***Trans*-(4-butyl-2-oxotetrahydrofuran-3-yl)-phosphonic acid diethyl ester (10a).**

Copper(I) Iodide (0.05g, 0.026mmol) was added to a solution of n-butylmagnesium bromide prepared from n-butyl bromide (2.05g, 15.0mmol) and magnesium (0.389g, 16mmol) in tetrahydrofuran (10ml) and then a solution of lactone **8a** (1.10g, 5.0mmol) in tetrahydrofuran (25ml) was added dropwise at -78°C. The mixture was allowed to warm up to room temperature and then stirred at this temperature for 2.5h. The reaction was acidified to pH 1 with 1N HCl and tetrahydrofuran was evaporated under reduced pressure. The residue was extracted with dichloromethane (3x15ml). The combined organic layers were washed with water (20ml) and then dried ($MgSO_4$), and evaporated under reduced pressure. The residue was purified by column chromatography (hexane-AcOEt 50:50, R_f = 0.23) to give the lactone **10a** as colourless oil (0.89 g, 64 %); ν_{max} 1748, 1224, 1042; δ_P ($CDCl_3$) 20.76; δ_H ($CDCl_3$) 0.91 (t, $^3J_{HH}$ = 6.7 Hz, 3H, C*H*$_3$); 1.27 ÷ 1.34 (m, 6H, 3xC*H*$_2$); 1.36 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 1.37 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 1.60 ÷ 1.74 (m, 1H, C*H*CP); 2.76 (dd, $^2J_{PH}$ = 22.5 Hz, $^3J_{HH}$ = 5.0 Hz, 1H, PC*H*); 4.01 (dd, $^2J_{HH}$ = 9.0 Hz, $^3J_{HH}$ = 4.0 Hz, 1H, C*H*H); 4.12 ÷ 4.28 (m, 4H, 2xC*H*$_2$OP); 4.56 (dd, $^2J_{HH}$ = 9.0 Hz, $^3J_{HH}$ = 7.0 Hz, 1H, CH*H*); δ_C ($CDCl_3$) 13.41 (s, *C*H$_3$); 15.90 (d, $^3J_{PC}$ = 6.0 Hz, *C*H$_3$CH$_2$OP); 15.93 (d, $^3J_{PC}$ = 5.9 Hz, *C*H$_3$CH$_2$OP); 21.94 (s, *C*H$_2$); 28.20 (s, *C*H$_2$); 33.19 (d, $^3J_{PC}$ = 8.7 Hz, *C*H$_2$); 37.28 (d, $^2J_{PC}$ = 2.5 Hz, *C*H); 44.68 (d, $^1J_{PC}$ = 141.0 Hz, *C*HP); 62.45 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$OP); 63.16 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$OP); 72.26 (d, $^3J_{PC}$ = 6.0 Hz, *C*H$_2$O); 171.63 (d, $^2J_{PC}$ = 2.9 Hz, *C*=O). $C_{12}H_{23}O_5P$: requires C 51.79, H 8.33; found C 51.90, H 8.31.

***Trans*-(4-phenyl-2-oxotetrahydrofuran-3-yl)-phosphonic acid diethyl ester (10b).**

Starting from bromobenzene (2.35g, 15.0mmol) and following the procedure described above the lactone **10b** was obtained (0.86 g, 58 %) as colourless oil. (hexane-AcOEt 50:50, R_f = 0.18); ν_{max} 1764, 1262, 1040; δ_P ($CDCl_3$) 19.52; δ_H ($CDCl_3$) 1.26 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.7 Hz, 3H, C*H*$_3$CH$_2$OP); 1.31 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.7 Hz, 3H, C*H*$_3$CH$_2$OP); 3.16 (dd, $^2J_{PH}$ = 23.7 Hz, $^3J_{HH}$ = 6.0 Hz, 1H, C*H*P); 3.65 (dd, $^3J_{HH}$ = 6.5 Hz, $^3J_{PH}$ = 6.0 Hz, 1H, C*H*CP); 4.08 ÷ 4.19 (m, 4H, 2xC*H*$_2$OP); 4.33 (dd, $^2J_{HH}$ = 9.2 Hz, $^3J_{HH}$ = 6.5 Hz, 1H, C*H*H); 4.76 (dd, $^2J_{HH}$ = 9.2 Hz, $^3J_{HH}$ = 6.5 Hz, 1H, CH*H*); 7.21 ÷ 7.37 (m, 5H, *Ar*); δ_C ($CDCl_3$) 15.82 (d, $^3J_{PC}$ = 6.7 Hz, *C*H$_3$CH$_2$OP); 15.87 (d, $^3J_{PC}$ = 6.5 Hz, *C*H$_3$CH$_2$OP); 43.24 (d, $^2J_{PC}$ = 1.8 Hz, *C*H); 46.77 (d, $^1J_{PC}$ = 142.2 Hz, *C*HP); 62.90 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$OP); 63.70 (d, $^2J_{PC}$ = 6.5 Hz, *C*H$_2$OP); 73.47 (d, $^3J_{PC}$ = 7.5 Hz, *C*H$_2$O); 126.53 (s, 2×*C*H$_{Ar}$); 128.97 (s, *C*H$_{Ar}$); 129.10 (s, 2×*C*H$_{Ar}$); 139.10 (d, $^3J_{PC}$ = 7.3 Hz, *C*$_{Ar}$); 171.27 (s, *C*=O). $C_{14}H_{19}O_5P$: requires C 56.38, H 6.42; found C 56.52, H 6.45.

***Trans*-(5,5-Dimethyl-2-oxo-4-phenyltetrahydrofuran-3-yl)-phosphonic acid diethyl ester (10c).**

Copper(I) iodide (0.05g, 0.026mmol) was added to a solution of phenylmagnesium bromide, prepared from bromobenzene (2.35g, 15.0mmol) and magnesium (0.389g, 16.0mmol) in diethyl ether (15ml) and then a solution of lactone **8b** in diethyl ether (25ml) was added dropwise at 0°C. The mixture was allowed to warm up to room temperature and then stirred at this temperature for 2.5h. The reaction was then acidified to pH 1 with 1N HCl and diethyl ether was evaporated under reduced pressure. The residue was extracted with dichloromethane (3x15ml). The combined organic layers were washed with water (20ml) and then dried ($MgSO_4$), and evaporated under reduced pressure. The residue was chromatographed (hexane-AcOEt 50:50, R_f = 0.31) to give the lactone **10c** as colourless oil (1.34 g, 82 %); ν_{max} 1768, 1260, 1024; δ_P ($CDCl_3$) 20.47; δ_H ($CDCl_3$) 0.97 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 1.06 (s, 3H, C*H*$_3$); 1.21 (t, $^3J_{HH}$ = 6.5 Hz, 3H, C*H*$_3$CH$_2$OP); 1.56 (s, 3H, C*H*$_3$); 3.66 (dd, $^2J_{PH}$ = 22.7 Hz, $^3J_{HH}$ = 12.2 Hz, 1H, C*H*P); 3.83 (dd, $^3J_{PH}$ = 15.7 Hz, $^3J_{HH}$ = 12.2 Hz, 1H, C*H*); 3.80 ÷ 3.90 (m, 2H, C*H*$_2$OP); 4.03 ÷ 4.20 (m, 2H, C*H*$_2$OP); 7.22 ÷ 7.40 (m, 5H, *Ar*); δ_C ($CDCl_3$) 15.78 (d, $^3J_{PC}$ = 6.5 Hz, *C*H$_3$CH$_2$OP); 16.13 (d, $^3J_{PC}$ = 6.4 Hz, *C*H$_3$CH$_2$OP); 20.96 (s, *C*H$_3$); 27.14 (s, *C*H$_3$); 44.54 (d, $^1J_{PC}$ = 151.2 Hz, C*H*P); 53.70 (s, C*H*); 62.45 (d, $^2J_{PC}$ = 6.6 Hz, *C*H$_2$OP); 63.59 (d, $^2J_{PC}$ = 6.3 Hz, *C*H$_2$OP); 86.42 (d, $^3J_{PC}$ = 13.6 Hz, *C*O); 127.91 (s, 2×*C*H$_{Ar}$); 128.13 (s, 2×*C*H$_{Ar}$); 128.52 (s, *C*H$_{Ar}$); 135.09 (s, *C*$_{Ar}$); 170.08 (s, *C*=O). $C_{16}H_{23}O_5P$: requires C 58.89, H 7.10; found C 58.70, H 7.14.

Diethyl 3,3-diethoxybutylphosphonate (12).

To a solution of oxophosponate **11** (15.0g, 0,072m) in triethyl orthoformate (60ml, 0.360m) Amberlist 15 (3.5g) was added and the reaction mixture was stirred at 0°C for 10h. Then Amberlist was removed by filtration and the residue was purified by distillation (b.p. 114 ÷ 120 °C / 0.2 mmHg) to give the phosphonate **12** as light yellow oil (20.00 g, 97 %); ν_{max} 1248, 1042; δ_P ($CDCl_3$) 32.70; δ_H ($CDCl_3$) 1.16 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2×C*H*$_3$CH$_2$O); 1.27 (s, 3H, C*H*$_3$); 1.32 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xC*H*$_3$CH$_2$OP); 1.68 ÷ 1.97 (m, 4H, 2xC*H*$_2$); 3.44 (q, $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$O); 3.45 (q, $^3J_{HH}$ = 7.0 Hz, 1H, C*H*$_2$O); 4.09 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP); 4.10 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP); δ_C ($CDCl_3$) 13.33 (s, 2×*C*H$_3$CH$_2$O); 14.42 (d, $^3J_{PC}$ = 5.9 Hz, *C*H$_3$CH$_2$OP); 16.02 (s, *C*H$_3$CH$_2$OP); 19.00 (d, $^1J_{PC}$ = 143.2 Hz, *C*H$_2$P); 28.02 (d, $^2J_{PC}$ = 4.3 Hz, *C*H$_2$); 34.32 (s, *C*H$_3$); 53.65 (s, 2×*C*H$_2$O); 59.41 (d, $^2J_{PC}$ = 6.7 Hz, 2x*C*H$_2$OP); 98.75 (d, $^3J_{PC}$ = 20.1 Hz, *C*). $C_{12}H_{27}O_5P$: requires C 51.05, H 9.64; found C 51.21, H 9.66.

