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for

Protein Farnesyltransferase-Catalyzed Isoprenoid Transfer to Peptide Depends on Lipid Size and Shape, not Hydrophobicity

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For Scheme 1

2-((*2E,6E*)-3,7-Dimethyl-9-phenyl-nona-2,6-dienyloxy)-tetrahydro-pyran (**14**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.28-1.85 (m, 12H), 2.00 (t, *J* = 8.4 Hz, 2H), 2.076 (t, *J* = 7.2Hz, 2H), 2.33 (t, *J* = 8.8 Hz, 2H), 2.60-2.68 (m, 2H), 3.46-3.51 (m, 1H), 3.97-4.02 (m, 1H), 4.10-4.23 (m, 1H), 4.57-4.61 (m, 1H), 5.07-5.11 (M, 1H), 5.27-5.34 (m, 1H), 7.09-7.26 (m, 5H)

(*2E,6E*)-3,7-Dimethyl-9-phenyl-nona-2,6-dien-1-ol (**15**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.63 (s, 3H), 1.64 (s, 3H), 1.98 (t, *J* = 8.4 Hz, 2H), 2.06-2.11 (m, 2H), 2.23-2.30 (m, 2H), 4.12 (d, *J* = 7.2 Hz, 2H), 5.07–5.10 (m, 1H), 5.34–5.39 (m, 1H), 7.13–7.16 (m, 3H), 7.23–7.26 (m, 2H).

(*2E,6E*)-3,7-Dimethyl-9-phenyl-nona-2,6-dien-1-diphosphate (**3**) ^{1}H NMR (400 MHz, D_2O) δ=1.51 (s, 3H), 1.53 (s, 3H), 1.86 (t, *J* = 8.0 Hz, 2H), 1.93-2.0 (m, 2H), 2.15 (t, *J* = 7.6 Hz, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 4.30 (t, *J* = 6.8 Hz, 2H), 5.0 (t, *J* = 6.8 Hz, 1H), 5.26 (t, *J* = 6.8 Hz, 1H), 7.06-7.21 (m, 5H); ^{31}P NMR (D_2O, 161.8 MHz) -6.37 (d, *J* = 22.0 Hz, 1P), -9.50 (d, *J* = 22.0Hz, 1P). LRMS: (EI) (M^+-H^+) 403 (M^+) 404.

For Scheme 2

(*2E,6E*)-3,7-Dimethyl-8-phenylsulfanyl-octa-2,6-dien-1-ol (**19a**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.62 (s, 3H), 1.73 (s, 3H), 1.93 (t, *J* = 8.0 Hz, 2H), 2.05-2.10 (m, 2H), 3.48 (s, 2H), 4.11 (d, *J* = 6.8 Hz, 2H), 7.17-7.32 (m, 5H).

(*2E,6E*)-8-Benzyloxy-3,7-dimethyl-octa-2,6-dien-1-ol (**19b**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.71 (s, 3H), 2.10-2.24 (m, 4H), 3.92 (s, 2H), 4.16 (d, *J* = 6.2 Hz, 2H), 4.48 (s, 2H), 5.41-5.49 (m, 2H), 7.33-7.38 (m, 5H).

(*2E,6E*)-3,7-Dimethyl-8-(3,4,5-trimethoxy-benzyloxy-octa-2,6-dien-1-ol (**19c**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.53 (s, 3H), 1.66 (s, 3H), 2.056 (t, *J* = 8.0 Hz, 2H), 2.14-2.20 (m, 2H), 3.81 (s, 3H), 3.84 (s, 6H), 6.37 (d, *J* = 0.8 Hz, 2H), 4.13 (d, *J* = 6.8 Hz, 2H), 4.35 (s, 2H), 5.38-5.41 (m, 2H), 6.54 (s, 2H).

(*2E,6E*)-3,7-Dimethyl-8-phenylsulfanyl-octa-2,6-dien-1-diphosphate (**4**) ^{1}H NMR (400 MHz, D_2O) δ=1.48 (s, 3H), 1.54 (s, 3H), 1.96 (t, *J* = 6.8 Hz, 2H), 2.05 (t, *J* = 7.2 Hz, 2H), 3.79 (s, 2H), 4.28 (t, *J* = 6.4 Hz, 2H), 5.28-5.33 (m, 2H), 7.21-7.25 (m, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ -7.20 (d, *J* = 20.2 Hz, 1P), -9.50 (d, *J* = 20.2 Hz, 1P). LRMS: (EI) (M^+-H^+) 421 (M^+) 422.

(*2E,6E*)-8-Benzyloxy-3,7-dimethyl-octa-2,6-dien-1-diphosphate (**5**) ^{1}H NMR (400 MHz, D_2O) δ=1.45 (s, 3H), 1.52 (s, 3H), 1.74 (t, *J* = 8.0 Hz, 2H), 1.87-1.90 (m, 2H), 3.38 (s, 2H), 4.25 (t, *J* = 6.8 Hz, 2H), 4.63 (s, 2H), 5.04 (t, *J* = 6.4 Hz, 1H), 5.15 (t, *J* = 6.4 Hz, 1H), 7.12-7.24 (m, 5H); ^{31}P NMR (D_2O, 161.8 MHz) -8.1 (d, *J* = 20.2 Hz, 1P), -9.30 (d, *J* = 20.2 Hz, 1P). LRMS: (EI) (M^+-H^+) 419 (M^+) 420.

(*2E,6E*)-3,7-Dimethyl-8-(3,4,5-trimethoxy-benzyloxy-octa-2,6-dien-1-diphosphate (**7**) ^{1}H NMR (400 MHz, D_2O) δ=1.46 (s, 3H), 1.54 (s, 3H), 1.191 (t, *J* = 6.4 Hz, 2H), 1.99-2.02 (m, 2H), 3.58 (s, 3H), 3.66 (s, 6H), 3.72 (s, 2H), 4.20 (s, 2H), 4.29 (t, *J* = 6.0 Hz, 2H), 5.24-5.32 (m, 2H), 6.50 (s, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ -6.41 (d, *J* = 20.7 Hz, 1P), -9.43 (d, *J* = 20.7 Hz, 1P). LRMS: (EI) (M^+-H^+) 509 (M^+) 510.

For Scheme 3

(*2E,6E*)-8-(-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24a**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.73(s, 3H), 2.08 (t, *J* = 8.0 Hz, 2H), 2.18-2.24 (m, 2H), 4.13 (d, *J* = 7.6 Hz, 2H), 4.37 (s, 2H), 5.37-5.42 (m, 1H), 5.49-5.53 (m, 1H), 6.90-6.95 (m, 2H), 7.24-7.28 (m, 2H).

(*2E,6E*)-8-(2-Fluoro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24b**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.66 (s, 3H), 1.73 (s, 3H), 2.06 (t, *J* = 8.0 Hz, 2H), 2.16-2.22 (m, 2H), 4.13 (d, *J* = 7.6 Hz, 2H), 4.44 (s, 2H), 5.36-5.40 (m, 1H), 5.49-5.52 (m, 1H), 6.84-7.08 (m, 4H).

(*2E,6E*)-8-(3-Fluoro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24c**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.65 (s, 3H), 1.70 (s, 3H), 2.06 (t, *J* = 7.6 Hz, 2H), 2.15-2.22 (m, 2H), 4.12 (d, *J* = 6.8 Hz, 2H), 4.34 (s, 2H), 5.38 (t, *J* = 6.0 Hz, 1H), 5.49 (t, *J* = 7.6 Hz, 1H), 6.60-6.70 (m, 3H), 7.20-7.25 (m, 1H).

(*2E,6E*)-8-(4-Fluoro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24d**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.71 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.22-2.16 (m, 2H), 4.13 (d, *J* = 6.8 Hz, 2H), 4.32 (s, 2H), 5.37-5.42 (m, 1H), 5.47-5.51 (m, 1H), 6.80-6.85 (m, 2H), 6.91-6.07 (m, 2H).

(*2E,6E*)-8-(2,6-Difluoro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24e**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.63 (s, 3H), 1.77 (s, 3H), 2.0 (t, *J* = 8.4 Hz, 2H), 2.11-2.17 (m, 2H), 4.12 (d, *J* = 6.8 Hz, 2H), 4.48 (s, 2H), 5.33-5.38 (m, 1H), 5.41-5.45 (m, 1H), 6.81-6.95 (m, 3H).

(*2E,6E*)-8-(3,4-Difluoro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24f**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.70 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.17-2.22 (m, 2H), 4.14 (d, *J* = 6.8 Hz, 2H), 4.31 (s, 2H), 5.38-5.42 (m, 1H), 5.47-5.51 (m, 1H), 6.56-6.60 (m, 1H), 6.67-6.73 (m, 1H), 6.98-7.06 (m, 1H).

(*2E,6E*)-8-(2, 3-Difluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24g**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.66 (s, 3H), 1.73 (s, 3H), 2.06 (t, *J* = 8.0 Hz, 2H), 2.16-2.22 (m, 2H), 4.14 (d, *J* = 6.8 Hz, 2H), 4.44 (s, 2H), 5.37-5.41 (m, 1H), 5.49-5.53 (m, 1H), 6.70-6.77 (m, 1H), 6.90-6.96 (m, 1H).

(*2E,6E*)-8-(3, 5-Difluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24h**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.66 (s, 3H), 2.06 (t, *J* = 8.0 Hz, 2H), 2.16-2.22 (m, 2H), 4.13 (d, *J* = 7.2 Hz, 2H), 4.32 (s, 2H), 5.36-5.41 (m, 1H), 5.47-5.50 (m, 1H), 6.34-6.43 (m, 3H).

(*2E, 6E*)-3,7-Dimethyl-8-(2,3,5,6-tetrafluoro-phenoxy)-oct-2-en-ol (**24i**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.64 (s, 3H), 1.75 (s, 3H), 2.00 (t, *J* = 8.0 Hz, 2H), 2.12-2.18 (m, 2H), 4.13 (d, *J* = 7.6 Hz, 2H), 4.57 (s, 2H), 5.35-5.38 (m, 1H), 5.44-5.48 (m, 1H), 6.70-6.79 (m, 1H).

3-(*2E,6E*)-8-Hydroxy-2,6-dimethyl-octa-2,6-dienyloxy)-benzonitrile (**24j**) ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.67 (s, 3H), 1.70 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.23-2.18 (m, 2H), 4.14 (d, *J* = 7.2 Hz, 2H), 4.39 (s, 2H), 5.37-5.42 (m, 1H), 5.49-5.53 (m, 1H), 7.11-7.13 (m, 2H), 7.20-7.23 (m, 1H), 7.32-7.36 (m, 1H).

4-((*2E,6E*)-8-Hydroxy-2,6-dimethyl-octa-2,6-dimethyl-octa-2,6-dienyloxy)-benzonitrile (**24k**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.71 (s, 3H), 2.07 (t, *J* = 8 Hz, 2H), 2.18-2.23 (m, 2H), 4.14 (d, *J* = 6.8 Hz, 2H), 4.41 (s, 2H), 5.42-5.37 (m, 1H), 5.53-5.50 (m, 1H), 6.93 (d, *J* = 8.8 Hz, 2H), 7.55 (d, *J* = 8.8 Hz, 2H).

(*2E,6E*)-8-(4-Nitro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24l**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.66 (s, 3H), 1.72 (s, 3H), 2.06

(t, *J* = 8.4 Hz, 2H), 2.24-2.18 (m, 2H), 4.14 (d, *J* = 6.8 Hz, 2H), 4.46 (s, 2H), 5.37-5.42 (m, 1H), 5.51-5.55 (m, 1H), 6.94 (d, *J* = 9.2 Hz, 2H), 8.17 (d, *J* = 9.2 Hz, 2H).

(*2E,6E*)-8-(2-Bromo-phenoxy)-3,7-dimethyl-oct-2-en-1-ol (**24m**) ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.67 (s, 3H), 1.75 (s, 3H), 2.08 (t, *J* = 8.0Hz, 2H), 2.2 (m, 2H), 4.13 (d, *J* = 6.8 Hz, 2H), 4.44 (s, 1H), 5.38-5.42 (m, 1H), 5.52-5.56 (m,1H), 6.8 (ddd, *J* = 1.6, 7.6, 7.6 Hz, 1H), 6.87 (d, *J* = 11.5 Hz, 1H), 7.19-7.24 (1H, m), 7.52 (dd, *J* = 1.6, 7.6 Hz, 1H).

(*2E,6E*)-8-(3-Bromo-phenoxy)-3,7-dimethyl-octa-2,6-dien-ol (**24n**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.70 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.16-2.23 (m, 2H), 4.13 (d, *J* = 7.6 Hz, 2H), 4.35 (s, 2H), 5.37-5.42 (m, 1H), 5.47-5.52 (m, 1H), 6.81-6.84 (m, 1H), 7.04-7.06 (m, 2H), 7.10 (t, *J* = 7.2 Hz, 1H).

(*2E,6E*)-8-(4-Bromo-phenoxy)-3,7-dimethyl-octa-2,6-dien-ol (**24o**) ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.67 (s, 3H), 1.70 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.17-2.21 (m, 2H), 4.13 (d, *J* = 7.2 Hz, 2H), 4.33 (s, 2H), 5.37-5.41 (m, 1H), 5.47-5.51 (m, 1H), 6.77 (d, *J* = 8.8 Hz, 2H), 7.34 (d, *J* = 8.8 Hz, 2H).

(*2E,6E*)-8-(3-Trifluoromethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24p**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.72 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.15-2.23 (m, 2H), 4.13 (d, *J* = 7.2 Hz, 2H), 4.40 (s, 2H), 5.38-5.42 (m, 1H), 5.51-5.54 (m, 1H), 7.04-7.19 (m, 3H), 7.35 (t, *J* = 8.0 Hz, 1H).

(*2E,6E*)-8-(3,5-dichloro-phenoxy)-3,7-dimethyl-oct-2-en-1-ol (**24q**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.70 (s, 3H), 2.07 (t, *J* = 8.4 Hz, 2H), 2.17-2.23 (m, 2H), 4.14 (d, *J* = 7.6 Hz, 2H), 4.34 (s, 2H), 5.38-5.42 (m, 1H), 5.47-5.52 (m, 1H), 6.79 (d, *J* = 1.6 Hz, 2H), 6.92 (t, *J* = 1.6Hz, 1H).

