Organocatalytic Enantioselective Synthesis of α -Hydroxy Phosphonates

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Supporting Information

General Experimental Procedure for the Synthesis of α -Hydroxy Phosphonate from α -Keto Phosphonate: To a stirred solution of the α -keto phosphonate (0.5 mmol) and dry acetone (2.0 mL) was added L-proline (0.25 mmol) at $-30\text{ }^{\circ}\text{C}$. The reaction mixture was stirred at this temperature for the time as specified in Table 1 (monitored by TLC) and then quenched by few drops of water. The mixture was extracted with ethyl acetate ($3 \times 10\text{ mL}$), and the combined extracts were washed with brine solution (2 mL), dried over MgSO_4 , and evaporated to give the crude product. The crude product was purified by column chromatography over silica gel (4:1 ethyl acetate/hexane) to furnish the desired α -hydroxy phosphonate as a pure compound, which is characterized by its ^1H and ^{13}C NMR spectroscopic data.

The reaction conditions were optimized and L-proline was identified as the best catalyst for this reaction (Table 2).

Table 2. Optimization of the reaction conditions for the cross aldol reaction of benzoylphosphonates with acetone^a

R	catalyst	loading (mol %)	time (h)	temp (°C)	yield (%) ^b	ee (%) ^c
Me	1	50	18	rt	91	35
Me	1	50	96	-30	76	92
Me	1	20	96	-30	66	95
Me	2	30	2	rt	89	60
Me	2	30	18	-25	93	73
Me	3	20	12	rt	96	13
Et	1	50	24	rt	81	83
Et	1	50	96	-30	67	86
Et	1	20	96	-30	65	87
Et	2	50	1.3	rt	73	54
Et	3	10	17	rt	93	34
<i>i</i> -Pr	1	50	96	-30	59	82
<i>i</i> -Pr	1	20	120	-30	60	96
<i>i</i> -Pr	2	30	18	rt	95	72
<i>i</i> -Pr	2	30	36	-25	34	63

^aAll reactions were carried out with the ketophosphonate (0.50 mmol) in dry acetone (2.0 mL), with the catalyst (loading as shown in the table) for the specified reaction time and temperature. ^bYield of isolated product after column chromatography. ^cEnantioselectivity was determined by HPLC analyses, the absolute configuration of major enantiomer not determined.

Table 3. Solvent effects on the cross aldol reaction of ethyl benzoylphosphonate with acetone^a

entry	solvent	time (h)	T (°C)	yield (%) ^b	ee (%) ^c
1	Acetone ^d	24	rt	85	71
2	Acetone ^d	96	-30	65	87
2	DMF	24	rt	87	48
3	DMSO	24	rt	77	38
4	CH ₂ Cl ₂	24	rt	75	74
5	CH ₂ Cl ₂	24	-30	-	-

^aAll reactions were carried out with the ketophosphonate (0.50 mmol), the catalyst (0.1 mmol, 20 mol %), and acetone (0.5 mL) in the solvent (2.0 mL) for the specified reaction time and temperature. ^bYield of isolated product after column chromatography. ^cEnantioselectivity was determined by HPLC analyses, the absolute configuration of major enantiomer not determined. ^d2.0 mL acetone was used.

Table 4. The cross aldol reaction of ketophosphonates with acetone with 50 mol % catalyst loading^a

entry	R ¹	R ²	T (°C)	time (h)	yield (%) ^b	ee (%) ^c
1	Ph	Et	rt	24	81	83
2	Ph	Et	-30	96	67	86
3	Ph	Me	-30	96	76	92
4	Ph	<i>i</i> -Pr	-30	96	59	82
5	<i>p</i> -Cl-C ₆ H ₄	Et	-30	96	72	86
6	<i>p</i> -Cl-C ₆ H ₄	<i>i</i> -Pr	-30	96	71	89
7	<i>p</i> -F-C ₆ H ₄	Et	-30	96	57	76
8	<i>p</i>-F-C₆H₄	<i>i</i>-Pr	-30	96	68	96
9	<i>p</i> -Br-C ₆ H ₄	Et	-30	96	73	74
10	<i>p</i> -I-C ₆ H ₄	Et	-30	96	78	77
11	<i>p</i> -Me-C ₆ H ₄	Et	-30	96	63	85
12	<i>p</i> -MeO-C ₆ H ₄	Et	-30	96	32	86
13	Me	Et	rt	24	94	93
14	Me	Et	-30	96	65	94
15	PhCH ₂	Et	rt	24	87	92
16	PhCH ₂ CH ₂	Et	rt	24	86	72
17	<i>trans</i> -CH ₃ CH=CH	Et	-30	96	78	93

^aAll reactions were carried out with the ketophosphonate (0.50 mmol) in dry acetone (2.0 mL), with L-proline (0.25 mmol, 50 mol %) as the catalyst for the specified reaction time and temperature. ^bYield of isolated product after column chromatography.

^cEnantioselectivity was determined by HPLC analyses, the absolute configuration of the major enantiomer not determined.

Diethyl (1-hydroxy-3-oxo-1-phenylbutyl)phosphonate (Table 1, entry 1): ¹H NMR (500 MHz, CDCl₃) δ 1.09 (t, *J* = 7.0 Hz, 3H), 1.32 (t, *J* = 7.0 Hz, 3H), 2.17 (s, 3H), 3.31-3.42 (m, 2H), 3.64-3.69 (m, 1H), 3.84-3.89 (m, 1H), 4.13-4.15 (m, 2H), 5.15 (br s, 1H), 7.27-7.38 (m, 3H), 7.60-7.62 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 16.4 (d, *J* = 5.25 Hz), 16.6 (d, *J* = 5.25 Hz), 32.4 (d, *J* = 1.87 Hz), 47.7 (d, *J* = 2.87 Hz), 63.8 (d, *J* = 7.62), 64.2 (d, *J* = 7.62), 75.5 (d, *J*_{cp} = 166.37 Hz), 126.1, 128.4, 128.5, 139.6, 210.1 (d, *J* = 12.37 Hz).

