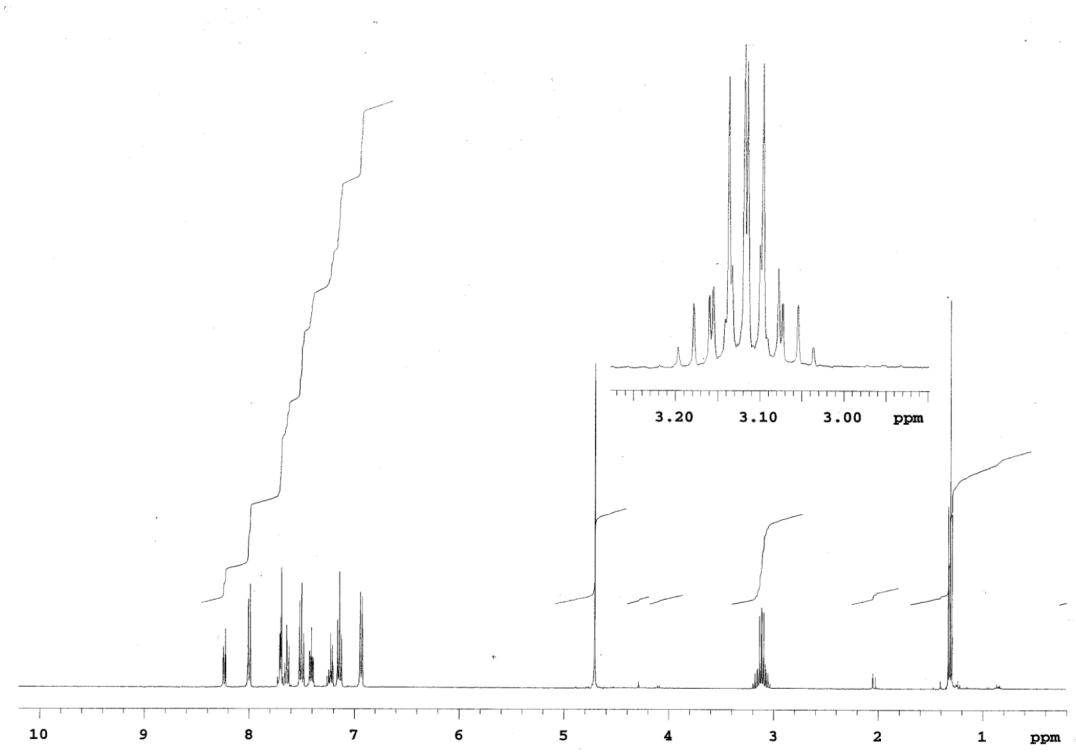


^{1}H and ^{13}C NMR spectra of Sulfoximine **17f**

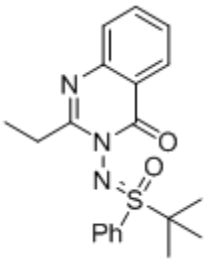


INDEX	FREQUENCY	PPM	HEIGHT
1	16306.721	162.221	14.0
2	16244.923	161.607	13.0
3	14763.295	146.867	15.0
4	13578.450	135.080	6.9
5	13497.578	134.276	50.9
6	13484.608	134.147	54.4
7	13196.980	131.285	111.3
8	13036.763	129.691	120.1
9	12990.986	129.236	119.8
10	12968.098	129.008	55.8
11	12952.839	128.856	5.8
12	12930.714	128.636	118.8
13	12868.153	128.014	21.9
14	12799.488	127.331	69.6
15	12753.712	126.876	62.1
16	12688.862	126.230	52.9
17	12195.240	121.320	17.3
18	7806.050	77.656	16.4
19	7774.006	77.337	16.9
20	7741.963	77.018	16.8
21	6293.141	62.605	38.6
22	2846.180	28.314	52.0
23	1100.575	10.949	37.5

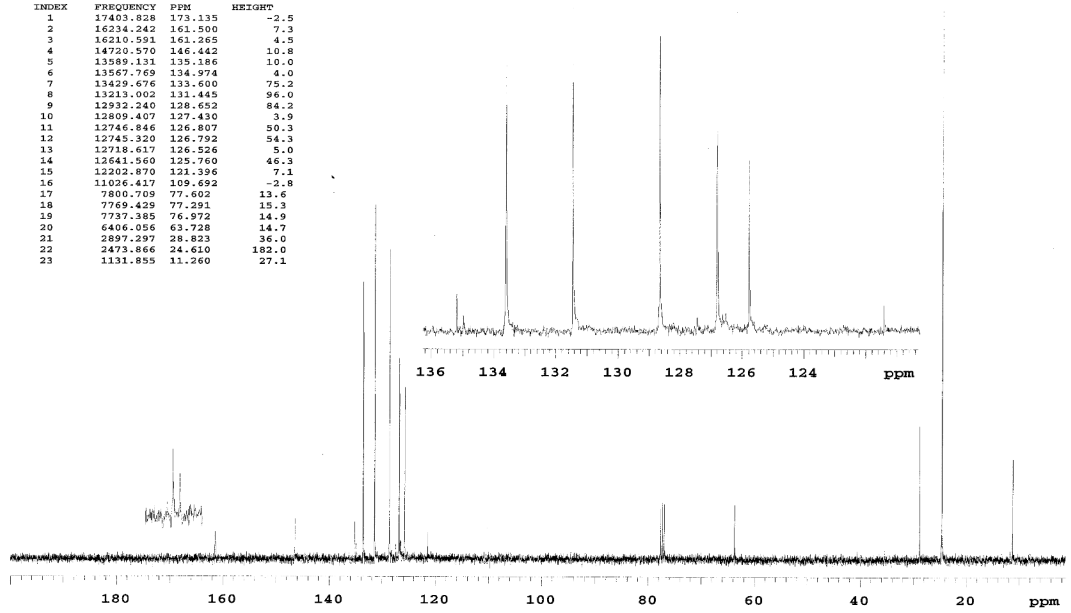


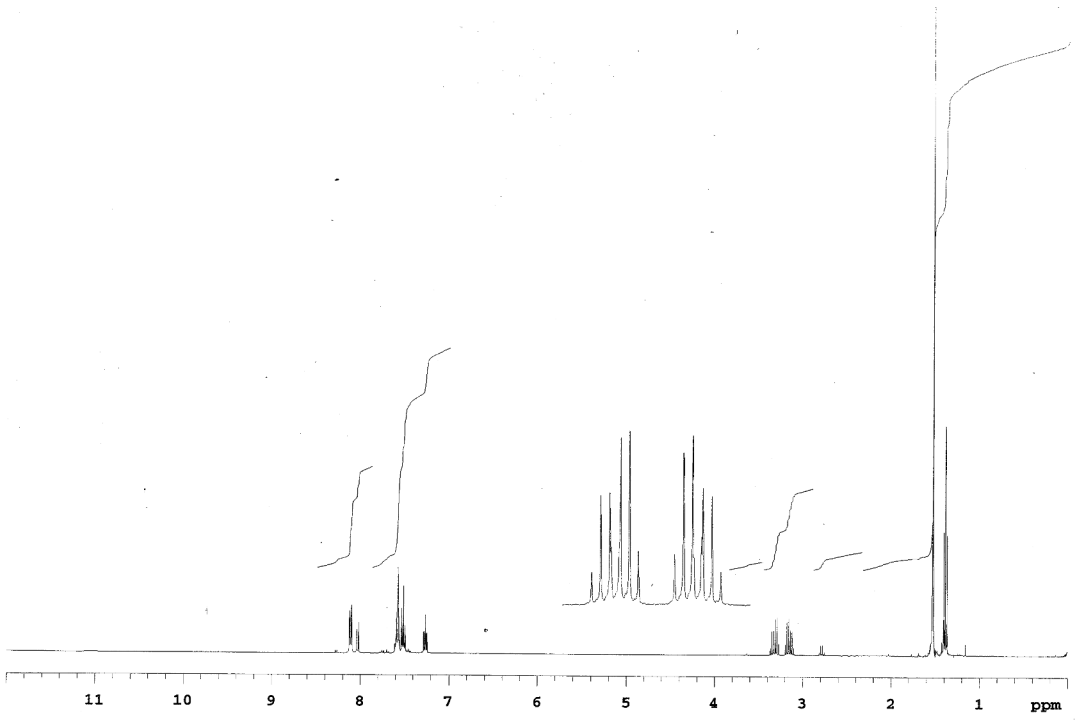


^{1}H and ^{13}C NMR spectra of Sulfoximine **17e**

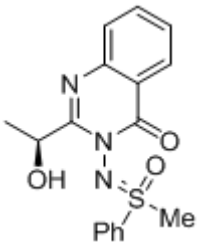


INDEX	FREQUENCY	PPM	HEIGHT
1	17403.828	173.135	-2.5
2	16234.242	161.500	7.3
3	16210.591	161.265	4.5
4	14720.570	146.442	10.8
5	13589.131	135.186	10.0
6	13567.769	134.974	4.0
7	13429.676	133.600	75.2
8	13213.002	131.445	96.0
9	12932.240	128.652	84.2
10	12809.407	127.430	3.9
11	12746.846	126.807	50.3
12	12745.320	126.792	54.3
13	12718.617	126.526	5.0
14	12641.560	125.760	46.3
15	12202.870	121.396	7.1
16	11026.417	109.692	-2.8
17	7800.709	77.602	13.6
18	7769.429	77.291	15.3
19	7737.385	76.972	14.9
20	6406.056	63.728	14.7
21	2897.297	28.823	36.0
22	2473.866	24.610	182.0
23	1131.855	11.260	27.1

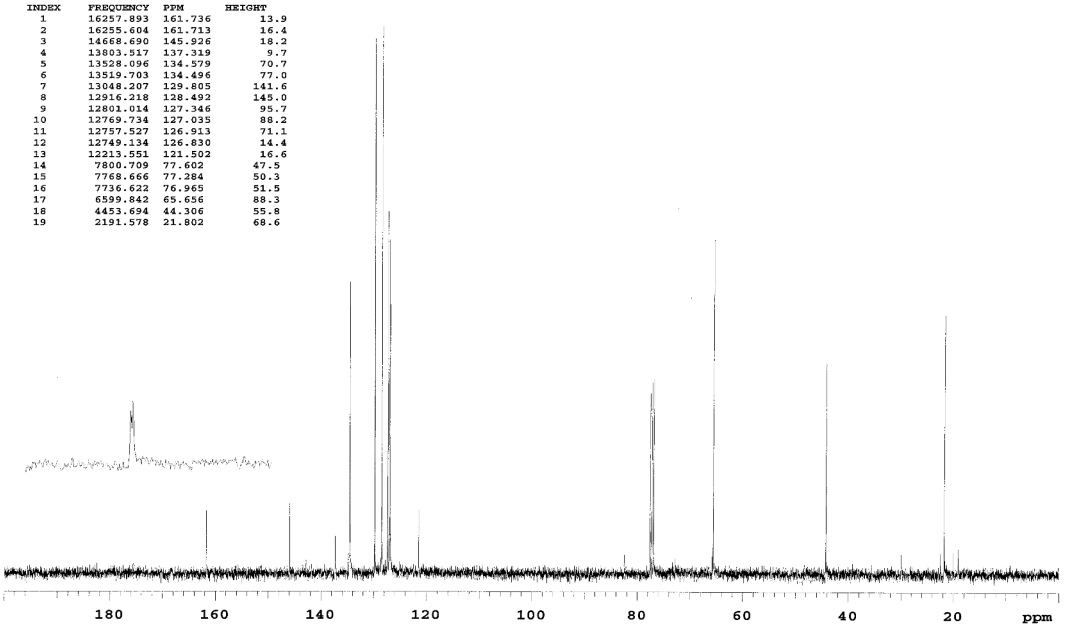


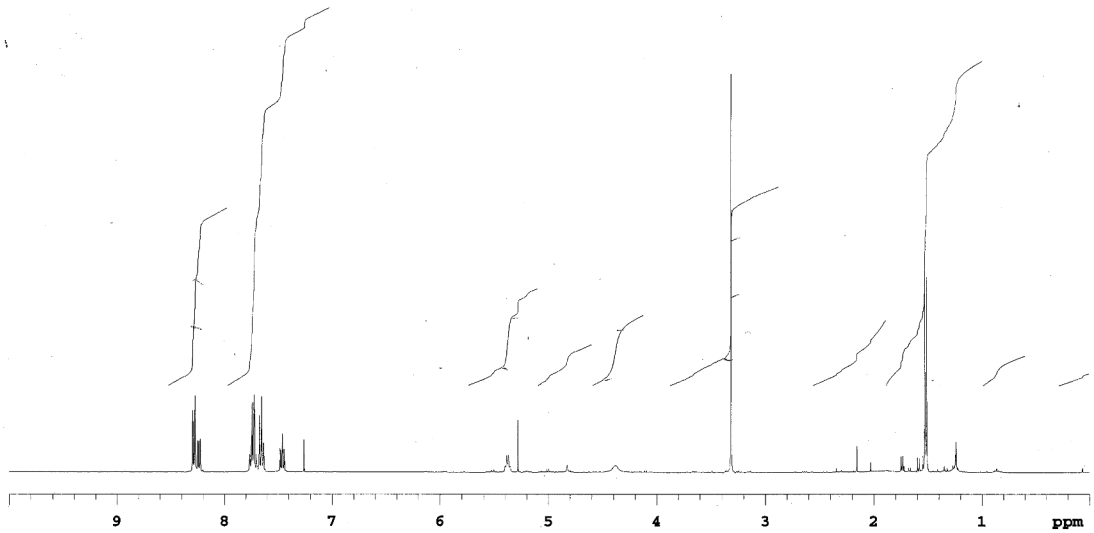


^{1}H and ^{13}C NMR spectra of Sulfoximine **17d**

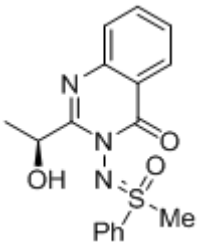


INDEX	FREQUENCY	PPM	HEIGHT
1	16257.893	161.736	13.9
2	16255.604	161.713	16.4
3	14668.690	145.926	18.2
4	13803.517	137.319	9.7
5	13528.096	134.579	70.7
6	13519.703	134.496	77.0
7	13048.207	129.805	141.6
8	12916.218	128.492	145.0
9	12801.014	127.346	95.7
10	12769.734	127.035	88.2
11	12757.527	126.913	71.1
12	12749.134	126.830	14.4
13	12213.551	121.502	16.6
14	7800.709	77.602	47.5
15	7768.666	77.284	50.3
16	7736.622	76.965	51.5
17	6599.842	65.656	88.3
18	4453.694	44.306	55.8
19	2191.578	21.802	68.6