(*2E,6E*)-8-(3,4-Dichloro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24r**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.70 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.23-2.16 (m, 2H), 4.14 (d, *J* = 7.2 Hz, 2H), 4.34 (s, 2H), 5.42-5.38 (m, 2H), 5.48-5.51 (m, 2H), 6.75 (dd, *J* = 2.8, 8.8 Hz, 1H), 6.98 (d, *J* = 2.8 Hz, 1H), 7.29 (d, *J* = 8.8 Hz, 1Hz).

(*2E,6E*)-8-(2-Iodo-phenoxy)-3,7-dimethyl-oct-2-en-1-ol (**24s**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.76 (s, 3H), 2.08 (t, *J* = 7.6 Hz), 2.24-2.19 (m, 2H), 4.14 (d, *J* = 6.8 Hz, 2H), 4.42 (s, 2H), 5.54-5.58 (m, 1H), 6.68 (ddd, *J* = 2.0, 8, 8Hz, 1H), 6.78 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.23-7.27 (m, 1H), 7.75 (dd, *J* = 8.0, 2.0 Hz, 1H).

(*2E,6E*)-8-(3-Iodo-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24t**) ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.67 (s, 3H), 1.70 (s, 3H), 2.07 (t, *J* = 8 Hz, 2H), 2.23-2.17 (m, 2H), 4.13 (d, *J* = 7.6 Hz, 2H), 4.34 (s, 2H), 5.38-5.42 (m, 2H), 5.52-5.48 (m, 2H), 6.84-6.87 (m, 1H), 6.96 (t, *J* = 8.0 Hz, 1H), 7.27-7.24 (m, 2H).

(*2E,6E*)-8-(4-Iodo-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24u**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.70 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.17-2.22 (m, 2H), 4.13 (d, *J* = 7.2 Hz, 2H), 4.33 (s, 2H), 5.37-5.41 (m, 1H), 5.47-5.50 (m, 1H), 6.67 (d, *J* = 8.8 Hz, 2H), 7.64 (d, *J* = 9.2 Hz, 2H).

(*2E, 6E*)-8-(2-Ethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24v**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.19 (t, *J* = 7.6 Hz, 3H), 1.68 (s, 3H), 1.73 (s, 3H), 2.08 (t, *J* = 8, 2H), 2.16-2.24 (m, 2H), 2.65 (q, *J* = 0.8, 7.2, 7.2 Hz, 2H), 4.13 (d, *J* = 6.8 Hz, 2H), 4.37 (s, 2H), 5.38-5.43 (m, 1H), 5.49-5.54 (m, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.87 (ddd, *J* = 0.8, 7.2, 7.2 Hz, 1Hz), 7.10-7.15 (m, 2H).

(*2E,6E*)-8-(3-Ethyl -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24w**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.22 (t, *J* = 7.6 Hz, 3H), 1.76 (s, 3H), 1.72 (s, 3H), 2.08 (t, *J* = 8.0 Hz, 2H), 2.17-2.23 (m, 2H), 2.61 (q, *J* = 7.6, 7.6 Hz), 4.13 (d, *J* = 6.8 Hz, 2H), 4.36 (s, 2H), 5.37-5.42 (m, 1H), 5.49-5.53 (m, 1H), 6.70-6.83 (m, 3H), 7.17 (t, *J* = 8.0 Hz, 1H).

(*2E,6E*)-8-(4-ethyl -phenoxy)-3,7-dimethyl-oct-2-en-1-ol (**24x**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.19 (t, 7.6 Hz, 3H), 1.67 (s, 3H), 1.71 (s, 3H), 2.07 (t, *J* = 8.4 Hz, 2H), 2.17-2.23 (m, 2H), 2.57 (q, *J* = 8.8 Hz, 1H), 4.13 (d, *J* = 7.6 Hz, 2H), 4.34 (s, 2H), 5.37-5.42 (m, 1H), 5.52-5.48 (m, 1H), 6.82 (d, *J* = 8.8 Hz, 2H), 7.08 (d, *J* = 8.8 Hz, 2H).

(*2E,6E*)-8-(3-Trifluoromethoxy -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24y**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.65 (s, 3H), 1.70 (s, 3H), 2.08 (t, *J* = 8.0 Hz, 2H), 2.15-2.20 (m, 2H), 4.10 (d, *J* = 6.8 Hz, 2H), 4.50 (s, 2H), 5.35 (t, *J* = 6.5 Hz, 1H), 5.48 (t, *J* = 6.5 Hz, 1H), 6.70-6.80 (m, 3H), 7.23-7.30 (m, 1H)

(*2E,6E*)-8-(4-Trifluoromethoxy- phenoxy)-3,7-dimethyl-oct-2-en-1-ol (**24z**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.71 (s, 3H), 2.08 (t, *J* = 8.4 Hz, 2H), 2.18-2.23 (m, 2H), 4.14 (d, *J* = 7.2 Hz, 2H), 4.35 (s, 2H), 5.38-5.42 (m, 1H), 5.48-5.52 (m, 1H), 6.87 (d, *J* = 9.2 Hz, 2H), 7.11 (d, *J* = 9.2 Hz, 2H).

(*2E,6E*)-8-(3-Isopropyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24aa**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.22 (d, *J* = 7.2Hz, 6H), 1.67 (s, 3H), 1.73 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.16-2.23 (m, 2H), 2.80-2.90 (m, 1H), 4.13 (d, *J* = 6.8 Hz, 2H), 4.36 (s, 2H), 5.38-5.42 (m, 1H), 5.49-5.53 (m, 1H), 6.70-6.73 (m, 1H), 6.78-6.80 (m, 1H), 8.0-8.1 (m, 1H), 7.17 (t, *J* = 7.2 Hz, 1H).

(*2E,6E*)-8-(4-Isopropyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24ab**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.67 (s, 3H), 1.72 (s, 3H), 2.07 (t, *J* = 8.0 Hz, 2H), 2.16-2.23 (m, 2H), 2.79-2.89 (m, 1H), 4.13 (d, *J* = 6.8 Hz, 2H), 4.34 (s, 2H), 5.37-5.42 (m, 1H), 5.48-5.52 (m, 1H), 6.83 (d, *J* = 8.8 Hz, 2H), 7.11 (d, *J* = 8.4 Hz, 2H).

(*2E,6E*)-8-(2-phenyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24ac**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.62 (s, 3H), 1.65 (s, 3H), 2.03 (t, *J* = 8.4 Hz, 2H), 2.12-2.17 (m, 2H), 4.10 (dd, *J* = 0.8, 7.2 Hz, 2H), 4.35 (s, 2H), 5.34-5.43 (m, 2H), 6.94-7.03 (m, 2H), 7.25-7.40 (m, 5H), 7.52-7.54 (m, 2H).

(*2E,6E*)-8-(4-benzyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (**24ad**) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.65 (s, 3H), 1.70 (s, 3H), 2.05 (t, *J* = 8.4 Hz, 2H), 2.15-2.21 (m, 2H), 8.90 (s, 2H), 4.11 (d, *J* = 5.6 Hz, 2H), 4.32 (s, 2H), 5.35-5.40 (m, 1H), 5.45-5.50 (m, 1H), 6.80-6.83 (m, 2H), 7.04-7.07 (m, 2H), 7.14-7.18 (m, 3H), 7.23-7.27 (m, 2H).

(*2E,6E*)-3,7-Dimethyl-8-phenoxy-octa-2,6-dien-1-diphosphate (**6a**) ^{1}H NMR (400 MHz, D_2O) δ=1.53 (s, 6H), 1.95 (t, *J* = 7.2 Hz, 2H), 2.02-2.10 (m, 2H), 4.30 (t, *J* = 6.4 Hz, 2H), 4.40 (s, 2H), 5.23 (t, *J* = 6.4 Hz, 1H), 5.46 (t, *J* = 6.4 Hz, 1H), 6.90-6.95 (m, 3H), 7.19-7.22 (m, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ -5.51 (d, *J* = 20.7 Hz, 1P), -9.20 (d, *J* = 20.7 Hz, 1P). LRMS: (EI) (M^+-H^+) 405 (M^+) 406.

(*2E,6E*)-8-(2-Fluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (**6b**) ^{1}H NMR (400 MHz, D_2O) δ=1.50 (s, 3H), 1.53 (s, 3H), 1.91 (t, *J* = 7.6 Hz, 2H), 2.01-2.06 (m, 2H), 4.26 (t, *J* = 6.4 Hz, 2H), 4.41 (s, 2H), 5.23 (t, *J* = 7.2 Hz, 1H), 5.40 (t, *J* = 7.2 Hz, 1H), 6.80-6.86 (m, 1H), 6.94-7.03 (m, 3H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.84 (d, *J* = 20.0 Hz, 1P), -9.58 (d, *J* = 20.0Hz, 1P). LRMS: (EI) (M^+-H^+) 423 (M^+) 424.

(*2E,6E*)-8-(3-Fluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (**6c**) ^{1}H NMR (400 MHz, D_2O) δ 1.52 (s, 6H), 1.94 (t, *J* = 7.2 Hz, 2H), 2.03 - 2.08 (m, 2H), 4.28 (t, *J* = 6.8 Hz, 2H), 4.33 (s, 2H), 5.25 (t, *J* = 6.8 Hz, 1H), 5.41 (t, *J* = 6.8 Hz, 1H), 6.58-6.66

(m, 3H), 7.12-7.18 (m, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-9.29 (d, *J* = 19.5 Hz, 1P), -9.05 (d, *J* = 19.5Hz, 1P). LRMS: (EI) (M^+-H^+) 423 (M^+) 424.

(*2E,6E*)-8-(4-Fluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6d) ^{1}H NMR (400 MHz, D_2O) δ=1.52 (s, 6H), 1.93 (t, *J* = 7.6 Hz, 2H), 2.02-2.07 (m, 2H), 4.28 (t, *J* = 6.4 Hz, 2H), 4.30 (s, 2H), 5.26 (t, *J* = 6.8 Hz, 1H), 5.40 (t, *J* = 6.4 Hz, 6.80-6.84 (m, 2H), 6.87-6.93 (m, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.28 (d, *J* = 22.0 Hz, 1P), -9.46 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 423 (M^+) 424.

(*2E,6E*)-8-(2, 6-Difluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6e) ^{1}H NMR (400 MHz, D_2O) δ=1.48 (s, 3H), 1.61 (s, 3H), 1.84 (t, *J* = 7.6 Hz, 2H), 1.97-2.02 (m, 2H), 4.27 (t, *J* = 6.0 Hz, 2H), 4.42 (s, 2H), 5.20 (t, *J* = 7.2 Hz, 1H), 5.31 (t, *J* = 6.8 Hz, 1H), 6.84-7.00 (m, 3H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.14 (d, *J* = 22.0 Hz, 1P), -9.47 (d, *J* = 22.0Hz, 1P). LRMS: (EI) (M^+-H^+) 441 (M^+) 442.

(*2E,6E*)-8-(3, 4-Difluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6f) ^{1}H NMR (400 MHz, D_2O) δ 1.54 (s, 6H), 1.96 (t, *J* = 7.6 Hz, 2H), 2.05-2.10 (m, 2H), 4.31 (t, *J* = 7.2 Hz, 2H), 4.32 (s, 2H), 5.27 (t, *J* = 7.2 Hz, 1H), 5.42 (t, *J* = 6.8 Hz, 1H), 6.60-6.63 (m, 1H), 6.74-6.80 (m, 1H), 7.00-7.10 (m, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-8.74 (d, *J* = 18.2 Hz, 1P), -9.68 (d, *J* = 20.8 Hz, 1P). LRMS: (EI) (M^+-H^+) 441 (M^+) 442.

(*2E,6E*)-8-(2, 3-Difluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6g) ^{1}H NMR (400 MHz, D_2O) δ=1.54 (s, 3H), 1.55 (s, 3H), 1.95 (t, *J* = 7.6 Hz, 2H), 2.05-2.10 (m, 2H), 4.30 (t, *J* = 6.4 Hz, 2H), 4.42 (s, 2H), 5.27 (t, *J* = 6.0 Hz, 1H), 5.46 (t, *J* = 6.8 Hz, 1H), 7.07-7.10 (m, 1H), 7.20 (m, 2H), 7.35 (t, *J* = 8.0 Hz, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ -7.16 (d, *J* = 22.0 Hz, 1P), -9.56 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 441 (M^+) 442.

(*2E,6E*)-8-(3, 5-Difluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6h) ^{1}H NMR (400 MHz, D_2O) δ=1.54 (s, 6H), 1.96 (t, *J* = 6.8 Hz, 2H), 2.04-2.11 (m, 2H), 4.30 (t, *J* = 6.4 Hz, 2H), 4.33 (s, 2H), 5.29 (t, *J* = 6.4 Hz, 2H), 5.44 (t, *J* = 6.8 Hz, 1H), 6.38-6.48 (m, 3H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.25 (d, *J* = 22.0 Hz, 1P), -9.52 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 441 (M^+) 442.

(*2E,6E*)-8-(2,3,5,6-Difluoro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6i) ^{1}H NMR (400 MHz, D_2O) δ=1.50 (s, 3H), 1.60 (s, 3H), 1.85 (t, *J* = 7.2 Hz, 2H), 1.97-2.04 (m, 2H), 4.27 (t, *J* = 6.4 Hz, 2H), 4.49 (s, 2H), 5.23 (t, *J* = 7.6 Hz, 1H), 5.37 (t, *J* = 6.8 Hz, 1H), 6.86-6.96 (m, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-5.89 (t, *J* = 22.0 Hz, 1P), -9.50 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 478 (M^+) 478.

(*2E,6E*)-8-(3-Cyano-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6j) ^{1}H NMR (400 MHz, D_2O) δ=1.53 (s, 3H), 1.54 (s, 3H), 1.94 (t, *J* = 6.8 Hz, 2H), 2.04-2.1 (m, 2H), 4.28 (t, *J* = 7.2 Hz, 2H), 4.38 (s, 2H), 5.26 (t, *J* = 6.4 Hz, 1H), 5.43 (t, *J* = 6.4 Hz, 1H), 7.13- 7.23 (m, 3H), 7.31 (t, *J* = 8.0 Hz, 1H) ; ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.60 (d, *J* = 19.5 Hz, 1P), -9.40 (d, *J* = 19.5 Hz, 1P). LRMS: (EI) (M^+-H^+) 430 (M^+) 431.