Dimethyl (1-hydroxy-3-oxo-1-phenylbutyl)phosphonate (Table 1, entry 3): ^1H NMR (300 MHz, CDCl_3) δ 2.14 (s, 1H), 3.28 (m, 2H), 3.44 (dd, $J_1 = 2.4$ Hz, $J_2 = 10.2$ Hz, 3H), 3.79 (dd, $J_1 = 2.4$ Hz, $J_2 = 10.2$ Hz, 3H), 5.14 (d, $J = 19.5$ Hz, 1H), 7.25-7.39 (m, 3H), 7.58-7.62 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 32.5 (d, $J = 2.32$ Hz), 47.8 (d, $J = 2.85$ Hz), 54.5 (d, $J = 7.27$ Hz), 54.8 (d, $J = 7.12$ Hz), 75.9 (d, $J_{cp} = 166.42$ Hz), 126.0 (d, $J = 4.57$ Hz), 128.1 (d, $J = 3.15$ Hz), 128.6 (d, $J = 2.85$ Hz), 139.4, 209.8 (d, $J = 12.9$ Hz).

Diisopropyl (1-hydroxy-3-oxo-1-phenylbutyl)phosphonate (Table 1, entry 4): ^1H NMR (300 MHz, CDCl_3) δ 0.92 (d, $J = 6.3$ Hz, 3H), 1.19 (d, $J = 6.3$ Hz, 3H), 1.23 (d, $J = 6.3$ Hz, 3H), 1.30 (d, $J = 6.3$ Hz, 3H), 2.11 (s, 3H), 3.29-3.34 (m, 2H), 4.29-4.35 (m, 1H), 4.69-4.75 (m, 1H), 4.96 (d, $J = 18.0$ Hz, 1H), 7.22-7.38 (m, 3H), 7.58-7.61 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 23.5 (d, $J = 5.7$ Hz), 24.2 (d, $J = 4.87$ Hz), 24.3 (d, $J = 3.75$ Hz), 24.6 (d, $J = 2.85$ Hz), 32.6 (d, $J = 2.32$ Hz), 48.3 (d, $J = 3.15$ Hz), 72.3 (d, $J = 7.72$ Hz), 72.8 (d, $J = 7.42$ Hz), 75.4 (d, $J_{cp} = 167.85$ Hz), 126.3 (d, $J = 4.27$ Hz), 127.7 (d, $J = 3.15$ Hz), 128.2 (d, $J = 2.55$ Hz), 139.8, 209.8 (d, $J = 12.3$ Hz).

Diethyl [1-(4-chlorophenyl)-1-hydroxy-3-oxobutyl]phosphonate (Table 1, entry 5): ^1H NMR (300 MHz, CDCl_3) δ 1.16 (t, $J = 6.9$ Hz, 3H), 1.35 (t, $J = 6.9$ Hz, 3H), 2.18 (s, 3H), 3.39-3.69 (m, 2H), 3.73-3.81 (m, 1H), 3.89-3.97 (m, 1H), 4.14-4.24 (m, 2H), 5.16 (d, $J = 18.3$ Hz, 1H), 7.54 (d, $J = 9.0$ Hz, 2H), 7.54-7.58 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.7 (d, $J = 5.47$ Hz), 16.8 (d, $J = 5.47$ Hz), 32.6 (d, $J = 2.25$ Hz), 47.8 (d, $J = 2.92$ Hz), 64.0 (d, $J = 7.42$ Hz), 64.3 (d, $J = 7.42$ Hz), 76.3 (d, $J_{cp} = 166.95$ Hz), 127.6 (d, $J = 4.27$ Hz), 128.6 (d, $J = 2.85$ Hz), 134.0 (d, $J = 3.45$ Hz), 138.5, 209.7 (d, $J = 12.3$ Hz).

Diisopropyl [1-(4-chlorophenyl)-1-hydroxy-3-oxobutyl]phosphonate (Table 1, entry 6): ^1H NMR (300 MHz, CDCl_3) δ 1.01 (d, $J = 6.3$ Hz, 3H), 1.20 (d, $J = 6.3$ Hz, 3H), 1.28 (d, $J = 6.3$ Hz, 3H), 1.31 (d, $J = 6.3$ Hz, 3H), 2.11 (s, 3H), 3.25-3.31 (m, 3H), 4.34-4.44 (m, 1H), 4.66-4.76 (m, 1H), 5.11 (d, $J = 16.8$ Hz, 1H), 7.27-7.30 (m, 2H), 7.49-7.55 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 23.7 (d, $J = 5.7$ Hz), 24.2 (d, $J = 5.17$ Hz), 24.4 (d, $J = 3.67$ Hz), 24.5 (d, $J = 2.85$ Hz), 72.5 (d, $J = 8.02$ Hz), 72.8 (d, $J = 7.72$ Hz), 75.09 (d, $J_{cp} = 168.45$ Hz), 127.8 (d, $J = 4.27$ Hz), 128.3 (d, $J = 2.62$ Hz), 133.7 (d, $J = 3.75$ Hz), 138.6, 209.5 (d, $J = 12.6$ Hz).

Diethyl [1-(4-fluorophenyl)-1-hydroxy-3-oxobutyl]phosphonate (Table 1, entry 7): ^1H NMR (300 MHz, CDCl_3) δ 1.10 (t, $J = 7.2$ Hz, 3H), 1.30 (t, $J = 7.2$ Hz, 3H), 2.15 (s, 3H), 3.29-3.32 (m, 2H), 3.67-4.18 (m, 4H), 5.18 (br s, 1H), 6.99-7.05 (m, 2H), 7.53-7.58 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.6 (d, $J = 5.17$ Hz), 16.8 (d, $J = 5.47$ Hz), 32.5 (d, $J = 1.95$ Hz), 47.9 (d, $J = 3.15$ Hz), 63.9 (d, $J = 7.72$ Hz), 64.1 (d, $J = 7.42$ Hz),

75.3 (d, J_{cp} = 167.02 Hz), 115.1 (d, J = 2.85 Hz), 115.4 (d, J = 2.85 Hz), 127.9 (d, J = 4.27 Hz), 128.0 (d, J = 4.27 Hz), 135.5 (d, J = 3.15 Hz), 162.4 (dd, J_I = 3.15 Hz, J_2 = 245.17 Hz), 209.6 (d, J = 12.3 Hz).