Diethyl 2-(2-phenyl-1,3-dioxolan-2-yl)ethylphosphonate (16).

A mixture of oxophosphonate **15** (20.0g, 0.075m), ethylene glycol (12.5ml, 0.022m) and p-toluenesulfonic acid (1.2g, 7.5mmol) in toluene (200ml) was heated at reflux under Dean-Stark water separator. After the phosphonate was completely consumed, the reaction mixture was washed with 10% $NaHCO_3$ (50ml) and water (50ml). Organic layer was dried ($MgSO_4$) and evaporated under reduced pressure. The residue was purified by distillation (b.p. 146 ÷ 150 °C / 0.1 mmHg) to give phosphonate **16** as light yellow oil (22.40 g, 95 %); ν_{max} 1248, 1024; δ_P ($CDCl_3$) 32.79; δ_H ($CDCl_3$) 1.29 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xCH_3CH$_2$OP); 1.88 (ddd, $^2J_{PH}$ = 22.5 Hz, $^3J_{HH}$ = 7.2 Hz, $^3J_{HH}$ = 4.0 Hz, 2H, CH_2P); 2.13 (ddd, $^3J_{PH}$ = 15.0 Hz, $^3J_{HH}$ = 7.2 Hz, $^3J_{HH}$ = 4.0 Hz, 2H, CH_2); 3.76 ÷ 3.81 (m, 2H, CH_2O); 3.99 ÷ 4.11 (m, 6H, 2xCH_2OP, CH_2O); 7.28 ÷ 7.46 (m, 5H, CH_{Ar}); δ_C ($CDCl_3$) 15.86 (d, $^3J_{PC}$ = 5.6 Hz, 2x*C*H$_3$CH$_2$OP); 19.49 (d, $^1J_{PC}$ = 143.0 Hz, *C*H$_2$P); 32.90 (d, $^2J_{PC}$ = 3.4 Hz, *C*H$_2$); 60.84 (d, $^2J_{PC}$ = 6.2 Hz, 2x*C*H$_2$OP); 64.17 (s, 2x*C*H$_2$O); 108.80 (d, $^3J_{PC}$ = 19.2 Hz, *C*H$_2$); 125.05 (s, 2×*C*H$_{Ar}$); 127.52 (s, *C*H$_{Ar}$); 127.64 (s, 2×*C*H$_{Ar}$); 141.38 (s, *C*$_{Ar}$). $C_{15}H_{23}O_5P$: requires C 57.32, H 7.38; found C 57.46, H 7.36.

General procedure for the preparation of ethyl alkanoates 13, 17 and 20.

A solution of ketal **12**, **16** or **20** (0.070m) in tetrahydrofuran (15ml) was added at -78°C to a stirred solution of LDA prepared from n-BuLi (12.0ml, 0.075m) and diisopropylamine (10.0ml, 0.076m) in tetrahydrofuran (50ml). Stirring was continued for 0.5h at this temperature. Then diethyl carbonate (8.5ml, 0.070m) in tetrahydrofuran (20ml) was added and the reaction mixture was allowed to warm up to room temperature and stirred for 20h then the mixture was acidified to pH 2 with 3N HCl and tetrahydrofuran was evaporated under reduced pressure. The residue was extracted with chloroform (3x35ml). The combined organic layers were washed with saturated NaCl solution (25ml), dried ($MgSO_4$) and evaporated under reduced pressure. The crude products were purified by vaccum distillation.

2-(Diethoxy-hosphoryl)-4,4-diethoxypentanoic acid ethyl ester (13).

Purification (bp = 150 ÷ 154°C / 0.1 mmHg) gave the phosphonate **13** as light yellow oil (21.20g, 85 %); ν_{max} 1764, 1248, 1042; δ_P ($CDCl_3$) 23.73; δ_H ($CDCl_3$) 1.18 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2×CH_3CH$_2$O); 1.26 (s, 3H, CH_3); 1.30 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xCH_3CH$_2$OP); 1.68 ÷ 1.97 (m, 2H, CH_2); 3.30 (ddd, $^2J_{PH}$ = 23.2 Hz, $^3J_{HH}$ = 10.2 Hz, $^3J_{HH}$ = 3.2 Hz, 1H, C*H*P); 3.45 (q, $^3J_{HH}$ = 7.0 Hz, 2H, CH_2O); 3.46 (q, $^3J_{HH}$ = 7.0 Hz, 2H, CH_2O); 4.08 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, CH_2OP); 4.10 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, CH_2OP); δ_C ($CDCl_3$) 13.28 (s, 2×*C*H$_3$CH$_2$O); 14.40 (d, $^3J_{PC}$ = 5.9 Hz, *C*H$_3$CH$_2$OP); 16.02 (s, *C*H$_3$CH$_2$OP); 30.00 (d, $^2J_{PC}$ = 3.2 Hz, *C*H$_2$); 34.32 (s, *C*H$_3$); 40.08 (d, $^1J_{PC}$ = 128.55 Hz, *C*HP); 52.65 (s, 2×*C*H$_2$O); 58.89 (s, *C*H$_2$O); 60.42 (d, $^2J_{PC}$ = 6.7 Hz, 2x*C*H$_2$OP); 167.75 (d, $^2J_{PC}$ = 4.8 Hz, *C*=O). $C_{15}H_{31}O_7P$: requires C 50.84, H 8.82; found C 51.00, H 8.78.

2-(Diethoxyphosphoryl)-3-(2-phenyl-[1,3]dioxolan-2-yl)-propionic acid ethyl ester (17).

Purification (bp = 178 ÷ 182°C / 0.05 mmHg) gave the phosphonate **17** as light yellow oil (24.30g, 90 %); ν_{max} 1764, 1240, 1020; δ_P ($CDCl_3$) 23.53; δ_H ($CDCl_3$) 1.29 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3CH$_2$O); 1.31 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xCH_3CH$_2$OP); 2.35 (ddd, $^2J_{HH}$ = 14.7 Hz, $^3J_{PH}$ = 13.0 Hz, $^3J_{HH}$ = 1.5 Hz, 1H, CH*H*); 2.69 (ddd, $^2J_{HH}$ = 14.7 Hz, $^3J_{HH}$ = 11.2 Hz, $^3J_{PH}$ = 3.0 Hz, 1H, C*H*H); 3.30 (ddd, $^2J_{PH}$ = 23.2 Hz, $^3J_{HH}$ = 11.2 Hz, $^3J_{HH}$ = 1.5 Hz, 1H, C*H*P); 3.69 ÷ 3.81 (m, 4H, 2xCH_2O); 4.08 ÷ 4.18 (m, 4H, 2xCH_2OP); 4.24 (q, $^3J_{HH}$ = 7.0 Hz, 2H, CH_2O); 7.27 ÷ 7.48 (m, 5H, CH_{Ar}); δ_C ($CDCl_3$) 13.71 (s, *C*H$_3$CH$_2$O); 15.86 (d, $^3J_{PC}$ = 5.9 Hz, 2x*C*H$_3$CH$_2$OP); 37.03 (d, $^2J_{PC}$ = 3.7 Hz, *C*H$_2$); 40.47 (d, $^1J_{PC}$ = 129.7 Hz, *C*HP); 60.67 (s, *C*H$_2$O); 62.12 (d, $^2J_{PC}$ = 6.6 Hz, *C*H$_2$OP); 62.44 (d, $^2J_{PC}$ = 6.3 Hz, *C*H$_2$OP); 64.21 (s, *C*H$_2$O); 64.30 (s, *C*H$_2$O); 108.61 (d, $^3J_{PC}$ = 18.1 Hz, *C*); 125.16 (s, 2×*C*H$_{Ar}$); 127.21 (s, *C*H$_{Ar}$); 127.76 (s, 2×*C*H$_{Ar}$); 141.35 (s, *C*$_{Ar}$); 168.75 (d, $^2J_{PC}$ = 5.0 Hz, *C*=O). $C_{18}H_{27}O_7P$: requires C 55.95, H 7.04; found C 55.80, H 7.07.

2-(Diethoxyphosphoryl)-4,4-diethoxybutyric acid ethyl ester (20).

Purification (bp = 142 ÷ 144°C / 0.1 mmHg) gave the phosphonate **20** as light yellow oil (20.20g, 85 %); ν_{max} 1744, 1264, 1042; δ_P ($CDCl_3$) 23.14; δ_H ($CDCl_3$) 1.18 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2×CH_3CH$_2$O); 1.30 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3CH$_2$O); 1.32 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xCH_3CH$_2$OP); 1.85 ÷ 2.05 (m, 1H, C*H*H); 2.24 ÷ 2.47 (m, 1H, CH*H*); 3.08 (ddd, $^2J_{PH}$ = 20.5 Hz, $^3J_{HH}$ = 7.0 Hz, $^3J_{HH}$ = 6.0 Hz, 1H, C*H*P); 3.50 (q, $^3J_{HH}$ = 7.0 Hz, 1H, C*H*HO); 3.52 (q, $^3J_{HH}$ = 7.0 Hz, 1H, CH*H*O); 3.63 (q, $^3J_{HH}$ = 7.0 Hz, 1H, C*H*HO); 3.66 (q, $^3J_{HH}$ = 7.0 Hz, 1H, CH*H*O); 4.11 ÷ 4.23 (m, 6H, CH_2O, 2xCH_2OP); 4.50 (t, $^3J_{HH}$ = 5.5 Hz, 1H, C*H*); δ_C ($CDCl_3$) 13.78 (s, *C*H$_3$CH$_2$O); 14.86 (s, 2×*C*H$_3$CH$_2$O); 16.00 (d, $^3J_{PC}$ = 6.2 Hz, *C*H$_3$CH$_2$OP); 16.02 (d, $^3J_{PC}$ = 6.0 Hz, *C*H$_3$CH$_2$OP); 30.65 (d, $^2J_{PC}$ = 4.5 Hz, *C*H$_2$); 41.10 (d, $^1J_{PC}$ = 131.2 Hz, *C*HP); 60.96 (s, *C*H$_2$O); 61.31 (s, *C*H$_2$O); 61.84 (s, *C*H$_2$O); 62.31 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$OP); 62.47 (d, $^2J_{PC}$ = 6.4 Hz, *C*H$_2$OP); 100.78 (d, $^3J_{PC}$ = 16.7 Hz, *C*H); 168.64 (d, $^2J_{PC}$ = 5.2 Hz, *C*=O). $C_{14}H_{29}O_7P$: requires C 49.41, H 8.59; found C 49.30, H 8.62.

