Supporting Information

Rapid syntheses of benzopyrans from *o*-OBOC salicylaldehydes and salicylalcohols — a three-component reaction

Ryan M. Jones, Carolyn Selenski, and Thomas R. R. Pettus*

Department of Chemistry and Biochemistry, University of California, Santa Barbara, California 93106-9510

pettus@chem.ucsb.edu

Table of Contents

- S2-S4: Compound 5, Spectral data, ¹H NMR, ¹³C NMR, MS, IR
- S5-S7: Compound 6, Spectral data, ¹H NMR, ¹³C NMR, MS, IR, NOE
- S8-S10: Compound 7, Spectral data, ¹H NMR, ¹³C NMR, MS, IR
- S11-S13: Compound 8, Spectral data, ¹H NMR, ¹³C NMR, MS, IR, NOE
- S14-S16: Compound 9, Spectral data, ¹H NMR, ¹³C NMR, MS, IR, NOE
- S17-S19: Compound 10, Spectral data, ¹H NMR, ¹³C NMR, MS, IR
- S20-S23: Compound 11, Spectral data, ¹H NMR, ¹³C NMR, MS, IR, NOE
- S24-S26: Compound 12, Spectral data, ¹H NMR, ¹³C NMR, MS, IR
- S27-S29: Compound 13, Spectral data, ¹H NMR, ¹³C NMR, MS, IR
- S30-S32: Compound 14, Spectral data, ¹H NMR, ¹³C NMR, MS, IR
- S33-S36: Compound 15, Spectral data, ¹H NMR, ¹³C NMR, MS, IR, NOE
- S37-S40: Compound 16, Spectral data, ¹H NMR, ¹³C NMR, MS, IR, NOE, ¹H decoupled NMR

Compound 5: ^1H NMR [CDCl_3 , 400 MHz] δ 7.44-7.31 (m, 5H), 7.07 (d, 1H, $J = 8.24$ Hz), 6.75 (d, 1H, $J = 2.38$ Hz), 6.70 (dd, 1H, $J_1 = 8.24$ Hz, $J_2 = 2.38$ Hz), 5.06 (dd, 1H, $J_1 = 10.15$ Hz, $J_2 = 2.47$ Hz), 3.01-2.93 (m, 1H), 2.82-2.76 (m, 1H), 2.25-2.19 (m, 1H), 2.13-2.03 (m, 1H), 1.56 (s, 9H); ^{13}C NMR [CDCl_3 , 100 MHz] δ 155.7, 152.2, 150.3, 141.6, 130.0, 128.7, 128.1, 126.2, 119.6, 113.6, 110.2, 94.6, 83.6, 30.0, 27.9, 24.9; IR [CH_2Cl_2 solution, ν_{max} cm^{-1}] 3627, 2936, 1756, 1733; MS (EI) m/z 226 (78), 104 (24), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{20}\text{H}_{22}\text{O}_4$ 326.1518, found 326.1528.

	FREQUENCY	PPM	HEIGHT
1	15661.177	155.729	8.4
2	15307.925	152.216	5.5
3	15110.317	150.251	8.5
4	14239.777	141.595	10.4
5	13077.785	130.040	25.2
6	12947.318	128.743	50.8
7	12884.755	128.121	24.8
8	12889.437	128.179	45.8
9	12031.000	119.632	8.9
10	11421.393	113.570	23.9
11	11082.637	110.201	23.4
12	9515.512	94.618	6.4
13	8410.742	83.633	11.5
14	7852.253	78.080	25.5
15	7738.846	77.549	172.4
16	7766.801	77.230	174.1
17	7735.520	78.319	170.0
18	3016.595	29.996	21.8
19	2806.017	27.902	85.9
20	2506.936	24.928	21.6

File:PET1220B Ident:36_40 Mer Def 0.25 Acq:20-DEC-2000 08:41:33 Cal:CAL1220C_1
70S EI+ Magnet BpM:57 BpI:2736373 TIC:11191670 Flags:HALL Mass Range:40-600
File Text:JONES/PETTUS, RVIV-41, MW 326, LREI

Peaktable of RJ1190.IRS, 17
 Peaks
 Threshold: 95, Noise: 3, No
 Range Selection

No.	Pos. (1/cm)
1	909.4
28.660	
2	1002.0
91.402	
3	1018.3
83.582	
4	1045.3
88.885	
5	1112.9
89.575	
6	1141.8
49.685	
7	1249.8
58.829	
8	1255.6
20.422	
9	1273.9
40.960	
10	1372.3
87.426	
11	1500.5
93.177	
12	1732.9
82.655	
13	1756.1
77.062	
14	2330.8
88.376	
15	2359.7
81.635	
16	2936.4
93.473	
17	3626.9
94.464	

Compound 6: ^1H NMR [CDCl_3 , 400 MHz] δ 7.46-7.34 (m, 5H), 7.28-7.25 (m, 1H), 6.76-6.73 (m, 2H), 5.10-5.07 (m, 1H), 3.20-3.14 (m, 1H), 2.24-2.19 (m, 1H), 1.86-1.76 (m, 1H), 1.56 (s, 9H), 1.36 (d, 3H, $J = 6.78$ Hz); ^{13}C NMR [CDCl_3 , 100 MHz] δ 155.5, 152.2, 150.3, 141.6, 128.8, 128.2, 127.8, 126.3, 125.0, 113.7, 110.1, 94.6, 83.7, 40.0, 30.1, 27.9, 20.3; IR [CH_2Cl_2 solution, ν_{max} cm^{-1}] 3054, 2930, 1757; MS (EI) m/z 240 (57), 225 (59), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{21}\text{H}_{24}\text{O}_4$ 340.1675, found 340.1682.