Diisopropyl [1-(4-fluorophenyl)-1-hydroxy-3-oxo-butyl]phosphonate (Table 1, entry 8): ^1H NMR (300 MHz, CDCl_3) δ 0.98 (t, J = 6.3 Hz, 3H), 1.21 (d, J = 6.3 Hz, 3H), 1.30 (d, J = 6.3 Hz, 3H), 1.33 (d, J = 6.3 Hz, 3H), 2.13 (s, 3H), 3.28 (d, J = 9.3 Hz, 2H), 4.33-4.40 (m, 1H), 4.69-4.76 (m, 1H), 5.05 (d, J = 17.7 Hz, 1H), 6.99-7.05 (m, 2H), 7.55-5.60 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 23.6 (d, J = 5.40 Hz), 24.2 (d, J = 4.87 Hz), 24.3 (d, J = 3.6 Hz), 24.6 (d, J = 2.85 Hz), 32.7 (d, J = 1.95 Hz), 48.2 (d, J = 2.85 Hz), 72.4 (d, J = 7.72 Hz), 72.8 (d, J = 7.42 Hz), 75.1 (d, J_{cp} = 168.97 Hz), 115.0 (d, J = 2.55 Hz), 115.3 (d, J = 2.55 Hz), 128.1 (d, J = 3.97 Hz), 128.2 (d, J = 4.27 Hz), 135.6 (d, J = 2.85 Hz), 162.3 (dd, J_I = 3.07 Hz, J_2 = 244.91), 209.8 (d, J = 12.3 Hz).

Diethyl [1-(4-bromophenyl)-1-hydroxy-3-oxobutyl]phosphonate (Table 1, entry 9): ^1H NMR (300 MHz, CDCl_3) δ 1.12 (t, J = 6.9 Hz, 3H), 1.30 (t, J = 6.9 Hz, 3H), 2.14 (s, 3H), 3.28-3.32 (m, 2H), 3.67-3.93 (m, 2H), 4.08-4.17 (m, 2H), 5.14 (d, J = 16.80 Hz, 1H), 7.46 (br s, 5H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.6 (d, J = 5.4 Hz), 16.8 (d, J = 5.4 Hz), 32.5 (d, J = 2.25 Hz), 47.7 (d, J = 2.85 Hz), 63.9 (d, J = 7.72 Hz), 64.3 (d, J = 7.42 Hz), 75.4 (d, J_{cp} = 166.72 Hz), 122.1 (d, J = 3.97 Hz), 127.9 (d, J = 4.35 Hz), 131.5 (d, J = 2.62 Hz), 138.9, 209.6 (d, J = 12.3 Hz).

Diethyl [1-hydroxy-1-(4-iodophenyl)-3-oxobutyl]phosphonate (Table 1, entry 10): ^1H NMR (300 MHz, CDCl_3) δ 1.11 (dt, J_I = 2.1 Hz, J_2 = 6.9 Hz, 3H), 1.30 (dt, J_I = 2.4 Hz, J_2 = 6.9 Hz, 3H), 2.14 (s, 3H), 3.30 (d, J = 9.9 Hz, 2H), 3.70-3.91 (m, 2H), 4.10-4.17 (m, 2H), 5.11 (d, J = 18.3 Hz, 1H), 7.25-7.34 (m, 2H), 7.64-7.67 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.6 (d, J = 5.40 Hz), 16.8 (d, J = 5.40 Hz), 32.5 (d, J = 2.25 Hz), 47.7 (d, J = 2.62 Hz), 63.9 (d, J = 7.72 Hz), 64.3 (d, J = 7.12 Hz), 75.4 (d, J_{cp} = 166.42 Hz), 93.9 (d, J = 4.27 Hz), 128.1 (d, J = 4.35 Hz), 137.4 (d, J = 2.62 Hz), 139.7, 209.6 (d, J = 12.6 Hz).

Diethyl [1-hydroxy-1-(4-methylphenyl)-3-oxobutyl]phosphonate (Table 1, entry 11): ^1H NMR (300 MHz, CDCl_3) δ 1.09 (t, J = 7.2 Hz, 3H), 1.29 (t, J = 7.2 Hz, 3H), 2.10 (s, 3H), 2.30 (s, 3H), 3.25-3.39 (m, 2H), 3.63-3.89 (m, 2H), 4.07-4.17 (m, 2H), 5.02 (d, J = 18.6 Hz, 1H), 7.12 (d, J = 8.1 Hz, 2H), 7.42-7.46 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.6 (d, J = 5.47 Hz), 16.8 (d, J = 5.47 Hz), 21.4 (d, J = 1.2 Hz), 32.6 (d, J = 2.25 Hz), 47.9 (d, J = 3.15 Hz), 63.7 (d, J = 7.72 Hz), 64.1 (d, J = 7.20 Hz), 75.5 (d, J_{cp} = 167.25 Hz), 125.9 (d, J = 4.27 Hz), 129.1 (d, J = 2.85 Hz), 136.5, 137.5 (d, J = 3.15 Hz), 209.8 (d, J = 12.9 Hz).