General procedure for the preparation of oxoalkanoates 14, 18 and 21.

A solution of ketal **13**, **17** or **20** (0.05m) in tetrahydrofuran (20ml) and 3N HCl (5ml) was stirred at room temperature for 24h. Then tetrahydrofuran was evaporated under reduced pressure and the residue was extracted with chloroform (3x20ml). The combined organic layers were dried ($MgSO_4$) and evaporated under reduced pressure to give the product which was spectroscopically pure.

2-(Diethoxyphosphoryl)-4-oxopentanoic acid ethyl ester (14).[10]

Light yellow oil (13.70g, 96 %); δ_P ($CDCl_3$) 22.72; δ_H ($CDCl_3$) 1.29 (t, $^3J_{HH}$ = 7.2 Hz, 3H, C*H*$_3$CH$_2$O); 1.33 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 1.36 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 2.20 (s, 3H, C*H*$_3$); 2.90 (ddd, $^2J_{HH}$ = 18.0 Hz, $^3J_{PH}$ = 9.0 Hz, $^3J_{HH}$ = 3.0 Hz, 1H, CH*H*); 3.26 (ddd, $^2J_{HH}$ = 18.0 Hz, $^3J_{HH}$ = 11.0 Hz, $^3J_{PH}$ = 6.2 Hz, 1H, C*H*H); 3.49 (ddd, $^2J_{PH}$ = 23.7 Hz, $^3J_{HH}$ = 11.0 Hz, $^3J_{HH}$ = 3.0 Hz, 1H, C*H*P); 4.12 (q, $^3J_{HH}$ = 7.2 Hz, 2H, C*H*$_2$O); 4.14 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP); 4.17 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP).

2-(Diethoxyphosphoryl)-4-oxo-4-phenylbutyric acid ethyl ester (18).[11]

Light yellow oil (23.50g, 95 %); δ_P ($CDCl_3$) 23.10; δ_H ($CDCl_3$) 1.30 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$O); 1.35 (t, $^3J_{HH}$ = 7.2 Hz, 3H, C*H*$_3$CH$_2$OP); 1.38 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 3.45 (ddd, $^2J_{HH}$ = 16.7 Hz, $^3J_{HH}$ = 11.0 Hz, $^3J_{PH}$ = 1.5 Hz, 1H, CH*H*); 3.64 (ddd, $^2J_{HH}$ = 16.7 Hz, $^3J_{PH}$ = 8.0 Hz, $^3J_{HH}$ = 5.7 Hz, 1H, C*H*H); 3.82 (ddd, $^2J_{PH}$ = 22.7 Hz, $^3J_{HH}$ = 11.0 Hz, $^3J_{HH}$ = 5.7 Hz, 1H, C*H*P); 4.18 (q, $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$O); 4.21 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.2 Hz, 2H, C*H*$_2$OP); 4.22 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP); 7.44 ÷ 7.50 (m, 2H, C*H*$_{Ar}$); 7.56 ÷ 7.59 (m, 1H, C*H*$_{Ar}$); 7.97 ÷ 8.01 (m, 2H, C*H*$_{Ar}$).

2-(Diethoxyphosphoryl)-4-oxobutyric acid ethyl ester (21).[12]

Light yellow oil (13.00g, 95 %); δ_P ($CDCl_3$) 22.72; δ_H ($CDCl_3$) 1.29 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$O); 1.32 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP); 1.33 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 3.00 (ddd, $^3J_{PH}$ = 8.5 Hz, $^3J_{HH}$ = 6.5 Hz, $^3J_{HH}$ = 3.0 Hz, 1H, C*H*H); 3.29 (ddd, $^2J_{PH}$ = 18.7 Hz, $^3J_{HH}$ = 7.2 Hz, $^3J_{HH}$ = 6.5 Hz, 1H, C*H*P); 3.46 (ddd, $^3J_{PH}$ = 22.5 Hz, $^3J_{HH}$ = 7.2 Hz, $^3J_{HH}$ = 3.0 Hz, 1H, CH*H*); 4.10 ÷ 4.24 (m, 6H, C*H*$_2$O, 2xC*H*$_2$OP); 9.76 (td, $^3J_{HH}$ = 3.0 Hz, $^4J_{PH}$ = 1.5 Hz, 1H, C*H*O).

General procedure for the preparation of phosphonates 23a and 23b.

To a stirred suspension of sodium hydride (1.2g, 0.05m) in tetrahydrofuran (30ml) alkanoate **22a** or **22b** (0.05m) was added followed by allyl bromide (4.35ml, 0.05m) in tetrahydrofuran (10ml) at room temperature .The reaction mixture was stirred for 24h and then acidified to pH 2 with 1N HCl. Tetrahydrofuran was evaporated under reduced pressure and the residue was extracted with chloroform (3x25ml). The combined organic layers were washed with saturated NaCl solution (20ml) and dried ($MgSO_4$). Evaporation of solvent afforded a crude product, which was purified by vacuum distillation.

2-(Diethoxyphosphoryl)-2-methylbut-3-enoic acid ethyl ester (23a).

Purification (bp = 82 ÷ 86°C / 0.1 mmHg) gave the phosphonate **23a** as light yellow oil (11.40g, 82 %); ν_{max} 1740, 1248 1020; δ_P ($CDCl_3$) 26.48; δ_H ($CDCl_3$) 1.28 (t, $^3J_{HH}$ = 7.2 Hz, 3H, C*H*$_3$CH$_2$O); 1.30 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xC*H*$_3$CH$_2$OP); 1.41 (d, $^3J_{PH}$ = 14.0 Hz, 3H, C*H*$_3$); 2.40 (ddd, $^3J_{PH}$ = 13.7 Hz, $^2J_{HH}$ = 8.7 Hz, $^3J_{HH}$ = 9.0 Hz, 1H, C*H*H); 2.84 ÷ 2.96 (m, 1H, CH*H*); 4.09 ÷ 4.22 (m, 6H, C*H*$_2$O, 2xC*H*$_2$OP); 5.10 (dd, $^3J_{HH}$ = 10.2 Hz, $^4J_{HH}$ = 1.0 Hz, 1H, C*H*H); 5.12 (dd, $^3J_{HH}$ = 15.5 Hz, $^4J_{HH}$ = 2.0 Hz, 1H, CH*H*); 5.60 ÷ 5.74 (m, 1H, C*H*); δ_C ($CDCl_3$) 13.82 (s, *C*H$_3$CH$_2$O); 16.22 (d, $^3J_{PC}$ = 5.7 Hz, 2x*C*H$_3$CH$_2$OP); 16.83 (d, $^2J_{PC}$ = 4.4 Hz, *C*H$_3$); 38.05 (d, $^3J_{PC}$ = 3.6 Hz, *C*H$_2$); 47.68 (d, $^1J_{PC}$ = 134.9 Hz, *C*P); 61.18 (s, *C*H$_2$O); 62.64 (d, $^2J_{PC}$ = 7.2 Hz, *C*H$_2$OP); 62.91 (d, $^2J_{PC}$ = 7.1 Hz, *C*H$_2$OP); 118.61 (s, *C*H$_2$); 131.98 (d, $^3J_{PC}$ = 13.7 Hz, *C*H); 170.48 (d, $^2J_{PC}$ = 3.5 Hz, *C*=O). $C_{12}H_{23}O_5P$: requires C 51.79, H 8.33; found C 51.57, H 8.35.

2-(Diethoxyphosphoryl)-2-phenylbut-3-enoic acid ethyl ester (23b).