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2981.422	7.455	3.2	40	1260.685	3.152	1.8
2	2979.591	7.450	4.8	41	1254.093	3.136	1.1
3	2977.760	7.445	1.8	42	894.454	2.236	2.2
4	2972.816	7.493	11.6	43	892.439	2.231	2.4
5	2971.534	7.430	10.2	44	888.777	2.222	2.3
6	2968.787	7.423	6.7	45	886.946	2.218	2.4
7	2967.689	7.420	9.7	46	880.903	2.203	2.9
8	2965.674	7.415	2.7	47	878.889	2.198	2.8
9	2961.648	7.405	7.3	48	875.226	2.188	2.7
10	2960.730	7.403	13.5	49	873.212	2.183	3.7
11	2959.082	7.399	4.6	50	742.101	1.855	2.8
12	2954.888	7.388	2.6	51	730.199	1.826	5.2
13	2952.856	7.383	4.7	52	728.368	1.821	2.7
14	2943.517	7.380	2.8	53	718.479	1.796	2.8
15	2941.888	7.356	5.0	54	716.648	1.792	4.5
16	2940.038	7.351	2.5	55	704.746	1.782	2.3
17	2937.475	7.345	1.9	56	643.585	1.609	1.0
18	2934.728	7.338	4.3	57	624.358	1.561	110.8
19	2931.981	7.331	1.4	58	623.442	1.559	264.5
20	2929.418	7.324	1.1	59	566.493	1.416	1.6
21	2927.770	7.320	1.9	60	559.352	1.399	2.1
22	2926.305	7.317	1.0	61	549.097	1.373	26.8
23	2907.827	7.270	75.0	62	542.322	1.356	26.5
24	2899.387	7.249	4.1				
25	2898.471	7.247	5.8				
26	2897.555	7.245	4.7				
27	2703.453	6.759	4.9				
28	2700.889	6.753	7.7				
29	2695.213	6.739	1.9				
30	2692.649	6.732	16.9				
31	2692.100	6.731	13.1				
32	2690.269	6.727	5.0				
33	2640.574	5.102	3.6				
34	2638.560	5.097	3.9				
35	2628.855	5.073	4.0				
36	2627.024	5.068	3.7				
37	1279.548	3.199	1.0				
38	1272.770	3.182	1.8				
39	1268.381	3.166	1.9

INDEX	FREQUENCY	PPM	HEIGHT
1	15644.391	155.562	5.9
2	15308.688	152.224	4.6
3	15110.317	150.251	5.7
4	14239.777	141.595	7.5
5	12845.844	128.758	34.0
6	12835.911	128.212	18.0
7	12657.286	127.646	17.8
8	12637.066	126.255	39.3
9	12370.414	124.885	7.7
10	11433.600	113.681	18.1
11	11073.482	110.110	15.8
12	8517.038	84.634	4.1
13	8847.156	87.873	-3.6
14	8413.030	83.656	8.2
15	7898.505	78.520	14.5
16	7789.845	77.548	219.0
17	7766.801	77.230	220.2
18	7734.757	76.911	211.8
19	4022.180	39.885	16.3
20	3024.225	30.072	15.7
21	3006.985	29.920	1.0
22	2806.017	27.892	63.3
23	2524.484	25.102	3.8
24	2044.580	20.331	17.0
25	124.203	1.235	4.2

File: PET091001A Ident: 28_30 Mer Def 0.25 Acq: 10-SEP-2001 07:32:01 Cal: CAL091001B_1
 70S EI+ Magnet BpM: 57 BpI: 1114581 TIC: 5734785 Flags: HALL Mass Range: 40-850
 File Text: jones/pettus, exp523, mw340, lrei

Peaktable of EXP523.IRS.
16 Peaks
Threshold: 95, Noise:
2, No Range Selection

No.	Inten. (%T)	Pos. (1/cm)
1		1053.1
90.151		
2		1123.5
81.254		
3		1140.8
48.742		
4		1261.4
48.617		
5		1279.7
81.106		
6		1371.3
93.428		
7		1427.2
97.382		
8		1456.2
97.297		
9		1497.6
90.368		
10		1587.3
96.011		
11		1612.4
94.388		
12		1757.0
76.381		
13		2854.5
94.816		
14		2929.7
89.620		
15		2963.4
90.787		
16		3054.1
92.367

Compound 7: ^1H NMR [CDCl_3 , 400 MHz] δ 7.35-7.30 (m, 2H), 7.28-7.24 (m, 1H), 7.21-7.19 (m, 2H), 6.75 (d, 1H, $J = 2.38$ Hz), 6.69 (dd, 1H, $J_1 = 8.42$ Hz, $J_2 = 0.92$ Hz), 6.60 (dd, 1H, $J_1 = 8.42$ Hz, $J_2 = 2.38$ Hz), 5.27 (dd, 1H, $J_1 = 8.61$ Hz, $J_2 = 2.38$ Hz), 4.18-4.14 (m, 1H), 4.11-4.03 (m, 1H), 3.71-3.63 (m, 1H), 2.41-2.36 (m, 1H), 2.23-2.15 (m, 1H), 1.56 (s, 9H), 1.27 (t, 3H, $J = 7.05$ Hz); ^{13}C NMR [CDCl_3 , 100 MHz] δ 154.2, 152.1, 150.6, 144.0, 130.1, 128.8, 127.0, 123.6, 114.0, 110.2, 99.9, 94.6, 83.7, 64.7, 41.2, 37.3, 27.9, 15.4; IR [CH_2Cl_2 solution, ν_{max} cm^{-1}] 2982, 1757; MS (EI) m/z 311 (34), 224 (56), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{22}\text{H}_{26}\text{O}_5$ 370.1780, found 370.1783.