(*2E,6E*)-8-(4-Cyano-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6k) ^{1}H NMR (400 MHz, D_2O) δ=1.60 (s, 6H), 1.97 (t, *J* = 7.6 Hz, 2H), 2.07-2.13 (m, 2H), 4.32 (t, *J* = 6.8 Hz, 2H), 4.43 (s, 2H), 5.29 (t, *J* = 7.6 Hz, 1H), 5.46 (t, *J* = 6.4 Hz, 1H), 6.95 (d, *J* = 8.8 Hz, 2H), 7.57 (d, *J* = 8.8 Hz, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-8.22 (d, *J* = 20.0 Hz, 1P), -9.68 (d, *J* = 20.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 430 (M^+) 431.

(*2E,6E*)-8-(4-Nitro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6l) ^{1}H NMR (400 MHz, D_2O) δ=1.53 (s, 3H), 1.55 (s, 3H), 1.95 (t, *J* = 7.2 Hz, 2H), 2.05-2.10 (m, 2H), 4.29 (t, *J* = 6.4 Hz, 2H), 4.45 (s, 2H), 5.26 (t, *J* = 6.4 Hz, 1H), 5.45 (t, *J* = 6.8 Hz, 1H), 6.93 (d, *J* = 9.2 Hz, 2H); ^{31}P NMR (D_2O, 161.8MHz) δ=-8.41 (d, *J* = 20.7 Hz, 1P), -9.66 (d, *J* = 20.7 Hz, 1P). LRMS: (EI) (M^+-H^+) 450 (M^+) 451.

(*2E,6E*)-8-(2-Bromo-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6m) ^{1}H NMR (400 MHz, D_2O) δ=1.52 (s, 3H), 1.56 (s, 3H), 1.93 (t, *J* = 7.6 Hz, 2H), 2.03-2.08 (m, 2H), 4.28 (t, *J* = 6.8 Hz, 2H), 4.43 (s, 2H), 5.24 (t, *J* = 6.4 Hz, 1H), 5.42 (t, *J* = 6.8 Hz, 1H), 6.77-6.81 (m, 1H), 6.95 (d, *J* = 8.0 Hz, 1H), 7.17-7.21 (m, 1H), 7.46 (dd, *J* = 1.6, 8.0 Hz, 1H); ^{31}P NMR (D_2O, 161.8MHz) δ=-9.58 (d, *J* = 22.0 Hz, 1P), -7.00 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 483 (M^+) 484.

(*2E,6E*)-8-(3-Bromo-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6n) ^{1}H NMR (400 MHz, D_2O) δ=1.53 (s, 6H), 1.94 (d, *J* = 7.2 Hz, 2H), 2.04-2.09 (m, 2H), 4.28 (d, *J* = 6.4 Hz, 2H), 4.35 (s, 2H), 5.25 (t, *J* = 6.8 Hz, 1H), 5.41 (t, *J* = 5.6 Hz, 1H), 6.80-6.84 (m, 1H), 7.02-7.10 (m, 3H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-9.73 (d, *J* = 18.2 Hz, 1P), -9.11 (d, *J* = 18.2.0Hz, 1P). LRMS: (EI) (M^+-H^+) 483 (M^+) 484.

(*2E,6E*)-8-(4-Bromo-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6o) ^{1}H NMR (400 MHz, D_2O) δ=1.54 (s, 6H), 1.96 (t, *J* = 7.6 Hz, 2H), 2.01-2.10 (m, 2H), 4.31 (t, *J* = 6.4 Hz, 2H), 4.35 (s, 2H), 5.28 (t, *J* = 5.6 Hz, 1H), 5.43 (t, *J* = 6.8 Hz, 1H), 6.80 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 8.8 Hz, 2H)**;** ^{31}P NMR (D_2O, 161.8 MHz) δ=-8.60 (d, *J* = 20.8Hz, 1P), -9.62 (d, *J* = 20.8 Hz, 1P) . LRMS: (EI) (M^+-H^+) 483 (M^{+2}) 484.

(*2E,6E*)-8-(3-Trifluoromethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6p) ^{1}H NMR (400 MHz, D_2O) δ=1.55 (s, 3H), 1.56 (s, 3H), 1.97 (t, *J* = 7.6 Hz, 2H), 2.11-2.06 (m, 2H), 4.31 (t, *J* = 6.4 Hz, 2H), 4.35 (s, 2H), 5.30 (t, *J* = 6.8 Hz, 1H), 5.45 (t, *J* = 7.2 Hz, 1H), 6.84-6.86 (m, 3H), 6.96 (t, *J* = 1.6 Hz, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.53 (d, *J* = 21.8 Hz, 1P), -9.50 (d, *J* = 21.8 Hz, 1P) . LRMS: (EI) (M^+-H^+) 473 (M^+) 474.

(*2E,6E*)-8-(3,5-Dichloro-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6q) ^{1}H NMR (400 MHz, D_2O) δ=1.54 (s, 3H), 1.57 (s, 3H), 1.95 (t, *J* = 7.2 Hz, 2H), 2.05-2.11 (m, 2H), 4.30 (t, *J* = 6.8 Hz, 6.8Hz, 2H), 4.46 (s, 2H), 5.28 (t, *J* = 7.2 Hz, 1H), 5.45 (t, *J* = 7.2 Hz, 1H), 6.74-6.83 (m, 2H), 6.92-7.0 (m, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ 6.62 (d, *J* = 20.7 Hz, 1P), 7.53 (d, *J* = 20.7 Hz, 1P). LRMS: (EI) (M^+-H^+) 474 (M^+) 475.

(*2E,6E*)-8-(3, 4-Dichloro -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6r) ^{1}H NMR (400 MHz, D_2O) δ=1.55 (s, 6H), 1.96 (t, *J* = 7.6 Hz, 2H), 2.05 - 2.11 (m, 2H), 4.31 (t, *J* = 6.8 Hz, 2H), 4.35 (s, 3H), 5.29 (t, *J* = 6.8 Hz, 1H), 5.44 (t, *J* = 6.8 Hz, 1H), 6.78 (dd, *J* = 3.2, 7.6 Hz, 1H), 7.03 (d, *J* = 2.8 Hz, 1H), 7.30 (d, *J* = 9.2 Hz, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-7.1 (d, *J* = 22.0 Hz, 1P), -9.53 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 474 (M^+) 475.

(*2E,6E*)-8-(2-Iodo -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6s) ^{1}H NMR (400 MHz, D_2O) δ=1.52 (s, 3H), 1.56 (s, 3H), 1.94 (t, *J* = 7.6 Hz, 2H), 2.03-2.08 (m, 2H), 4.28 (t, *J* = 6.8 Hz, 2H), 4.39 (s, 2H), 5.25 (t, *J* = 6.8 Hz, 1H), 5.44 (t, *J* = 9.0 Hz, 1H), 6.65 (t, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 1H), 7.21 (t, *J* = 8.0 Hz, 1H), 7.68 (dd, *J* = 2.0, 8.0 Hz, 1H) ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.47 (d, *J* = 20.7 Hz, 1P), -9.51 (d, *J* = 20.70 Hz, 1P). LRMS: (EI) (M^+-H^+) 531 (M^+) 532.

(*2E,6E*)-8-(3-Iodo -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6t) ^{1}H NMR (400 MHz, D_2O) δ=1.50 (s, 3H), 1.52 (s, 3H), 1.92 (t, *J* = 7.6 Hz, 2H), 2.03 - 2.08 (m, 2H), 4.25 - 4.33 (m, 2H), 5.24 (t, *J* = 6.8 Hz, 1H), 5.37 (t, *J* = 6.8 Hz, 1H), 6.78 - 6.92 (m, 2H), 7.18 - 7.26 (m, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-8.93(d, *J* = 22.0 Hz, 1P), -9.73(d, *J* = 22.0Hz, 1P). LRMS: (EI) (M^+-H^+) 474 (M^+) 475.

(*2E,6E*)-8-(4-Iodo -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6u) ^{1}H NMR (400 MHz, D_2O) δ=1.50 (s, 3H), 1.51 (s, 3H), 1.91 (t, *J* = 7.6 Hz, 2H), 2.00 - 2.10 (m, 2H), 4.27 (s, 2H), 4.28 (t, 2H, *J* = 6.4 Hz, 2H), 5.25 (t, *J* = 5.6 Hz, 1H), 5.37 (t, *J* = 5.6 Hz, 1H), 6.61 (d, *J* = 8.8 Hz, 2H), 7.45 (8.8 Hz, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-8.03 (d, *J* = 20.8 Hz, 1P), -9.64 (d, *J* = 20.8 Hz, 1P). LRMS: (EI) (M^+-H^+) 474 (M^+) 475.

(*2E, 6E*)-8-(2-Ethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6v) ^{1}H NMR (400 MHz, D_2O) δ=1.00 (t, *J* = 7.2 Hz, 3H), 1.53 (s, 3H), 1.56 (s, 3H), 1.95 (t, *J* = 7.2 Hz, 2H), 2.04-2.10 (m, 2H), 2.47 (q, *J* = 7.6, 7.6 Hz, 2H), 4.29 (t, *J* = 6.8 Hz, 2), 4.34 (s, 2H), 5.26 (t, *J* = 6.8 Hz, 1H), 5.42 (t, *J* = 6.8 Hz, 1H), 6.82-6.88 (m, 2H), 7.04-7.12 (m, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-9.51 (d, *J* = 20.7 Hz, 1P), -9.80 (d, *J* = 20.7 Hz, 1P). LRMS: (EI) (M^+-H^+) 433 (M^+) 434.

(*2E, 6E*)-8-(3-Ethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6w) ^{1}H NMR (400 MHz, D_2O) δ=1.00 (t, J = 7.6 Hz, 3H), 1.53 (s, 6H), 1.94 (t, *J* = 7.6 Hz, 2H), 2.03-2.08 (m, 2H), 2.44 (q, *J* = 7.6, 7.6 Hz, 2H), 4.29 (t, *J* = 6.8 Hz, 2H), 4.34 (s, 2H), 5.25 (t, *J* = 6.4 Hz, 1H), 5.41 (t, *J* = 6.0 Hz, 1H), 6.67 (dd, *J* = 2.4, 8.0 Hz, 1H), 6.74 – 6.80 (m, 2H), 7.09 – 7.13 (m, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-9.51 (d, *J* = 20.7 Hz, 1P), -9.80 (d, *J* = 19.6 Hz, 1P). LRMS: (EI) (M^+-H^+) 433 (M^+) 434.

(*2E,6E*)-8-(4-Ethyl -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6x) ^{1}H NMR (400 MHz, D_2O) δ=1.0 (t, *J* = 7.6 Hz, 3H), 1.52 (s, 6H), 1.94 (t, *J* = 7.6 Hz, 2H), 2.02 - 2.08 (m, 2H), 2.41 (q, *J* = 8.0, 8.0 Hz, 2H), 4.29 (t, *J* = 6.8 Hz, 2H), 4.32 (s, 2H), 5.25 (t, *J* = 6.0 Hz, 1H), 5.40 (t, *J* = 6.0 Hz, 1H), 6.79 (d, *J* = 8.8 Hz, 1H), 7.06 (d, *J* = 8.8 Hz, 2H); ^{31}P NMR (D_2O, 161.8MHz) δ=-9.75 (d, *J* = 20.7 Hz, 1P), -9.05 (d, *J* = 20.7 Hz, 1P). LRMS: (EI) (M^+-H^+) 433 (M^+) 434.

(*2E,6E*)-8-(3-Trifluoromethoxy -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6y) ^{1}H NMR (400 MHz, D_2O) δ=1.53 (s, 6H), 1.93 (t, *J* = 7.2 Hz, 2H), 2.02 - 2.10 (m, 2H), 4.28 (t, *J* = 6.4 Hz, 2H), 4.67 (s, 2H), 5.23 (t, *J* = 6.4 Hz, 1H), 5.42 (t, *J* = 6.4 Hz, 1H), 6.78 - 6.83 (m, 3H), 7.23 (t, *J* = 8.0 Hz, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-8.17(d, *J* = 21.0 Hz, 1P), -9.68(d, *J* = 21.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 489 (M^+) 490.

(*2E,6E*)-8-(4-Trifluoromethoxy-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6z) ^{1}H NMR (400 MHz, D_2O) δ=1.52 (s, 6H), 1.93 (t, *J* = 7.2 Hz, 2H), 2.03 - 2.08 (m, 2H), 2.90 (t, *J* = 6.8 Hz, 2H), 4.32 (s, 2H), 5.26 (t, *J* = 6.8 Hz, 1H), 5.40 (t, *J* = 6.8 Hz, 1H), 5.40 (t, *J* = 6.8 Hz, 1H), 7.3 (d, *J* = 9.2 Hz, 2H), 7.76 (d, *J* = 9.2 Hz, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-7.80 (d, *J* = 22.0 Hz, 1P), -9.63 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 489 (M^+) 490.

(*2E,6E*)-8-(3-Isopropyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6aa) ^{1}H NMR (400 MHz, D_2O) δ=1.02 (d, *J* = 6.8 Hz, 6H), 1.53 (s, 3H), 1.57 (s, 3H), 1.95 (t, *J* = 7.2 Hz, 2H), 2.05-2.10 (m, 2H), 3.12-3.19 (m, 1H), 4.29 (t, *J* = 6.0 Hz, 2H), 4.34 (s, 2H), 5.26 (t, *J* = 6.8 Hz, 1H), 5.42 (t, *J* = 6.8 Hz, 1H), 6.87-6.90 (m, 2H), 7.04-7.08 (m, 1H), 7.19 (dd, *J* = 1.6, 8.0 Hz, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-9.38 (d, *J* = 22.0 Hz, 1P), -9.72 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 447 (M^+) 448.

(*2E,6E*)-8-(4-Isopropyl -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6ab) ^{1}H NMR (400 MHz, D_2O) δ=1.02 (d, *J* = 6.5 Hz, 6H), 1.53 (s, 6H), 1.93 (t, *J* = 7.6 Hz, 2H), 2.03-2.08 (m, 2H), 2.67-2.74 (m, 2H), 4.30 (t, *J* = 6.8 Hz, 2H), 4.32 (s, 2H), 5.2 (t, *J* = 6.4 Hz, 1H), 5.41 (t, *J* = 7.2 Hz, 1H), 6.8 (d, *J* = 8.4 Hz, 2H), 7.10 (d, *J* = 8.4 Hz, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-8.41 (d, *J* = 22.0 Hz, 1P), -9.70 (d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 447 (M^+) 448.