Purification (bp = 146 ÷ 150°C / 0.6 mmHg) gave the phosphonate **23b** as light yellow oil (14.50g, 85%); ν_{max} 1764, 1660, 1224, 1048; δ_P ($CDCl_3$) 22.46; δ_H ($CDCl_3$) 1.19 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP); 1.20 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP); 1.28 (t, $^3J_{HH}$ = 7.2 Hz, 3H, C*H*$_3$CH$_2$O); 2.96 ÷ 3.11 (m, 1H, C*H*H); 3.15 ÷ 3.27 (m, 1H, CH*H*); 3.94 ÷ 4.10 (m, 4H, 2xC*H*$_2$OP); 4.27 (t, $^3J_{HH}$ = 7.2 Hz, 2H, C*H*$_2$O); 5.04 (dd, $^3J_{HH}$ = 10.2 Hz, $^4J_{HH}$ = 1.2 Hz, 1H, C*H*H); 5.09 (dd, $^3J_{HH}$ = 17.0 Hz, $^4J_{HH}$ = 2.0 Hz, 1H, CH*H*); 5.90 (ddt, $^3J_{HH}$ = 17.0 Hz, $^3J_{HH}$ = 10.2 Hz, $^3J_{HH}$ = 6.7 Hz, 1H, C*H*); 7.28 ÷ 7.40 (m, 5H, C*H*$_{Ar}$); δ_C ($CDCl_3$) 13.62 (s, *C*H$_3$CH$_2$O); 15.87 (d, $^3J_{PC}$ = 5.5 Hz, 2x*C*H$_3$CH$_2$OP); 37.91 (d, $^2J_{PC}$ = 3.4 Hz, *C*H$_2$); 58.18 (d, $^1J_{PC}$ = 137.6 Hz, *C*P); 61.16 (s, *C*H$_2$O); 62.62 (d, $^2J_{PC}$ = 7.1 Hz, *C*H$_2$OP); 62.98 (d, $^2J_{PC}$ = 7.3 Hz, *C*H$_2$OP); 118.00 (s, *C*H$_2$); 127.02 (d, $^4J_{PC}$ = 2.3 Hz, 2×*C*H$_{Ar}$); 127.57 (s, *C*H$_{Ar}$); 128.33 (d, $^3J_{PC}$ = 5.5 Hz, 2×*C*H$_{Ar}$); 133.06 (d, $^3J_{PC}$ = 8.0 Hz, *C*H); 135.22 (d, $^2J_{PC}$ = 6.7 Hz, *C*$_{Ar}$); 169.86 (d, $^2J_{PC}$ = 3.5 Hz, *C*=O). $C_{17}H_{25}O_5P$: requires C 59.95, H 7.40; found C 60.12, H 7.36.

2-(Diethoxyphosphoryl)-2-methyl-4-oxobutyric acid ethyl ester (24a).

Starting from phosphonate **23a** (5.56g, 0.02m) and following the procedure described in ref. 12, the phosphonate **24a** was obtained as light yellow oil (5.45g, 95 %). The crude product was spectroscopically pure. ν_{max} 1748, 1724, 1240, 1032; δ_P ($CDCl_3$) 24.75; δ_H ($CDCl_3$) 1.24 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0 .5 Hz, 3H, CH_3CH$_2$OP); 1.32 (td, $^3J_{HH}$ = 7.2 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, CH_3CH$_2$OP); 1.32 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3CH$_2$O); 1.58 (d, $^3J_{PH}$ = 12.2 Hz, 3H, CH_3); 2.78 (dd, $^2J_{HH}$ = 18.0 Hz, $^3J_{PH}$ = 9.2 Hz, 1H, C*H*H); 3.30 (dd, $^2J_{HH}$ = 18.0 Hz, $^3J_{PH}$ = 9.5 Hz, 1H, CH*H*); 4.10 ÷ 4.27 (m, 6H, CH_2O, 2xCH_2OP); 9.52 (td, $^3J_{HH}$ = $^4J_{PH}$ = 2.0 Hz, 1H, C*H*O); δ_C ($CDCl_3$) 13.49 (s, *C*H$_3$CH$_2$O); 14.69 (d, $^2J_{PC}$ = 19.1 Hz, *C*H$_3$); 15.87 (d, $^3J_{PC}$ = 5.8 Hz, 2x*C*H$_3$CH$_2$OP); 36.90 (d, $^2J_{PC}$ = 4.2 Hz, *C*H$_2$); 45.65 (d, $^1J_{PC}$ = 130.9 Hz, *C*P); 60.66 (s, *C*H$_2$O); 62.13 (s, *C*H$_2$OP); 62.24 (s, *C*H$_2$OP); 170.60 (d, $^2J_{PC}$ = 4.0 Hz, *C*=O); 197.80 (d, $^3J_{PC}$ = 15.0 Hz, *C*HO). $C_{11}H_{21}O_6P$: requires C 47.14, H 7.55; found C 47.00, H 7.59.

2-(Diethoxyphosphoryl)-4-oxo-2-phenylbutyric acid ethyl ester (24b).

Starting from phosphonate **23b** (6.80g, 0.02m) and following the procedure described in ref. 12, the phosphonate **24b** was obtained as light yellow oil (6.65g, 95 %). The crude product was spectroscopically pure. ν_{max} 1764, 1732, 1240, 1024; δ_P ($CDCl_3$) 20.93; δ_H ($CDCl_3$) 1.17 (t, $^3J_{HH}$ = 7.2 Hz, 3H, CH_3CH$_2$O); 1.23 (t, $^3J_{HH}$ = 7.2 Hz, 3H, CH_3CH$_2$OP); 1.32 (t, $^3J_{HH}$ = 7.2 Hz, 3H, CH_3CH$_2$OP); 3.30 (dd, $^3J_{PH}$ = 12.0 Hz, $^3J_{HH}$ = 2.0 Hz, CH_2); 4.03 (q, $^3J_{HH}$ = 7.2 Hz, 2H, CH_2O); 4.05 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.2 Hz, 2H, CH_2OP); 4.08 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.2 Hz, 2H, CH_2OP); 7.30 ÷ 7.51 (m, 5H, CH_{Ar}); 9.57 (dd, $^3J_{HH}$ = $^4J_{PH}$ = 2.0 Hz, 1H, C*H*O); δ_C ($CDCl_3$) 13.74 (s, *C*H$_3$CH$_2$O); 15.97 (d, $^3J_{PC}$ = 6.0 Hz, *C*H$_3$CH$_2$OP); 16.07 (d, $^3J_{PC}$ = 5.8 Hz, *C*H$_3$CH$_2$OP); 46.07 (d, $^2J_{PC}$ = 8.1 Hz, *C*H$_2$); 54.64 (d, $^1J_{PC}$ = 196.7 Hz, *C*P); 61.76 (s, CH$_3$*C*H$_2$O); 63.53 (d, $^2J_{PC}$ = 7.4 Hz, *C*H$_2$OP); 63.83 (d, $^2J_{PC}$ = 7.3 Hz, *C*H$_2$OP); 127.37 (s, 2×*C*H$_{Ar}$); 127.56 (s, *C*H$_{Ar}$); 127.80 (d, $^3J_{PC}$ = 8.8 Hz, 2×*C*H$_{Ar}$); 134.34 (d, $^2J_{PC}$ = 7.6 Hz, *C*$_{Ar}$); 171.57 (d, $^3J_{PC}$ = 3.9 Hz, *C*=O); 198.90 (d, $^3J_{PC}$ =11.3 Hz, *C*HO). $C_{16}H_{23}O_6P$: requires C 56.14, H 6.77; found C 56.31, H 6.74.

General procedure for the preparation of lactones 26a and 26b.

To a stirred solution of phosphonate **14** or **18** (0.035m) in methanol (20ml) potasium borohydride (2.70g, 0.05m) was added at 0°C. Stirring was continued for 75min. The resulting mixture was neutralized to pH 2 with 3N HCl. Methanol was evaporated under reduced pressure and the residue was extracted with chloroform (4x20ml). The combined organic layers were washed with water (20ml) and dried ($MgSO_4$). Evaporation of the solvent under reduced pressure afforded a crude product.

2-Oxo-5-methyltetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26a).

Purification by vaccum distillation (b.p. 158 ÷ 160°C / 0.05 mmHg) gave the lactone **26a** as light yellow oil (8.0g, 96%) diaA : diaB = 56 : 44; δ_P ($CDCl_3$) 21.12 (diaA); 20.96 (diaB); δ_H ($CDCl_3$) 1.36 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xCH_3CH$_2$OP, diaA); 1.37 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xCH_3CH$_2$OP, diaB); 1.42 (d, $^3J_{HH}$ = 6.2 Hz, 3H, CH_3, diaA); 1.48 (d, $^3J_{HH}$ = 6.2 Hz, 3H, CH_3, diaB); 2.02 ÷ 2.81 (m, 1H, C*H*H, diaA + diaB); 2.56 ÷ 2.81 (m, 1H, CH*H*, diaA + diaB); 3.14 (ddd, $^2J_{PH}$ = 23.7 Hz, $^3J_{HH}$ = 10.2 Hz, $^3J_{HH}$ = 3.7 Hz, 1H, C*H*P, diaA); 3.18 (dt, $^2J_{PH}$ = 23.5 Hz, $^3J_{HH}$ = 9.7 Hz, 1H, C*H*P, diaB); 4.14 ÷ 4.31 (m, 4H, 2xCH_2OP, diaA + diaB); 4.60 (ddq, $^3J_{HH}$ = 8.7 Hz, $^3J_{HH}$ = 6.5 Hz, $^3J_{HH}$ = 6.2 Hz, 1H, C*H*, diaB); 4.83 (ddq, $^3J_{HH}$ = 8.7 Hz, $^3J_{HH}$ = 6.5 Hz, $^3J_{HH}$ = 6.2 Hz, 1H, C*H*, diaA).

(2-Oxo-5-phenyltetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26b).