INDEX	FREQUENCY	PPM	HEIGHT
1	15510.110	154.228	17.9
2	15293.429	152.072	9.4
3	15146.940	150.815	13.8
4	14476.585	143.969	18.7
5	13886.074	139.063	42.8
6	12952.659	128.796	177.0
7	12774.889	127.028	47.8
8	12426.215	123.561	18.0
9	11461.967	113.964	46.5
10	11081.111	110.188	47.8
11	10847.297	99.906	42.5
12	8513.886	94.603	2.5
13	8416.083	83.686	17.1
14	7798.846	77.549	42.3
15	7766.801	77.230	44.5
16	7734.757	76.911	42.9
17	6504.861	64.682	39.0
18	4753.099	47.263	2.6
19	4147.306	41.239	40.1
20	3748.277	37.271	37.9
21	2803.729	27.879	148.0
22	1544.077	15.354	39.4

Peaktable of EXP221.IRS, 16
Peaks
Threshold: 40, Noise: 2,
No Range Selection

No.	Pos. (1/cm)
1	888.2
14.856	
2	960.5
34.866	
3	1005.8
16.311	
4	1027.0
16.239	
5	1038.6
24.148	
6	1054.0
18.389	
7	1065.6
15.917	
8	1113.8
12.717	
9	1135.0
0.257	
10	1244.0
0.606	
11	1279.7
1.493	
12	1371.3
14.771	
13	1496.7
15.009	
14	1592.1
39.513	
15	1757.0
0.815	
16	2981.7
29.332	

Compound 8: ^1H NMR [CDCl_3 , 400 MHz] δ 7.09 (dt, 1H, $J_1 = 8.06$ Hz, $J_2 = 0.92$ Hz), 6.72-6.69 (m, 2H), 5.95-5.86 (m, 1H), 5.22-5.09 (m, 3H), 4.03-3.95 (m, 1H), 3.67-3.59 (m, 1H), 3.53-3.47 (m, 1H), 2.23-2.17 (m, 1H), 1.97-1.89 (m, 1H) 1.56 (s, 9H), 1.25 (t, 3H, $J = 7.05$ Hz); ^{13}C NMR [CDCl_3 , 100 MHz] δ 153.1, 152.1, 150.7, 141.3, 129.8, 121.8, 115.9, 113.9, 110.3, 98.8, 83.7, 64.6, 39.2, 34.2, 27.9, 15.4; IR [CH_2Cl_2 solution, $\nu_{\text{max}}\text{cm}^{-1}$] 2980, 1757, 1614; MS FAB m/z 261 (24), 174 (54), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{18}\text{H}_{24}\text{O}_5$ 320.1624, found 320.1616.

INDEX	FREQUENCY	PPM	HEIGHT
1	15401.769	153.148	18.8
2	15294.191	152.079	13.5
3	15155.332	150.699	18.2
4	14206.207	141.281	39.4
5	13052.807	129.790	47.7
6	12251.498	121.824	18.2
7	11659.437	115.937	37.1
8	11458.778	113.942	44.9
9	11096.267	110.277	48.1
10	9937.430	98.814	44.1
11	8415.319	83.879	21.5
12	7788.846	77.548	139.4
13	7766.801	77.230	136.9
14	7735.520	76.819	133.0
15	6481.890	64.553	38.0
16	3942.089	39.188	36.9
17	3440.039	34.208	38.0
18	2805.254	27.894	180.4
19	1546.368	15.378	41.7

File:PET0724B Ident:54_56 Mer Def 0.25 Acq:24-JUL-2001 06:04:30 Cal:CAL0724A_1
70S FAB+ Magnet BpM:57 BpI:6126142 TIC:22024976 Flags:HALL Mass Range:40-750
File Text:jones/pettus, exp420, mw 320, lrei

Peaktable of EXP222.IRS, 18
Peaks
Threshold: 50, Noise: 2,
No Range Selection

No.	Pos. (1/cm)
1	888.2
34.449	
2	909.4
24.668	
3	1001.0
47.059	
4	1023.2
27.799	
5	1057.9
30.239	
6	1109.0
34.304	
7	1142.7
1.265	
8	1246.9
2.733	
9	1260.4
42.627	
10	1264.3
38.671	
11	1277.8
7.831	
12	1371.3
31.269	
13	1497.6
38.156	
14	1588.3
63.639	
15	1614.3
59.121	
16	1757.0
6.979	
17	2933.5
57.581	
18	2980.8
44.845

Compound 9. ^1H NMR [CDCl_3 , 400 MHz] δ 7.54-7.52 (m, 2H), 7.38-7.36 (m, 3H), 6.75 (dd, 1H, $J_1 = 8.42$ Hz, $J_2 = 0.73$ Hz), 6.66 (d, 1H, $J = 2.38$ Hz), 6.57 (dd, 1H, $J_1 = 8.42$ Hz, $J_2 = 2.56$ Hz), 5.09-5.07 (m, 1H), 4.00-3.92 (m, 1H), 3.66-3.58 (m, 1H), 2.64-2.61 (m, 1H), 2.15-2.09 (m, 1H), 2.06-2.00 (m, 1H), 1.55 (s, 9H), 1.25 (t, 3H, $J = 7.05$ Hz) 0.32 (s, 3H), 0.26 (s, 3H); ^{13}C NMR [CDCl_3 , 100 MHz] δ 152.7, 152.1, 149.2, 139.5, 134.3, 129.1, 128.5, 128.0, 122.6, 113.6, 110.4, 98.3, 83.5, 64.4, 29.2, 27.9, 22.3, 15.3, -2.8, -3.9; IR [CH_2Cl_2 solution, $\nu_{\text{max}} \text{cm}^{-1}$] 2983, 1757, 1613; MS (EI) m/z 148 (67), 135 (77), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{24}\text{H}_{32}\text{O}_5\text{Si}$ 428.2019, found 428.2018.