Diethyl [1-hydroxy-1-(4-methoxyphenyl)-3-oxobutyl]phosphonate (Table 1, entry 12): ^1H NMR (300 MHz, CDCl_3) δ 1.11 (t, $J = 7.2$ Hz, 3H), 1.31 (t, $J = 7.2$ Hz, 3H), 2.13 (s, 3H), 3.22-3.40 (m, 2H), 3.65-3.71 (m, 1H), 3.78 (s, 1H), 3.79-3.90 (m, 1H), 4.10-4.16 (m, 2H), 5.03 (d, $J = 18.9$ Hz, 1H), 6.86 (d, $J = 8.40$ Hz, 2H), 7.47-7.51 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.7 (d, $J = 5.17$ Hz), 16.8 (d, $J = 5.70$ Hz), 32.6 (d, $J = 2.25$ Hz), 47.9 (d, $J = 3.45$ Hz), 55.5, 63.6 (d, $J = 7.72$ Hz), 64.1 (d, $J = 7.5$ Hz), 75.5 (d, $J_{cp} = 167.55$ Hz), 113.8 (d, $J = 2.62$ Hz), 127.4 (d, $J = 4.35$ Hz), 131.5, 159.2 (d, $J = 3.15$ Hz), 209.9 (d, $J = 12.82$ Hz).

Diethyl (1-hydroxy-1-methyl-3-oxobutyl)phosphonate (Table 1, entry 13) is known compound.¹

Diethyl (1-benzyl-1-hydroxy-3-oxobutyl)phosphonate (Table 1, entry 15): ^1H NMR (300 MHz, CDCl_3) δ 1.30-1.38 (m, 6H), 2.17 (s, 3H), 2.23-2.36 (m, 1H), 2.85-2.96 (m, 2H), 3.19-3.26 (m, 1H), 4.11-4.24 (m, 4H), 5.21 (d, $J = 3.9$ Hz, 1H), 7.22-7.27 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.8, 32.7, 42.1 (d, $J = 4.57$ Hz), 44.0 (d, $J = 5.70$ Hz), 63.3 (d, $J = 7.42$ Hz), 63.6 (d, $J = 7.42$ Hz), 74.9 (d, $J_{cp} = 165.82$ Hz), 127.1, 128.3, 131.4, 135.3 (d, $J = 12.9$ Hz), 211.5 (d, $J = 4.39$ Hz).

Diethyl [1-hydroxy-3-oxo-1-(2-phenylethyl)butyl]phosphonate (Table 1, entry 16): ^1H NMR (300 MHz, CDCl_3) δ 1.33 (t, $J = 7.5$ Hz, 6H), 1.96-2.21 (m, 2H), 2.25 (s, 3H), 2.56-3.08 (m, 4H), 4.10-4.24 (m, 4H), 5.06 (d, $J = 6.3$ Hz, 1H), 7.13-7.28 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.8, 16.9, 29.6, 32.7, 39.1 (d, $J = 3.75$ Hz), 45.4 (d, $J = 5.7$ Hz), 63.3 (d, $J = 2.62$ Hz), 63.4 (d, $J = 2.62$ Hz), 74.4 (d, $J_{cp} = 164.70$ Hz), 126.1, 128.6, 128.7, 141.9, 210.8 (d, $J = 6.3$ Hz).

Diethyl [*trans*-1-hydroxy-1-(2-oxopropyl)-2-butenyl]phosphonate (Table 1, entry 17): ^1H NMR (300 MHz, CDCl_3) δ 1.26-1.34 (m, 6H), 1.69-1.74 (m, 3H), 2.16 (s, 3H), 2.76-3.04 (m, 2H), 4.04-4.18 (m, 4H), 4.66 (d, $J = 16.20$ Hz, 1H), 5.51-5.58 (m, 1H), 5.81-5.93 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 16.7 (d, $J = 3.75$ Hz), 16.8 (d, $J = 3.37$ Hz), 18.2 (d, $J = 2.55$ Hz), 32.5 (d, $J = 2.02$ Hz), 47.0, 63.5 (d, $J = 7.72$ Hz), 63.9 (d, $J = 7.42$ Hz), 74.4 (d, $J_{cp} = 168.07$ Hz), 128.2 (d, $J = 9.97$ Hz), 28.9 (d, $J = 2.85$ Hz), 209.5 (d, $J = 12.6$ Hz).

Dimethyl [1-hydroxy-1-phenyl-3-oxopentyl]phosphonate (Scheme 1): ^1H NMR (CDCl_3 , 300 MHz) δ 0.97 (t, $J = 7.2$ Hz, 3H), 2.30-2.59 (m, 2H), 3.31-3.35 (m, 2H), 3.43 (d, $J = 10.2$ Hz, 3H), 3.80 (d, $J = 10.2$ Hz, 3H), 5.30 (d, $J = 19.2$ Hz, 1H), 7.25-7.39 (m, 3H), 7.58-7.62 (m, 2H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 7.5, 38.6, 46.7, 54.5 (d, $J = 7.27$ Hz), 54.8 (d, $J = 7.12$ Hz), 76.01 (d, $J_{cp} = 166.65$), 126.0 (d, $J = 4.27$ Hz), 128.1 (d, $J = 3.15$ Hz), 128.5 (d, $J = 2.92$ Hz), 139.5, 212.7 (d, $J = 12.00$ Hz).

Diethyl [1-hydroxy-1-phenyl-3-oxopentyl]phosphonate (Scheme 1): ^1H NMR (CDCl_3 , 300 MHz) δ 0.96 (t, $J = 7.50$ Hz, 3H), 1.08 (t, $J = 7.20$ Hz, 3H), 1.30 (t, $J = 7.20$ Hz, 3H), 2.30-2.60 (m, 2H), 3.30-3.34 (m, 2H), 3.60-3.69 (m, 1H), 3.81-3.89 (m, 1H), 4.11-4.21 (m, 2H), 5.27 (d, $J = 18.3$ Hz, 1H), 7.23-7.27 (m, 3H), 7.58-7.62 (m, 2H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 7.5, 16.6 (d, $J = 5.40$ Hz), 16.8 (d, $J = 5.4$ Hz), 38.6, 46.8 (d, $J = 3.15$ Hz), 63.8 (d, $J = 7.95$ Hz), 64.2 (d, $J = 7.95$ Hz), 75.7 (d, $J_{cp} = 166.12$ Hz), 126.1 (d, $J = 4.35$ Hz), 127.9 (d, $J = 3.15$ Hz), 128.4 (d, $J = 2.85$ Hz), 139.7, 212.7 (d, $J = 11.70$ Hz).