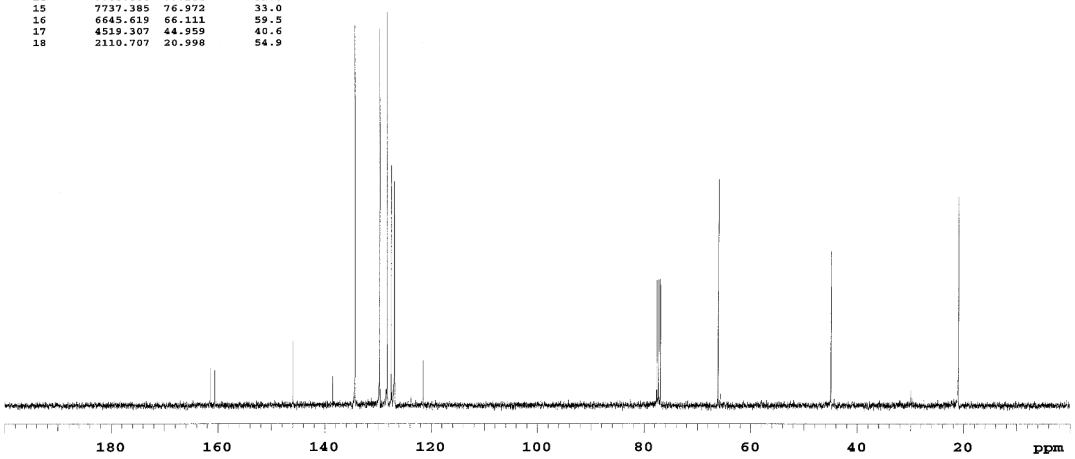


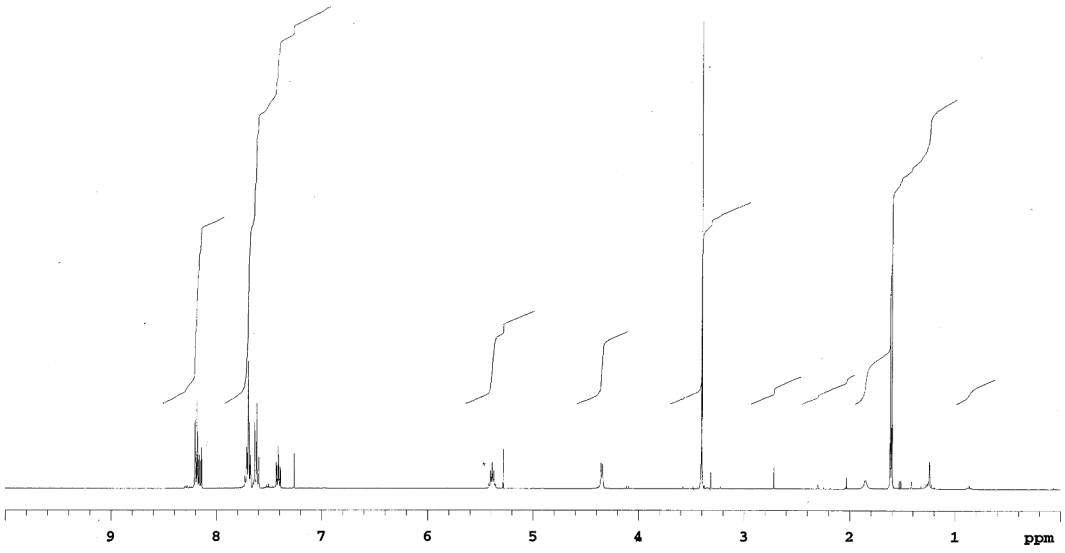


^{1}H and ^{13}C NMR spectra of Sulfoximine **20a**

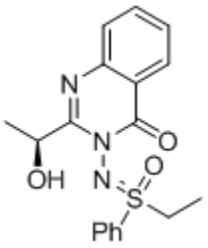


INDEX	FREQUENCY	PPM	HEIGHT
1	16228.901	161.447	9.9
2	16139.637	160.559	9.2
3	14675.557	145.994	16.9
4	13921.772	138.496	7.6
5	13500.630	134.306	100.4
6	13048.207	129.805	6.0
7	13033.711	129.661	99.2
8	12891.804	128.249	103.7
9	12813.984	127.475	63.3
10	12759.053	126.929	58.9
11	12750.660	126.845	50.0
12	12211.262	121.479	11.8
13	7800.709	77.602	32.9
14	7768.666	77.284	32.8
15	7737.385	76.972	33.0
16	6645.619	66.111	59.5
17	4519.307	44.959	40.6
18	2110.707	20.998	54.9

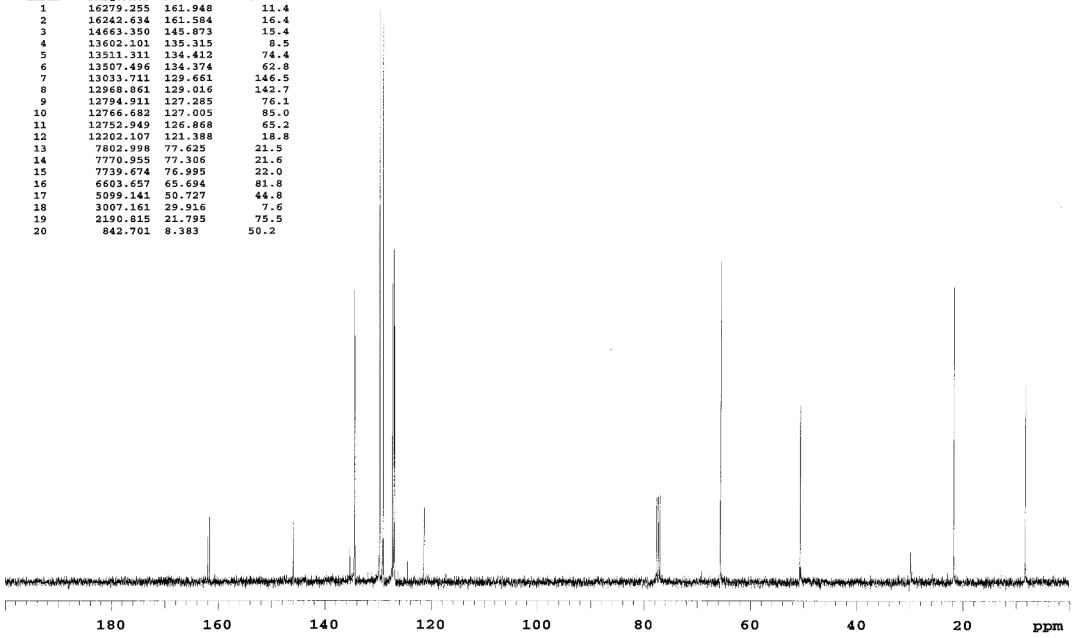


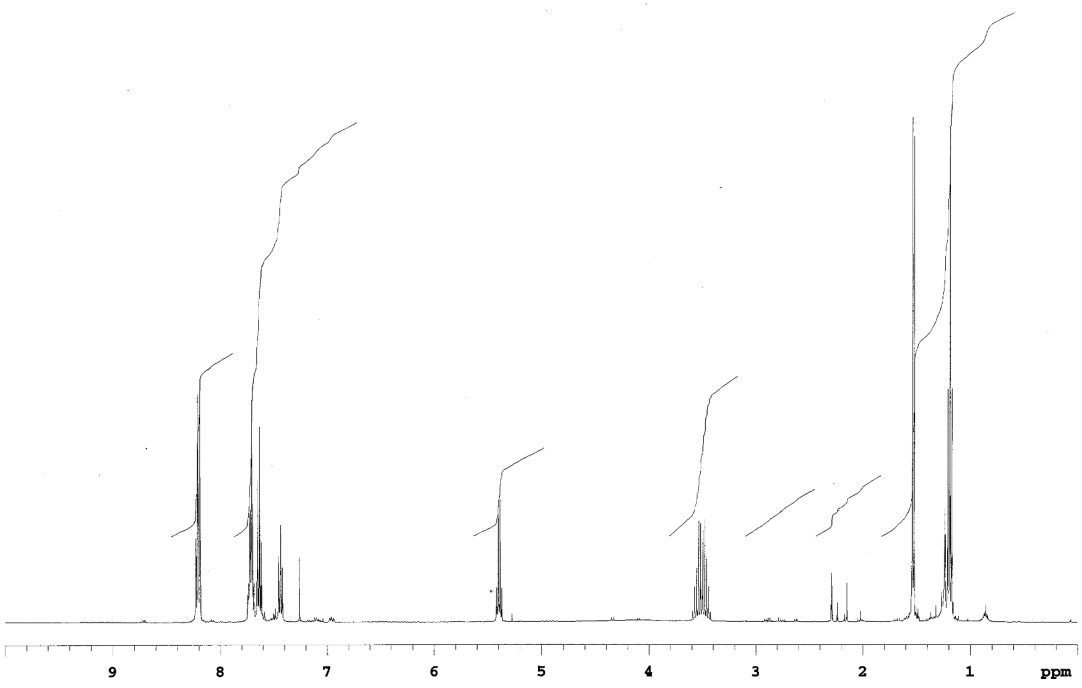


^{1}H and ^{13}C NMR spectra of Sulfoximine **20b**

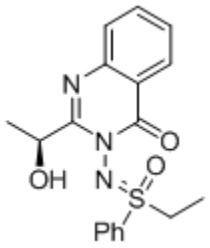


INDEX	FREQUENCY	PPM	HEIGHT
1	16279.255	161.948	11.4
2	16242.634	161.584	16.4
3	14663.350	145.873	15.4
4	13602.101	135.315	8.5
5	13511.311	134.412	74.4
6	13507.496	134.374	62.8
7	13033.711	129.661	146.5
8	12968.861	129.016	142.7
9	12794.911	127.285	76.1
10	12766.682	127.005	85.0
11	12752.949	126.868	65.2
12	12202.107	121.388	18.8
13	7802.998	77.625	21.5
14	7770.955	77.306	21.6
15	7739.674	76.995	22.0
16	6603.657	65.694	81.8
17	5099.141	50.727	44.8
18	3007.161	29.916	7.6
19	2190.815	21.795	75.5
20	842.701	8.383	50.2