INDEX	FREQUENCY	PPM	HEIGHT
1	15355.991	152.694	9.0
2	15300.295	152.140	7.5
3	15006.555	149.219	8.5
4	14035.014	139.539	7.9
5	13501.992	134.258	44.2
6	12387.755	129.145	18.9
7	12319.088	128.462	18.9
8	12874.074	128.015	43.2
9	12331.607	122.621	8.7
10	11424.444	113.600	22.0
11	11097.898	110.353	21.4
12	8880.971	88.252	18.8
13	8394.719	83.474	9.9
14	7798.846	77.549	137.0
15	7766.801	77.230	141.3
16	7734.757	76.911	136.7
17	7721.786	76.782	-6.3
18	6475.105	64.386	18.9
19	2934.195	29.178	15.2
20	2808.780	27.910	73.1
21	2802.203	27.884	-8.1
22	2248.003	22.333	18.3
23	1538.736	15.301	19.2
24	-282.456	-2.808	17.9
25	-387.745	-3.856	18.5

File:PET1218A Ident:78_85-97_104 Mer Def 0.25 Acq:18-DEC-2000 08:59:38 Cal:CAL1218B_1
 70S EI+ Magnet BpM:57 BpI:594211 TIC:3938745 Flags:HALL Mass Range:40-750
 File Text:jones/pettus, rj-iv38, mw 428, lrei

Peaktable of EXP338.IRS, 15
 Peaks
 Threshold: 75, Noise: 2,
 No Range Selection

No.	Pos. (1/cm)
1	1011.6
64.389	
2	1060.8
53.403	
3	1104.2
50.453	
4	1142.7
13.358	
5	1247.9
22.943	
6	1279.7
43.055	
7	1371.3
64.838	
8	1427.2
77.692	
9	1496.7
66.494	
10	1587.3
85.701	
11	1613.3
89.935	
12	1757.0
30.562	
13	2255.6
92.556	
14	2380.0
95.512	
15	2982.7
71.958

Compound 10. ^1H NMR [CDCl_3 , 400 MHz] δ 7.11 (dt, 1H, $J_1 = 7.69$ Hz, $J_2 = 1.28$ Hz), 6.73-6.70 (m, 2H), 5.48 (d, 1H, $J = 2.56$ Hz), 4.00-3.94 (m, 1H), 3.77-3.73 (m, 1H), 3.14 (quintet, 1H, $J = 6.41$ Hz), 2.04-2.00 (m, 1H), 1.77-1.65 (m, 1H), 1.64-1.58 (m, 3H), 1.56 (s, 9H), 1.30 (d, 3H, $J = 7.14$ Hz); ^{13}C NMR [CDCl_3 , 100 MHz] δ 154.3, 152.2, 150.6, 127.2, 122.3, 113.8, 109.2, 97.5, 83.7, 61.1, 38.0, 32.0, 27.9, 24.7, 17.6, 15.4; IR [CH_2Cl_2 solution, ν_{max} cm^{-1}] 2978, 1758, 1613; MS (EI) m/z 220 (53), 205 (75), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{18}\text{H}_{24}\text{O}_5$ 320.1624, found 320.1617.

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2907.627	7.270	78.9	40	894.125	1.736	7.2
2	2848.664	7.123	14.0	41	890.463	1.726	4.0
3	2847.382	7.119	18.5	42	887.899	1.720	3.8
4	2846.466	7.117	9.9	43	886.068	1.715	3.6
5	2840.973	7.103	11.0	44	881.307	1.703	3.7
6	2839.691	7.100	20.6	45	860.432	1.651	6.7
7	2838.592	7.097	16.3	46	855.305	1.638	14.6
8	2690.818	6.728	14.6	47	844.887	1.612	12.4
9	2688.437	6.722	23.7	48	833.697	1.584	5.6
10	2680.747	6.703	72.1	49	831.133	1.578	67.4
11	2679.282	6.699	20.3	50	824.987	1.562	810.7
12	2193.659	5.485	18.4	51	824.926	1.312	88.6
13	2191.095	5.478	19.5	52	817.785	1.295	88.8
14	1603.844	4.010	5.0	53	810.825	1.277	6.8
15	1601.463	4.004	6.1	54	803.868	1.260	16.0
16	1590.476	3.977	11.5				
17	1579.672	3.950	7.4				
18	1577.292	3.944	6.7				
19	1506.426	3.767	8.9				
20	1504.778	3.762	6.4				
21	1501.482	3.754	9.7				
22	1495.073	3.738	7.7				
23	1493.608	3.734	5.3				
24	1491.777	3.730	5.2				
25	1490.312	3.726	7.3				
26	1263.065	3.158	6.3				
27	1256.656	3.142	6.4				
28	1250.430	3.126	6.8				
29	814.798	2.037	6.3				
30	812.051	2.030	6.7				
31	810.403	2.026	5.1				
32	806.375	2.016	5.2				
33	803.145	2.009	5.6				
34	801.797	2.005	6.8				
35	799.850	1.998	6.9				
36	794.472	1.986	3.7				
37	708.508	1.771	6.7				
38	703.447	1.759	5.4				
39	698.886	1.747	5.1