(*2E,6E*)-8-(2-Phenyl -phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6ac) ^{1}H NMR (400 MHz, D_2O) δ=1.45(s, 3H), 1.52 (s, 3H), 1.91 (t, *J* = 6.8 Hz, 2H), 1.99-2.05 (m, 2H), 4.28 (t, *J* = 6.8 Hz, 2H), 4.30 (s, 2H), 5.23 (t, *J* = 7.2 Hz, 1H), 5.30 (t, *J* = 7.2 Hz, 1H), 6.97-7.03 (m, 2H), 7.22-7.29 (m, 3H), 7.33-7.40 (m, 6H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-8.78 (d, *J* = 22.0 Hz, 1P), -9.73(d, *J* = 22.0 Hz, 1P). LRMS: (EI) (M^+-H^+) 481 (M^+) 482.

(*2E,6E*)-8-(4-Benzyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (6ad) ^{1}H NMR (400 MHz, D_2O) δ=1.51 (s, 6H), 1.93 (t, *J* = 6.8 Hz, 2H), 2.01-2.06 (m, 2H), 3.76 (s, 3H), 4.29 (t, *J* = 6.0 Hz, 2H), 4.30 (s, 2H), 5.25 (t, *J* = 6.4 Hz, 1H), 5.38 (t, *J* = 6.4 Hz, 1H), 6.77 (d, *J* = 8.4 Hz, 1H), 7.05-7.20 (m, 8H); ^{31}P NMR (D_2O, 161.8 MHz) δ -8.17 (d, *J* = 20.8 Hz, 1P), -9.68 (d, *J* = 20.9 Hz, 1P). LRMS: (EI) (M^+-H^+) 495 (M^+) 496.

For Scheme 4

2-((*2E,6E*)-2,6-Dimethyl-8-(tetrahydro-pyran-2-yloxy)-octa-2,6-dienyloxy)-benzoic acid methyl ester (27a) ^{1}H NMR (400 MHz, $CDCl_3$) δ= 1.49-1.61 (m, 4H), 1.67 (s, 3H), 1.74 (s, 3H), 1.76-1.84 (m, 1H), 2.08 (d, *J* = 8.4 Hz, 2H), 2.18-2.23 (m, 2H), 3.47-3.53 (m, 1H), 3.85-3.91 (m, 4H, OMe protons merged with other proton), 3.98-4.03 (m, 1H), 4.21-4.25 (m, 1H), 4.50 (s, 2H), 4.60-4.62 (m, 1H), 5.34-5.38 (m, 1H), 5.54-5.57 (m, 1H), 6.92-6.97 (m, 1H), 7.39-7.43 (m, 1H), 7.77 (dd, *J* = 2.0, 8.0 Hz, 1H).

3-((*2E,6E*)-2,6-Dimethyl-8-(tetrahydro-pyran-2-yloxy)-octa-2,6-dienyloxy)-benzaldehyde (27b) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.47-1.59 (m, 4H), 1.67 (s, 3H), 1.69-1.70 (m, 1H), 1.72 (s, 3H), 1.79 -1.85 (m, 1H), 2.1 (t, *J* = 8.4 Hz, 2H), 2.18-2.23 (m, 2H), 3.47-3.54 (m, 1H), 3.85-3.90 (m, 1H), 3.97-4.02 (m, 1H), 4.20-4.24 (m, 1H), 4.44 (s, 2H), 4.58-4.62 (m, 1H), 5.35 (t, *J* = 7.2 Hz, 1H), 5.54 (t, *J* = 6.8 Hz, 1H), 7.16-7.19 (m, 1H), 7.38 -7.44 (m, 3H), 9.95 (s, 1H).

4-((*2E,6E*)-2,6-Dimethyl-8-(tetrahydro-pyran-2-yloxy)-octa-2,6-dienyloxy)-benzaldehyde (27c) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.50-1.60 (m, 5H), 1.65 (s, 3H), 1.69-1.70 (m, 1H), 1.71 (s, 3H), 2.1 (t, *J* = 8.4 Hz, 2H), 2.15-2.22 (m, 2H), 3.44-3.54 (m, 1H), 3.81-3.92 (m, 1H), 3.93-4.03 (m, 1H), 4.17-4.26 (m, 1H), 4.44 (s, 2H), 4.58-4.62 (m, 1H), 5.34 (t, *J* = 7.2 Hz, 1H), 5.52 (t, *J* = 6.8 Hz, 1H), 7.00 (d, *J* = 8.8Hz, 2H), 7.80 (d, *J* = 8.8Hz, 2H) 9.85 (s, 1H).

(2-((*2E,6E*)-2,6-Dimethyl-8-(tetrahydro-pyran-2-yloxy)-nona-2,6-dienyl)-phenyl)-methanol (28a) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.49-1.61 (m, 4H), 1.67 (s, 3H), 1.72 (s, 3H), 1.80-1.85 (m, 1H), 2.08 (t, *J* = 8.0 Hz, 2H), 2.16-2.22 (m, 2H), 3.47-3.53 (m, 1H), 3.85-3.90 (m, 1H), 4.00-4.03 (m, 1H), 4.21-4.24 (m, 1H), 4.42 (s, 2H), 4.60-4.62 (m, 1H), 4.70 (s, 2H), 5.34-5.39 (m, 1H), 5.50-5.54 (m, 1H), 6.82-6.87 (m, 1H), 6.90-6.94 (m, 1H), 7.18-7.28 (m, 2H).

(3-((*2E,6E*)-2,6-Dimethyl-8-(tetrahydro-pyran-2-yloxy)-octa-2,6-dienyloxy)-phenyl)-methanol (28b) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.49-1.60 (m, 4H), 1.66 (s, 3H), 1.70 (s, 3H), 1.79-1.84 (m, 1H), 3.47-3.53 (m, 1H), 3.85-3.97 (m, 1H), 4.00-4.05 (m, 1H), 4.20-4.23 (m, 1H), 4.38 (s, 2H), 4.60-4.61 (m, 1H), 4.65 (s, 2H), 5.32-5.35 (m, 1H), 5.48-5.53 (m, 1H), 6.81-6.84 (m, 1H), 6.90-6.93 (m, 2H), 7.22-7.30 (m, 1H).

(4-((*2E,6E*)-2,6-Dimethyl-8-(tetrahydro-pyran-2-yloxy)-octa-2,6-dienyloxy)-phenyl)-methanol (28c) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.56 (s, 6H), 1.70 (t, *J* = 8.0 Hz, 2H), 1.78-1.82 (m, 2H), 3.48-3.52 (m, 2H), 3.86-3.91 (m, 2H), 4.08-4.13 (m, 1H), 4.23-4.25 (m, 1H), 4.25-4.42 (m, 1H), 4.42 (d, *J* = 2.8 Hz, 2H), 4.44-4.48 (m, 1H), 4.48-4.76 (m, 4H), 5.75-5.79 (m, 2H), 6.80-6.84 (m, 1H), 6.92-6.93 (m, 2H), 7.21-7.24 (m, 2H).

(*2E,6E*)-8-(2-Hydroxymethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (29a) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.65(s, 3H), 1.70 (s,

3H), 2.03 (t, *J* = 8.0 Hz, 2H), 2.19-2.02 (m, 2H), 4.10 (d, *J* = 6.8 Hz, 2H), 4.40(s, 2H), 4.70 (s, 2H), 6.82 (d, *J* = 8.0 Hz, 1H), 6.91(m, 1H), 7.22-7.28 (m, 1H).

(*2E,6E*)-8-(3-Hydroxymethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-dien-1-ol (29b) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.66 (s, 3H), 1.71 (s, 3H), 2.07 (t, *J* = 8.0Hz, 2H), 2.17-2.23 (m, 2H), 4.11 (d, *J* = 6.8 Hz, 2H), 4.39 (s, 2H), 4.65 (s, 2H), 5.35 (t, *J* = 6.8 Hz, 1H), 5.49 (t, *J* = 6.8 Hz, 1H), 6.81-6.84 (m, 1H), 6.90-6.93 (m, 2H), 7.22-7.26 (m, 2H).

(*2E,6E*)-8-(4-Hydroxymethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-ol (29c) ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.64 (s, 3H), 1.70 (s, 3H), 2.06 (t, *J* = 8.0 Hz, 2H), 2.16-2.22 (m, 2H), 4.07 (dd, *J* = 2.8, 6.8 Hz, 2H), 4.38 (s, 2H), 4.60 (s, 2H), 5.28-5.33 (m, 1H), 5.45-5.49 (m, 1H), 6.88 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H).

Compound 30a ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.56-1.75 (m, 15H), 2.04-2.90 (m, 2H), 2.15-2.18 (m, 2H), 3.50-3.56 (m, 1H), 3.88-4.03 (m, 1H), 4.37 (s, 2H), 4.52-4.64 (m, 2H), 4.75-4.81 (m, 1H), 5.11-5.23 (m, 1H), 5.30-5.36 (m, 1H), 5.45-5.48 (m, 1H), 6.20-6.26 (m, 1H), 6.81-6.86 (m, 1H), 6.90-6.94 (m, 1H), 7.18-7.30 (m, 1H), 7.40-7.45 (m, 2H), 7.60-7.70 (m, 2H), 7.94-8.00 (m, 1H).

Compound 30b ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.50-1.72 (m, 15H), 2.05-2.25 (m, 4H), 3.40-3.57 (m, 2H), 3.84 -4.06 (m, 2H), 4.39 (s, 2H), 4.56-4.62 (m, 1H), 5.10-5.27 (m, 1H), 5.28-5.47 (m, 2H), 6.22-6.29 (m, 1H), 6.82-6.97 (m, 2H), 7.18-7.31 (m, 1H), 7.40-7.46 (m, 1H), 7.59-7.73 (m, 2H), 7.83-8.00 (m, 2H).

Compound 30c ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.57-1.92 (m, 15H), 2.08-2.19 (m, 4H), 3.44-3.60 (m, 1H), 3.84-4.07 (m, 2H), 4.20-4.29 (m, 1H), 4.37 (s, 2H), 4.55-4.73 (m, 2H), 5.30-5.52 (m, 2H), 6.18-6.29 (m, 1H), 6.88 (d, *J* = 13.2 Hz, 2H), 7.23-7.29 (m, 3H), 7.39-7.48 (m, 1H), 7.63-7.70 (m, 1H), 7.83-7.99 (m, 1H).

Compound 31a ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.66 (s, 3H), 1.67 (s, 3H), 1.68 (s, 3H), 2.06 (t, *J* = 7.6 Hz, 2H), 2.15-2.20 (m, 2H), 4.13 (d, *J* = 6.8Hz, 2H), 4.37 (s, 3H), 5.11-5.24 (m, 2H), 5.37-5.41 (m, 1H), 5.45-5.49 (m, 1H), 6.23-6.28 (m, 1H), 6.84 (d, *J* = 8.4Hz, 1H), 6.90 (t, *J* = 7.6Hz, 1H), 7.24-7.28 (m, 2H), 7.41-7.45 (m, 1H), 7.60-7.71 (m, 2H), 7.96 (dd, *J* = 1.6, 8.4Hz, 1H)

Compound 31b ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.66 (s, 3H), 1.67 (s, 3H), 1.71 (s, 3H), 2.07 (t, *J* = 8.4 Hz, 2H), 2.16-2.22 (m, 2H), 4.13 (t, *J* = 6.8 Hz, 2H), 4.34 (s, 3H), 5.04 (dd, *J* = 12, 12 Hz, 2H), 5.37-5.41 (m, 1H), 5.48-5.52 (m, 1H), 6.24 (dd, *J* = 6.8 Hz, 1H), 6.84-6.89 (m, 3H), 7.21-7.25 (m, 2H), 7.41-7.45 (m, 1H), 7.60-7.69 (m, 2H), 7.95-7.97 (m, 1H).

Compound 31c ^{1}H NMR (400 MHz, $CDCl_3$) δ=1.65 (s, 3H), 1.67 (s, 3H), 1.71 (s, 3H), 2.07 (t, *J* = 7.6 Hz, 2H), 2.17-2.23 (m, 2H), 4.13 (d, *J* = 6.8 Hz, 2H), 4.36 (s, 2H), 5.01 (dd, *J* = 12.4, 12.4 Hz, 2H), 5.38 (t, *J* = 7.2 Hz, 1H), 5.50 (t, *J* = 7.2 Hz, 1H), 6.21-6.26 (m, 1H), 6.86 (d, *J* = 8.8 Hz, 2H), 7.25 (d, *J* = 8.8 Hz, 2H), 7.41-7.45 (m, 1H), 7.60-7.68 (m, 2H), 7.95-7.97 (m, 1H).

Compound 32a ^{1}H NMR (400 MHz, D_2O) δ=1.45 (s, 3H), 1.49 (s, 3H), 1.50 (s, 3H), 1.87 (t, *J* = 6.4 Hz, 2H), 1.95-2.00 (m, 2H), 4.25 (s, 2H), 4.28 (t, *J* = 6.4 Hz, 2H), 4.50 (dd, *J* = 11.6, 11.6 Hz, 1H), 6.80-6.88 (m, 2H), 7.12 (d, *J* = 7.6 Hz, 1H), 7.22 (t, *J* = 7.2 Hz, 1H), 7.37 (t, *J* = 8.4 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.80 (d, *J* = 8.4 Hz, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-7.64 (d, *J* = 22.0 Hz, 1P), -9.66 (d, *J* = 22.0 Hz, 1P).

Compound 32b ^{1}H NMR (400 MHz, D_2O) δ=1.46 (s, 9H), 1.86 (t, *J* = 7.2 Hz, 2H), 1.95-2.05 (m, 2H), 4.24 (s, 2H), 4.63 (s, 2H, merged with D_2O peak), 4.88 (s, 2H), 5.18-5.24 (m, 1H), 5.30-5.36 (m, 1H), 5.90-5.96 (m, 1H), 6.62-6.66 (m, 1H), 6.70-6.80 (m, 2H), 7.00-7.15 (m, 1H), 7.28-7.35 (m, 1H), 7.36-7.42 (m, 1H), 7.45-7.51 (m, 1H), 7.75-7.80 (m, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ =-7.77 (d, *J* = 20.2 Hz, 1P), -9.67 (d, *J* = 20.2 Hz, 1P).