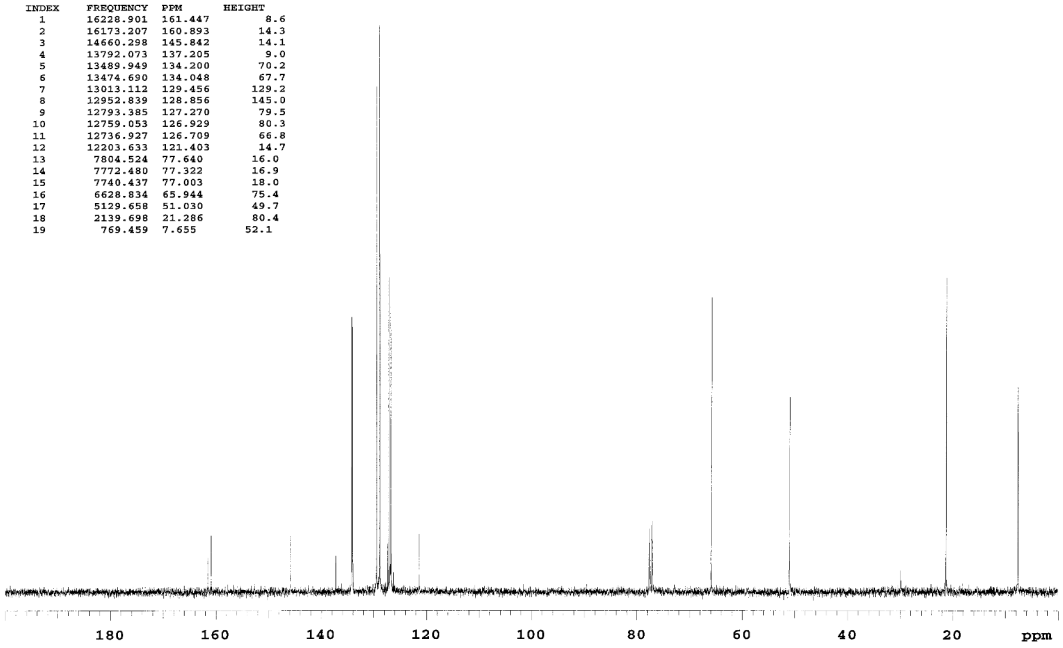


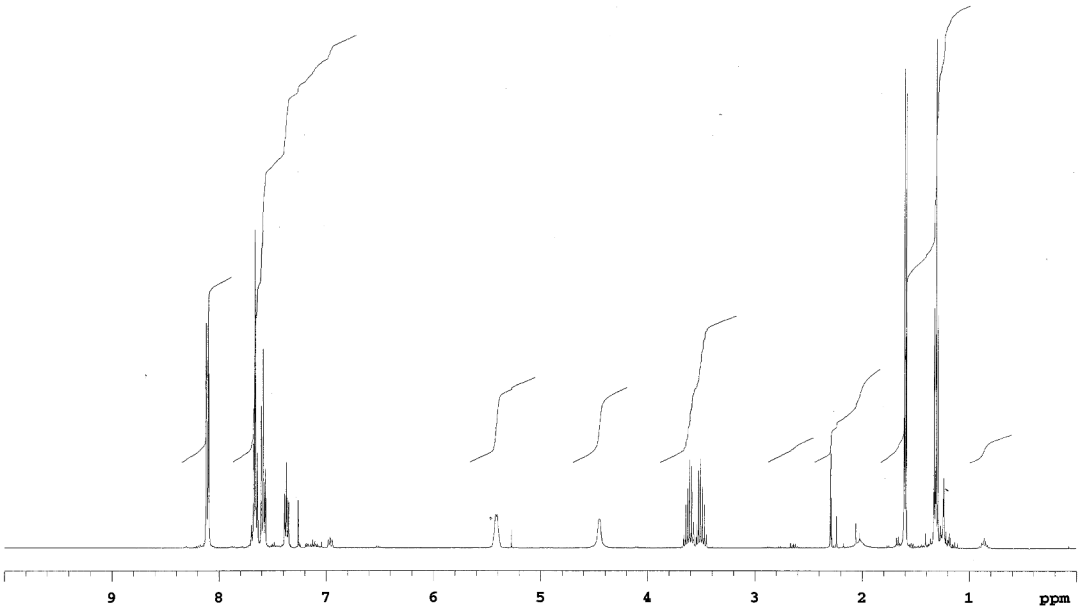


^{1}H and ^{13}C NMR spectra of Sulfoximine **21a**

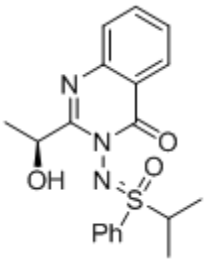


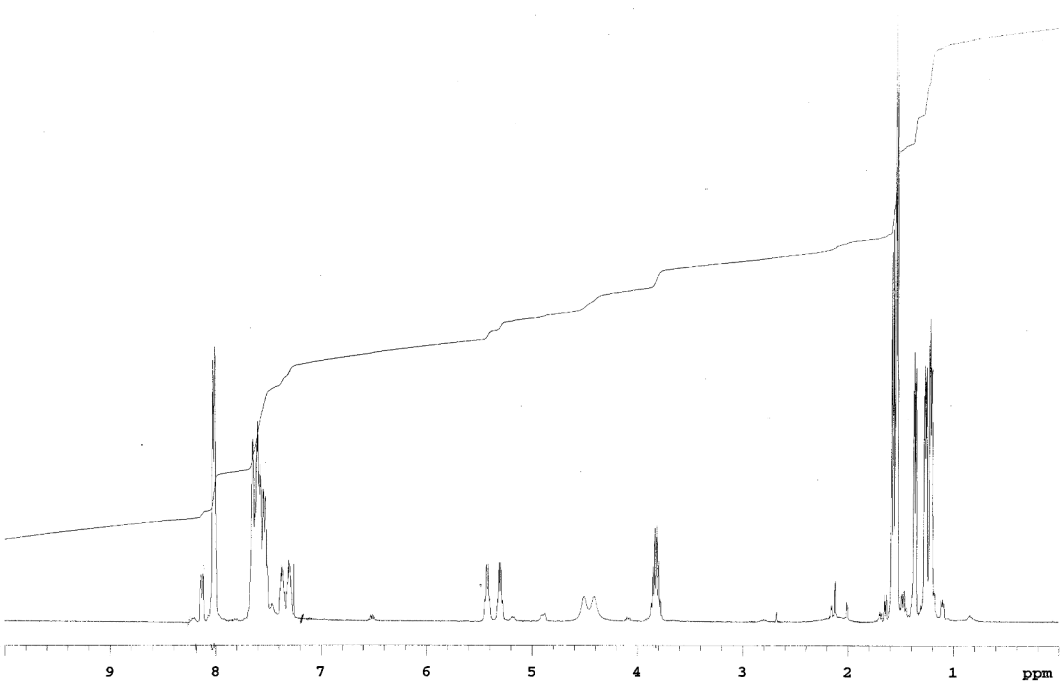
INDEX	FREQUENCY	PPM	HEIGHT
1	16228.901	161.447	8.6
2	16173.207	160.893	14.3
3	14660.298	145.842	14.1
4	13792.073	137.205	9.0
5	13489.949	134.200	70.2
6	13474.690	134.048	67.7
7	13013.112	129.456	129.2
8	12952.839	128.856	145.0
9	12793.385	127.270	79.5
10	12759.053	126.929	80.3
11	12736.927	126.709	66.8
12	12203.633	121.403	14.7
13	7804.524	77.640	16.0
14	7772.480	77.322	16.9
15	7740.437	77.003	18.0
16	6628.834	65.944	75.4
17	5129.658	51.030	49.7
18	2139.698	21.286	80.4
19	769.459	7.655	52.1



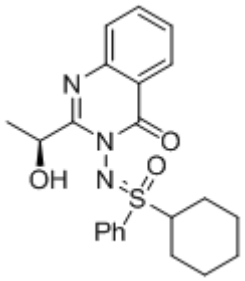


^{1}H and ^{13}C NMR spectra of Sulfoximine **21b**

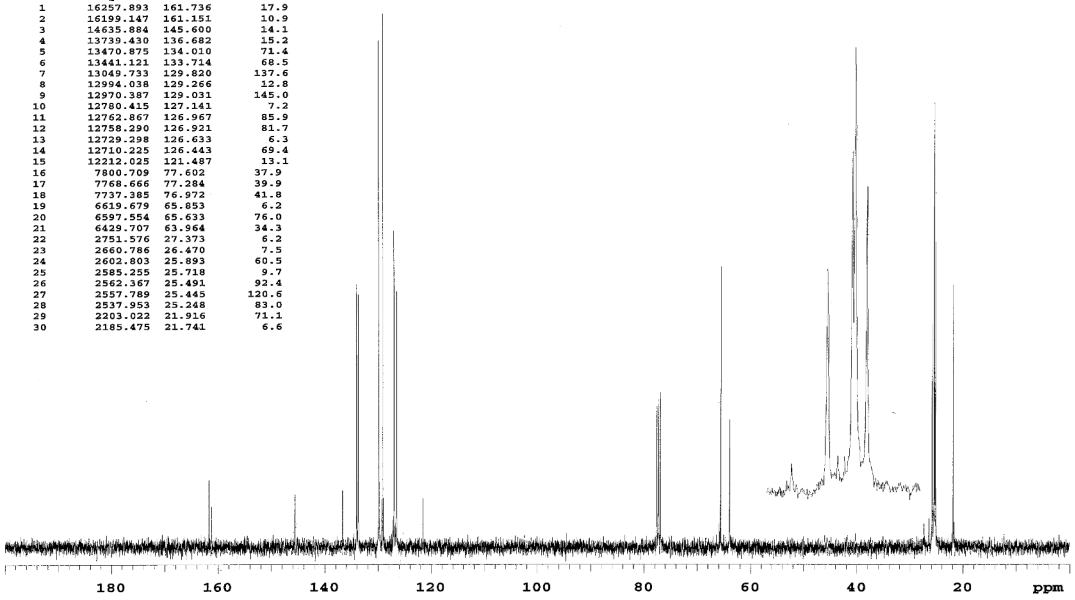


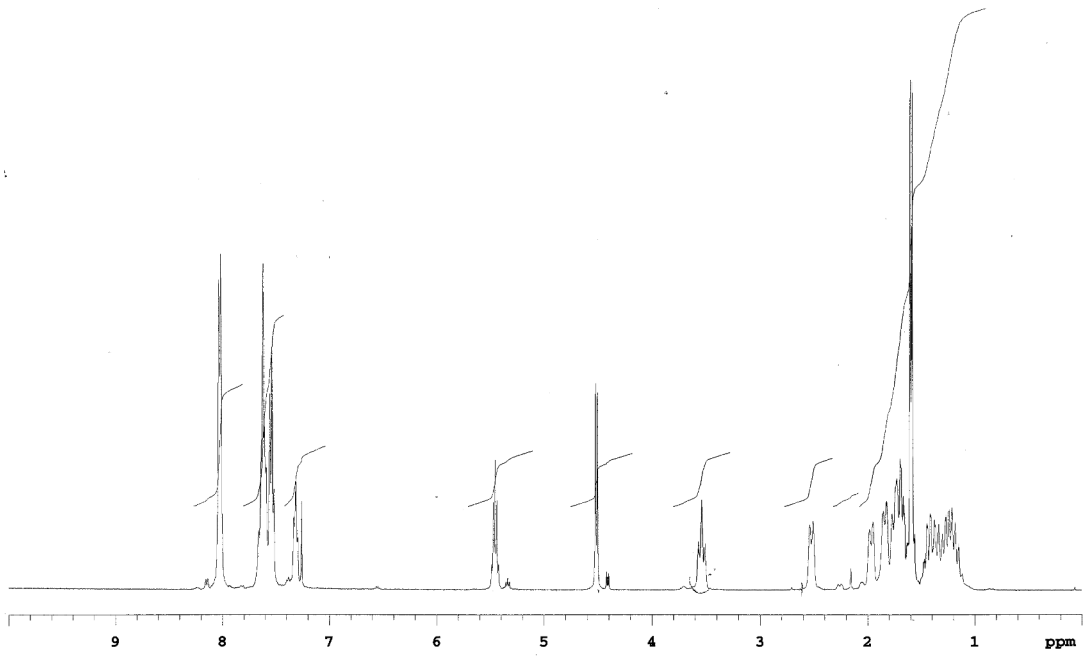


^{1}H NMR spectrum of Sulfoximine **22**

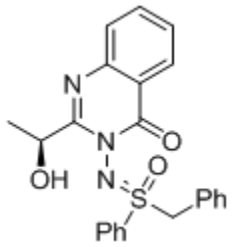


INDEX	FREQUENCY	PPM	HEIGHT
1	16257.893	161.736	17.9
2	16199.147	161.151	10.9
3	14635.884	145.600	14.1
4	13739.430	136.682	15.2
5	13470.875	134.010	71.4
6	13441.121	133.714	68.5
7	13049.733	129.820	137.6
8	12994.038	129.266	12.8
9	12970.387	129.031	145.0
10	12780.415	127.141	7.2
11	12762.867	126.967	85.9
12	12758.290	126.921	81.7
13	12729.298	126.633	6.3
14	12710.225	126.443	69.4
15	12212.025	121.487	13.1
16	7800.709	77.602	37.9
17	7768.666	77.284	39.9
18	7737.385	76.972	41.8
19	6619.679	65.853	6.2
20	6597.554	65.633	76.0
21	6429.707	63.964	34.3
22	2751.576	27.373	6.2
23	2660.786	26.470	7.5
24	2602.803	25.893	60.5
25	2585.255	25.718	9.7
26	2562.367	25.491	92.4
27	2557.789	25.445	120.6
28	2537.953	25.248	83.0
29	2203.022	21.916	71.1
30	2185.475	21.741	6.6

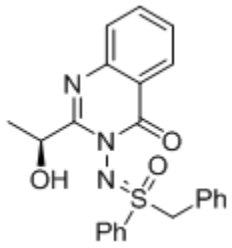




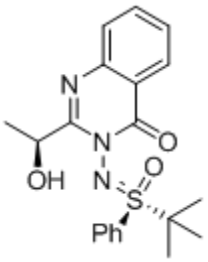
^{1}H and ^{13}C NMR spectra of Sulfoximine **25**



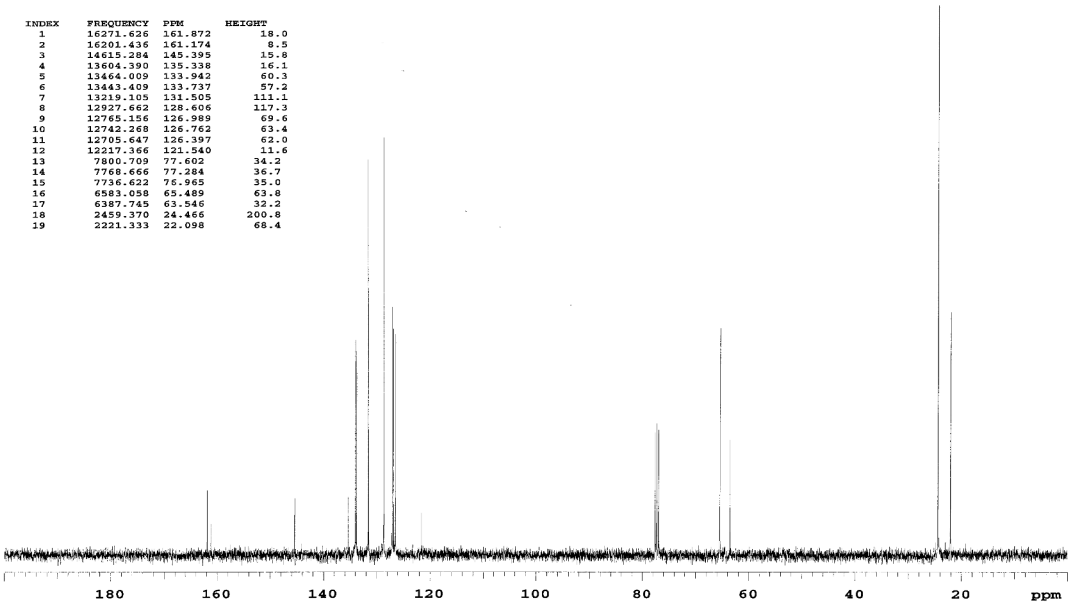
^{1}H and ^{13}C NMR spectra of Sulfoximine **24a**



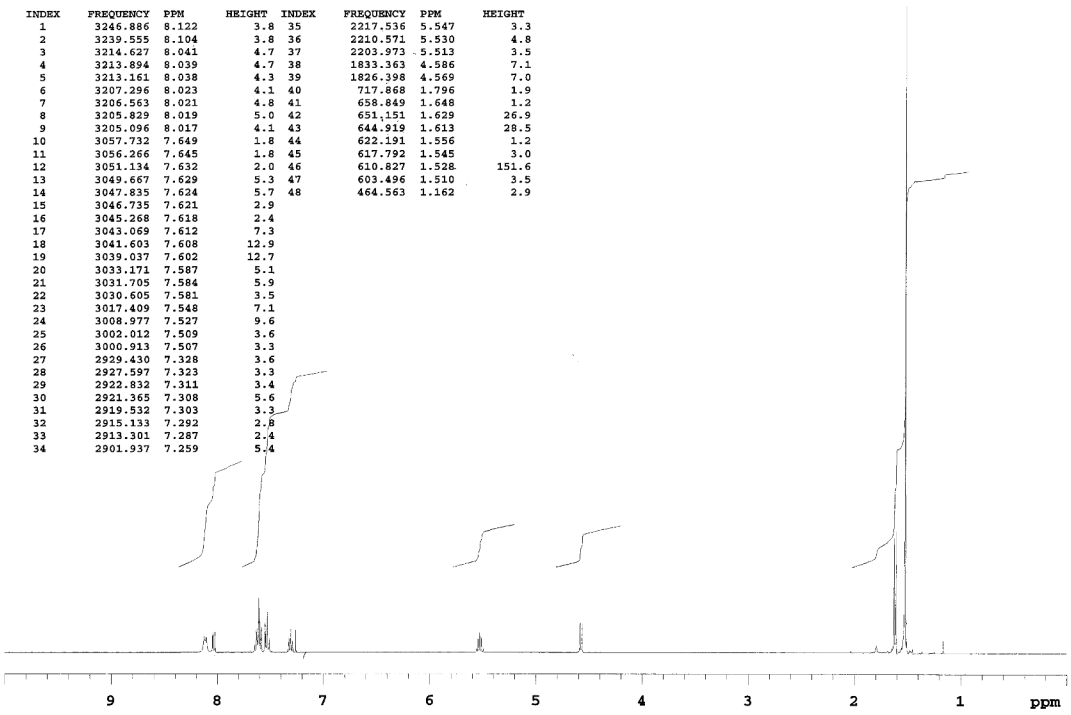
^{1}H and ^{13}C NMR spectra of Sulfoximine **24b**



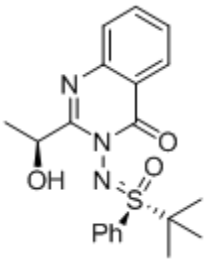
INDEX	FREQUENCY	PPM	HEIGHT
1	16271.626	161.872	18.0
2	16201.436	161.174	8.5
3	14615.284	145.395	15.8
4	13604.390	135.338	16.1
5	13464.009	133.942	60.3
6	13443.409	133.737	57.2
7	13219.105	131.505	111.1
8	12927.662	128.606	117.3
9	12765.156	126.989	69.6
10	12742.268	126.762	63.4
11	12705.647	126.397	62.0
12	12217.366	121.540	11.6
13	7800.709	77.602	34.2
14	7768.666	77.284	36.7
15	7736.622	76.965	35.0
16	6583.058	65.489	63.8
17	6387.745	63.546	32.2
18	2459.370	24.466	200.8
19	2221.333	22.098	68.4