INDEX	FREQUENCY	PPM	HEIGHT
1	15514.888	154.272	10.0
2	15302.584	152.183	8.0
3	15142.382	150.570	9.8
4	12788.622	127.165	33.5
5	12300.326	122.310	12.0
6	11440.467	113.759	33.0
7	10978.874	109.170	32.1
8	9800.860	97.456	33.0
9	8416.845	83.684	16.8
10	7798.848	77.549	182.3
11	7768.801	77.230	171.8
12	7734.757	76.911	169.9
13	6144.742	61.101	31.2
14	5818.489	57.989	31.9
15	5218.017	51.999	28.9
16	2806.017	27.902	118.5
17	2479.469	24.655	33.5
18	1766.099	17.561	30.7
19	1553.233	15.445	27.7

File:PET0105A Ident:35_38 Mer Def 0.25 Acq: 5-JAN-2001 07:30:27 Cal:CAL0105A_1
 70S EI+ Magnet BpM:57 BpI:2431144 TIC:11574912 Flags:HALL Mass Range:40-600
 File Text:jones/pettus, rj-iv-39b, mw 320, lrei

Peaktable of EXP339.IRS, 16
Peaks
Threshold: 70, Noise: 2,
No Range Selection

No.	Pos. (1/cm)
1	1036.7
71.112	
2	1092.6
43.831	
3	1121.5
13.842	
4	1146.6
17.457	
5	1210.2
68.878	
6	1249.8
27.303	
7	1269.1
59.598	
8	1279.7
34.551	
9	1371.3
65.406	
10	1431.1
74.551	
11	1499.6
63.667	
12	1591.2
74.037	
13	1613.3
76.313	
14	1758.0
29.861	
15	2941.2
74.298	
16	2977.9
72.186	

Compound 11. Major isomer: ^1H NMR [CDCl_3 , 400 MHz] δ 7.61-7.28 (m, 5H), 7.17 (dd, 1H, $J_1 = 8.42$ Hz, $J_2 = 0.92$ Hz), 6.89 (d, 1H, $J = 2.38$ Hz), 6.79 (dd, 1H, $J_1 = 8.42$ Hz, $J_2 = 2.38$ Hz), 3.13 (s, 3H), 2.73 (sextet, 1H, $J = 6.78$ Hz), 2.15 (qd, 2H, $J_1 = 42.3$ Hz, $J_2 = 13.73$ Hz, $J_3 = 6.23$ Hz) 1.58 (s, 9H), 1.43 (d, 3H, $J = 7.14$ Hz); ^{13}C NMR [CDCl_3 , 100 MHz] δ 153.0, 152.2, 150.3, 141.2, 128.6, 127.5, 126.5, 125.6, 114.2, 110.2, 102.3, 101.0, 83.6, 50.3, 41.8, 27.9, 27.5, 22.2; IR [CH_2Cl_2 solution, ν_{max} cm^{-1}] 2967, 2935, 1758; MS (EI) m/z 255 (29), 223 (24), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{22}\text{H}_{26}\text{O}_5$ 370.1780, found 370.1790.

INDEX	FREQUENCY	PPM	HEIGHT
1	15384.984	152.982	9.6
2	15304.110	152.178	6.6
3	15114.885	150.297	11.2
4	14204.881	141.246	15.2
5	12932.059	128.591	54.3
6	12929.007	128.581	62.9
7	12909.170	128.384	35.8
8	12824.481	127.522	10.4
9	12721.481	126.497	82.4
10	12710.037	126.384	23.9
11	12639.740	125.825	13.0
12	11482.430	114.177	27.4
13	11098.859	110.361	12.6
14	11078.822	110.183	27.9
15	10287.630	102.296	12.5
16	8412.258	83.648	20.3
17	7798.846	77.549	153.7
18	7768.801	77.230	161.2
19	7734.757	76.911	163.7
20	5084.224	50.558	11.5
21	5082.881	50.343	25.3
22	4402.135	43.773	12.0
23	4208.817	41.831	24.4
24	2808.308	27.925	125.8
25	2783.292	27.477	30.0
26	2579.417	25.648	11.1
27	2229.981	22.174	28.3
28	1936.240	19.253	11.1

Peaktable of EXP582.IRS,
14 Peaks
Threshold: 80, Noise:
2, No Range Selection

No.	Inten. (%T)	Pos. (1/cm)
1		1023.2
74.963		
2		1046.3
67.002		
3		1120.6
44.525		
4		1143.7
13.186		
5		1248.8
18.160		
6		1280.6
38.200		
7		1371.3
72.974		
8		1425.3
79.280		
9		1498.6
69.823		
10		1591.2
80.984		
11		1613.3
82.760		
12		1758.0
34.714		
13		2934.5
80.259		
14		2967.3
79.656

Compound 12. NMR yield, 52%. ^1H NMR [CDCl_3 , 400 MHz] δ 7.61 (s, 1H), 7.18 (d, 1H, $J = 8.29$ Hz), 6.94 (dd, 1H, $J_1 = 8.29$ Hz, $J_2 = 2.3$ Hz), 6.87 (d, 1H, $J = 2.3$ Hz), 3.93 (q, 1H, $J = 6.76$ Hz), 2.32 (s, 3H), 1.57 (s, 9H), 1.27 (d, 3H, $J = 6.91$ Hz); ^{13}C NMR [CDCl_3 , 100.6 MHz] δ 195.9, 152.0, 150.1, 129.7, 124.3, 122.0, 118.2, 109.9, 84.1, 77.4, 29.9, 27.9, 27.3, 25.6, 25.5; IR [CH_2Cl_2 , $\nu_{\text{max}} \text{cm}^{-1}$] 2928, 1759, 1644, 1587, 1501; MS (EI) m/z 204 (21), 189 (90), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{17}\text{H}_{20}\text{O}_5$, 304.1311, found 304.1315.