(*2E,6E*)-8-(2-Hydroxymethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (9a) ^{1}H NMR (400 MHz, D_2O) δ 1.52 (s, 3H), 1.54 (s, 3H), 1.94 (t, *J* = 6.8 Hz, 2H), 2.03-2.07 (m, 2H), 4.28 (t, *J* = 6.8 Hz, 2H), 4.37 (s, 2H), 4.50 (s, 2H), 5.24 (t, *J* = 6.0 Hz, 1H), 5.41(t, *J* = 7.2 Hz, 1H), 6.85-6.90 (m, 2H), 7.15-7.20 (m, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-9.00 (d, *J* = 20.0 Hz, 1P), -9.474 (d, *J* = 20.0 Hz, 1P).

(*2E,6E*)-8-(3-Hydroxymethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (9b) ^{1}H NMR (400 MHz, D_2O) δ=1.53 (s, 3H), 1.54 (s, 3H), 1.94 (t, *J* = 7.6 Hz, 2H), 2.03 – 2.083 (m, 2H), 4.28 (t, *J* = 6.4 Hz, 2H), 4.36 (s, 2H), 4.43 (s, 2H), 5.26 (t, *J* = 6.0 Hz, 1H), 5.42 (t, J = 6.0Hz, 1H), 6.78 – 6.86 (m, 3H), 7.16 – 7.20 (m, 1H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.40 (d, *J* = 22.0 Hz, 1P), -9.474 (d, *J* = 22.0 Hz, 1P).

(*2E,6E*)-8-(4-Hydroxymethyl-phenoxy)-3,7-dimethyl-octa-2,6-dien-1-diphosphate (9c) ^{1}H NMR (400 MHz, D_2O) δ=1.52 (s, 3H), 1.54 (s, 3H), 1.94 (t, *J* = 7.2Hz, 2H), 2.04-2.09 (m, 2H), 4.28 (t, *J* = 6.4 Hz, 2H), 4.36 (s, 2H), 4.40 (s, 2H), 5.25 (t, *J* = 6.8 Hz, 1H), 5.42 (t, *J* = 6.8 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H); ^{31}P NMR (D_2O, 161.8 MHz) δ=-6.856 (d, *J* = 22.0 Hz, 1P), -9.52 (d, *J* = 22.0 Hz, 1P).

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TS-VI-118H-May25-2007

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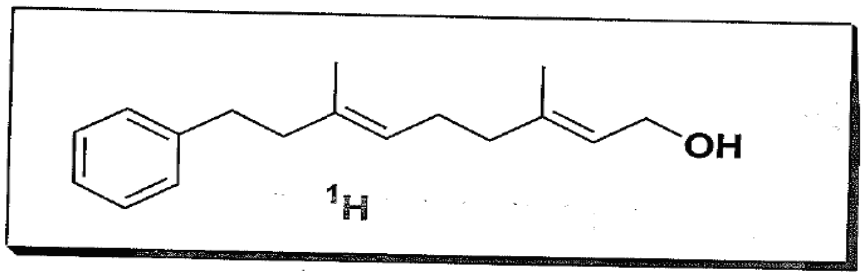
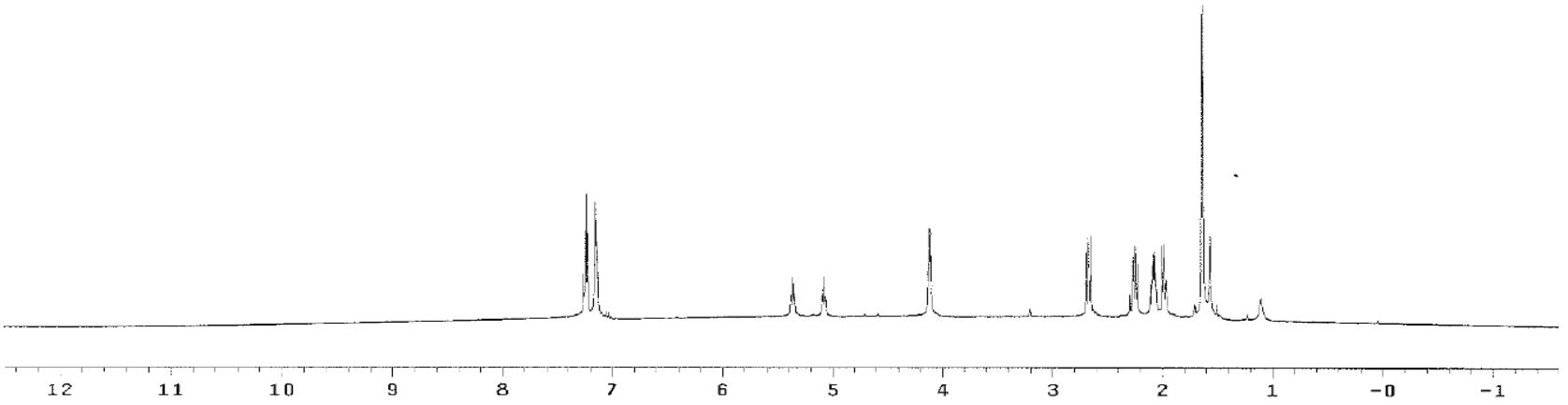


Proton NMR of **15**



TS-II-80PP-Dec10-2004

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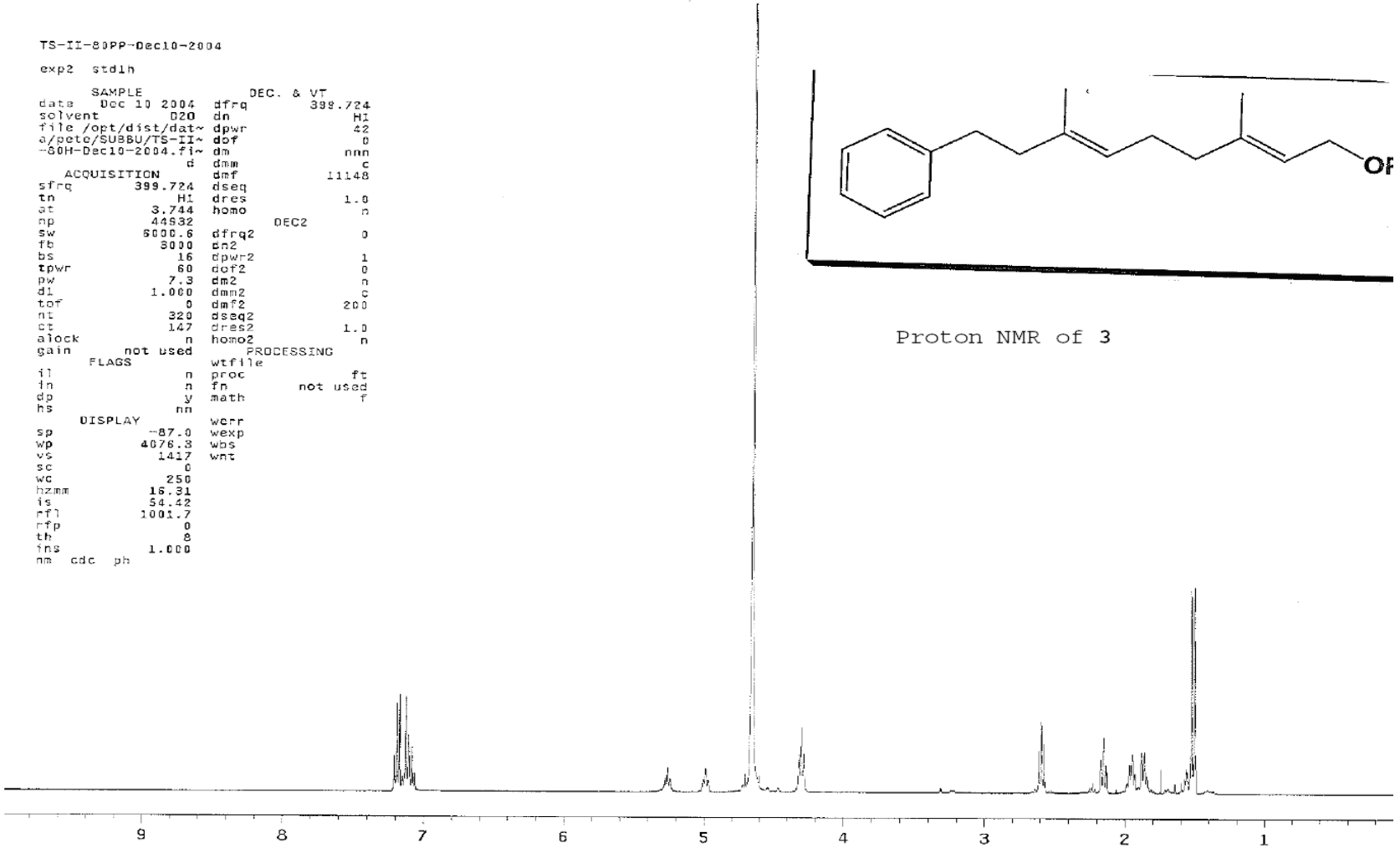
nt	320	dseq2	
ct	147	dres2	1.0
alock	n	homo2	n
gain	not used	**PROCESSING**	
FLAGS		wtfile	
il	n	proc	ft
in	n	fn	not used
dp	y	math	f
hs	nn		
DISPLAY		werr	
sp	-87.0	wexp	
wp	4076.8	wbs	
vs	1417	wnt	
sc	0		
wc	250		
hzmm	16.31		
is	54.42		
rfl	1001.7		
rfp	0		
th	8		
ins	1.000		
nm cdc ph			

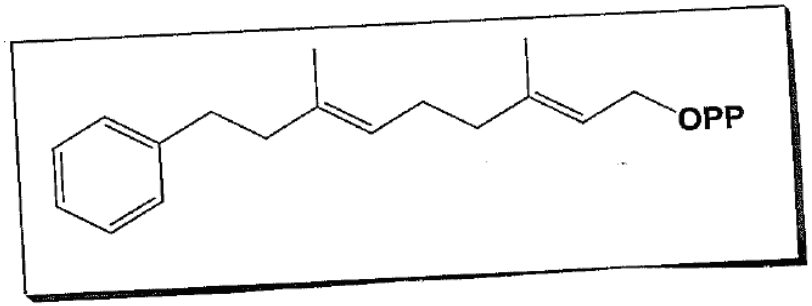
Proton NMR of 3



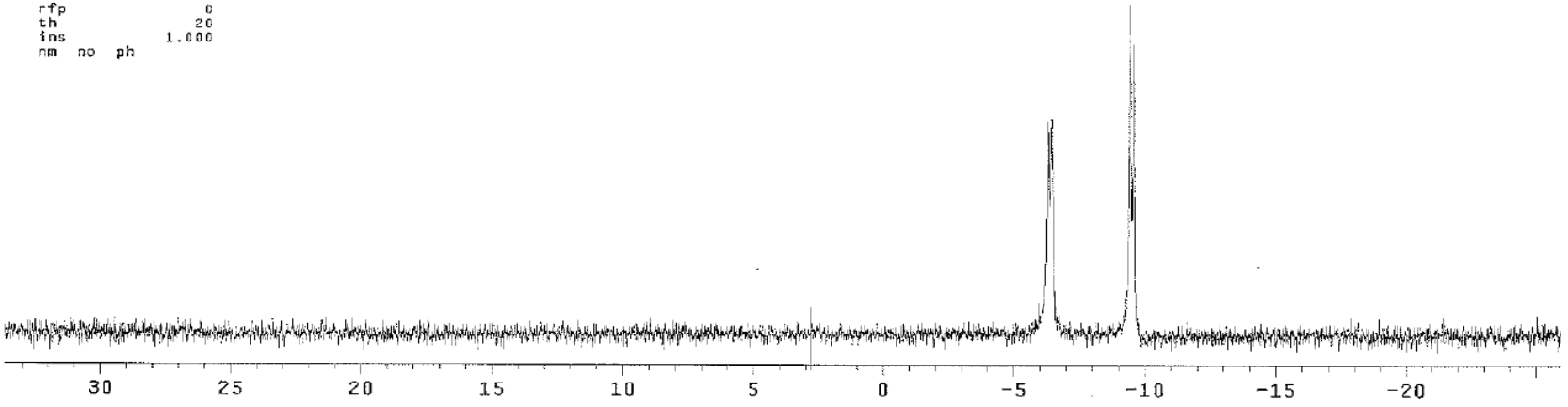
TS-II-80PP-Dec10-2004

exp2 s2pul

SAMPLE		DEC. & VT	
date	Dec 10 2004	dfrq	399.724
solvent	D2O	dn	H1
file	/opt/dist/dat~	dpwr	42
	a/pete/SUBBU/TS-II~	dof	0
	-80P-Dec10-2004.fi~	dm	yyy
	a	dmm	w
ACQUISITION		dmf	11148
sfrq	161.811	dseq	
tn	P31	dres	1.0
at	0.800	homo	n
np	16000	DEC2	
sw	10000.0	dfrq2	undefined
fb	6000	dn2	undefined
bs	64	dpwr2	undefined
tpwr	55	dof2	undefined
pw	10.0	dm2	undefined
d1	0	dmm2	undefined
tof	0	dmf2	undefined
nt	640	dseq2	undefined
ct	640	dres2	undefined
alock	n	homo2	undefined
gain	not used	PROCESSING	
FLAGS		lb	1.00
il	n	wtfile	
in	n	proc	ft
dp	y	fn	not used
hs	nn	math	f
DISPLAY			
sp	-4546.0	werr	
wp	9998.8	wexp	
vs	55	wbs	
sc	0	wnt	
wc	250		
hzmm	20.00		
is	13281.79		
rfl	4547.2		
rfp	0		
th	20		
ins	1.000		
nm no ph			