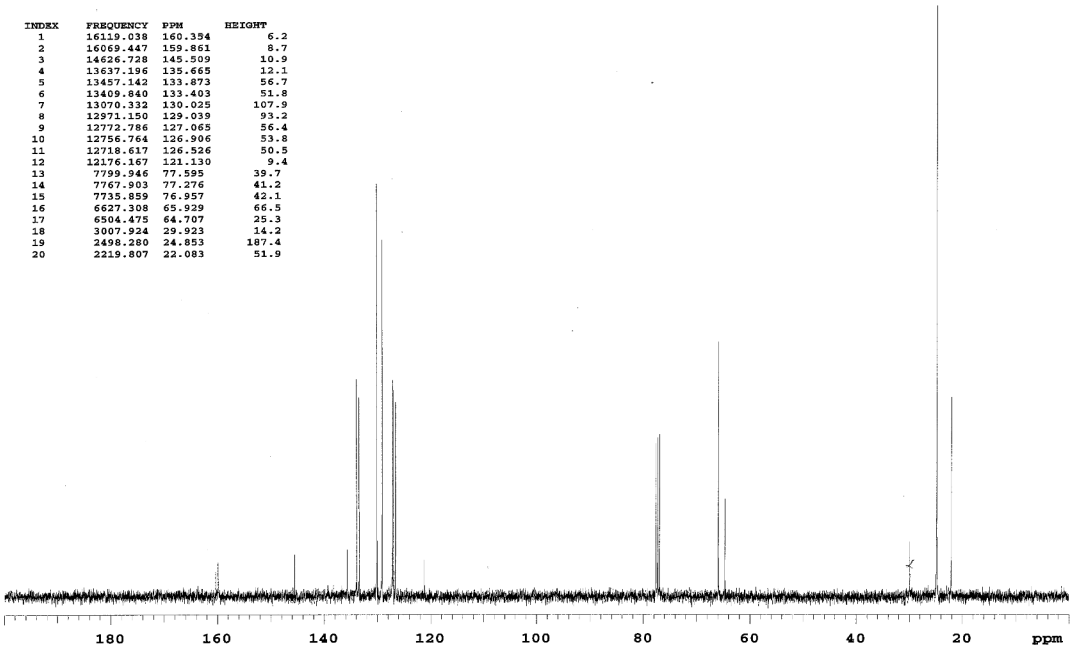
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3246.886	8.122	3.8	35	2217.536	5.547	3.3
2	3239.555	8.104	3.8	36	2210.571	5.530	4.8
3	3214.627	8.041	4.7	37	2203.973	5.513	3.5
4	3213.894	8.039	4.7	38	1833.363	4.586	7.1
5	3213.161	8.038	4.3	39	1826.398	4.569	7.0
6	3207.296	8.023	4.1	40	717.868	1.796	1.9
7	3206.563	8.021	4.8	41	658.849	1.648	1.2
8	3205.829	8.019	5.0	42	651.151	1.629	26.9
9	3205.096	8.017	4.1	43	644.919	1.613	28.5
10	3057.732	7.649	1.8	44	622.191	1.556	1.2
11	3056.266	7.645	1.8	45	617.792	1.545	3.0
12	3051.134	7.632	2.0	46	610.827	1.528	151.6
13	3049.667	7.629	5.3	47	603.496	1.510	3.5
14	3047.835	7.624	5.7	48	464.563	1.162	2.9
15	3046.735	7.621	2.9				
16	3045.268	7.618	2.4				
17	3043.069	7.612	7.3				
18	3041.603	7.608	12.9				
19	3039.037	7.602	12.7				
20	3033.171	7.587	5.1				
21	3031.705	7.584	5.9				
22	3030.605	7.581	3.5				
23	3017.409	7.548	7.1				
24	3008.977	7.527	9.6				
25	3002.012	7.509	3.6				
26	3000.913	7.507	3.3				
27	2929.430	7.328	3.6				
28	2927.597	7.323	3.3				
29	2922.832	7.311	3.4				
30	2921.365	7.308	5.6				
31	2919.532	7.303	3.3				
32	2915.133	7.292	2.8				
33	2913.301	7.287	2.4				
34	2901.937	7.259	5.4				



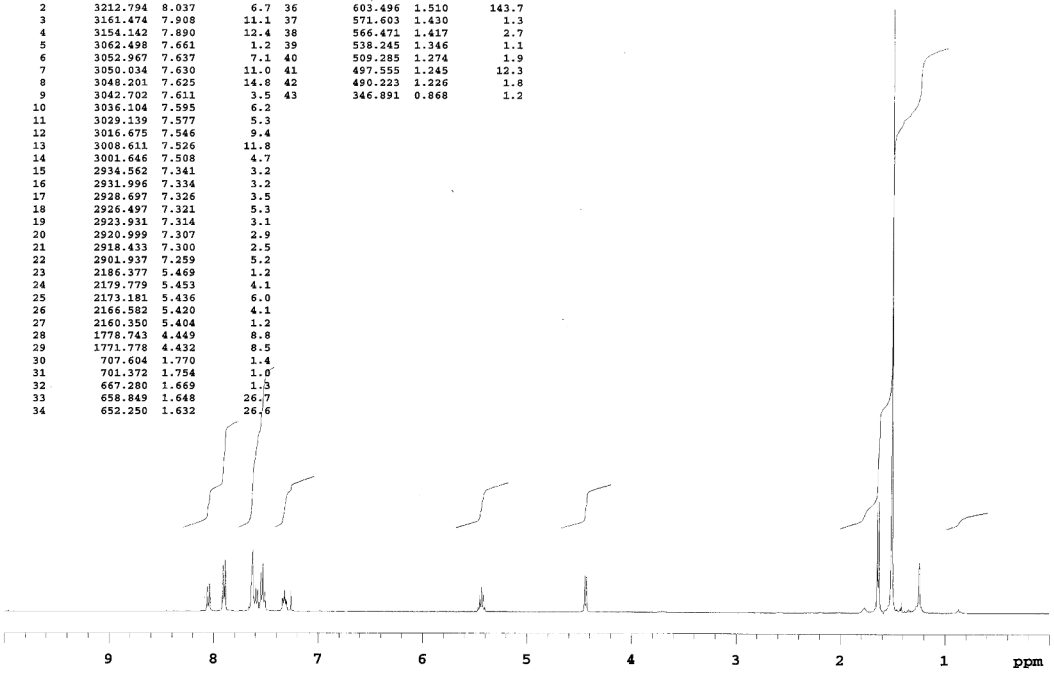
^{1}H and ^{13}C NMR spectra of Sulfoximine **23a**



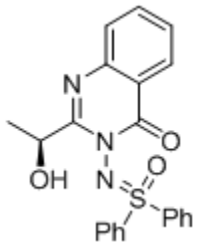
INDEX	FREQUENCY	PPM	HEIGHT
1	16119.038	160.354	6.2
2	16069.447	159.861	8.7
3	14626.728	145.509	10.9
4	13637.196	135.665	12.1
5	13457.142	133.873	56.7
6	13409.840	133.403	51.8
7	13070.332	130.025	107.9
8	12971.150	129.039	93.2
9	12772.786	127.065	56.4
10	12756.764	126.906	53.8
11	12718.617	126.526	50.5
12	12176.167	121.130	9.4
13	7799.946	77.595	39.7
14	7767.903	77.276	41.2
15	7735.859	76.957	42.1
16	6627.308	65.929	66.5
17	6504.475	64.707	25.3
18	3007.924	29.923	14.2
19	2498.280	24.853	187.4
20	2219.807	22.083	51.9



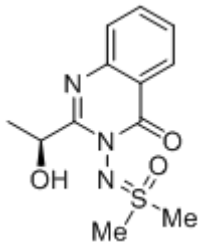
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3220.859	8.057	5.9	35	617.426	1.544	1.9
2	3212.794	8.037	6.7	36	603.496	1.510	143.7
3	3161.474	7.908	11.1	37	571.603	1.430	1.3
4	3154.142	7.890	12.4	38	566.471	1.417	2.7
5	3062.498	7.661	1.2	39	538.245	1.346	1.1
6	3052.967	7.637	7.1	40	509.285	1.274	1.9
7	3050.034	7.630	11.0	41	497.555	1.245	12.3
8	3048.201	7.625	14.8	42	490.223	1.226	1.8
9	3042.702	7.611	3.5	43	346.891	0.868	1.2
10	3036.104	7.595	6.2				
11	3029.139	7.577	5.3				
12	3016.675	7.546	9.4				
13	3008.611	7.526	11.8				
14	3001.646	7.508	4.7				
15	2934.562	7.341	3.2				
16	2931.996	7.334	3.2				
17	2928.697	7.326	3.5				
18	2926.497	7.321	5.3				
19	2923.931	7.314	3.1				
20	2920.999	7.307	2.9				
21	2918.433	7.300	2.5				
22	2901.937	7.259	5.2				
23	2186.377	5.469	1.2				
24	2179.779	5.453	4.1				
25	2173.181	5.436	6.0				
26	2166.582	5.420	4.1				
27	2160.350	5.404	1.2				
28	1778.743	4.449	8.8				
29	1771.778	4.432	8.5				
30	707.604	1.770	1.4				
31	701.372	1.754	1.8				
32	667.280	1.669	1.3				
33	658.849	1.648	26.7				
34	652.250	1.632	26.6				



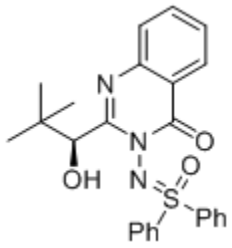
^{1}H and ^{13}C NMR spectra of Sulfoximine **23b**



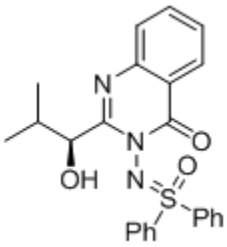
^{1}H and ^{13}C NMR spectra of Sulfoximine **26**



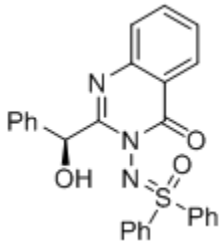
^{1}H and ^{13}C NMR spectra of Sulfoximine **27**



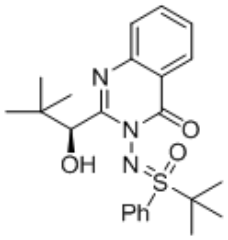
^{1}H and ^{13}C NMR spectra of Sulfoximine **33**



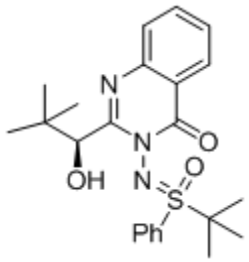
^{1}H and ^{13}C NMR spectra of Sulfoximine **34**

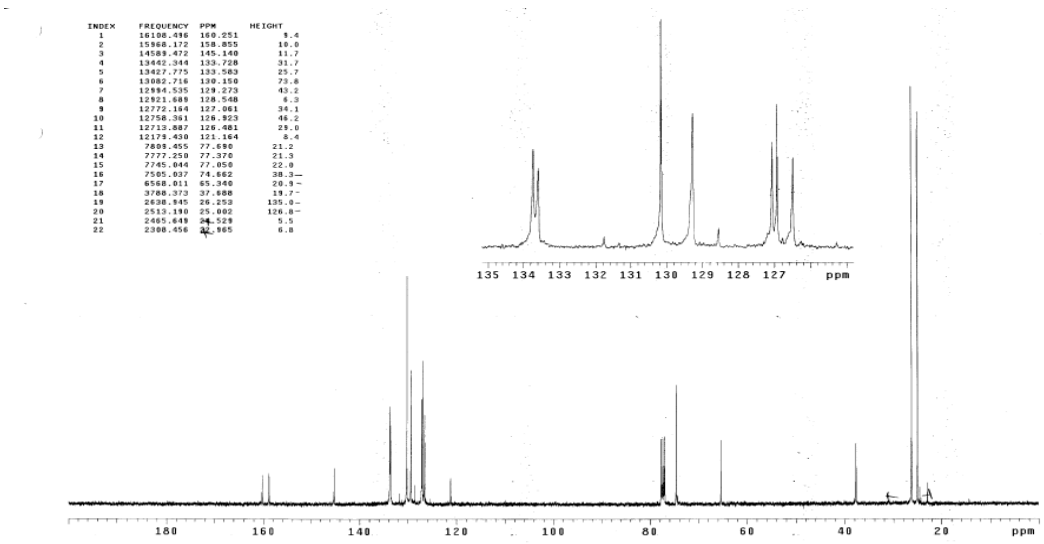


^{1}H and ^{13}C NMR spectra of Sulfoximine **35**

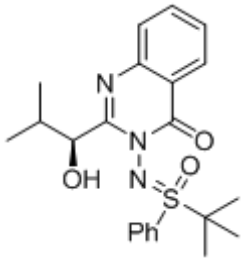


^{1}H and ^{13}C NMR spectra of Sulfoximine **36a**

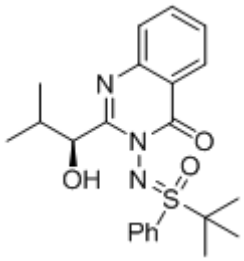




^{1}H and ^{13}C NMR spectra of Sulfoximine **36b**



^{1}H and ^{13}C NMR spectra of Sulfoximine **37a**



^{1}H and ^{13}C NMR spectra of Sulfoximine **37b**

Data File E:\SABRI\SU000400.D Sample Name: SU0004

2.4 helyum 110 derece sabit

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Acq. Instrument : Instrument 2                   Inj Volume : Manually
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Last changed    : 2/18/2008 2:43:47 PM by murat
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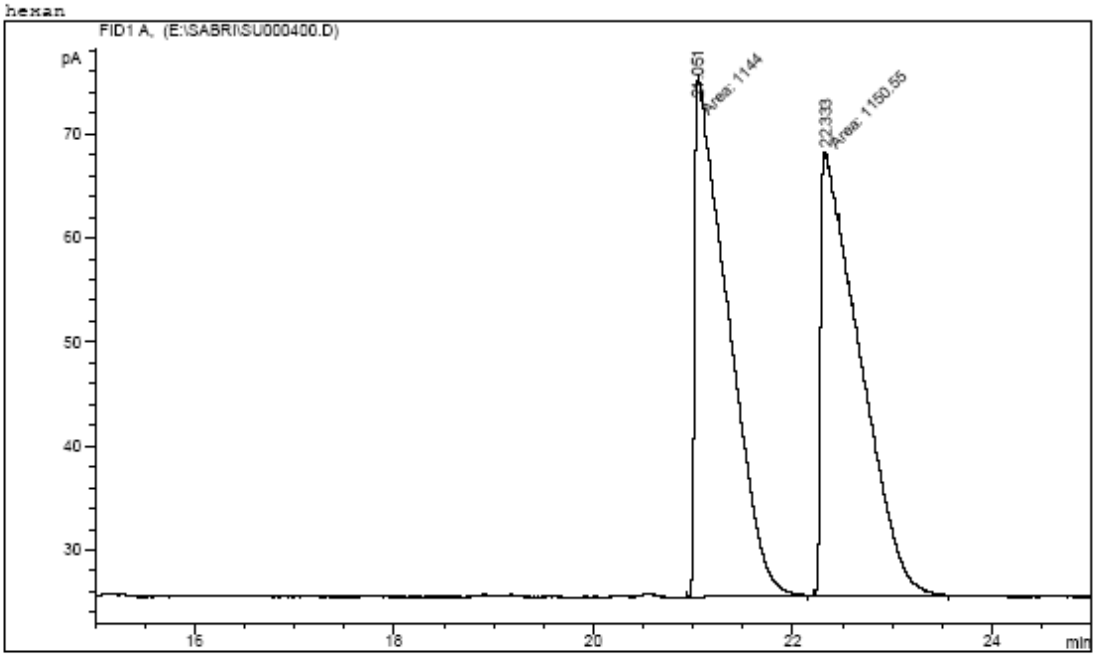
hexan