INDEX	FREQUENCY	PPM	HEIGHT
1	3043.213	7.609	7.6
2	2807.627	7.270	19.2
3	2877.531	7.195	3.0
4	2889.239	7.174	3.5
5	2780.793	6.953	2.3
6	2778.488	6.947	2.6
7	2772.501	6.932	1.9
8	2770.044	6.926	2.3
9	2747.825	6.870	3.4
10	2745.322	6.864	2.8
11	1675.408	3.939	2.0
12	1588.851	3.822	2.0
13	1555.981	3.390	1.4
14	827.417	2.319	25.9
15	627.222	1.568	80.0
16	624.819	1.562	10.8
17	512.518	1.281	11.6
18	505.609	1.264	15.3

INDEX	FREQUENCY	PPM	HEIGHT
1	12686.012	195.889	2.1
2	15285.789	151.986	6.3
3	15098.480	158.054	1.9
4	13041.825	129.684	5.0
5	12504.837	134.385	2.2
6	12272.859	122.636	1.5
7	11800.615	119.236	6.6
8	11048.830	109.075	5.9
9	8460.334	84.128	3.3
10	7788.848	77.848	140.1
11	7787.401	77.435	6.0
12	7786.801	77.230	151.2
13	7734.757	76.911	140.4
14	5809.728	39.828	3.1
15	2804.491	27.887	21.9
16	2756.321	27.348	6.0
17	2571.025	25.565	6.4
18	2566.447	25.520	6.0

Peaktable of 252B2.IRS, 11
Peaks
Threshold: 100, Noise:
2, No Range Selection

No.	Pos. (1/cm)
1	1102.2
99.523	
2	1149.5
72.106	
3	1193.9
83.575	
4	1248.8
83.475	
5	1280.6
91.302	
6	1371.3
98.544	
7	1501.5
96.380	
8	1587.3
99.221	
9	1644.2
88.693	
10	1759.0
85.395	
11	2928.7
98.939	

Compound 13. NMR yield, 56%. ^1H -NMR [CDCl_3 , 400 MHz] δ 7.95 (d, 2H, $J = 7.37$ Hz), 7.58 (t, 1H, $J = 7.37$ Hz), 7.45 (t, 2H, $J = 7.7$ Hz), 7.18 (d, 1H, $J = 8.29$ Hz), 6.76-6.72 (m, 2H), 3.78-3.73 (m, 1H), 3.47 (dd, 1H, $J_1 = 18.77$ Hz, $J_2 = 9.52$ Hz), 3.29 (dd, 1H, $J_1 = 18.58$ Hz, $J_2 = 3.22$ Hz), 1.54 (s, 9H), 1.41 (d, 3H, $J = 7.06$ Hz); ^{13}C -NMR [CDCl_3 , 100.6 MHz] δ 201.8, 154.8, 152.2, 150.3, 136.2, 134.0, 130.8, 128.9, 128.6, 127.1, 114.1, 111.2, 83.6, 48.7, 27.9, 26.2, 21.7; IR [CH_2Cl_2 , $\nu_{\text{max}}\text{cm}^{-1}$] 3686, 3053, 2960, 2928, 2855, 1755, 1733, 1671, 1599; MS (EI) m/z 281 (60), 57 (74), 43 (100); HRMS (EI) m/z ($\text{M}+\text{Na}$) $^+$ calcd for $\text{C}_{21}\text{H}_{24}\text{O}_5\text{Na}^+$ 379.1521, found 379.1527.

INDEX	FREQUENCY	PPM	HEIGHT
1	20293.123	201.787	8.8
2	15586.588	154.788	12.4
3	15303.347	152.170	7.6
4	15110.317	150.251	8.9
5	13895.785	138.185	7.9
6	13489.629	134.046	22.9
7	13149.503	130.753	11.5
8	12959.526	128.864	48.9
9	12829.007	128.581	48.5
10	12779.486	127.074	23.7
11	11475.563	114.106	24.5
12	11186.400	111.233	29.1
13	8411.505	83.641	11.8
14	7798.848	77.549	129.1
15	7766.801	77.230	132.1
16	7734.757	76.911	127.5
17	4895.772	48.682	21.0
18	2804.481	27.867	81.8
19	2630.538	26.157	20.0
20	2167.255	21.749	21.0

File: PET030802B Ident: 62_90 Mer Def 0.25 Acq: 8-MAR-2002 09:00:39 Cal: CAL030802B_1
70S EI+ Magnet BpM: 43 BpI: 1059494 TIC: 7551739 Flags: HALL Mass Range: 40-600
File Text: selenski/pettus, cs253b, mw 356, lrei