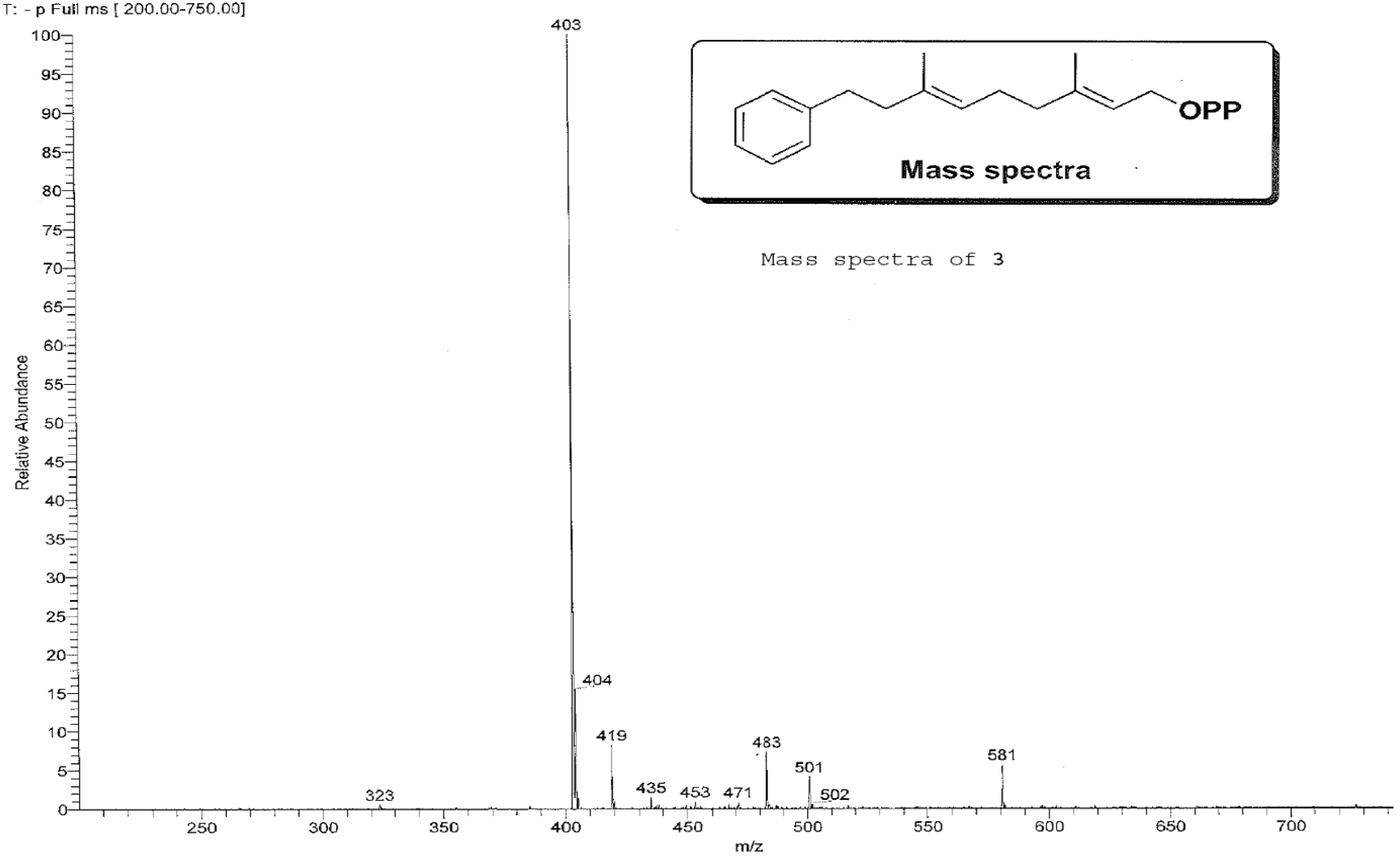


Phosphorus NMR of 3



\\lcq\data\UKMSF\08-0046 01/25/2008 11:14:26 AM TS-II-80-3PP

08-0046 #3-18 RT: 0.13-0.85 AV: 16 NL: 1.13E4
T: - p Full ms [200.00-750.00]

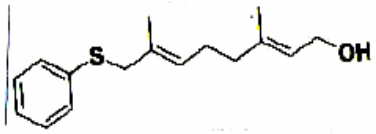


Mass spectra of 3

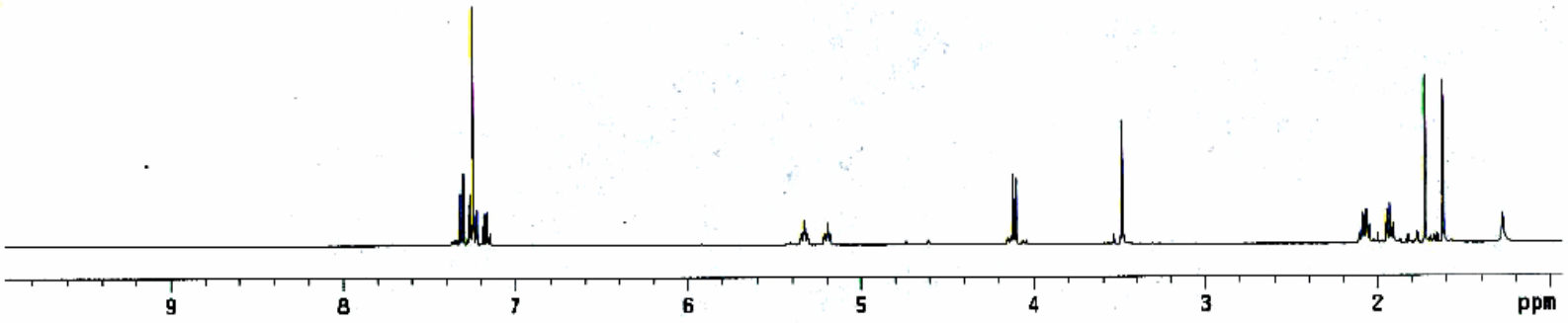
TS-II-41-oct3-2004

expl stdin

SAMPLE		DEC. & VT	
date	Oct 3 2004	dfrq	399.723
solvent	CDCl3	dn	H1
file	/opt/dist/dat~	dpwr	42
a/pete/SUSBU/TS-II~		dof	0
-41-Oct3-2004.fid		dm	nnn
ACQUISITION		dmm	c
sfrq	399.723	dmf	11145
tn	H1	dseq	
at	3.744	dres	1.0
np	44932	homo	n
sw	6000.6	DEC2	
fb	3004	dfrq2	0
bs	16	dn2	
tpwr	60	dpwr2	1
pw	7.3	dof2	0
d1	1.000	dm2	n
tof	0	dmm2	c
nt	160	dmf2	200
ct	80	dseq2	
alock	n	dres2	1.0
gain	not used	homo2	n
FLAGS		PROCESSING	
il	n	wtfile	
in	n	proc	ft
dp	y	fn	not used
hs	nn	math	f
DISPLAY			
sp	378.4	werr	
wp	3611.2	wexp	
vs	36	wbs	
sc	0	wnt	
wc	250		
hzmm	14.44		
is	441.72		
rfl	1801.7		
rfp	0		
th	18		
ins	1.000		
nm cdc ph			

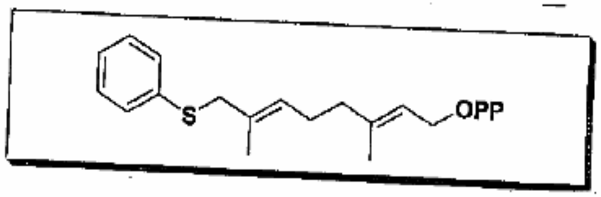


Proton NMR of 19a

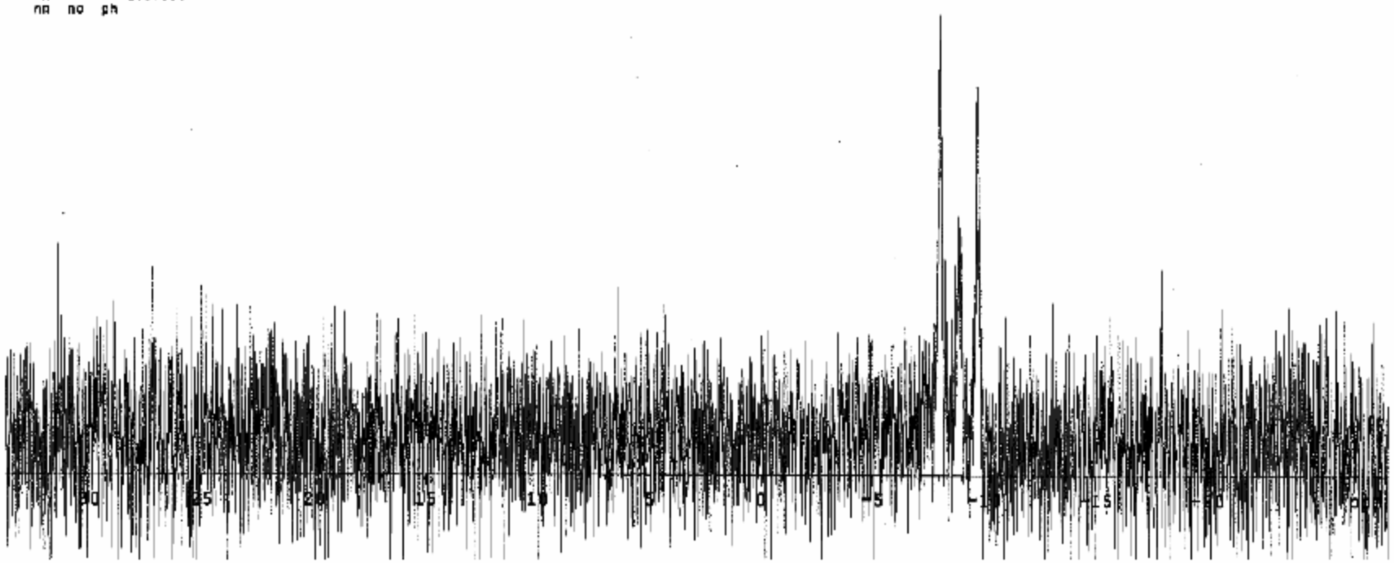


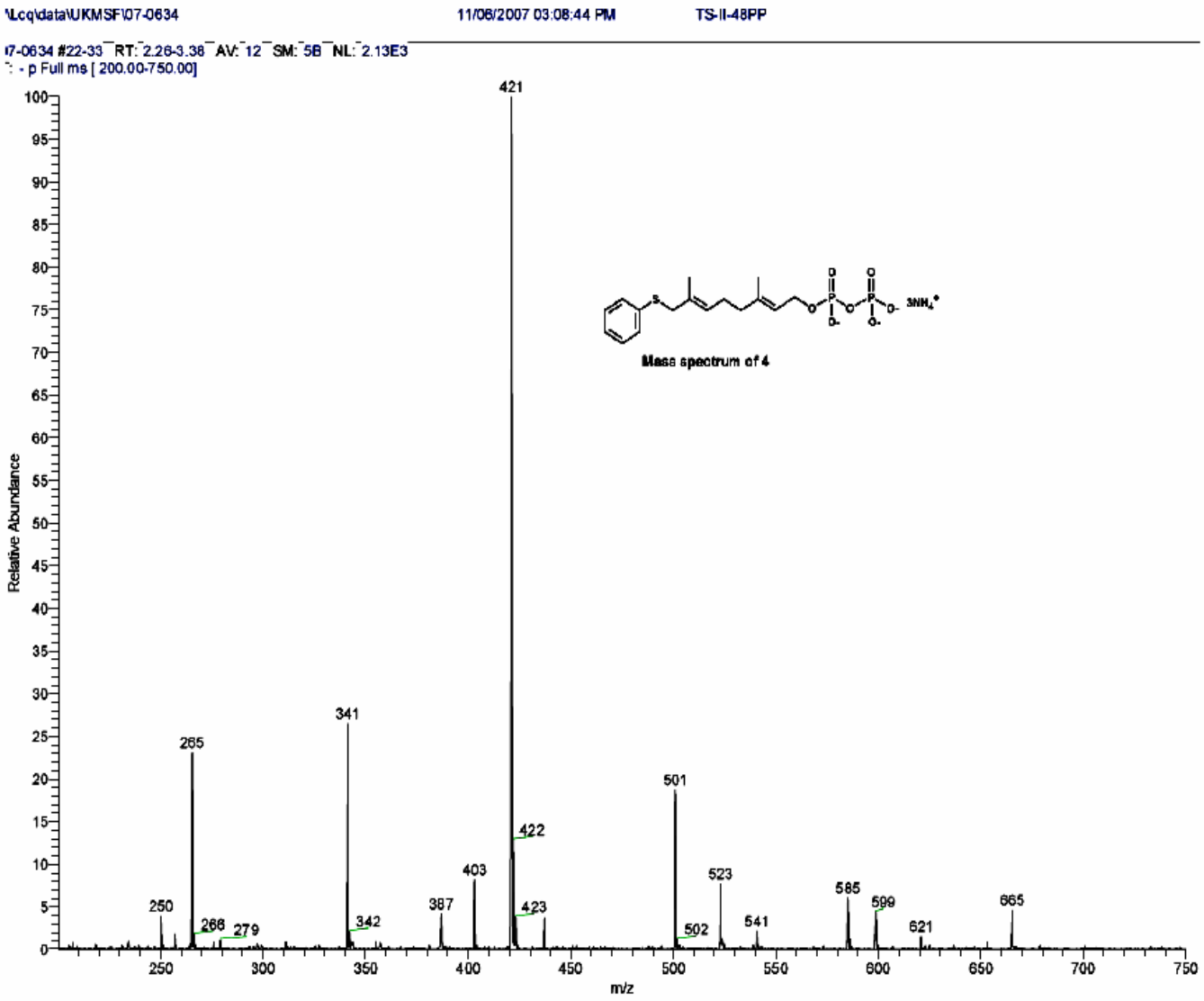
```
exp1  s2pul

      SAMPLE               DEC. & VT
date   May 23 2008   dfrq      399.724
solvent        D2O   dn             H1
file           exp   dpwr           42
   ACQUISITION       dof             0
sfrq       161.811   dm            yyy
tn             P31   dmm             w
at           0.800   dmf         11148
np           16190   dseq
sw         10001.0   dres          1.0
fb            6100   homo            n
bs              64      PROCESSING
tpwr            55   lb           1.00
pw            13.0   wtfile
d1               0   proc           ft
tof              0   fn       not used
nt            3200   math            f
ct             303
alock            n   werr
gain      not used   wexp
      FLAGS          wbs
il               n   wnt
in               n
dp               y
hs              nn
     DISPLAY
sp         -4547.2
wp         10003.0
vs              73
sc               0
wc             250
hzmm         20.00
is          364.60
rfl         4547.2
rfp              0
th              20
ins        100.000
nm  no  ph
```