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Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
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   2  22.333 MM    0.4496 1150.55176   42.64870  50.1428

Totals :                  2294.54810   92.84703
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Data File E:\HPLCAN~1\2\DATA\SEMS\SK046800.D Sample Name: SK0468

2.4 helyum 110 derece sabit 1-fenil-1-propanol

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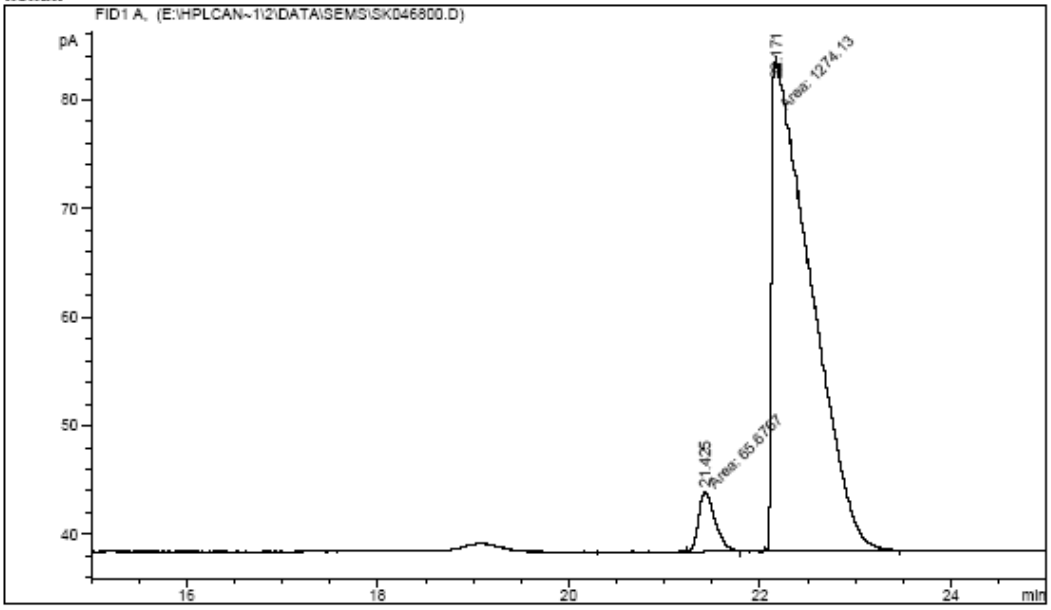


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Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
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Totals :                  1339.81074   51.16091
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Data File E:\HPLCAN~1\2\DATA\SEMS\SK049201.D Sample Name: 2-metoksiBenz

2.4 helyum 135 derece sabit

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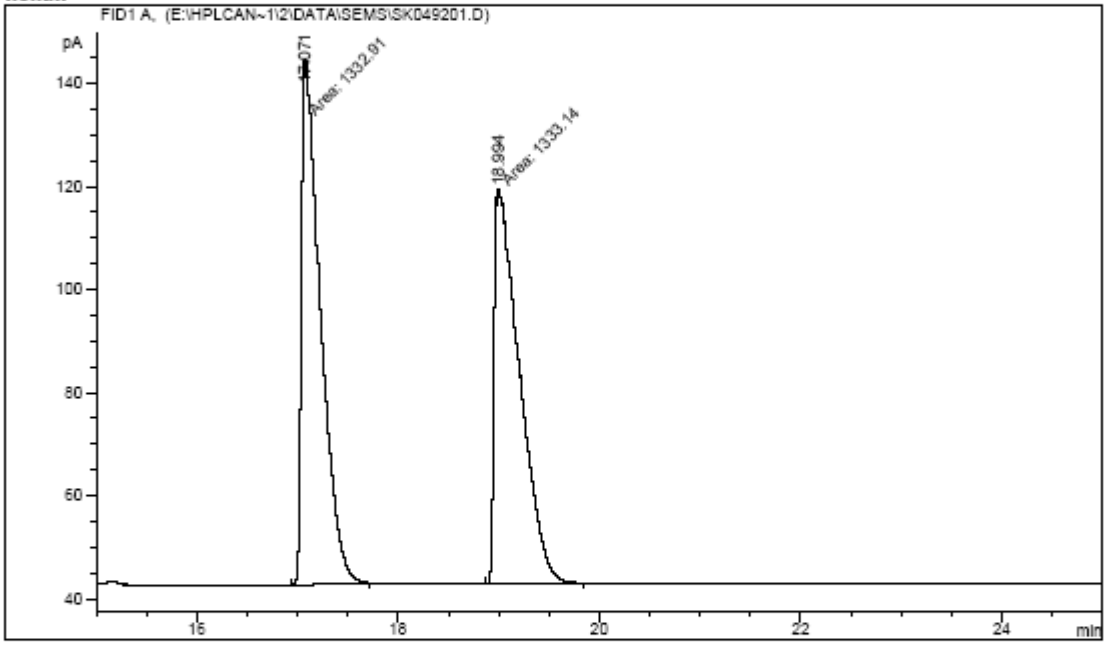
hexan

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Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

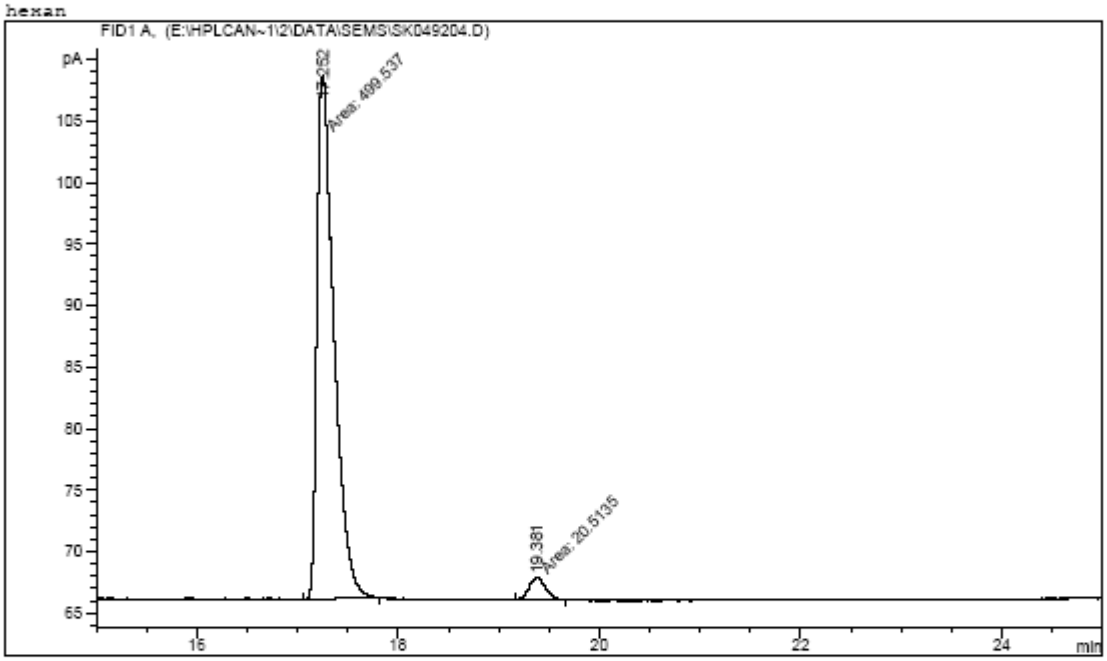
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]     [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  17.071 MM    0.2177 1332.90955  102.03021  49.9958
   2  18.994 MM    0.2902 1333.13599   76.57037  50.0042

Totals :                  2666.04553  178.60058
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049204.D Sample Name: SK0492

2.4 helyum 135 derece sbt 2-metoksi metoduna göre

```
==========================================================================
Injection Date  : 30.04.2008 11:22:29
Sample Name     : SK0492                          Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                   Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\2METOKSI.M
Last changed    : 4/25/2008 3:04:15 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/6/2010 2:12:24 PM
                  (modified after loading)
hexan
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  17.252 MM    0.1956  499.53687   42.56927  96.0555
   2  19.381 MM    0.1887   20.51348    1.81162   3.9445

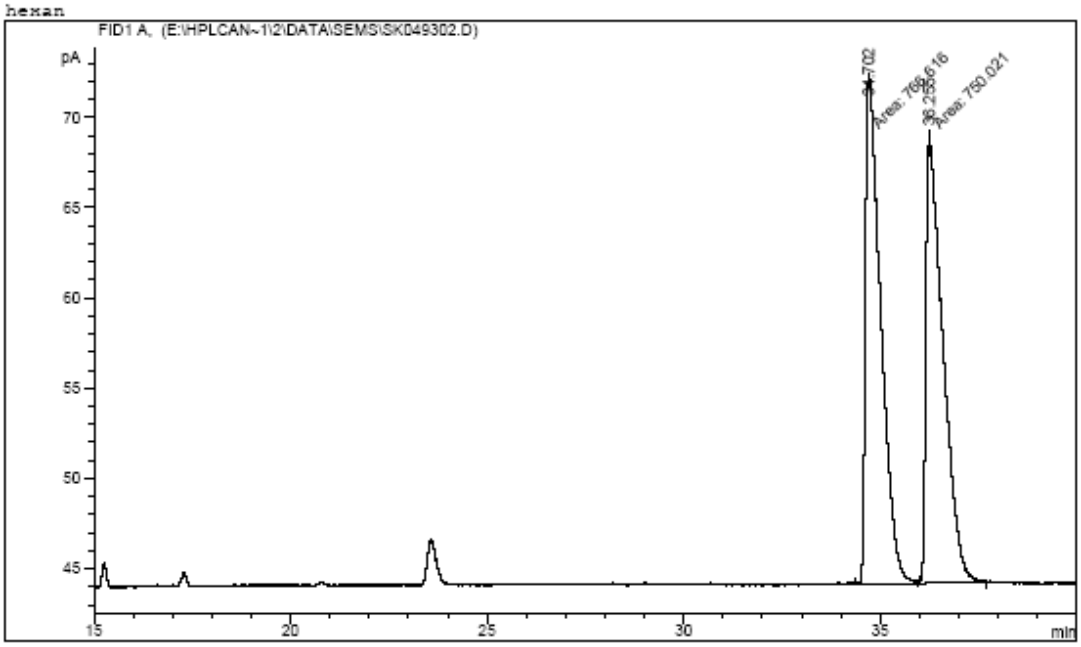
Totals :                   520.05034   44.38089
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049302.D Sample Name: 3-metoksiBenz

2.4 helyum 135 derece sabit

```
======================================================================
Injection Date  : 26.04.2008 11:08:05
Sample Name     : 3-metoksiBenz                    Location : Vial 1
Acq. Operator   : murat                               Inj :   1
Acq. Instrument : Instrument 2                   Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\3METOKSI.M
Last changed    : 4/26/2008 11:06:12 AM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/6/2010 11:30:57 PM
                  (modified after loading)
```

hexan




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  34.702 MM    0.4529  766.61609   28.21136  50.5471
   2  36.255 MM    0.4982  750.02051   25.08920  49.4529

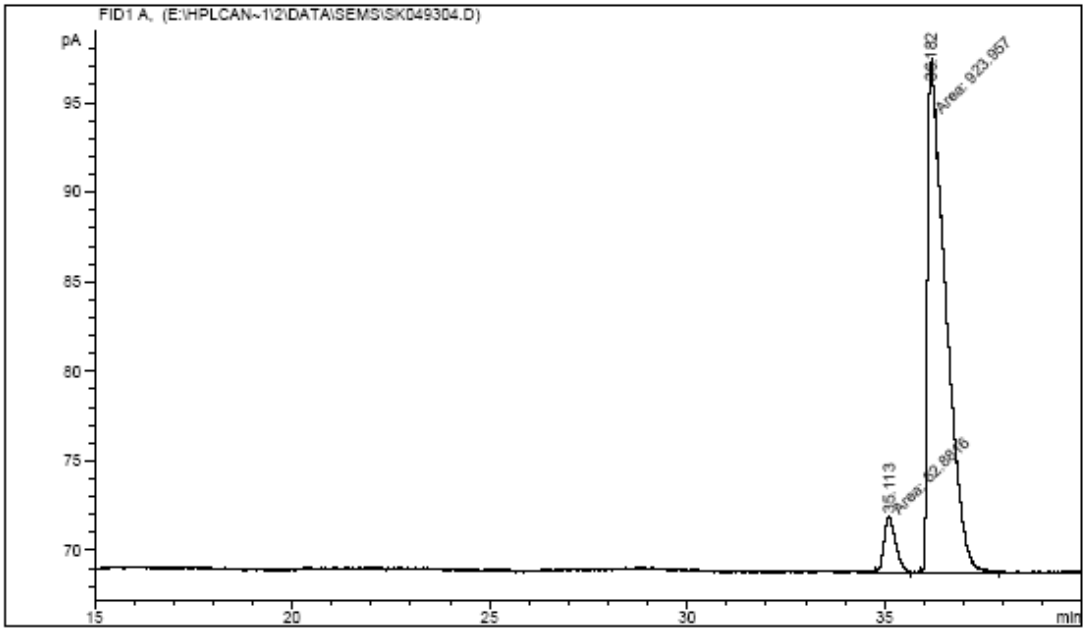
Totals :                  1516.63660   53.30055
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049304.D Sample Name: 3-metoksi bnzald

2.4 helyum 127 derece sabit 3-metoksi benzaldehite die
til cinko katýlma ürünü