Peaktable of 253B.IRS, 13
Peaks
Threshold: 100, Noise:
2, No Range Selection

No.	Inten. (%T)	Pos. (1/cm)
1		1142.7
89.415		
2		1252.7
71.693		
3		1276.8
70.998		
4		1372.3
95.267		
5		1598.9
96.781		
6		1671.2
97.192		
7		1732.9
93.546		
8		1755.1
94.528		
9		2855.4
95.540		
10		2927.7
91.253		
11		2959.6
92.246		
12		3053.1
95.084		
13		3685.7
98.115		

Compound 14. ^1H NMR [CDCl_3 , 400 MHz] δ 7.06 (dt, 1H, $J_1 = 8.06$ Hz, $J_2 = 0.916$ Hz) 6.67-6.64 (m, 2H), 5.87-5.78 (m, 1H), 5.22-5.14 (m, 2H), 3.70-3.60 (m, 4H), 3.36-3.32 (m, 1H), 2.82-2.71 (m, 4H), 2.15-2.10 (m, 1H), 1.84-1.72 (m, 3H), 1.66-1.56 (m, 12H), 1.49 (s, 1H) 1.42-1.36 (m, 1H); ^{13}C NMR [CDCl_3 , 100 MHz] δ 153.4, 152.2, 150.9, 142.1, 130.3, 120.8, 116.3, 113.1, 109.8, 91.6, 83.4, 67.6, 45.0, 44.0, 38.0, 29.9, 28.0, 27.8, 22.6, 22.1; IR [CH_2Cl_2 solution, ν_{max} cm^{-1}] 2936, 2858, 1757, 1610, 1591, 1498; MS (EI) m/z 167 (100), 57 (14), 43 (15); HRMS (EI) m/z calcd for $\text{C}_{24}\text{H}_{33}\text{NO}_5$ 415.2359, found 415.2348.

INDEX	FREQUENCY	PPM	HEIGHT
1	15430.761	153.437	4.8
2	15304.872	152.106	11.0
3	15178.220	150.926	14.1
4	14288.369	142.088	10.2
5	13100.673	130.268	32.3
6	12144.680	120.762	9.5
7	11742.598	116.764	-4.2
8	11699.110	116.331	5.5
9	11612.132	115.468	-4.1
10	11377.903	113.137	30.7
11	11198.318	111.332	-4.2
12	11043.725	109.814	36.6
13	10886.244	108.070	-6.1
14	9212.615	91.607	5.2
15	8475.304	84.255	-5.4
16	8387.852	83.405	14.8
17	8169.645	81.236	-5.9
18	7786.845	77.549	244.5
19	7766.800	77.230	254.1
20	7734.756	76.911	251.6
21	7087.763	70.478	-5.7
22	6600.128	67.618	38.4
23	6465.949	64.295	-5.4
24	4529.548	45.040	41.9
25	4420.446	43.855	4.8
26	4210.831	41.869	-4.5
27	3816.942	37.954	8.4
28	3010.490	29.935	4.6
29	2817.461	28.018	145.0
30	2782.203	27.765	29.3
31	2274.994	22.622	43.4
32	2223.076	22.113	6.1

File: PET022802D Ident: L15 Mer Def 0.25 Acq: 28-FEB-2002 10:59:22 Cal: CAL022802A_1
 70S EI+ Magnet BpM: 167 BpI: 1979091 TIC: 7571479 Flags: HALL Mass Range: 40-600
 File Text: jones/pettus, rjVI-98, mw 415, lrei

Peaktable of 12.IRS, 14
Peaks
Threshold: 80, Noise:
1, No Range Selection

No.	Pos. (1/cm)
1	1028.9
2	1123.5
3	1142.7
4	1249.8
5	1269.1
6	1280.6
7	1371.3
8	1497.6
9	1591.2
10	1609.5
11	1711.7
12	1757.0
13	2858.3
14	2936.4

Compound 15. ¹H NMR [CDCl₃, 400 MHz] δ 7.77-7.75 (m, 2H), 7.45-7.41 (m, 2H), 7.35-7.29 (m, 5H), 7.25-7.22 (m, 1H), 6.99 (d, 1H, J = 8.42 Hz), 6.89 (d, 1H, J = 2.38 Hz), 6.75 (dd, 1H, J₁ = 8.42 Hz, J₂ = 2.38 Hz), 6.09 (s, 1H), 3.86- 3.79 (m, 2H), 3.34 (d, 1H, J = 14.83 Hz), 1.59 (s, 9H), 1.55 (d, 1H, J = 6.96 Hz); ¹³C NMR [CDCl₃, 100 MHz] δ 154.8, 152.3, 150.3, 139.6, 138.2, 129.3, 128.5, 128.4, 128.2, 128.1, 127.0, 126.7, 123.0, 114.1, 110.2, 86.0, 83.8, 52.4, 49.8, 27.9, 24.1; IR [CH₂Cl₂ solution, ν_{max}cm⁻¹] 2975, 2935, 1758, 1617; MS FAB *m/z* 136 (27), 91 (100) 57 (82); HRMS (EI) *m/z* calcd for C₂₇H₂₉NO₄ 431.2097, found 430.2113.