Purification by column chromatography (hexane-AcOEt 50:50, R_f = 0.35) gave the lactone **26b** as light yellow oil (10.2g, 96%) diaA : diaB = 55 : 45; ν_{max} 1768, 1248, 1020; δ_P ($CDCl_3$) 20.84 (diaA); 21.09 (diaB); δ_H ($CDCl_3$) 1.34 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3CH$_2$OP, diaA); 1.35 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3CH$_2$OP, diaA); 1.39 (t, $^3J_{HH}$ = 7.0 Hz, 6H, 2xCH_3CH$_2$OP, diaB); 2.42 ÷ 2.61 (m, 2H, 2xC*H*H, diaA + diaB); 2.78 (dddd, $^2J_{HH}$ = 13.2 Hz, $^3J_{HH}$ = 9.2 Hz, $^3J_{HH}$ = 6.7 Hz, $^3J_{PH}$ = 4.0 Hz, 1H, CH*H*, diaA); 3.00 (dddd, $^3J_{PH}$ = 17.5 Hz, $^2J_{HH}$ = 13.2 Hz, $^3J_{HH}$ = 6.7 Hz, $^3J_{HH}$ = 3.7 Hz, 1H, CH*H*, diaB); 3.24 (ddd, $^2J_{PH}$ = 23.5 Hz, $^3J_{HH}$ = 9.7 Hz, $^3J_{HH}$ = 3.7 Hz, 1H, C*H*P, diaB); 3.32 (ddd, $^2J_{PH}$ = 23.2 Hz, $^3J_{HH}$ = 11.7 Hz, $^3J_{HH}$ = 9.5 Hz, 1H, C*H*P, diaA); 4.21 (q, $^3J_{HH}$ = 7.0 Hz, 2H, CH_2OP, diaA + diaB); 4.22 ÷ 4.33 (m, 2H, CH_2OP, diaA + diaB); 5.43 (dd, $^3J_{HH}$ = 9.5 Hz, $^3J_{HH}$ = 6.7 Hz, 1H, C*H*, diaB); 5.72 (dd, $^3J_{HH}$ = 9.7 Hz, $^3J_{HH}$ = 6.7 Hz, 1H, C*H*, diaA); 7.35 ÷ 7.40 (m, 10H, 2xCH_{Ar}, diaA + diaB); δ_C ($CDCl_3$) 16.16 (d, $^3J_{PC}$ = 6.0 Hz, *C*H$_3$CH$_2$OP, diaA + diaB); 30.10 (d, $^2J_{PC}$ = 7.5 Hz, *C*H$_2$, diaA); 30.15 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$, diaB); 40.01 (d, $^1J_{PC}$ = 145.1 Hz, *C*HP, diaB); 40.02 (d, $^1J_{PC}$ = 152.1 Hz, *C*HP, diaA); 62.65 (d, $^2J_{PC}$ = 6.6 Hz, *C*H$_2$OP, diaA); 62.86 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$OP, diaB); 63.26 (d, $^2J_{PC}$ = 6.5 Hz, *C*H$_2$OP, diaA); 63.53 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$OP, diaB); 80.05 (d, $^3J_{PC}$ = 11.9 Hz, *C*HO, diaA); 80.24 (d, $^3J_{PC}$ = 3.1 Hz, *C*HO, diaB); 125.14 (s, 2×*C*H$_{Ar}$, diaB); 125.52 (s, 2×*C*H$_{Ar}$, diaA); 128.44 (s, *C*H$_{Ar}$, diaB); 128.53 (s, *C*H$_{Ar}$, diaA); 128.54 (s, 2×*C*H$_{Ar}$, diaB); 128.59 (s, 2×*C*H$_{Ar}$, diaA); 138.17 (s, *C*$_{Ar}$, diaB); 138.43 (s, *C*$_{Ar}$, diaA); 170.99 (s, *C*=O, diaA); 171.21 (d, $^2J_{PC}$ = 4.3 Hz, *C*=O, diaB). $C_{14}H_{19}O_5P$: requires C 56.38, H 6.42; found C 56.51, H 6.45.

General procedure for the preparation of lactones 26 c-h.

To a stirred solution of alkylmagnesium bromide (15.0mmol) in diethyl ether (15ml) a solution of phosphonate **21**, **24a** or **24b** (5.0mmol) in diethyl ether (25ml) was added at 0°C. The reaction mixture was allowed to warm up to room temperature and stirred for 2.5h. Then the reaction mixture was acidified to pH 1 with 3N HCl and extracted with methylene chloride (4x15ml). The combined organic layers were washed with water (20ml), dried ($MgSO_4$) and evaporated under reduced pressure to give a crude product, which was purified by column chromatography.

5-Butyl-2-oxotetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26c).

Purification (AcOEt, R_f = 0.36) gave the lactone **26c** as light yellow oil (0,97g, 70 %) diaA : diaB = 56 : 44; ν_{max} 1748, 1248, 1040; δ_P ($CDCl_3$) 21.22 (diaA); 21.03 (diaB); δ_H ($CDCl_3$) 0.92 (t, $^3J_{HH}$ = 6.7 Hz, 3H, C*H*$_3$, diaA + diaB); 1.36 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 6H, 2xC*H*$_3$CH$_2$OP, diaA + diaB); 1.24 ÷ 1.40 (m, 4H, 2xC*H*$_2$, diaA + diaB); 1.50 ÷ 1.70 (m, 2H, C*H*$_2$, diaA + diaB); 2.05 ÷ 2.30 (m, 1H, C*H*H, diaA + diaB); 2.59 (dddd, $^3J_{PH}$ = 19.5 Hz, $^2J_{HH}$ = 13.5 Hz, $^3J_{HH}$ = 6.7 Hz, $^3J_{HH}$ = 3.5 Hz, 1H, CH*H*, diaA); 2.70 (dddd, $^3J_{PH}$ = 19.7 Hz, $^2J_{HH}$ = 13.5 Hz, $^3J_{HH}$ = 6.5 Hz, $^3J_{HH}$ = 3.5 Hz, 1H, CH*H*, diaB); 3.12 (ddd, $^2J_{PH}$ = 23.7 Hz, $^3J_{HH}$ = 7.2 Hz, $^3J_{HH}$ = 3.5 Hz, 1H, C*H*P, diaA + diaB); 4.08 ÷ 4.30 (m, 4H, 2xC*H*$_2$OP, diaA + diaB); 4.38 ÷ 4.48 (m, 1H, C*H*, diaB); 4.62 ÷ 4.74 (m, 1H, C*H*, diaA); δ_C ($CDCl_3$) 13.57 (s, *C*H$_3$, diaA + diaB); 16.07 (d, $^3J_{PC}$ = 5.9 Hz, 2x*C*H$_3$CH$_2$OP, diaA + diaB); 22.08 (s, *C*H$_2$, diaA + diaB); 26.97 (s, *C*H$_2$, diaA + diaB); 30.23 (d, $^2J_{PC}$ = 3.1 Hz, *C*H$_2$, diaB); 30.38 (d, $^2J_{PC}$ = 3.9 Hz, *C*H$_2$, diaA); 34.77 (s, *C*H$_2$, diaB); 34.82 (s, *C*H$_2$, diaA); 39.58 (d, $^1J_{PC}$ = 151.3 Hz, *C*HP, diaB); 39.85 (d, $^1J_{PC}$ = 138.6 Hz, *C*HP, diaA); 62.44 (d, $^2J_{PC}$ = 6.5 Hz, *C*H$_2$OP, diaA); 62.66 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$OP, diaB); 63.14 (d, $^2J_{PC}$ = 6.8 Hz, *C*H$_2$OP, diaB); 63.25 (d, $^2J_{PC}$ = 7.0 Hz, *C*H$_2$OP, diaA); 79.55 (d, $^3J_{PC}$ = 10.8 Hz, *C*HO, diaB); 79.92 (d, $^3J_{PC}$ = 2.9 Hz, *C*HO, diaA); 171.20 (s, *C*=O, diaA); 171.36 (d, $^2J_{PC}$ = 4.3 Hz, *C*=O, diaB). $C_{12}H_{23}O_5P$: requires C 51.79, H 8.33; found C 51.99, H 8.36.

(5-Benzyl-2-oxotetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26d).

Purification (AcOEt, R_f = 0.30) gave the lactone **26d** as colourless oil (1.06g, 68 %) diaA : diaB = 67 : 33; ν_{max} 1760, 1248, 1032; δ_P ($CDCl_3$) 20.95 (diaA); 20.70 (diaB); δ_H ($CDCl_3$) 1.34 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP, diaA); 1.36 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP, diaA); 1.38 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaB); 1.39 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaB); 2.39 ÷ 2.50 (m, 1H, C*H*H, diaA + diaB); 2.49 (dddd, $^3J_{PH}$ = 16.5 Hz, $^2J_{HH}$ = 10.0 Hz, $^3J_{HH}$ = 6.5 Hz, $^3J_{HH}$ = 3.2 Hz, 1H, CH*H*, diaA + diaB); 2.58 (dddd, $^3J_{PH}$ = 17.5 Hz, $^2J_{HH}$ = 13.2 Hz, $^3J_{HH}$ = 6.7 Hz, $^3J_{HH}$ = 4.2 Hz, 1H, C*H*P, diaA); 2.90 ÷ 3.37 (m, 3H, C*H*$_2$, diaA + diaB, C*H*P, diaB); 4.18 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP, diaA + diaB); 4.20 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP, diaA + diaB); 4.60 ÷ 4.69 (m, 1H, C*H*, diaB); 4.87 ÷ 4.97 (m, 1H, C*H*, diaA); 7.21 ÷ 7.30 (m, 5H, C*H*$_{Ar}$, diaA + diaB); δ_C ($CDCl_3$) 16.07 (s, 2x*C*H$_3$CH$_2$OP, diaA + diaB); 29.40 (d, $^2J_{PC}$ = 2.9 Hz, *C*H$_2$, diaA + diaB); 38.21 (s, *C*H$_2$, diaB); 38.48 (s, *C*H$_2$, diaA); 39.47 (d, $^1J_{PC}$ = 158.4 Hz, *C*HP, diaB); 39.61 (d, $^1J_{PC}$ = 141.6 Hz, *C*HP, diaA); 62.72 (d, $^2J_{PC}$ = 6.3 Hz, 2x*C*H$_2$OP, diaB); 63.22 (d, $^2J_{PC}$ = 6.8 Hz, 2x*C*H$_2$OP, diaA); 79.42 (s, *C*HO, diaB); 79.58 (s, *C*HO, diaA); 126.68 (s, 2×*C*H$_{Ar}$, diaB); 126.78 (s, 2×*C*H$_{Ar}$, diaA); 128.30 (s, *C*H$_{Ar}$, diaB); 128.39 (s, *C*H$_{Ar}$, diaA); 129.11 (s, 2×*C*H$_{Ar}$, diaB); 129.19 (s, 2×*C*H$_{Ar}$, diaA); 135.14 (s, *C*$_{Ar}$, diaB); 135.48 (s, *C*$_{Ar}$, diaA); 171.00 (s, *C*=O, diaA); 171.16 (d, $^2J_{PC}$ = 4.1 Hz, *C*=O, diaB). $C_{15}H_{21}O_5P$: requires C 57.69, H 6.78; found C 57.45, H 6.80.