```
==========================================================================
Injection Date  : 27.04.2008 14:48:30
Sample Name     : 3-metoksi bnzald              Location : Vial 1
Acq. Operator   : murat                            Inj :   1
Acq. Instrument : Instrument 2                 Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\3METOKSI.M
Last changed    : 4/27/2008 3:23:43 PM by murat
                  (modified after loading)
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/6/2010 11:30:57 PM
                  (modified after loading)
```

hexan




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  35.113 MM    0.3376   62.68158    3.10455   6.3720
   2  36.182 MM    0.5349  923.95721   28.78959  93.6280

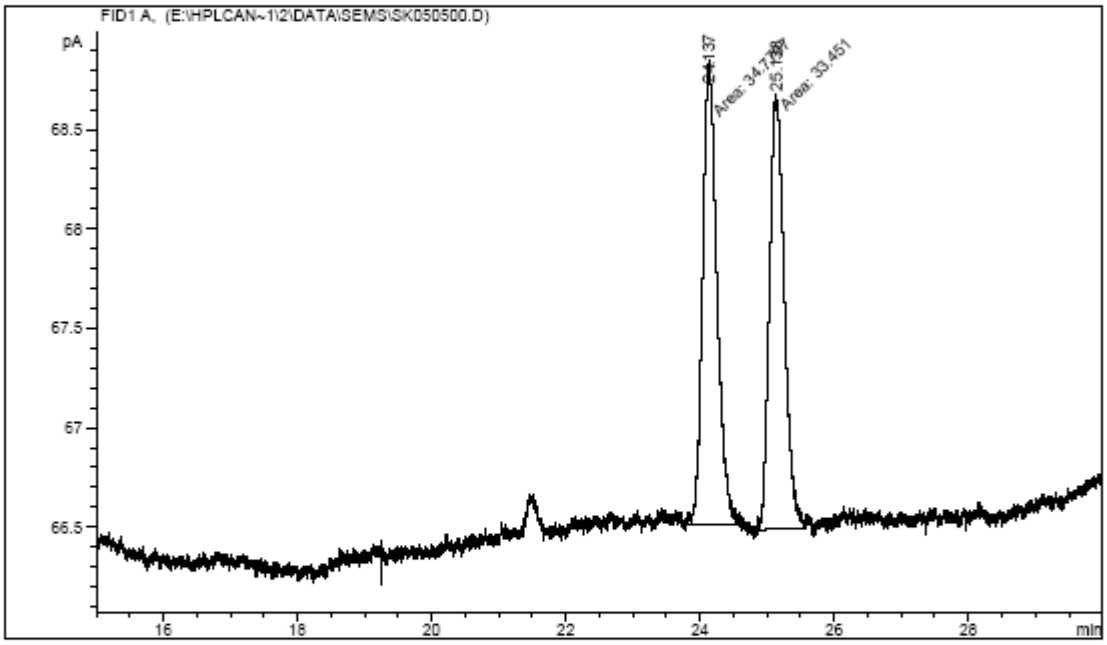
Totals :                   986.63879   31.89414
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK050500.D Sample Name: 4-metoksi benz

2.4 helyum 135 derece sabit aldehit'in kendisi 4-metoks
i

```
======================================================================
Injection Date  : 27.04.2008 11:36:56
Sample Name     : 4-metoksi benz                  Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                   Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\4METOKSI.M
Last changed    : 4/27/2008 12:14:30 PM by murat
                  (modified after loading)
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 1:03:33 AM
                  (modified after loading)
```

hexan




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  24.137 MM    0.2473   34.77974    2.34412  50.9737
   2  25.138 MM    0.2553   33.45103    2.18418  49.0263

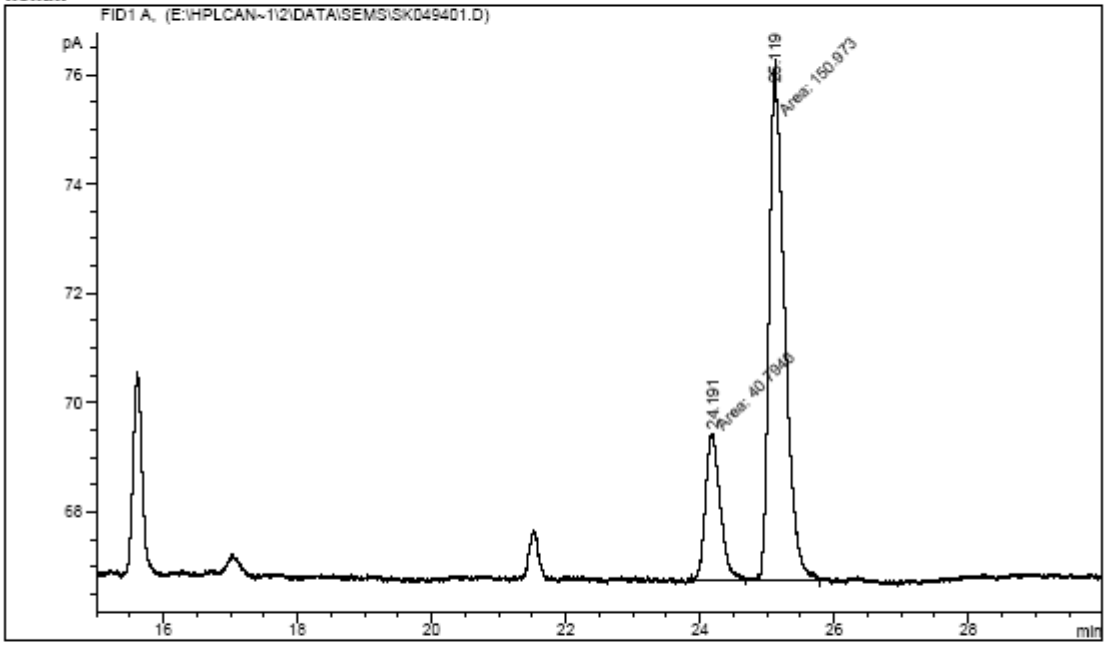
Totals :                    68.23077    4.52830
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049401.D Sample Name: SK0494

2.4 helyum 4-metoksi metoduna göre

```
==========================================================================
Injection Date  : 29.04.2008 12:09:51
Sample Name     : SK0494                          Location : Vial 1
Acq. Operator   : sems                               Inj :   1
Acq. Instrument : Instrument 2                  Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\4METOKSI.M
Last changed    : 4/27/2008 12:26:21 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/6/2010 11:48:24 PM
                  (modified after loading)
```

hexan




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  24.191 MM    0.2521   40.79426    2.69694  21.2728
   2  25.119 MM    0.2639  150.97269    9.53492  78.7272

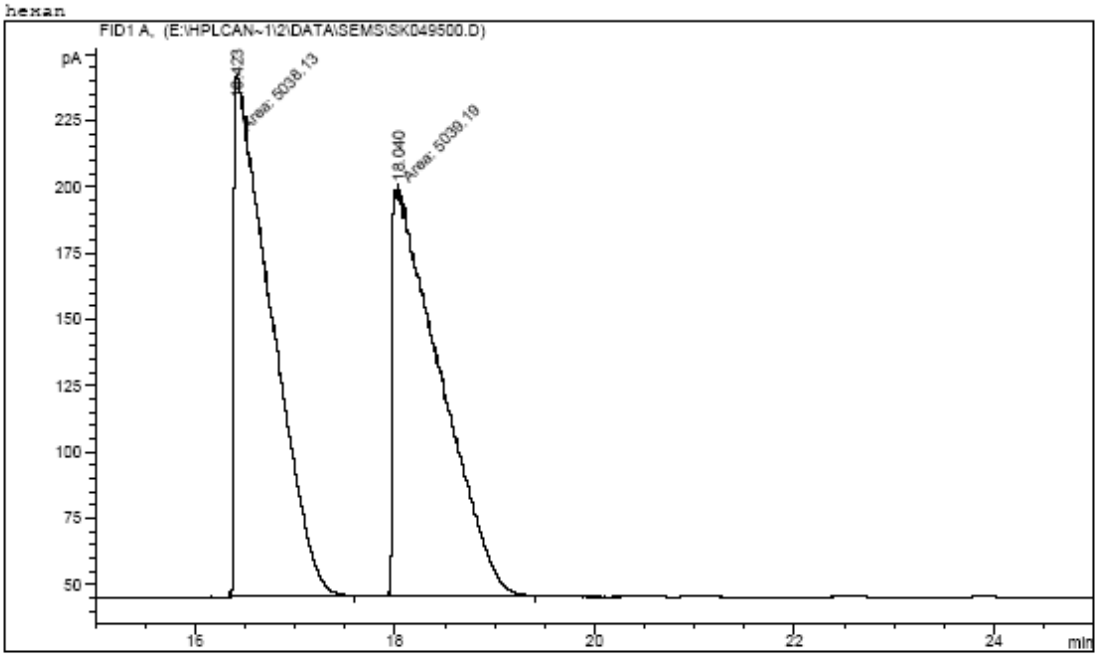
Totals :                   191.76694   12.23186
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049500.D Sample Name: 2-KloroiBenz

2.4 helyum 135 derece sabit

```
==========================================================================
Injection Date  : 26.04.2008 12:52:02
Sample Name     : 2-KloroiBenz                    Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                  Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\2KLORO.M
Last changed    : 4/25/2008 3:04:15 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/6/2010 11:57:15 PM
                  (modified after loading)
```

hexan




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  16.423 MM    0.4267 5038.13281  196.78612  49.9948
   2  18.040 MM    0.5414 5039.18896  155.12448  50.0052

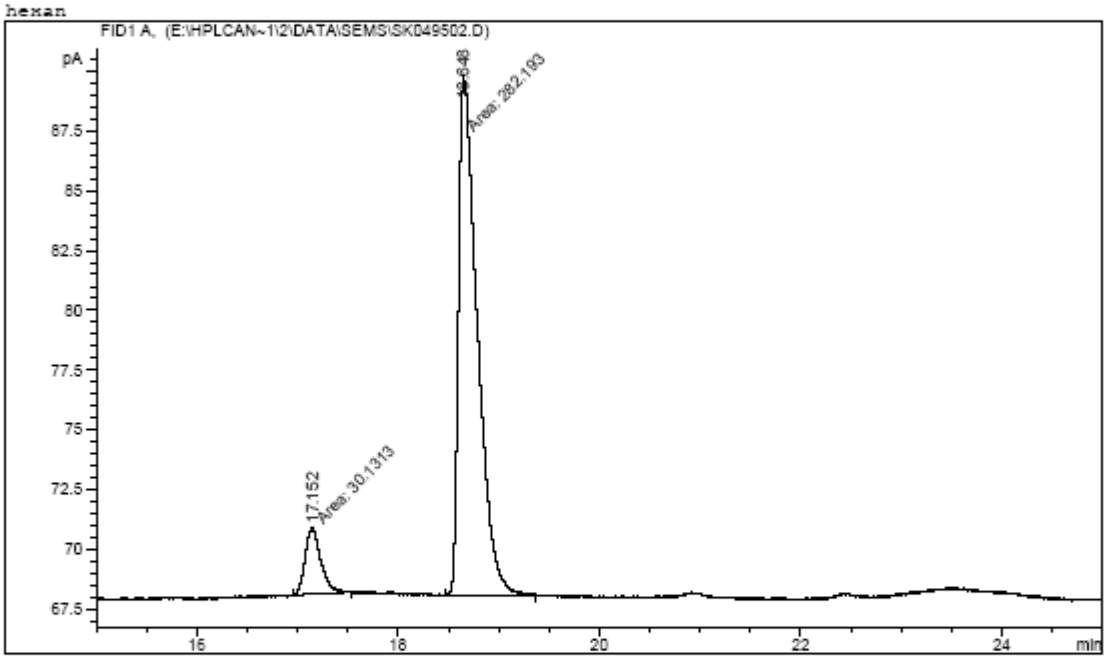
Totals :                  1.00773e4   351.91060
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049502.D Sample Name: 2-kloro bnzald

2.4 helyum 135 derece sabit 2-kloro benzaldehite dieti
1 cinko katýlma ürünü

```
==========================================================================
Injection Date  : 27.04.2008 16:09:53
Sample Name     : 2-kloro bnzald                  Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                   Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\2KLORO.M
Last changed    : 4/26/2008 12:51:54 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/6/2010 11:57:15 PM
                  (modified after loading)
```

hexan




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

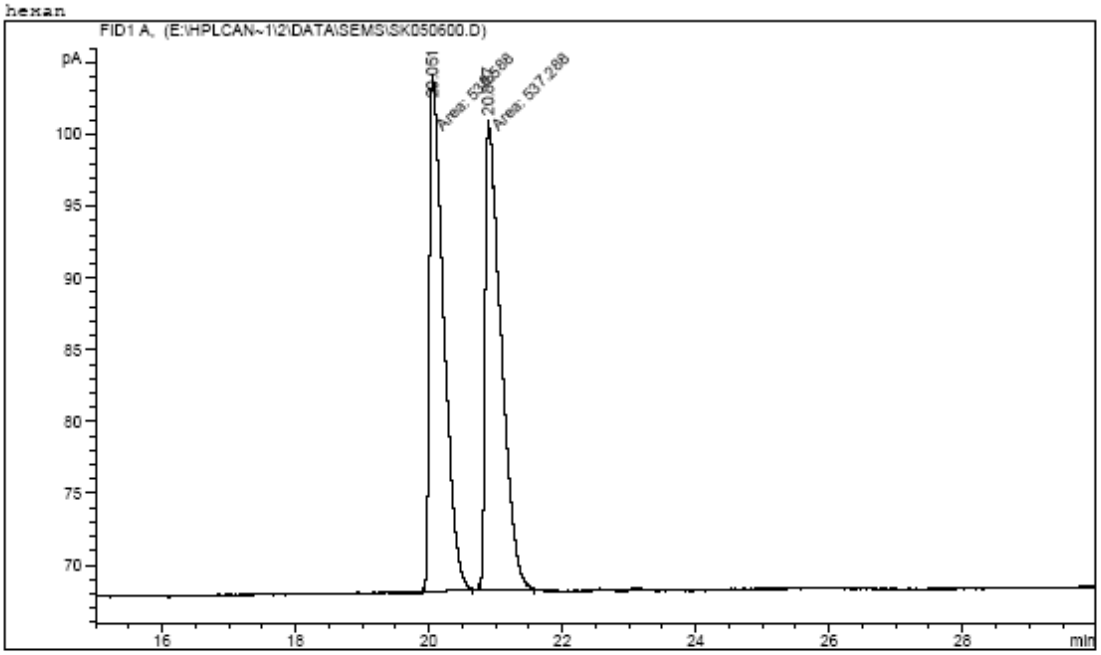
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  17.152 MM    0.1780   30.13128    2.82091   9.6474
   2  18.648 MM    0.2156  282.19296   21.81289  90.3526

Totals :                   312.32425   24.63380
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK050600.D Sample Name: 4-metoksi benz

2.4 helyum 135 derece sabit aldehit'in kendisi 4-metoks
i