Diisopropyl [1-hydroxy-1-phenyl-3-oxopentyl]phosphonate (Scheme 1): ^1H NMR (CDCl_3 , 500 MHz) δ 0.90 (t, $J = 6.5$ Hz, 3H), 0.92 (d, $J = 6.00$ Hz, 3H), 1.16 (d, $J = 6.00$ Hz, 3H), 1.27 (d, $J = 6.5$ Hz, 3H), 1.30 (d, $J = 6.5$ Hz), 2.23-2.24 (m, 1H), 2.46-2.53 (m, 1H), 3.24-3.28 (m, 2H), 4.28-4.32 (m, 1H), 4.69-4.73 (m, 1H), 5.21 (d, $J = 17.50$ Hz, 1H), 7.22-7.33 (m, 3H), 7.58-7.61 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 7.2, 23.3 (d, $J = 5.62$ Hz), 24.1 (d, $J = 4.87$ Hz), 24.2 (d, $J = 3.87$ Hz), 24.4 (d, $J = 2.87$ Hz), 38.5 (d, $J = 1.87$ Hz), 47.0 (d, $J = 3.37$ Hz), 72.2 (d, $J = 8.12$ Hz), 72.7 (d, $J = 7.62$ Hz), 75.4 (d, $J_{cp} = 168.37$ Hz), 126.3 (d, $J = 4.25$ Hz), 127.7 (d, $J = 2.87$ Hz), 128.2 (d, $J = 2.87$ Hz), 139.9, 212.8 (d, $J = 11.87$ Hz).

Diisopropyl [1-hydroxy-1-phenyl-3-oxo-4-methoxybutyl]phosphonate (Scheme 1): ^1H NMR (CDCl_3 , 500 MHz) δ 0.94 (d, $J = 5.5$ Hz, 3H), 1.18 (d, $J = 6.00$ Hz, 3H), 1.27 (d, $J = 6.00$ Hz, 3H), 1.31 (d, $J = 6.5$ Hz), 3.22-3.53 (m, 5H), 3.82 (d, $J = 18.00$ Hz, 1H), 3.92 (d, $J = 18.00$ Hz, 1H), 4.32-4.37 (m, 1H), 4.68-4.73 (m, 1H), 4.88 (d, $J = 15.5$ Hz, 1H), 7.24-7.33 (m, 3H), 7.59-7.60 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 23.4 (d, $J = 5.62$ Hz), 24.0 (d, $J = 4.75$ Hz), 24.2 (d, $J = 3.37$ Hz), 24.4 (d, $J = 2.62$ Hz), 44.7 (d, $J = 3.37$ Hz), 59.4, 72.4 (d, $J = 8.12$ Hz), 72.8 (d, $J = 7.62$ Hz), 75.2 (d, $J_{cp} = 168.25$ Hz), 78.5 (d, $J = 2.37$ Hz), 126.4 (d, $J = 4.25$ Hz), 127.9 (d, $J = 2.75$ Hz), 128.3 (d, $J = 2.50$ Hz), 139.3, 209.0 (d, $J = 12.87$ Hz).

Additional Reference:

1) Kreutzkamp, N.; Mengel, W. *Ann.* **1962**, 657, 19-24.

STANDARD PROTON PARAMETERS

Archive directory: /home/samanta/vnmrSYS/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13

Ambient temperature

File: III-SS-130

Mercury-300BB "Varian300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7996.0 Hz

5 repetitions

OBSERVE H1, 499.7420463 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 16 sec

STANDARD CARBON PARAMETERS
III-SS-130carbon

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
User: 1-14-87
File: III-SS-130carbon
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
2000 repetitions
OBSERVE C13, 125.6602075 MHz
DECOUPLE H1, 499.7445451 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 1 hr, 20 min, 24 sec

STANDARD 1H OBSERVE

iv-ss-7

Archive directory: /home/samanta/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
8 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

13C OBSERVE

iv-ss-7c

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
File: iv-ss-7c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
7000 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min, 0 sec

STANDARD 1H OBSERVE
IV-SS-22

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: IV-SS-22

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
32 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 10 min, 24 sec

13C OBSERVE

IV-SS-22carbon

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDC13
Ambient temperature
File: IV-SS-22carbon
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
3328 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 16 hr, 11 min, 18 sec

STANDARD 1H OBSERVE

iv-ss-38

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
File: iv-ss-38
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
10 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 31 sec

¹³C OBSERVE

iv-ss-38carbon

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
File: iv-ss-38carbon
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
1280 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 26 min, 6 sec

STANDARD 1H OBSERVE

iv-ss-105

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: iv-ss-105

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
24 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 15 sec

13C OBSERVE

iv-ss-105c

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
File: iv-ss-105c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
640 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 37 min, 36 sec

STANDARD 1H OBSERVE

iv-ss-56

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13

Ambient temperature

File: iv-ss-56

Mercury-300BB "Varian300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.001 sec

Width 4789.3 Hz

10 repetitions

OBSERVE H1, 299.7452618 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 31 sec

13C OBSERVE

iv-ss-56carbon

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
File: iv-ss-56carbon
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
2368 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 26 min, 6 sec

STANDARD 1H OBSERVE

iv-ss-98

Archive directory: /home/samanta/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
16 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 0 sec

iv-ss-98c

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
6000 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 4 hr, 51 min, 44 sec

STANDARD 1H OBSERVE

iv-ss-95

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: iv-ss-95

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
20 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 3 sec