(5-Butyl-3-methyl-2-oxotetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26e).

Purification (AcOEt, R_f = 0.25) gave the lactone **26e** as colourless oil (0.99g, 68 %) diaA : diaB = 67 : 33; ν_{max} 1748, 1224, 1032; δ_P ($CDCl_3$) 24.30 (diaA); 24.12 (diaB); δ_H ($CDCl_3$) 0.90 (t, $^3J_{HH}$ = 6.7 Hz, 3H, C*H*$_3$, diaA + diaB); 1.30 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP, diaA + diaB); 1.32 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaA + diaB); 1.24 ÷ 1.48 (m, 4H, 2xC*H*$_2$, diaA + diaB); 1.52 (d, 3H, $^3J_{HP}$=18.0 Hz, C*H*$_3$), 1.54 ÷ 1.65 (m, 2H, C*H*$_2$, diaA + diaB); 2.23 (ddd, $^3J_{PH}$ = 22.7 Hz, $^2J_{HH}$ = 13.0 Hz, $^3J_{HH}$ = 9.0 Hz, 1H, C*H*H, diaA + diaB); 3.40 (ddd, $^3J_{PH}$ = 17.2 Hz, $^2J_{HH}$ = 13.0 Hz, $^3J_{HH}$ = 6.2 Hz, 1H, CH*H*, diaA + diaB); 4.10 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 1H, CH*H*OP, diaA + diaB); 4.11 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 1H, CH*H*OP, diaA + diaB); 4.14 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP, diaA + diaB); 4.52 ÷ 4.64 (m, 1H, C*H*O, diaA + diaB); δ_C ($CDCl_3$) 12.85 (s, *C*H$_3$, diaA + diaB); 15.11 (d, $^3J_{PC}$ = 6.0 Hz, *C*H$_3$CH$_2$OP, diaA + diaB); 15.15 (d, $^3J_{PC}$ = 6.1 Hz, *C*H$_3$CH$_2$OP, diaA + diaB); 18.21 (d, $^2J_{CP}$=12.2 Hz, *C*H$_3$), 21.00 (s, *C*H$_2$, diaA + diaB); 26.14 (s, *C*H$_2$, diaA + diaB); 34.19 (s, *C*H$_2$, diaA + diaB); 38.75 (d, $^2J_{PC}$ = 6.0 Hz,*C*H$_2$, diaA + diaB); 52.42 (d, $^1J_{PC}$ = 140.2 Hz, *C*P, diaA + diaB); 62.24 (d, $^2J_{PC}$ = 7.5 Hz, *C*H$_2$OP, diaA + diaB); 63.27 (d, $^2J_{PC}$ = 7.6 Hz, *C*H$_2$OP, diaA + diaB); 77.99 (s, *C*HO, diaA + diaB); 171.48 (d, $^2J_{PC}$ = 3.7 Hz, *C*HO, diaA + diaB). $C_{13}H_{25}O_5P$: requires C 53.42, H 8.62; found C 53.65, H 8.60.

5-Benzyl-3-methyl-2-oxotetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26f).

Purification (AcOEt, R_f = 0.42) gave the lactone **26f** as colourless oil (0.99g, 68 %) diaA : diaB = 90 : 10; ν_{max} 1764, 1224, 1044; δ_P ($CDCl_3$) 25.11 (diaA); 24.70 (diaB); δ_H ($CDCl_3$) 1.28 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaA + diaB); 1.30 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaA + diaB); 1.58 (d, $^3J_{PH}$ = 17.0 Hz, 3H, C*H*$_3$, diaA + diaB); 2.50 ÷ 2.65 (m, 1H, C*H*H, diaA + diaB); 2.78 (ddd, $^3J_{PH}$ = 18.0 Hz, $^2J_{HH}$ = 9.2 Hz, $^3J_{HH}$ = 4.2 Hz, 1H, CH*H*, diaA + diaB); 2.94 ÷ 3.08 (m, 2H, C*H*$_2$, diaA + diaB); 4.09 ÷ 4.24 (m, 5H, C*H*O, 2xC*H*$_2$OP, diaA + diaB); 7.23 ÷ 7.33 (m, 5H, C*H*$_{Ar}$, diaA + diaB); δ_C ($CDCl_3$) 16.08 (s, 2x*C*H$_3$CH$_2$OP, diaA + diaB); 18.04 (d, $^2J_{PC}$ = 8.5 Hz, *C*H$_3$, diaA + diaB); 36.87 (s, *C*H$_2$, diaA + diaB); 38.96 (S, *C*H$_2$); 45.86 (d, $^1J_{PC}$ = 132.1 Hz, *C*P, diaA + diaB); 62.51 (d, $^2J_{PC}$ = 7.5 Hz, 2x*C*H$_2$OP, diaA + diaB); 67.28 (s, *C*HO, diaA + diaB); 126.57 (s, 2×*C*H$_{Ar}$, diaA + diaB); 127.51 (s, *C*H$_{Ar}$, diaA + diaB); 128.01 (s, 2×*C*H$_{Ar}$, diaA + diaB); 137.32 (s, *C*$_{Ar}$, diaA + diaB); 170.77 (s, *C*=O, diaA + diaB). $C_{17}H_{25}O_5P$: requires C 59.99, H 7.40; found C 60.15, H 7.36.

(5-Butyl-2-oxo-3-phenyltetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26g).

Purification (AcOEt, R_f = 0.29) gave the lactone **26g** as colourless oil (0.99g, 68 %) diaA : diaB = 80 : 20; ν_{max} 1748, 1224, 1032; δ_P ($CDCl_3$) 20.39 (diaA); 20.52 (diaB); δ_H ($CDCl_3$) 0.89 (t, $^3J_{HH}$ = 6.7 Hz, 3H, C*H*$_3$, diaA + diaB); 1.10 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP, diaA + diaB); 1.32 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaA + diaB); 1.26 ÷ 1.48 (m, 4H, 2xC*H*$_2$, diaA + diaB); 1.54 ÷ 1.65 (m, 2H, C*H*$_2$, diaA + diaB); 2.38 (ddd, $^3J_{PH}$ = 22.7 Hz, $^2J_{HH}$ = 13.2 Hz, $^3J_{HH}$ = 9.5 Hz, 1H, C*H*H, diaA + diaB); 3.40 (ddd, $^3J_{PH}$ = 17.7 Hz, $^2J_{HH}$ = 13.2 Hz, $^3J_{HH}$ = 6.2 Hz, 1H, CH*H*, diaA + diaB); 4.15 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 1H, C*H*HOP, diaA + diaB); 4.16 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 1H, CH*H*OP, diaA + diaB); 4.20 (dq, $^3J_{PH}$ = $^3J_{HH}$ = 7.0 Hz, 2H, C*H*$_2$OP, diaA + diaB); 4.73 (ddt, $^3J_{HH}$ = 9.5 Hz, $^3J_{HH}$=6.2 Hz, $^3J_{HH}$ = 4.7 Hz, 1H, C*H*O, diaA + diaB); 7.33 ÷ 7.37 (m, 3H, C*H*$_{Ar}$, diaA + diaB); 7.77 ÷ 7.81 (m, 2H, C*H*$_{Ar}$, diaA + diaB); δ_C ($CDCl_3$) 12.85 (s, *C*H$_3$, diaA + diaB); 15.14 (d, $^3J_{PC}$ = 6.0 Hz, *C*H$_3$CH$_2$OP, diaA + diaB); 15.25 (d, $^3J_{PC}$ = 6.1 Hz, *C*H$_3$CH$_2$OP, diaA + diaB); 21.36 (s, *C*H$_2$, diaA + diaB); 26.28 (s, *C*H$_2$, diaA + diaB); 34.29 (s, *C*H$_2$, diaA + diaB); 38.93 (d, $^2J_{PC}$ = 5.1 Hz, *C*H$_2$, diaA + diaB); 52.42 (d, $^1J_{PC}$ = 129.7 Hz, *C*P, diaA + diaB); 62.81 (d, $^2J_{PC}$ = 7.5 Hz, *C*H$_2$OP, diaA + diaB); 63.45 (d, $^2J_{PC}$ = 7.6 Hz, *C*H$_2$OP, diaA + diaB); 76.68 (s, *C*HO, diaA + diaB); 126.50 (s, 2×*C*H$_{Ar}$, diaA + diaB); 126.84 (s, *C*H$_{Ar}$, diaA + diaB); 127.28 (d, $^3J_{PC}$ = 2.6 Hz, 2×*C*H$_{Ar}$, diaA + diaB); 134.14 (d, $^2J_{PC}$ = 7.3 Hz, *C*$_{Ar}$, diaA + diaB); 171.48 (d, $^2J_{PC}$ = 3.2 Hz, *C*=O, diaA + diaB). $C_{19}H_{29}O_5P$: requires C 61.94, H 7.93; found C 61.75, H 7.96.

(5-Benzyl-2-oxo-3-phenyltetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26h).