INDEX	FREQUENCY	PPM	HEIGHT
1	15563.517	154.757	6.9
2	15314.791	152.284	5.3
3	15111.060	150.259	7.8
4	14037.592	139.504	10.6
5	13901.021	138.226	9.1
6	13006.829	129.335	13.8
7	12927.481	128.548	30.6
8	12919.851	128.470	29.2
9	12896.200	128.235	30.6
10	12850.096	128.174	15.7
11	12774.888	127.028	14.5
12	12738.792	126.879	29.7
13	12372.044	123.023	7.4
14	11479.378	114.146	13.6
15	11066.452	110.239	12.6
16	8650.312	86.015	13.5
17	8430.578	83.830	7.7
18	7786.848	77.549	225.3
19	7786.801	77.230	226.3
20	7734.757	76.811	218.0
21	5532.083	55.009	-5.5
22	5270.387	52.407	9.6
23	5010.217	49.820	11.5
24	2809.089	27.432	47.8
25	2428.351	24.147	7.8

File:PET0724A Ident:165_180 Mer Def 0.25 Acq:24-JUL-2001 04:20:52 Cal:CAL0724A_1
70S FAB+ Magnet BpM:91 BpI:5782259 TIC:28015978 Flags:HALL Mass Range:40-750
File Text:jones/pettus, exp429, mw 431, lrei

Compound 16. ^1H NMR [CDCl_3 , 400 MHz] δ 7.15 (dd, 1H, $J_1 = 8.24$ Hz, $J_2 = 1.28$ Hz), 6.83 (dd, 1H, $J_1 = 8.24$ Hz, $J_2 = 2.38$ Hz), 6.71 (d, 1H, $J = 2.38$ Hz), 6.39-6.37 (m, 1H), 5.5 (ddd, 1H, $J_1 = 8.24$ Hz, $J_2 = 2.56$ Hz, $J_3 = 0.92$ Hz), 5.07 (td, 1H, $J_1 = 2.56$ Hz, $J_2 = 0.37$ Hz), 4.92 (dd, 1H, $J_1 = 8.24$ Hz, $J_2 = 2.93$ Hz), 3.08 (qd, 1H, $J_1 = 6.96$ Hz, $J_2 = 2.93$ Hz), 1.58-1.53 (m, 12H); ^{13}C NMR [CDCl_3 , 100 MHz] δ 155.2, 152.0, 151.8, 150.4, 126.6, 126.2, 115.1, 111.9, 101.5, 85.2, 83.6, 82.3, 31.1, 27.9, 13.7; IR [CH_2Cl_2 solution, $\nu_{\text{max}}\text{cm}^{-1}$] 2975, 2935, 1758, 1612; MS (EI) m/z 189 (46), 136 (27), 57 (100); HRMS (EI) m/z calcd for $\text{C}_{17}\text{H}_{20}\text{O}_5$ 304.1311, found 304.1316.

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2907.627	7.270	82.8	40	620.696	1.552	401.0
2	2863.862	7.161	8.9	41	613.005	1.533	46.3
3	2862.580	7.157	8.9	42	507.530	1.269	2.5
4	2855.439	7.140	10.0	43	504.600	1.262	3.0
5	2854.340	7.137	10.2				
6	2736.597	6.842	8.3				
7	2734.216	6.836	9.0				
8	2728.173	6.821	7.2				
9	2725.793	6.815	8.1				
10	2686.972	6.718	15.3				
11	2684.592	6.712	13.8				
12	2553.298	6.384	7.4				
13	2552.749	6.383	7.6				
14	2551.650	6.380	6.4				
15	2550.551	6.377	7.6				
16	2550.002	6.376	7.6				
17	2222.591	5.557	5.2				
18	2221.876	5.555	5.5				
19	2220.028	5.551	5.5				
20	2219.112	5.548	5.5				
21	2214.534	5.537	5.6				
22	2213.619	5.535	5.8				
23	2211.970	5.531	5.8				
24	2211.055	5.528	5.8				
25	2030.869	5.078	7.4				
26	2028.122	5.071	14.2				
27	2025.559	5.065	7.1				
28	1972.455	4.932	8.6				
29	1964.342	4.924	8.8				
30	1964.215	4.911	6.2				
31	1961.285	4.904	6.3				
32	1237.795	3.095	2.9				
33	1234.685	3.088	3.0				
34	1230.637	3.077	3.0				
35	1227.724	3.070	3.1				
36	822.308	2.056	2.4				
37	630.950	1.578	6.0				
38	628.119	1.573	2.7				
39	625.274	1.563	52.8

INDEX	FREQUENCY	PPM	HEIGHT
1	15605.480	155.175	14.2
2	15285.038	151.808	10.8
3	15267.488	151.814	53.0
4	15120.989	150.357	12.8
5	12728.822	126.550	13.3
6	12694.014	126.224	47.0
7	11573.222	115.080	50.4
8	11254.304	111.908	48.8
9	10212.097	101.545	45.7
10	8572.480	85.241	46.5
11	8404.838	83.572	17.7
12	8277.988	82.313	52.6
13	7788.848	77.549	146.3
14	7768.801	77.230	150.8
15	7794.757	76.911	148.4
16	3123.410	31.058	50.3
17	2806.017	27.902	173.6
18	1380.803	13.730	47.7

File: PET082801A Ident: 35_37-4_17 Mer Def 0.25 Acq: 28-AUG-2001 03:49:48 Cal: CAL082801A_1
 70S EI+ Magnet BpM: 57 BpI: 930901 TIC: 3555857 Flags: HALL Mass Range: 40-850
 File Text: jones/pettus, exp501, mw 304, lrei

Peaktable of EXP501.IRS,
16 Peaks
Threshold: 80, Noise:
4, No Range Selection

No.	Pos. (1/cm)
1	1018.3
2	1059.8
3	1118.6
4	1139.9
5	1162.0
6	1249.8
7	1278.7
8	1371.3
9	1426.3
10	1496.7
11	1595.0
12	1612.4
13	1758.0
14	2883.4
15	2934.5
16	2975.0