¹³C OBSERVE

iv-ss-95c

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
File: iv-ss-95c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
2688 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 26 min, 6 sec

STANDARD 1H OBSERVE

iv-ss-80

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13

Ambient temperature

File: iv-ss-80

Mercury-300BB "Varian300"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.001 sec

Width 4789.3 Hz

16 repetitions

OBSERVE H1, 299.7452618 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 49 sec

¹³C OBSERVE

iv-ss-80c

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
File: iv-ss-80c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
1920 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 37 min, 36 sec

STANDARD 1H OBSERVE

iv-ss-60

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
File: iv-ss-60
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
12 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 37 sec

iv-ss-60c
13C OBSERVE

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
File: iv-ss-60c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
3000 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 26 min, 6 sec

STANDARD 1H OBSERVE

iv-ss-101

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: iv-ss-101

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
24 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 15 sec

¹³C OBSERVE

iv-ss-101c

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: iv-ss-101c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
1472 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 37 min, 36 sec

STANDARD 1H OBSERVE

iv-ss-benzyl-diethylphosphonate

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
File: iv-ss-benzyl-diethylphosphonate
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
10 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 31 sec

¹³C OBSERVE

iv-ss benzyldiethylphosphonatecarbon

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13

Ambient temperature

File: iv-ss-benzyldiethylphosphonatecarbon
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
20000 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 16 hr, 11 min, 18 sec

STANDARD 1H OBSERVE

iv-ss 29

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: iv-ss-29

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
10 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 31 sec

13C OBSERVE

iv-ss-29carbon

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3
Ambient temperature
File: IV-SS-29carbon
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
20000 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 16 hr, 11 min, 18 sec

STANDARD 1H OBSERVE

iv-ss-68

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: iv-ss-68

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
24 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 15 sec

iv-ss-68
13C OBSERVE

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
File: iv-ss-68c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
1792 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 26 min, 6 sec

STANDARD 1H OBSERVE

v-ss-22

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: v-ss-22

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
8 repetitions
OBSERVE H1, 299.7452618 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

¹³C OBSERVE
V-ss-22c

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul1

Solvent: CDC13
Ambient temperature
File: v-ss-22c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
960 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 49 min, 2 sec

STANDARD 1H OBSERVE

1v-ss-126

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: 1v-ss-126

Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.001 sec
Width 4789.3 Hz
5 repetitions
OBSERVE H1, 299.7452605 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 16 sec

¹³C OBSERVE

iv-ss-126c

Archive directory: /home/samanta/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl₃
Ambient temperature
File: iv-ss-126c
Mercury-300BB "Varian300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.815 sec
Width 18832.4 Hz
128 repetitions
OBSERVE C13, 75.3709880 MHz
DECOUPLE H1, 299.7467965 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 26 min, 6 sec

STANDARD PROTON PARAMETERS
v-ss-36

Archive directory: /home/samanta/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pu1
Solvent: CDC13
Ambient temperature
INOVA-500 "Varian500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
8 repetitions
OBSERVE H1, 499.6931887 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec

STANDARD CARBON PARAMETERS
v-ss-36c

Archive directory: /home/samanta/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
User: 1-14-87
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
2000 repetitions
OBSERVE C13, 125.6479222 MHz
DECOUPLE H1, 499.6956872 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 1 hr, 16 min, 58 sec

STANDARD PROTON PARAMETERS

v-ss-52

Archive directory: /home/samanta/vnmrsys/data

Sample directory:

File: PROTON

Pulse Sequence: s2pu1

Solvent: CDC13

Ambient temperature

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

10 repetitions

OBSERVE H1, 499.6931887 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 29 sec

STANDARD CARBON PARAMETERS

Archive directory: /home/samanta/vnmrsys/data
 Sample directory:
 File: CARBON

Pulse Sequence: s2pu1

Solvent: CDCl3
 Ambient temperature
 User: 1-14-87
 INOVA-500 "Inova500"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31409.5 Hz
 1500 repetitions
 OBSERVE C13, 125.6479222 MHz
 DECOUPLE H1, 499.6956872 MHz
 Power 36 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072
 Total time 57 min, 44 sec

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\III-SS-131RACEMIC
User: System
Acquired: 11/24/2005 3:11:10 PM
Printed: 2/12/2006 2:29:00 PM

Racemic

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
5.400	5203093	50.64	298781	60.68
8.408	5071240	49.36	193629	39.32
Totals	10274333	100.00	492410	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-136B
User: System
Acquired: 3/6/2006 9:36:26 AM
Printed: 3/23/2006 11:29:56 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
4.883	155352	6.57	12957	17.85
8.342	2208011	93.43	59625	82.15
Totals	2363363	100.00	72582	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-06E
 User: System
 Acquired: 11/22/2005 2:40:31 PM
 Printed: 2/14/2006 1:12:29 PM

Page 1 of 1

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
6.408	2415034	9.21	92667	21.16
7.533	1747980	6.66	49108	11.21
9.142	1766615	6.73	52898	12.08
22.800	9972219	38.01	126050	28.78
26.100	10332412	39.39	117298	26.78
Totals	26234260	100.00	438021	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\iv-ss-150a
User: System
Acquired: 3/9/2006 1:18:18 PM
Printed: 3/23/2006 11:33:30 AM

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
23.675	325306	2.68	3552	3.28
27.300	11823199	97.32	104766	96.72
Totals	12148505	100.00	108318	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-38H
 User: System
 Acquired: 1/2/2006 10:15:44 AM
 Printed: 2/14/2006 1:18:29 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
0.000	0	0.00	0	0.00
18.575	16934559	48.06	258827	55.31
21.600	18299594	51.94	209132	44.69

Totals	35234153	100.00	467959	100.00
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Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\V-SS-6
User: System
Acquired: 3/12/2006 1:59:26 PM
Printed: 3/23/2006 11:52:31 AM