Purification (AcOEt, R_f = 0.42) gave the lactone **26h** as colourless oil (1.50g, 75%) diaA : diaB = 92 : 8; ν_{max} 1768, 1224, 1022; δ_P ($CDCl_3$) 20.54 (diaA); 20.21 (diaB); δ_H ($CDCl_3$) 1.12 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP, diaA + diaB); 1.27 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaA + diaB); 2.68 ÷ 2.86 (m, 1H, CH*H*, diaA + diaB); 2.88 ÷ 3.20 (m, 2H, C*H*$_2$, diaA + diaB); 3.40 (ddd, $^3J_{PH}$ = 19.5 Hz, $^2J_{HH}$=13.7 Hz, $^3J_{HH}$ = 5.7 Hz, 1H, CH*H*, diaA + diaB); 4.00 ÷ 4.22 (m, 4H, 2xC*H*$_2$O)P, diaA + diaB); 4.60 ÷ 4.70 (m, 1H, C*H*O, diaA + diaB); 7.21 ÷ 7.71 (m, 10H, C*H*$_{Ar}$, diaA + diaB); δ_C ($CDCl_3$) 15.19 (d, $^3J_{PC}$ = 6.0 Hz, *C*H$_3$CH$_2$OP, diaA + diaB); 15.29 (d, $^3J_{PC}$ = 6.1 Hz, *C*H$_3$CH$_2$OP, diaA + diaB); 32.98 (s, *C*H$_2$, diaA + diaB); 38.25 (s, *C*H$_2$, diaA + diaB); 50.78 (d, $^1J_{PC}$ = 146.2 Hz, *C*P, diaA + diaB); 63.07 (d, $^2J_{PC}$ = 6.7 Hz, *C*H$_2$OP, diaA + diaB); 63.41 (d, $^2J_{PC}$ = 6.6 Hz, *C*H$_2$OP, diaA + diaB); 74.26 (s, *C*HO, diaA + diaB); 126.48 (s, 4×*C*H$_{Ar}$, diaA + diaB); 126.94 (s, 2×*C*H$_{Ar}$, diaA + diaB); 127.14 (d, $^3J_{PC}$ = 2.9 Hz, 4×*C*H$_{Ar}$, diaA + diaB); 134.11 (d, $^2J_{PC}$ = 7.6 Hz, 2×*C*$_{Ar}$, diaA + diaB); 171.14 (d, $^2J_{PC}$ = 3.2 Hz, *C*=O, diaA + diaB). $C_{22}H_{27}O_5P$: requires C 65.66, H 6.76; found C 65.81, H 6.80.

General procedure for the preparation of lactones 26i-l.

A solution of lactone **26b**, **26c**, or **26d** (0.01m) in tetrahydrofuran (15ml) was added dropwise at -70°C to a stirred solution of LDA prepared from n-BuLi (1.6M, 0.62ml, 0.01m) and diisopropyloamine (1.06ml, 0.01m) in tetrahydrofuran (15ml). The reaction mixture was stirred for 15min at 0°C and then cooled to -70°C. Alkyl bromide (0.01m) in tetrahydrofuran (10ml) was added and stirring was continued for 1h at -70°C and then for 20h at room temperature. The reaction mixture was acidified to pH 1 with 3N HCl solution and tetrahydrofuran was evaporated under reduced pressure. The residue was extracted with chloroform (3x20ml). The combined organic layers were washed with saturated NaCl solution, dried ($MgSO_4$) and evaporated under reduced pressure to give a crude product, which was purified by column chromatography.

(3-Benzyl-5-butyl-2-oxotetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26i).

Purification (hexane-AcOEt 70:30, R_f = 0.20) gave the lactone **26i** as colourless oil (1.44g, 70%) diaA : diaB = 84 : 16; ν_{max} 1724, 1224, 1042; δ_P ($CDCl_3$) 24.22 (diaA); 23.96 (diaB); δ_H ($CDCl_3$) 0.82 (t, $^3J_{HH}$ = 6.5 Hz, 3H, C*H*$_3$, diaA); 0.82 (t, $^3J_{HH}$ = 6.7 Hz, 3H, C*H*$_3$, diaB); 1.10 ÷ 1.23 (m, 4H, 2xC*H*$_2$, diaA + diaB); 1.33 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP, diaB); 1.36 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, C*H*$_3$CH$_2$OP, diaB); 1.38 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.7 Hz, 3H, C*H*$_3$CH$_2$OP, diaA); 1.39 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.7 Hz, 3H, C*H*$_3$CH$_2$OP, diaA); 1.54 ÷ 1.63 (m, 2H, C*H*$_2$, diaA + diaB); 2.32 (ddd, $^3J_{PH}$ = 24.2 Hz, $^2J_{HH}$ = 13.7 Hz, $^3J_{HH}$ = 8.0 Hz, 1H, C*H*H, diaA); 2.37 (ddd, $^2J_{HH}$ = 13.7 Hz, $^3J_{HH}$ = 8.2 Hz, $^3J_{PH}$ = 6.0 Hz, 1H, CH*H*, diaA); 2.41 ÷ 2.48 (m, 2H, C*H*$_2$, diaB); 2.95 (dd, $^2J_{HH}$ = 13.5 Hz, $^3J_{PH}$ = 9.7 Hz, 1H, CH*H*, diaB); 2.97 (dd, $^2J_{HH}$ = 13.2 Hz, $^3J_{PH}$ = 9.0 Hz, 1H, CH*H*, diaA); 3.51 (dd, $^2J_{HH}$ = 13.2 Hz, $^3J_{PH}$ = 6.2 Hz, 1H, C*H*H, diaA); 3.60 (dd, $^2J_{HH}$ = 13.5 Hz, $^3J_{PH}$ = 7.5 Hz, 1H, C*H*H, diaB); 4.07 ÷ 4.19 (m, 2H, C*H*$_2$OP, diaA + diaB); 4.20 ÷ 4.32 (m, 2H, C*H*$_2$OP, diaA + diaB); 4.38 ÷ 4.48 (m, 1H, C*H*O, diaB); 7.22 ÷ 7.32 (m, 5H, C*H*$_{Ar}$, diaA + diaB); δ_C ($CDCl_3$) 13.30 (s, *C*H$_3$, diaA); 13.43 (s, *C*H$_3$, diaB); 15.57 (s, *C*H$_3$CH$_2$OP, diaB); 15.68 (s, *C*H$_3$CH$_2$OP, diaB); 15.93 (s, *C*H$_3$CH$_2$OP, diaA); 16.02 (s, *C*H$_3$CH$_2$OP, diaA); 21.69 (s, *C*H$_2$, diaA); 21.78 (s, *C*H$_2$, diaB); 26.46 (s, *C*H$_2$, diaB); 26.51 (s, *C*H$_2$, diaA); 32.12 (d, $^2J_{PC}$ = 2.5 Hz, *C*H$_2$, diaB); 33.68 (d, $^2J_{PC}$ = 1.9 Hz, *C*H$_2$, diaA); 34.27 (s, *C*H$_2$, diaB); 34.84 (s, *C*H$_2$, diaA); 36.38 (d, $^2J_{PC}$ = 2.75 Hz, *C*H$_2$, diaB); 38.04 (d, $^2J_{PC}$ = 2.95 Hz, *C*H$_2$, diaA); 50.62 (d, $^1J_{PC}$ = 145.7 Hz, *C*P, diaA); 51.02 (d, $^1J_{PC}$ = 134.2 Hz, *C*P, diaB); 62.73 (d, $^2J_{PC}$ = 7.1 Hz, *C*H$_2$OP, diaA); 63.07 (d, $^2J_{PC}$ = 7.7 Hz, *C*H$_2$OP, diaB); 63.36 (d, $^2J_{PC}$ = 7.7 Hz, *C*H$_2$OP, diaA); 63.41 (d, $^2J_{PC}$ = 6.0 Hz, *C*H$_2$OP, diaB); 77.79 (d, $^3J_{PC}$ = 8.0 Hz, *C*HO, diaA); 78.03 (d, $^3J_{PC}$ = 6.3 Hz, *C*HO, diaB); 126.86 (s, 2×*C*H$_{Ar}$, diaB); 127.16 (s, 2×*C*H$_{Ar}$, diaA); 128.12 (s, 2×*C*H$_{Ar}$, diaB); 128.31 (s, 2×*C*H$_{Ar}$, diaA); 129.39 (s, *C*H$_{Ar}$, diaB); 130.09 (s, *C*H$_{Ar}$, diaA); 134.80 (d, $^3J_{PC}$ = 15.85 Hz, *C*$_{Ar}$, diaB); 134.87 (d, $^3J_{PC}$ = 19.7 Hz, *C*$_{Ar}$, diaA); 173.92 (d, $^2J_{PC}$ = 4.3 Hz, *C*=O, diaB); 174.55 (s, *C*=O, diaA). $C_{19}H_{29}O_5P$: requires C 61.94, H 7.93; found C 62.10, H 7.96.

(3-Benzyl-2-oxo-5-phenyltetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26j).

Purification (hexane-AcOEt 70:30, R_f = 0.36) gave the lactone **26j** as light yellow oil (1.45g, 75%) diaA : diaB = 93 : 7; ν_{max} 1732, 1240, 1024; δ_P ($CDCl_3$) 23.91 (diaA); 23.52 (diaB); δ_H ($CDCl_3$, only diaA) 1.36 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 1.39 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP); 2.67 (ddd, $^2J_{HH}$ = 13.0 Hz, $^3J_{PH}$ = 20.2 Hz, $^3J_{HH}$ = 7.5 Hz, 1H, C*H*H); 2.73 (ddd, $^2J_{HH}$ = 13.0 Hz, $^3J_{PH}$ = 9.2 Hz, $^3J_{HH}$ = 6.7 Hz, 1H, CH*H'*); 3.04 (dd, $^2J_{HH}$ = 13.2 Hz, $^3J_{PH}$ = 9.2 Hz, 1H, CH*H*); 3.60 (dd, $^2J_{HH}$ = 13.2 Hz, $^3J_{PH}$ = 9.2 Hz, 1H, C*H*H); 3.98 (dd, $^3J_{HH}$ = 7.5 Hz, $^3J_{HH}$ = 6.7 Hz, 1H, C*H*O); 4.21 ÷ 4.34 (m, 4H, 2xC*H*$_2$OP); 7.11 ÷ 7.16 (m, 2H, C*H*$_{Ar}$); 7.27 ÷ 7.37 (m, 3H, C*H*$_{Ar}$); δ_C ($CDCl_3$, only diaA) 16.37 (s, 2x*C*H$_3$CH$_2$OP); 37.11 (s, *C*H$_2$); 38.45 (s, *C*H$_2$); 51.42 (d, $^1J_{PC}$ = 147.8 Hz, *C*P); 63.22 (s, *C*H$_2$OP); 62.86 (s, *C*H$_2$OP); 79.05 (d, $^3J_{PC}$ = 4.0 Hz, *C*HO); 125.66 (s, 2×*C*H$_{Ar}$); 127.81 (s, *C*H$_{Ar}$); 128.47 (s, 2×*C*H$_{Ar}$); 128.92 (s, 2×*C*H$_{Ar}$); 129.92 (s, 2×*C*H$_{Ar}$); 128.59 (s, *C*H$_{Ar}$); 135.13 (d, $^3J_{PC}$ = 15.6 Hz, *C*$_{Ar}$); 139.01 (s, *C*$_{Ar}$); 174.93 (s, *C*=O). $C_{21}H_{25}O_5P$: requires C 64.94, H 6.49; found C 64.75, H 6.45.