Phosphorus NMR of **4**



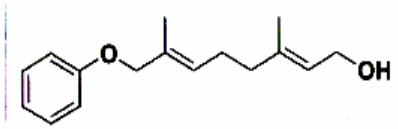


Mass spectrum of 4

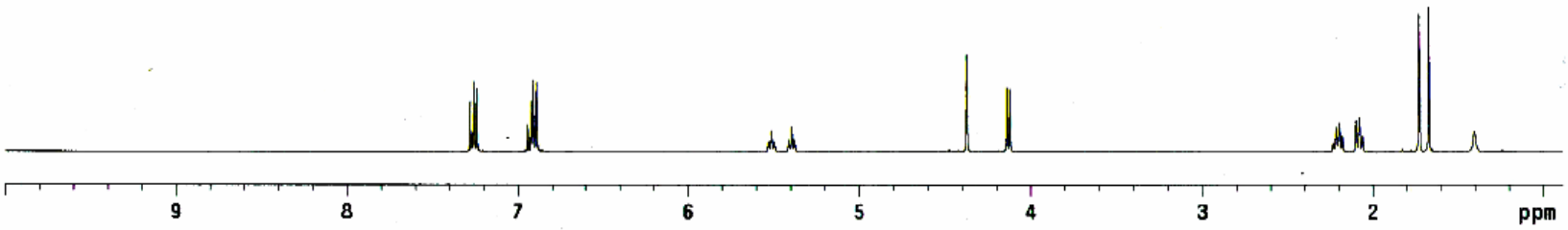
TS-II-23-sept4-2004

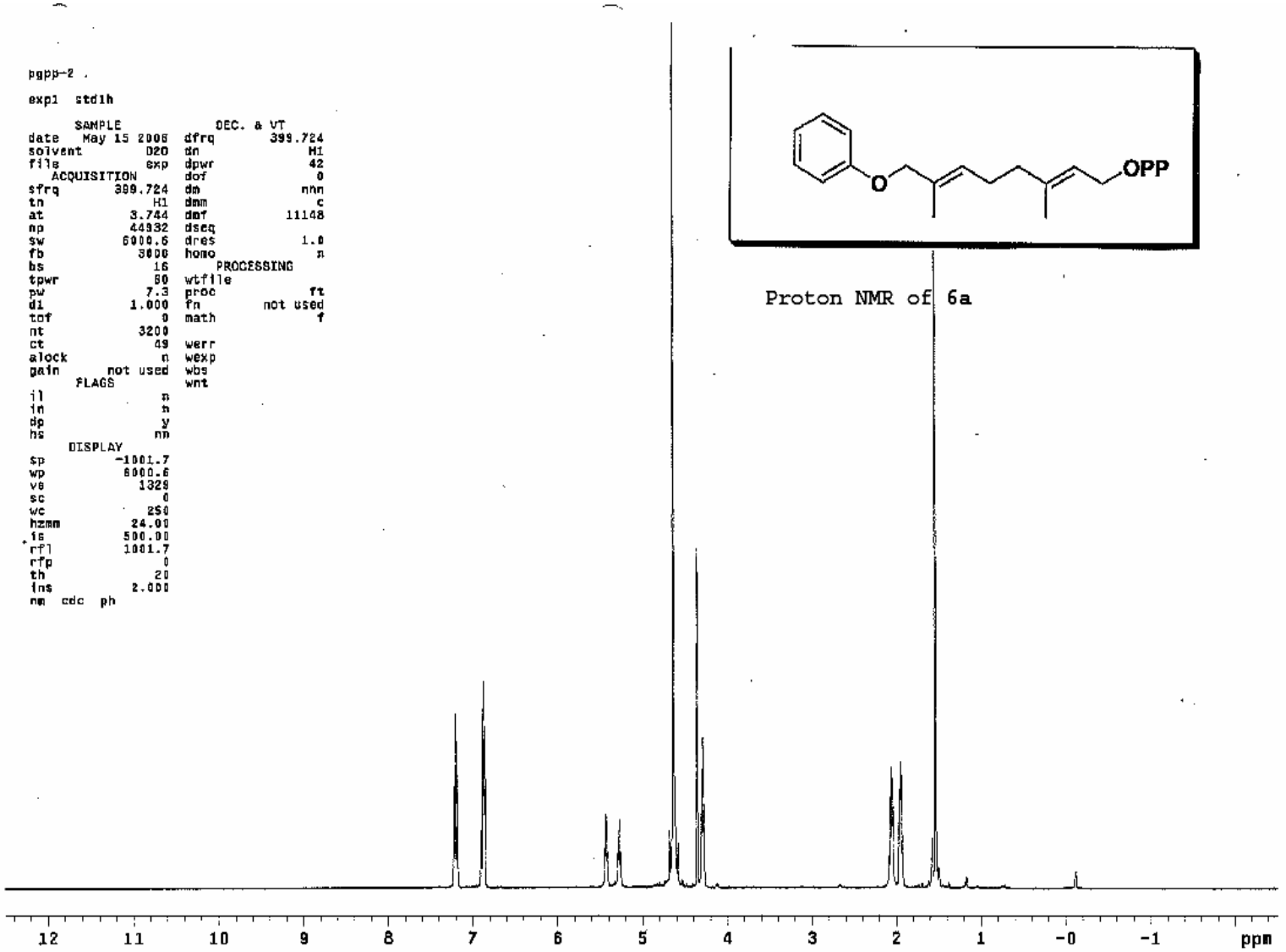
expl std1h

SAMPLE		DEC. & VT	
date	Sep 4 2004	dfrq	399.723
solvent	CDCl3	dn	H1
file	/opt/dist/dat~	dpwr	42
	a/pete/SUBBU/TS-II~	dof	0
	-23-Sept4-2004.fid	dm	nnn
ACQUISITION		dmm	c
sfrq	399.723	dmf	11148
tn	H1	dseq	
at	3.744	dres	1.0
np	44932	homo	n
sw	6000.6	DEC2	
fb	3000	dfrq2	0
bs	16	dn2	
tpwr	60	dpwr2	1
pw	7.3	dof2	0
d1	1.000	dm2	n
tof	0	dmm2	c
nt	32	dmf2	200
ct	32	dseq2	
alock	n	dres2	1.0
gain	not used	homo2	n
FLAGS		PROCESSING	
il	n	wtfile	
in	n	proc	ft
dp	y	fn	not used
hs	nn	math	f
DISPLAY			
sp	354.5	werr	
wp	3642.7	wexp	
vs	22	wbs	
sc	0	wnt	
wc	250		
hzmm	14.57		
is	212.40		
rfl	1001.7		
rfp	0		
th	10		
ins	2.000		
nm	cdc ph		



Proton NMR of 19c

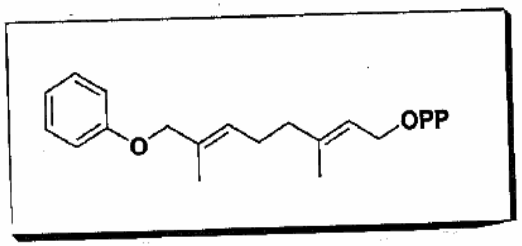



pgpp-2
expl stdlh
SAMPLE
date May 15 2006
solvent D2O
file exp
ACQUISITION
sfrq 399.724
tn H1
at 3.744
np 44932
sw 6000.6
fb 3000
bs 16
tpwr 60
pw 7.3
dl 1.000
tof 0
nt 3200
ct 49
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1001.7
wp 6000.6
vs 1329
sc 0
wc 250
hzmm 24.00
is 500.00
rfl 1001.7
rfp 0
th 20
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.724
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
O
OPP
Proton NMR of 6a
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

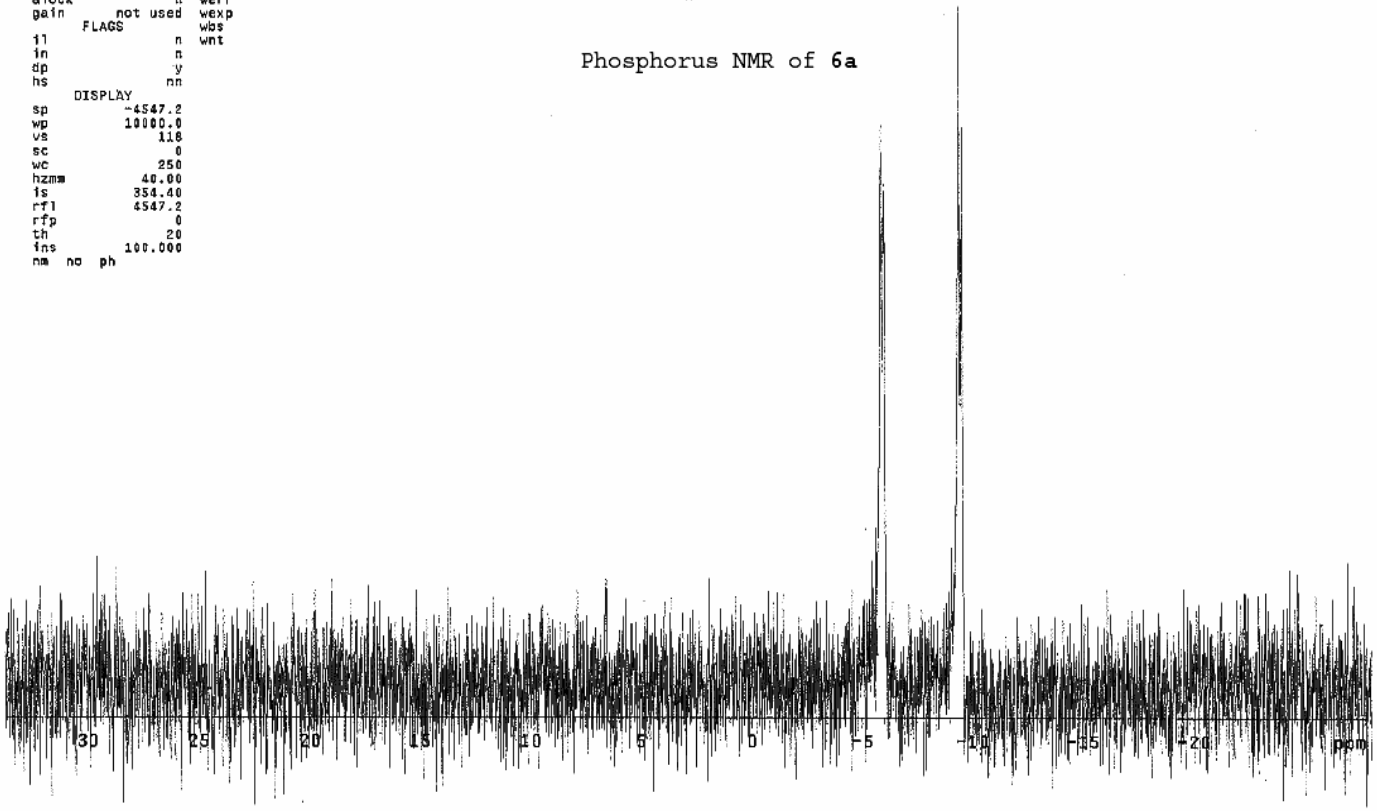
```
pgpp-2

exp1  s2pul

      SAMPLE                    DEC. & VT
date    May 15 2006   dfrq          399.724
solvent         D2O   dn               H1
file            exp   dpwr               42
    ACQUISITION       dof                 0
sfrq        161.811   dm                yyy
tn              P31   dmm                 w
at            0.800   dmf             11148
np            16000   dseq
sw          10000.0   dres              1.0
fb             8000   homo                n
bs               64       PROCESSING
tpwr             55   lb                1.00
pw             10.0   wtfile
d1                0   proc                ft
tof               0   fn            not used
nt             3200   math                f
ct              128
alock             n   werr
gain      not used    wexp
      FLAGS           wbs
i1                n   wnt
in                n
dp                y
hs               nn
     DISPLAY
sp           -4547.2
wp           10000.0
vs               118
sc                 0
wc               250
hzmm           40.00
is            354.40
rfl           4547.2
rfp                0
th                20
ins          100.000
nm  no  ph
```