SPD-10AVvp
 Ch1-215nm
 Results

Retention Time	Area	Area %	Height	Height %
18.342	53812200	95.43	792663	95.79
21.750	2574335	4.57	34848	4.21
Totals	56386535	100.00	827511	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-23B
 User: System
 Acquired: 12/20/2005 11:00:31 AM
 Printed: 2/14/2006 1:15:31 PM

Racemic

Page 1 of 1

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
4.867	16261830	49.11	1265046	57.93
5.600	16851696	50.89	918691	42.07

Totals	33113526	100.00	2183737	100.00
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Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\v-ss-11e
 User: System
 Acquired: 3/13/2006 1:33:36 PM
 Printed: 3/23/2006 11:41:48 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
4.400	408760	2.30	32428	4.38
5.508	17367364	97.70	708246	95.62
Totals	17776124	100.00	740674	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-105C
User: System
Acquired: 2/6/2006 12:56:32 PM
Printed: 2/14/2006 1:20:02 PM

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Racemic

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.200	8165473	49.92	474973	58.14
7.308	8192887	50.08	342011	41.86

Totals	16358360	100.00	816984	100.00
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Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-8
User: System
Acquired: 3/12/2006 1:08:07 PM
Printed: 3/23/2006 11:40:17 AM

SPD-10AVvp

Ch1-215nm

Results

Retention Time	Area	Area %	Height	Height %
6.342	187860	2.57	7608	3.40
7.725	7133108	97.43	216456	96.60
Totals	7320968	100.00	224064	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-57D
User: System
Acquired: 1/13/2006 1:59:03 PM
Printed: 2/14/2006 1:21:17 PM

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SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
6.983	3815896	49.71	253309	54.08
7.708	3859675	50.29	215110	45.92
Totals	7675571	100.00	468419	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\v-ss-5a
User: System
Acquired: 3/17/2006 10:18:37 AM
Printed: 3/23/2006 11:39:20 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
7.425	795807	10.23	43679	10.92
8.308	6984668	89.77	356331	89.08
Totals	7780475	100.00	400010	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-98D
User: System
Acquired: 2/2/2006 4:11:17 PM
Printed: 2/14/2006 1:22:13 PM

Page 1 of 1

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
6.392	2171011	49.75	111977	56.33
7.500	2193212	50.25	86827	43.67
Totals	4364223	100.00	198804	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\v-ss-40a
User: System
Acquired: 3/18/2006 2:25:07 PM
Printed: 3/23/2006 11:43:29 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
6.475	423963	3.58	23535	4.96
7.867	11429759	96.42	451136	95.04

Totals	11853722	100.00	474671	100.00
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Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-95D
 User: System
 Acquired: 1/31/2006 5:04:43 PM
 Printed: 2/14/2006 1:23:18 PM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
29.733	31448805	49.82	529870	53.10
31.883	31678506	50.18	467985	46.90
Totals	63127311	100.00	997855	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\v-ss-9e
User: System
Acquired: 3/14/2006 12:45:56 PM
Printed: 3/23/2006 11:40:45 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
28.300	32650	0.13	845	0.24
30.542	24922629	99.87	355352	99.76
Totals	24955279	100.00	356197	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-81B
User: System
Acquired: 1/25/2006 9:56:15 AM
Printed: 2/14/2006 1:24:58 PM

Racemic

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
14.708	7517208	50.33	279751	53.03
15.917	7417555	49.67	247777	46.97
Totals	14934763	100.00	527528	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\v-ss-10b
User: System
Acquired: 3/16/2006 3:59:49 PM
Printed: 3/23/2006 11:41:16 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
14.942	167822	3.24	7063	4.73
15.700	5006796	96.76	142281	95.27
Totals	5174618	100.00	149344	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-60C
 User: System
 Acquired: 1/16/2006 11:55:54 AM
 Printed: 2/14/2006 1:26:15 PM

Page 1 of 1

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
15.792	5133083	49.62	176723	52.01
17.083	5211965	50.38	163050	47.99

Totals	10345048	100.00	339773	100.00
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Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-62
 User: System
 Acquired: 1/19/2006 1:53:54 PM
 Printed: 2/14/2006 1:26:32 PM

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SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
15.483	9882750	92.22	336379	92.77
16.775	833485	7.78	26199	7.23

Totals	10716235	100.00	362578	100.00
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Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-101C
User: System
Acquired: 2/6/2006 11:29:30 AM
Printed: 2/14/2006 1:26:52 PM

Page 1 of 1

SPD-10AVvp
Ch1-220nm
Results

Retention Time	Area	Area %	Height	Height %
8.867	5522052	49.76	248945	53.74
10.292	5495323	49.52	204763	44.20
10.875	79926	0.72	9570	2.07
Totals	11097301	100.00	463278	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-108
 User: System
 Acquired: 2/11/2006 1:42:10 PM
 Printed: 2/14/2006 1:27:15 PM

Page 1 of 1

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
8.892	6862325	93.12	320090	93.88
10.433	489961	6.65	18952	5.56
10.875	16806	0.23	1911	0.56
Totals	7369092	100.00	340953	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-71
 User: System
 Acquired: 1/20/2006 12:36:50 PM
 Printed: 2/14/2006 1:29:16 PM

Racemic

SPD-10AVvp

Ch1-280nm

Results

Retention Time	Area	Area %	Height	Height %
29.125	1632581	48.95	20295	58.24
31.875	1702769	51.05	14552	41.76

Totals	3335350	100.00	34847	100.00
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Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met

Data: C:\EZStart\Projects\Default\Data\v-ss-38a

User: System

Acquired: 3/19/2006 2:57:49 PM

Printed: 3/23/2006 11:42:59 AM

SPD-10AVvp

Ch1-280nm

Results

Retention Time	Area	Area %	Height	Height %
27.575	1048681	98.49	7651	97.45
30.125	16111	1.51	200	2.55
Totals	1064792	100.00	7851	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-45E
 User: System
 Acquired: 1/8/2006 3:44:04 PM
 Printed: 2/14/2006 1:33:30 PM