```
==========================================================================
Injection Date  : 27.04.2008 12:34:50
Sample Name     : 4-metoksi benz                  Location : Vial 1
Acq. Operator   : murat                             Inj :   1
Acq. Instrument : Instrument 2                  Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\3KLORO.M
Last changed    : 4/26/2008 12:51:54 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 1:03:33 AM
                  (modified after loading)
hexan
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

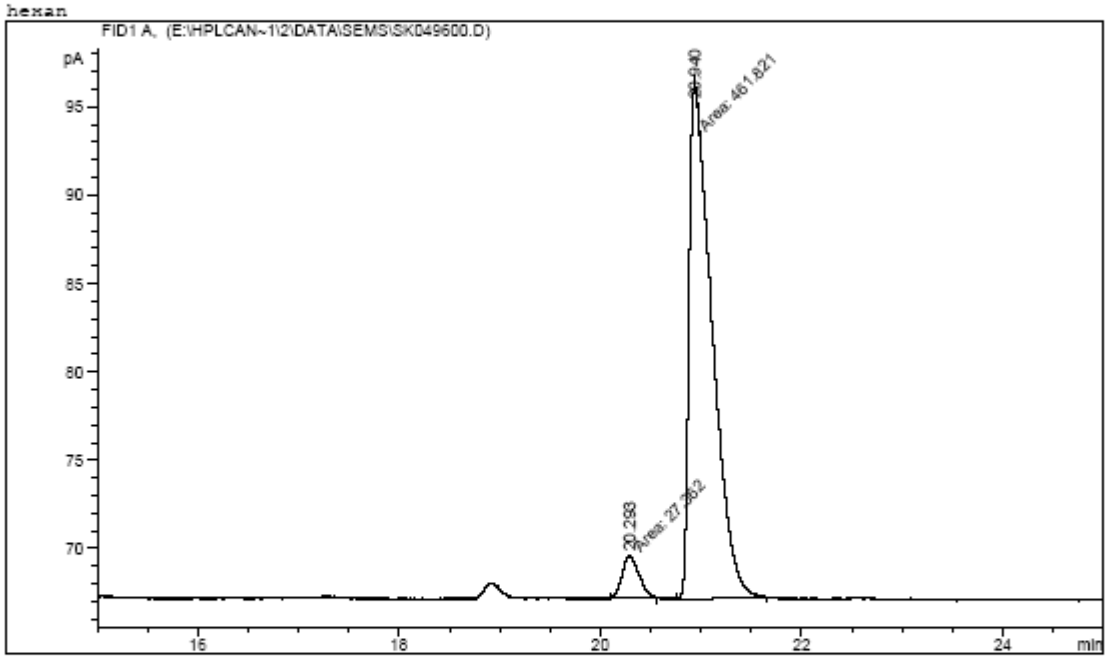
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  20.051 MM    0.2478  536.58832   36.09711  49.9674
   2  20.887 MM    0.2735  537.28839   32.73851  50.0326

Totals :                  1073.87671   68.83562
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049600.D Sample Name: 3-kloro benz

2.4 helyum 135 derece sabit 3-kloro benzaldehite dieti
l cinko katýlma ürünü

```
==========================================================================
Injection Date  : 27.04.2008 17:17:26
Sample Name     :  3-kloro benz                  Location : Vial 1
Acq. Operator   : murat                             Inj :   1
Acq. Instrument : Instrument 2                  Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\3KLORO.M
Last changed    : 4/27/2008 12:34:02 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/6/2010 11:57:15 PM
                  (modified after loading)
hexan
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

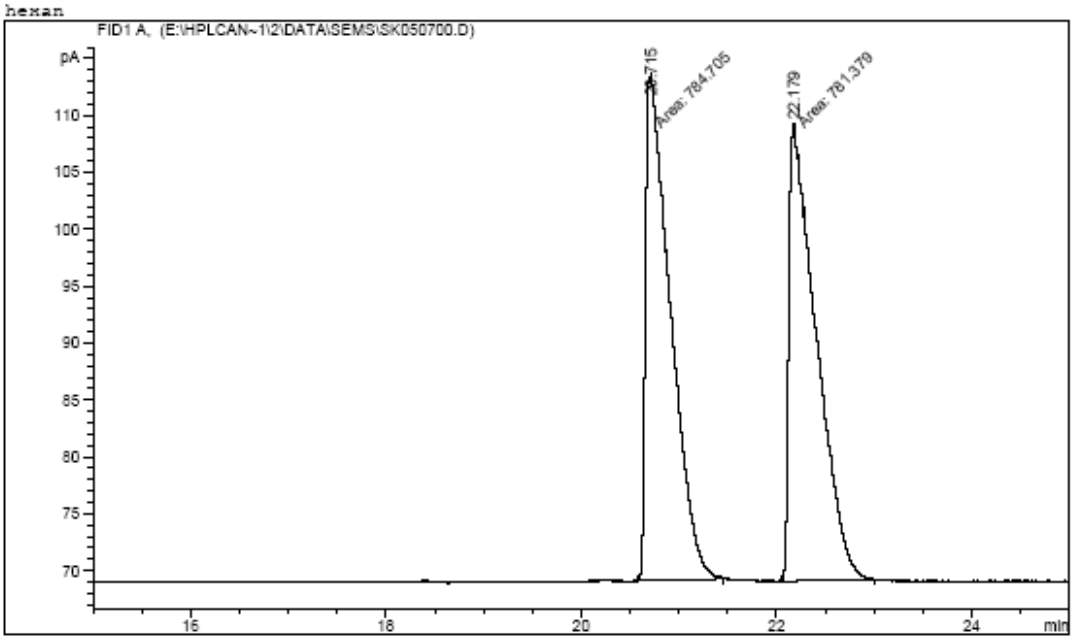
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  20.293 MM    0.1880   27.36200    2.42600   5.5934
   2  20.940 MM    0.2595  461.82095   29.65526  94.4066

Totals :                   489.18295   32.08126
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK050700.D Sample Name: 4-Kloro benz

2.4 helyum 135 derece sabit 4-Kloro benzaldehite dieti
1 cinko katýlma ürünü rasemik

```
======================================================================
Injection Date  : 27.04.2008 14:15:29
Sample Name     : 4-Kloro benz                    Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                  Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\4KLORO.M
Last changed    : 4/26/2008 12:51:54 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 1:07:42 AM
                  (modified after loading)
hexan
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

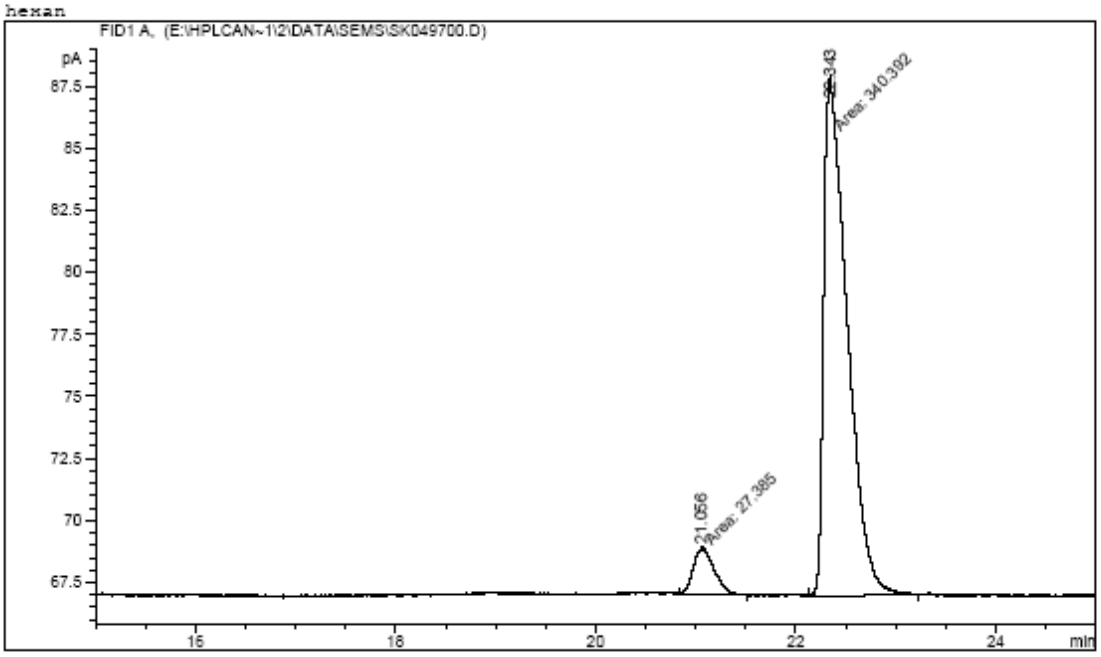
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [pA*s]     [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  20.715 MM    0.2933  784.70520   44.59794  50.1062
   2  22.179 MM    0.3242  781.37946   40.16769  49.8938

Totals :                  1566.08466   84.76563
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049700.D Sample Name: 4-kloro benz

2.4 helyum 135 derece sabit 4-kloro benzaldehite dieti
l cinko katýlma ürünü

```
==========================================================================
Injection Date  : 27.04.2008 17:44:46
Sample Name     :  4-kloro benz                  Location : Vial 1
Acq. Operator   : murat                             Inj :   1
Acq. Instrument : Instrument 2                 Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\4KLORO.M
Last changed    : 4/27/2008 2:12:33 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 12:11:58 AM
                  (modified after loading)
hexan
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

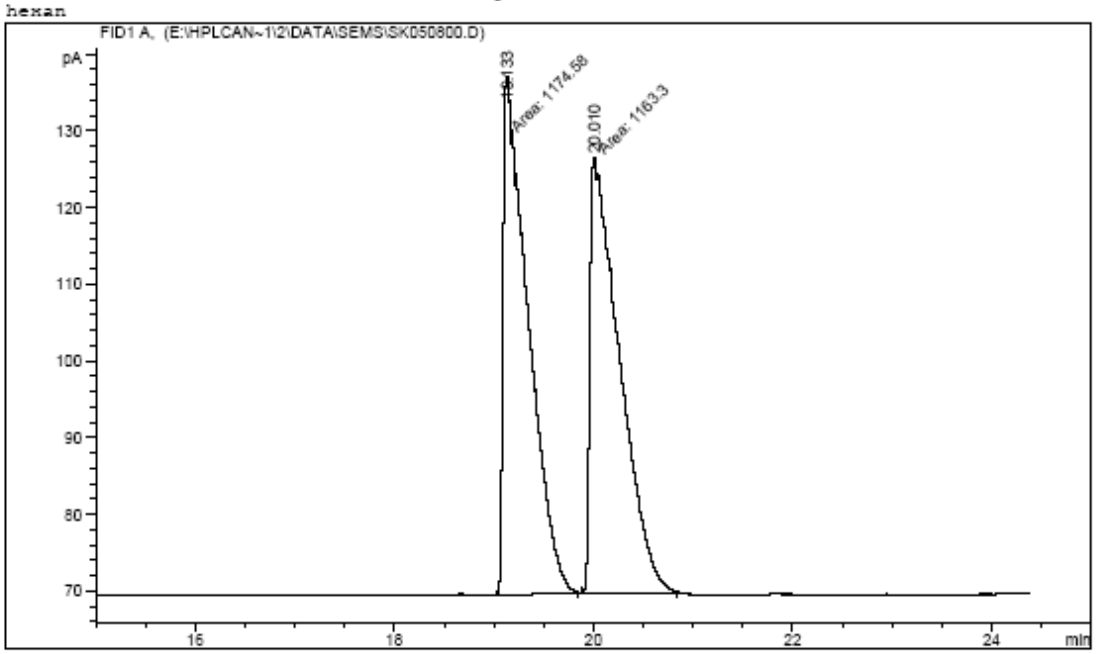
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  21.056 MM    0.2402   27.38501    1.90006   7.4461
   2  22.343 MM    0.2701  340.39243   21.00195  92.5539

Totals :                   367.77743   22.90201
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK050800.D Sample Name: 3-metil benz

2.4 helyum 135 derece sabit 3-metil benzaldehite dieti
1 cinko katýlma ürünü rasemik

```
======================================================================
Injection Date  : 27.04.2008 13:16:49
Sample Name     : 3-metil benz                    Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                 Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\4METIL.M
Last changed    : 4/27/2008 1:16:32 PM by murat
                  (modified after loading)
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 1:07:42 AM
                  (modified after loading)
hexan
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [pA*s]      [pA]       %
----|-------|----|-------|----------|----------|--------|
   1  19.133 MM    0.2890 1174.58142   67.74236  50.2413
   2  20.010 MM    0.3419 1163.29834   56.70794  49.7587

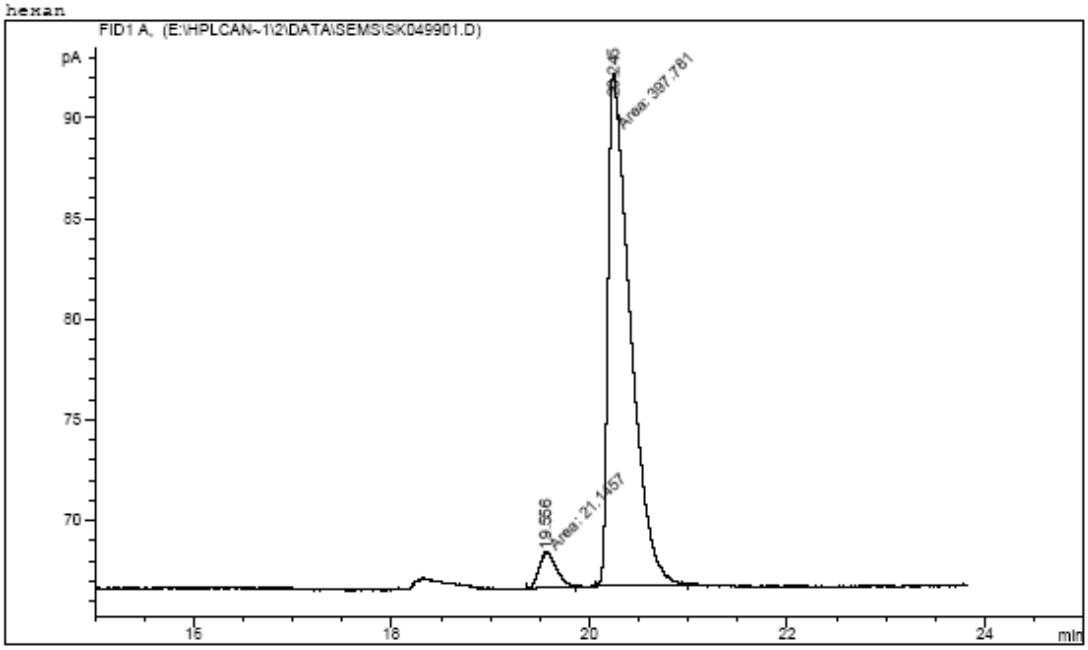
Totals :                  2337.87976  124.45029
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049901.D Sample Name: SK0499