[3-(3-Methylbut-2-enyl)-2-oxo-5-phenyltetrahydrofuran-3-yl]-phosphonic acid diethyl ester (26k).

Purification (hexane-AcOEt 80:20, R_f = 0.15) gave the lactone **26k** as colourless oil (1.63g, 89%) diaA : diaB = 70 : 30; ν_{max} 1760, 1648, 1248, 1040; δ_P ($CDCl_3$) 24.59 (diaA); 24.07 (diaB); δ_H ($CDCl_3$) 1.32 (t, $^3J_{HH}$ = 7.2 Hz, 3H, C*H*$_3$CH$_2$OP, diaA); 1.35 (t, $^3J_{HH}$ = 7.2 Hz, 3H, C*H*$_3$CH$_2$OP, diaA); 1.38 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaB); 1.43 (t, $^3J_{HH}$ = 7.0 Hz, 3H, C*H*$_3$CH$_2$OP, diaB); 1.73 (s, 3H, C*H*$_3$, diaA + diaB); 1.80 (s, 3H, C*H*$_3$, diaA + diaB); 2.27 (ddd, $^3J_{PH}$ = 24.2 Hz, $^2J_{HH}$ = 13.7 Hz, $^3J_{HH}$ = 11.0 Hz, 1H, C*H*H, diaA + diaB); 2.54 ÷ 3.04 (m, 3H, , C*H*H, C*H*$_2$CH=C(CH$_3$), diaA + diaB); 4.21 (dq, $^3J_{HH}$ = 7.0 Hz, 4H, 2xC*H*$_2$OP, diaB); 4.24 (dq, $^3J_{HH}$ = 7.2 Hz, 4H, 2xC*H*$_2$OP, diaA); 4.93 ÷ 5.00 (m, 1H, C*H*O, diaB); 5.04 ÷ 5.12 (m, 1H, C*H*O, diaA); 5.59 (dd, $^3J_{HH}$ = 9.7 Hz, $^3J_{HH}$ = 7.5 Hz, 1H, C*H*, diaA + diaB); 7.31 ÷ 7.51 (m, 5H, C*H*$_{Ar}$, diaA + diaB); δ_C ($CDCl_3$) 16.20 (d, $^3J_{PC}$ = 5.8 Hz, 2X*C*H$_3$CH$_2$OP, diaB); 16.25 (d, $^3J_{PC}$ = 5.8 Hz, 2X*C*H$_3$CH$_2$OP, diaA); 17.91 (s, 2x*C*H$_3$, diaA + diaB); 25.67 (s, *C*H$_3$, diaB); 25.87 (s, *C*H$_3$, diaA); 30.11 (d, $^2J_{PC}$ = 2.4 Hz, *C*H$_2$, diaB); 31.51 (d, $^2J_{PC}$ = 2.7 Hz, *C*H$_2$, diaA); 36.85 (d, $^2J_{PC}$ = 1.9 Hz, *C*H$_2$, diaB); 37.42 (d, $^2J_{PC}$ = 1.3 Hz, *C*H$_2$, diaA); 49.74 (d, $^1J_{PC}$ = 147.3 Hz, *C*P, diaA); 50.38 (d, $^1J_{PC}$ = 135.4 Hz, *C*P, diaB); 62.99 (d, $^2J_{PC}$ = 7.1 Hz, *C*H$_2$OP, diaA); 63.15 (d, $^2J_{PC}$ = 7.4 Hz, *C*H$_2$OP, diaB); 63.35 (d, $^2J_{PC}$ = 6.8 Hz, *C*H$_2$OP, diaA); 63.65 (d, $^2J_{PC}$ = 7.0 Hz, *C*H$_2$OP, diaB); 78.96 (s, *C*HO, diaB); 79.23 (d, $^3J_{PC}$ = 8.5 Hz, *C*HO, diaA); 116.72 (d, $^3J_{PC}$ = 13.4 Hz, *C*H); 117.07 (d, $^3J_{PC}$ = 12.5 Hz, *C*H); 125.63 (s, 2×*C*H$_{Ar}$, diaB); 125.67 (s, 2×*C*H$_{Ar}$, diaA); 128.37 (s, *C*H$_{Ar}$, diaA); 128.40 (s, *C*H$_{Ar}$, diaB); 128.50 (s, 2×*C*H$_{Ar}$, diaB); 128.55 (s, 2×*C*H$_{Ar}$, diaA); 137.54 (s, *C*, diaA); 137.60 (s, *C*, diaB); 139.23 (s, *C*$_{Ar}$, diaB); 139.30 (s, *C*$_{Ar}$, diaA); 174.09 (d, $^2J_{PC}$ = 3.5 Hz, *C*=O, diaB); 174.78 (s, *C*=O, diaA). $C_{19}H_{27}O_5P$: requires C 62.29, H 7.43; found C 62.41, H 7.48.

(3-Allyl-5-benzyl-2-oxotetrahydrofuran-3-yl)-phosphonic acid diethyl ester (26l).

Purification (AcOEt, R_f = 0.35) gave the lactone **26l** as colourless oil (1.00g, 79%) diaA : diaB = 84 : 16; ν_{max} 1748, 1664, 1248, 1044; δ_P ($CDCl_3$) 21.85 (diaA); 21.70 (diaB); δ_H ($CDCl_3$) 1.30 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, CH_3CH$_2$OP, diaA); 1.31 (td, $^3J_{HH}$ = 7.0 Hz, $^4J_{PH}$ = 0.5 Hz, 3H, CH_3CH$_2$OP, diaA); 1.32 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3CH$_2$OP, diaB); 1.33 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3CH$_2$OP, diaB); 2.40 ÷ 2.53 (m, 1H, C*H*H, diaA + diaB); 2.54 (dddd, $^3J_{PH}$ = 16.5 Hz, $^2J_{HH}$ = 12.0 Hz, $^3J_{HH}$ = 6.5 Hz, $^3J_{HH}$ = 3.2 Hz, 1H, C*H*H, diaA + diaB); 2.54 (ddd, $^2J_{HH}$ = 13.2 Hz, $^3J_{PH}$ = 8.2 Hz, $^3J_{HH}$ = 7.2 Hz, 1H, C*H*H, diaA + diaB); 2.57 (dd, $^2J_{HH}$ = 13.2 Hz, $^3J_{HH}$ = 6.2 Hz, 1H, CH*H*, diaB); 2.60 (ddd, $^2J_{HH}$ = 13.2 Hz, $^3J_{PH}$ =12.5 Hz, $^3J_{HH}$ = 6.2 Hz, 1H, CH*H*, diaA); 4.11 (dq, $^3J_{PH}$ = 7.0 Hz, $^3J_{HH}$ = 7.0 Hz, 2H, CH_2OP, diaA + diaB); 4.12 (dq, $^3J_{PH}$ = 7.0 Hz, $^3J_{HH}$ = 7.0 Hz, 2H, CH_2OP, diaA + diaB); 4.54 ÷ 4.62 (m, 1H, C*H*O, diaB); 4.76 ÷ 4.85 (m, 1H, C*H*O, diaA); 7.18 ÷ 7.27 (m, 5H, CH_{Ar}, diaA + diaB); δ_C ($CDCl_3$) 16.12 (s, 2x*C*H$_3$CH$_2$OP, diaA + diaB); 31.56 (d, $^2J_{PC}$ = 2.3 Hz, *C*H$_2$, diaA); 32.00 (d, $^2J_{PC}$ = 2.5 Hz, *C*H$_2$, diaA); 37.21 (s, *C*H$_2$, diaB); 37.48 (s, *C*H$_2$, diaA); 37.55 (d, $^2J_{PC}$ = 2.8 Hz, *C*H$_2$); 38.47 (d, $^1J_{PC}$ = 158.5 Hz, *C*P, diaB); 38.61 (d, $^1J_{PC}$ = 141.0 Hz, *C*P, diaA); 62.24 (d, $^2J_{PC}$ = 7.0 Hz, *C*H$_2$OP, diaB); 62.42 (d, $^2J_{PC}$ = 6.8 Hz, *C*H$_2$OP, diaA); 78.42 (s, *C*HO, diaB); 78.50 (s, *C*HO, diaA); 126.24 (s, 2×*C*H$_{Ar}$, diaB); 126.36 (s, 2×*C*H$_{Ar}$, diaA); 128.30 (s, *C*H$_{Ar}$, diaB); 128.32 (s, *C*H$_{Ar}$, diaA); 129.20 (s, 2×*C*H$_{Ar}$, diaB); 129.25 (s, 2×*C*H$_{Ar}$, diaA); 135.14 (s, *C*$_{Ar}$, diaB); 135.21 (s, *C*$_{Ar}$, diaA); 171.02 (s, *C*=O, diaA); 171.16 (d, $^2J_{PC}$ = 3.2 Hz, *C*=O, diaB). $C_{18}H_{33}O_5P$: requires C 59.98, H 9.23; found C 59.77, H 9.18.