Racemic

Page 1 of 1

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
19.042	4291756	48.80	106773	48.99
20.375	4230454	48.10	106698	48.95
21.308	272313	3.10	4486	2.06
Totals	8794523	100.00	217957	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-46E
 User: System
 Acquired: 1/11/2006 4:49:44 PM
 Printed: 2/14/2006 1:34:01 PM

Page 1 of 1

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
18.333	6235539	96.04	176341	96.15
20.383	256940	3.96	7064	3.85
Totals	6492479	100.00	183405	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-30L
 User: System
 Acquired: 12/28/2005 12:53:45 PM
 Printed: 2/14/2006 1:31:35 PM

Racemic

Page 1 of 1

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
0.000	0	0.00	0	0.00
21.750	1339704	49.47	33005	53.41
23.292	1368589	50.53	28796	46.59
Totals	2708293	100.00	61801	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\iv-ss-4
User: System
Acquired: 3/9/2006 2:18:11 PM
Printed: 3/23/2006 11:37:45 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
21.825	632608	9.73	15159	12.50
23.283	5865727	90.27	106092	87.50
Totals	6498335	100.00	121251	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
 Data: C:\EZStart\Projects\Default\Data\IV-SS-68H
 User: System
 Acquired: 1/19/2006 11:55:37 AM
 Printed: 2/14/2006 1:34:39 PM

Racemic

SPD-10AVvp
 Ch1-215nm
 Results

Retention Time	Area	Area %	Height	Height %
21.558	5755902	49.13	145518	51.52
23.217	5958571	50.87	136909	48.48
Totals	11714473	100.00	282427	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-148L1
User: System
Acquired: 3/11/2006 3:07:27 PM
Printed: 3/23/2006 11:32:41 AM

SPD-10AVvp
Ch1-215nm
Results

Retention Time	Area	Area %	Height	Height %
19.883	33202	2.93	898	4.24
21.167	1089203	96.22	19898	94.01
22.775	9620	0.85	370	1.75
Totals	1132025	100.00	21166	100.00

Area % Report**Method Name:** C:\EZStart\Projects\Default\Methods\AS.met**Data:** C:\EZStart\Projects\Default\Data\v-ss-22a**User:** System**Acquired:** 3/15/2006 10:06:21 AM**Printed:** 3/23/2006 11:22:16 AM **SPD-10AVvp****Ch1-220nm****Results**

Retention Time	Area	Area %	Height	Height %
17.333	9830858	49.85	97485	49.79
21.608	9890475	50.15	98294	50.21
Totals	19721333	100.00	195779	100.00

Area % Report**Method Name:** C:\EZStart\Projects\Default\Methods\AS.met**Data:** C:\EZStart\Projects\Default\Data\V-SS-41**User:** System**Acquired:** 3/21/2006 5:02:58 PM**Printed:** 3/23/2006 11:24:04 AM **SPD-10AVvp****Ch1-220nm****Results**

Retention Time	Area	Area %	Height	Height %
17.558	41623888	84.51	374629	83.00
22.183	7632144	15.49	76719	17.00
Totals	49256032	100.00	451348	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\IV-SS-133
User: System
Acquired: 3/3/2006 9:59:00 AM
Printed: 3/23/2006 11:20:08 AM

SPD-10AVvp

Ch1-220nm

Results

Retention Time	Area	Area %	Height	Height %
4.783	15689446	49.24	973916	76.52
10.867	1049788	3.29	75218	5.91
11.575	15126456	47.47	223694	17.57

Totals	31865690	100.00	1272828	100.00
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Area % Report**Method Name:** C:\EZStart\Projects\Default\Methods\AS.met**Data:** C:\EZStart\Projects\Default\Data\v-ss-37b**User:** System**Acquired:** 3/18/2006 3:47:44 PM**Printed:** 3/23/2006 11:21:16 AM **SPD-10AVvp****Ch1-220nm****Results**

Retention Time	Area	Area %	Height	Height %
4.792	1798822	15.72	121093	38.57
10.867	892436	7.80	57984	18.47
11.467	8753114	76.48	134866	42.96

Totals	11444372	100.00	313943	100.00
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Area % Report**Method Name:** C:\EZStart\Projects\Default\Methods\AS.met**Data:** C:\EZStart\Projects\Default\Data\v-ss-36b**User:** System**Acquired:** 3/20/2006 10:53:48 AM**Printed:** 3/23/2006 11:24:49 AM **SPD-10AVvp****Ch1-220nm****Results**

Retention Time	Area	Area %	Height	Height %
5.842	13386282	48.70	762312	60.57
10.667	9662888	35.15	268358	21.32
10.875	4438717	16.15	227933	18.11

Totals	27487887	100.00	1258603	100.00
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Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\V-SS-42A
User: System
Acquired: 3/21/2006 6:22:25 PM
Printed: 3/23/2006 11:25:18 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
5.867	1095597	13.19	32672	13.08
10.867	3565063	42.92	108516	43.45
10.875	3646183	43.89	108536	43.46
Totals	8306843	100.00	249724	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\V-SS-52B
User: System
Acquired: 3/23/2006 10:13:36 AM
Printed: 3/23/2006 11:18:42 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
5.942	21379260	49.71	989448	62.49
7.692	21630946	50.29	593810	37.51
Totals	43010206	100.00	1583258	100.00

Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS.met
Data: C:\EZStart\Projects\Default\Data\V-SS-53A
User: System
Acquired: 3/23/2006 11:03:02 AM
Printed: 3/23/2006 11:17:58 AM

SPD-10AVvp
 Ch1-220nm
 Results

Retention Time	Area	Area %	Height	Height %
6.000	1363287	7.30	64803	12.24
7.733	17302920	92.70	464708	87.76
Totals	18666207	100.00	529511	100.00