2.4 helyum 120 derece sbt 3-metil metoduna göre

```
==========================================================================
Injection Date  : 30.04.2008 14:55:30
Sample Name     : SK0499                          Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                   Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\3METIL.M
Last changed    : 4/27/2008 1:41:26 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 12:11:58 AM
                  (modified after loading)
```

hexan




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  19.556 MM    0.2005   21.14571    1.75734   5.0476
   2  20.245 MM    0.2607  397.78131   25.42587  94.9524

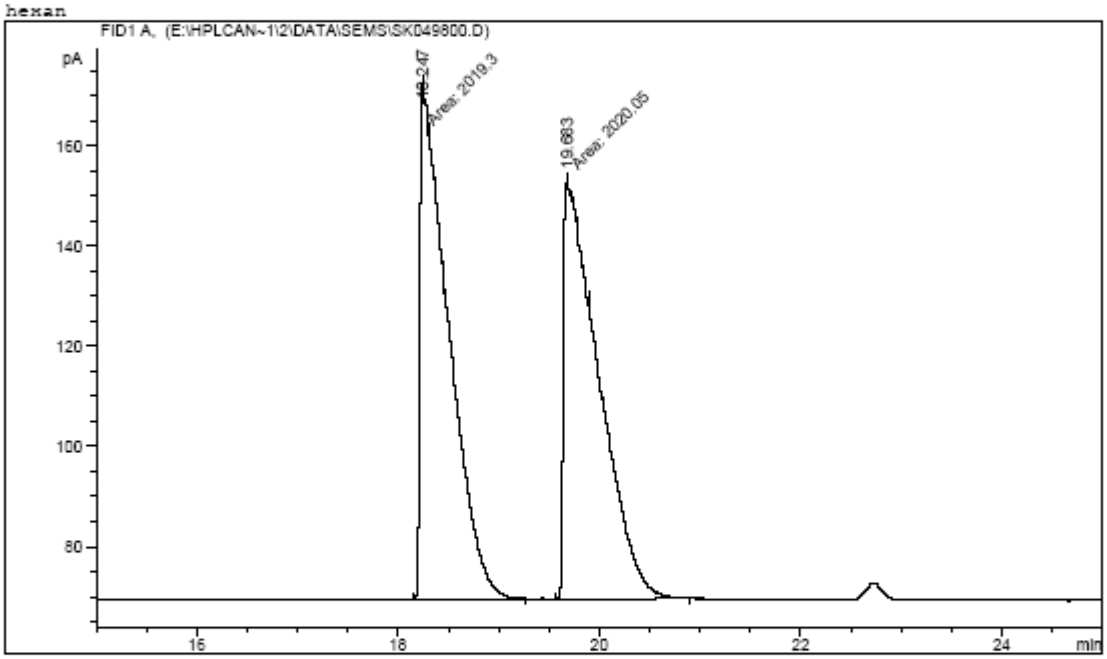
Totals :                   418.92702   27.18321
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049800.D Sample Name: 4-metil benz

2.4 helyum 135 derece sabit 4-metil benzaldehite dieti
l cinko katýlma ürünü rasemik

```
==================================================================
Injection Date  : 27.04.2008 13:46:35
Sample Name     : 4-metil benz                    Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                   Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\4METIL.M
Last changed    : 4/27/2008 1:43:30 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 12:11:58 AM
                  (modified after loading)
```

hexan




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

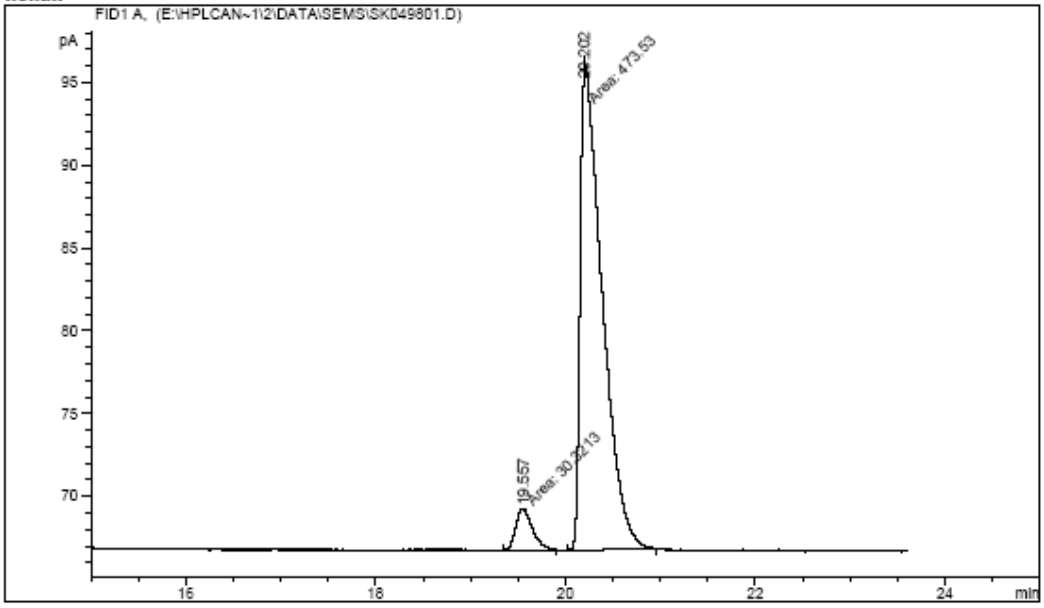
Peak RetTime Type  Width     Area      Height     Area
  #   [min]      [min]   [pA*s]      [pA]       %
----|-------|----|-------|---------|---------|--------|
   1  18.247 MM    0.3205 2019.30286  105.01093  49.9908
   2  19.683 MM    0.3947 2020.04871   85.29192  50.0092

Totals :                  4039.35156  190.30285
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK049801.D Sample Name: 4-metil benz

2.4 helyum 120 derece sabit 4-metil benzaldehite dieti
1 cinko katýlma ürünü

```
======================================================================
Injection Date  : 27.04.2008 18:17:57
Sample Name     :  4-metil benz                  Location : Vial 1
Acq. Operator   : murat                             Inj :   1
Acq. Instrument : Instrument 2                 Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\4METIL.M
Last changed    : 4/27/2008 1:43:30 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 12:11:58 AM
                  (modified after loading)
hexan
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  19.557 MM    0.2022   30.32131    2.49925   6.0179
   2  20.202 MM    0.2642  473.53000   29.87476  93.9821

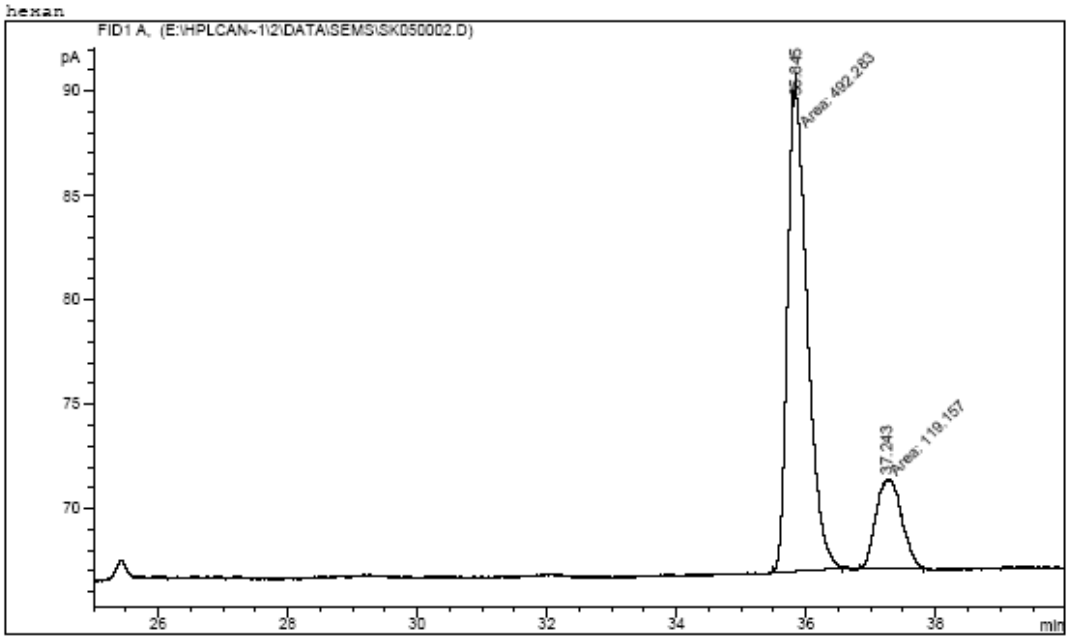
Totals :                   503.85131   32.37401
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK050002.D Sample Name: SK0500

2.4 helyum 160 derece

```
==========================================================================
Injection Date  : 30.04.2008 16:20:43
Sample Name     : SK0500                          Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                   Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\NAFTIL.M
Last changed    : 4/28/2008 5:32:11 PM by sems
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 12:43:24 AM
                  (modified after loading)
```

hexan




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

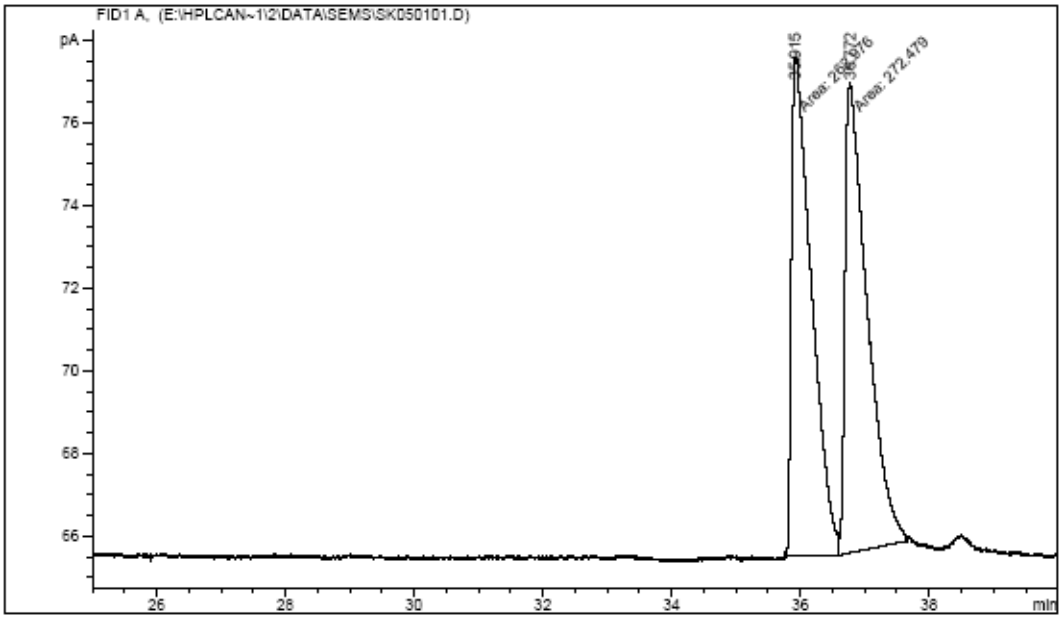
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  35.845 MM    0.3439  492.28284   23.85693  80.5121
   2  37.243 MM    0.4603  119.15686    4.31479  19.4879

Totals :                   611.43970   28.17172
```

Data File E:\HPLCAN~1\2\DATA\SEMS\SK050101.D Sample Name: SK0501

2.4 helyum piridin karbaldehýte dietil çinko katýlmasý
(initial 50 sonra 5oC/dak 100 dereceye cýk 50 dakýka be
kle 3oC/dak 140 a çýk 5 dakýka bekle)

```
==========================================================================
Injection Date  : 30.04.2008 18:43:41
Sample Name     : SK0501                          Location : Vial 1
Acq. Operator   : murat                              Inj :   1
Acq. Instrument : Instrument 2                   Inj Volume : Manually
Acq. Method     : C:\HPCHEM\2\METHODS\PIRIDIN.M
Last changed    : 4/30/2008 6:38:45 PM by murat
Analysis Method : E:\HPLCAN~1\2\DATA\XXXX\SEVDA\ALKOL100.D\RUN.M
Last changed    : 12/7/2010 12:43:24 AM
                  (modified after loading)
hexan
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1  35.915 MM    0.3626  262.97644   12.08604  49.1126
   2  36.772 MM    0.3986  272.47940   11.39236  50.8874

Totals :                   535.45584   23.47839
```

Crystal structure determination

For the crystal structure determination, the single-crystal of the compound **23a** was used for data collection on a four-circle Rigaku R-AXIS RAPID-S diffractometer (equipped with a two-dimensional area IP detector). The graphite-monochromatized Mo K_α radiation (λ=0.71073 Å) and oscillation scans technique with$\Delta\omega$=5˚ for one image were used for data collection. The lattice parameters were determined by the least-squares methods on the basis of all reflections with $F^2>2\sigma(F^2)$. Integration of the intensities, correction for Lorentz and polarization effects and cell refinement was performed using CrystalClear (Rigaku/MSC Inc., 2005) software.[1] The structures were solved by direct methods using SHELXS-97 [2] and refined by a full-matrix least-squares procedure using the program SHELXL-97.[2] Hydrogen atoms were added at calculated positions and refined using a riding model. Anisotropic thermal displacement parameters were used for all non-hydrogen atoms. The final difference Fourier maps showed no peaks of chemical significance. ***Crystal data for 23a***: Compound ***23a*** was prepared by crystallization from ethanol solution at room temperature. $C_{20}H_{23}N_3O_3S$, crystal system, space group: monoclinic, $P2_1$; (no:4); unit cell dimensions: a=10.3331(3), b=10.8017(3), c=18.4984(5)Å, α=90, β=102.951(45), γ=90˚; volume: 2012.2(4) $Å^3$; Z=4; calculated density: 1.27 g/cm^3; absorption coefficient: 0.185 mm^{-1}; F(000): 816; θ-range for data collection 2.1 – 30.6˚; refinement method: full-matrix least-square on F^2; data/parameters: 7693/498; goodness-of-fit on F^2: 1.073; final R indices [I>2σ(I)]: R_1= 0.052, wR_2=0.123; R indices (all data): R_1=0.084, wR_2=0.146; largest diff. peak and hole: 0.252 and -0.217 e $Å^{-3}$; Flack x parameter : -0.012(0.06); CCDC : 817919

Supplementary data

Crystallographic data (excluding structure factors) for the structures reported in this article have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC-818097. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; **e-mail: deposit@ccdc.cam.ac.uk).**

References

1. Rigaku/MSC, Inc., 9009 new Trails Drive, The Woodlands, TX 77381.

2. Sheldrick, G. M., *SHELXS97* and *SHELXL97*, University of Göttingen, Germany, 1997