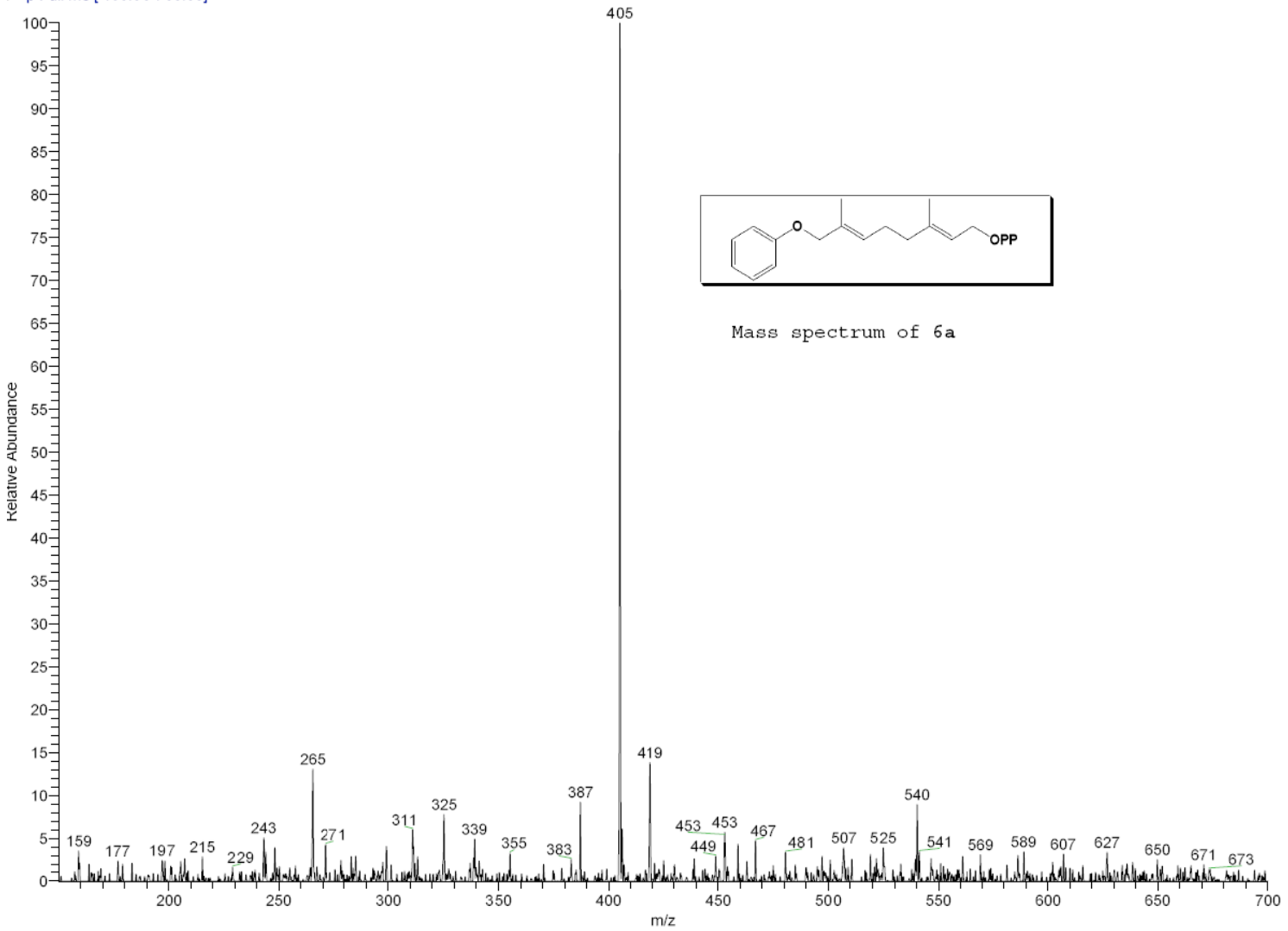


Phosphorus NMR of **6a**

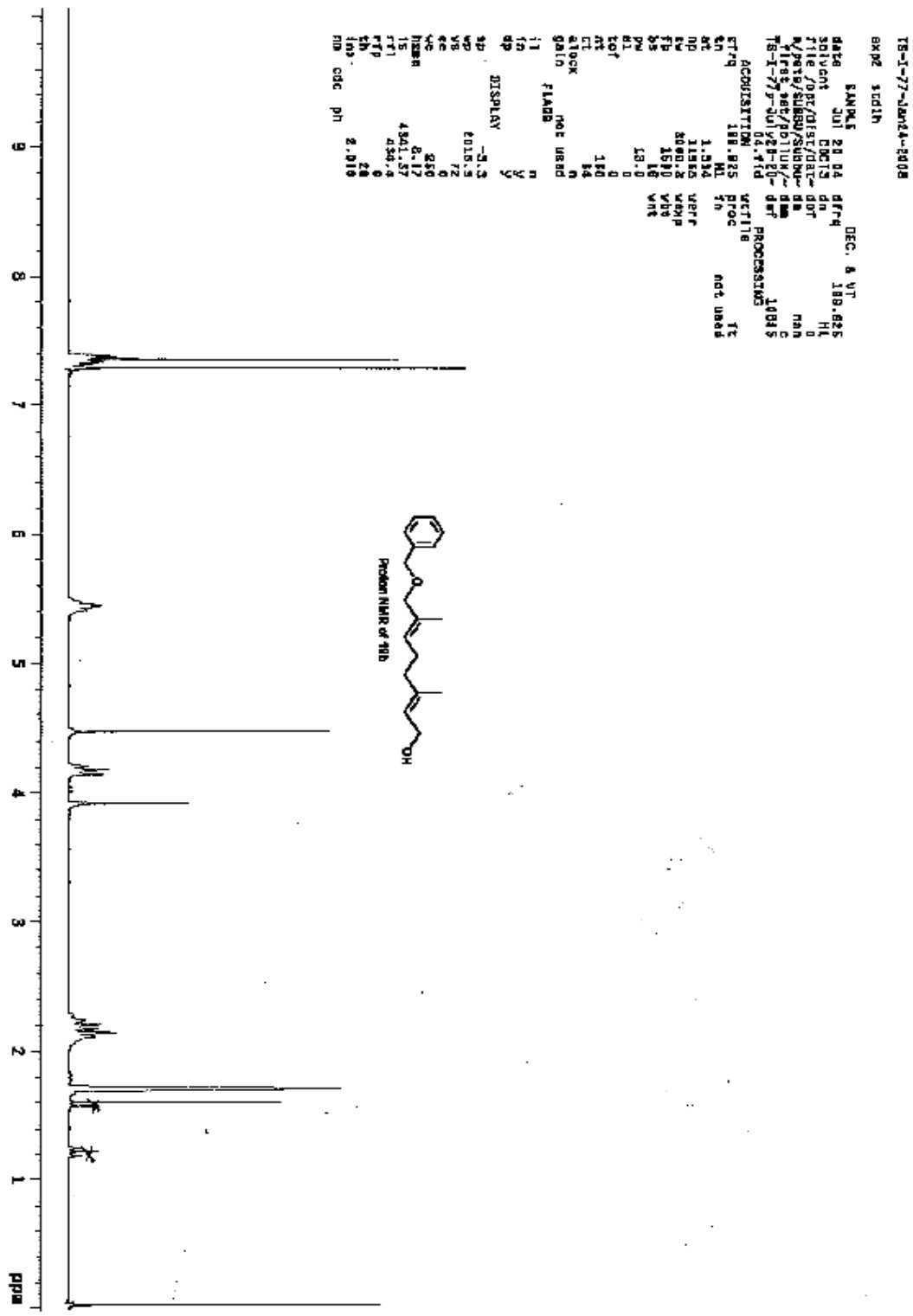


Lcq\data\UKMSF\07-0623 11/06/2007 10:17:27 AM TS-II-28PP

7-0623 #21-29 RT: 2.20-3.02 AV: 9 SM: 5B NL: 3.32E2
: - p Full ms [150.00-700.00]



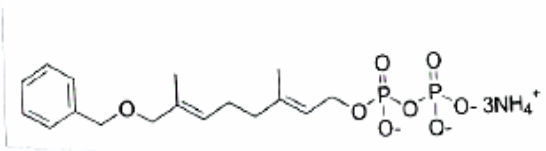
Mass spectrum of 6a


Proton NMR of 10b
OH
ppm

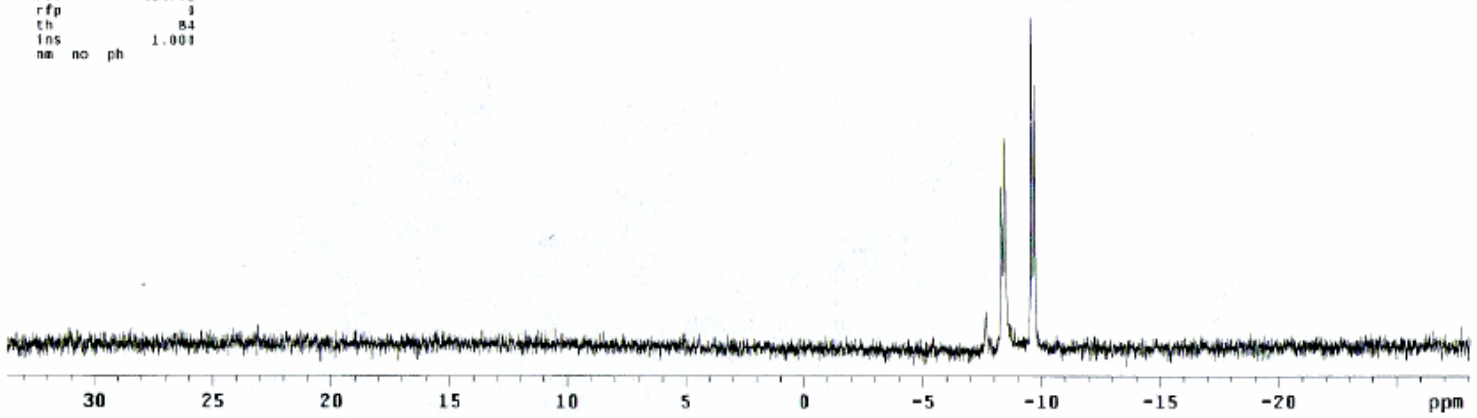
TS-benzyloxyOPP

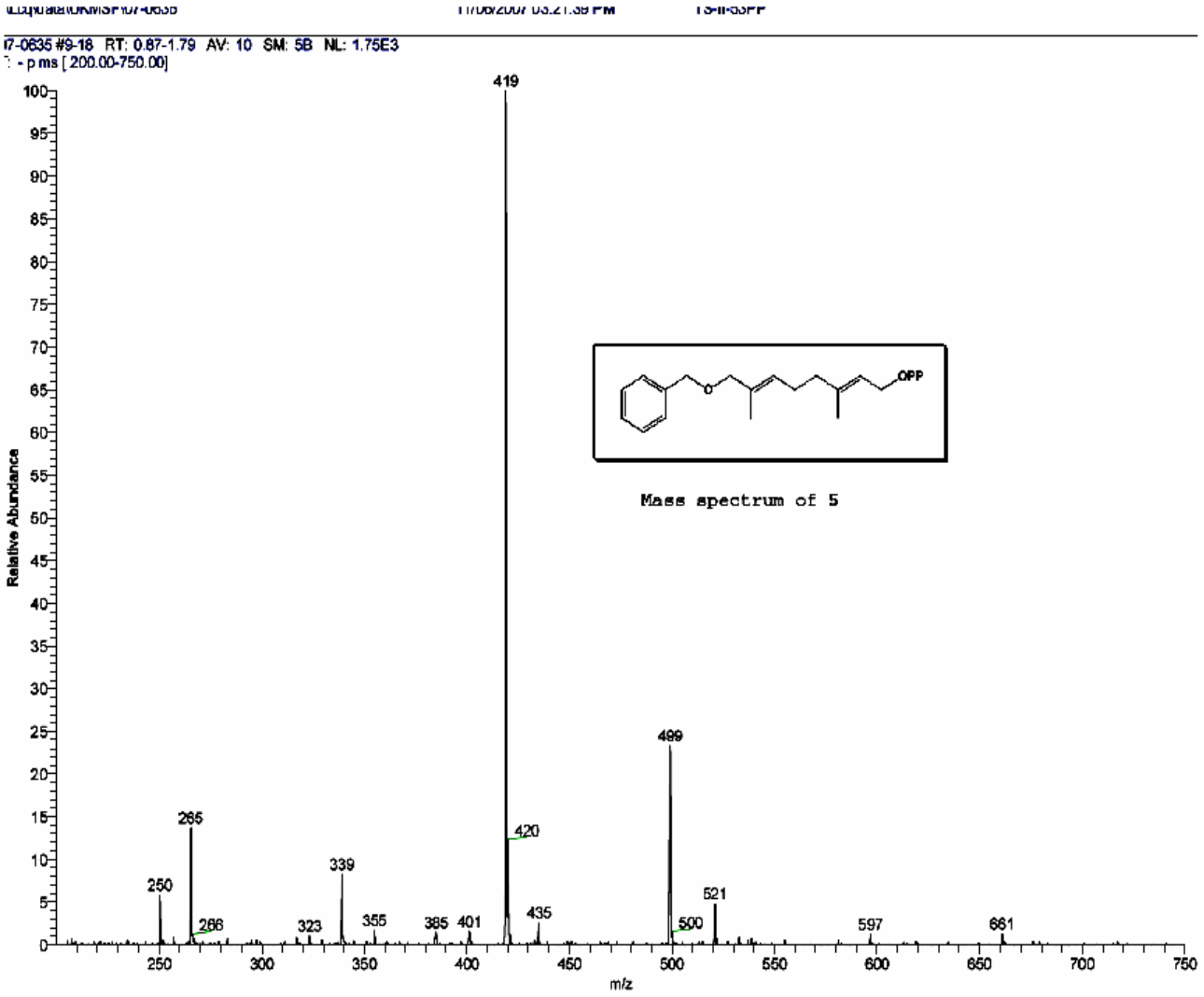
expl sZpul

SAMPLE		DEC. & VT	
date	Oct 9 2004	dfrq	399.724
solvent	D2O	dn	H1
file	/opt/dist/dat~	dpwr	42
a/pete/SUBBU/Benzy~		dof	0
loxyOPyrophophate.~		dm	yyy
	fid	dmm	w
ACQUISITION		dmf	11148
sfrq	161.811	dseq	
tn	P31	dres	1.0
at	0.804	homo	n
np	16008	DEC2	
sw	10000.0	dfrq2	undefined
fb	6000	dn2	undefined
bs	64	dpwr2	undefined
tpwr	55	dof2	undefined
pw	10.0	dm2	undefined
d1	0	dmm2	undefined
tof	0	dmf2	undefined
nt	160	dseq2	undefined
ct	160	dres2	undefined
alock	n	homo2	undefined
gain	not used	PROCESSING	
FLAGS		lb	1.00
fl	n	wtfile	
in	n	proc	ft
dp	y	fn	not used
hs	nn	math	f
DISPLAY			
sp	-4546.8	werr	
wp	9998.8	wexp	
vs	53	wbs	
sc	0	wnt	
wc	250		
hzmm	40.00		
is	13764.37		
rfl	4547.2		
rfp	1		
th	84		
ins	1.000		
nm no ph			



Phosphorus NMR of 5



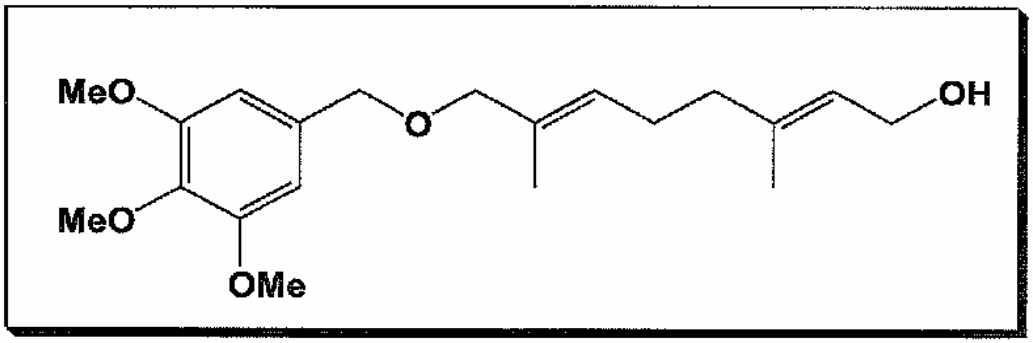


Mass spectrum of 5

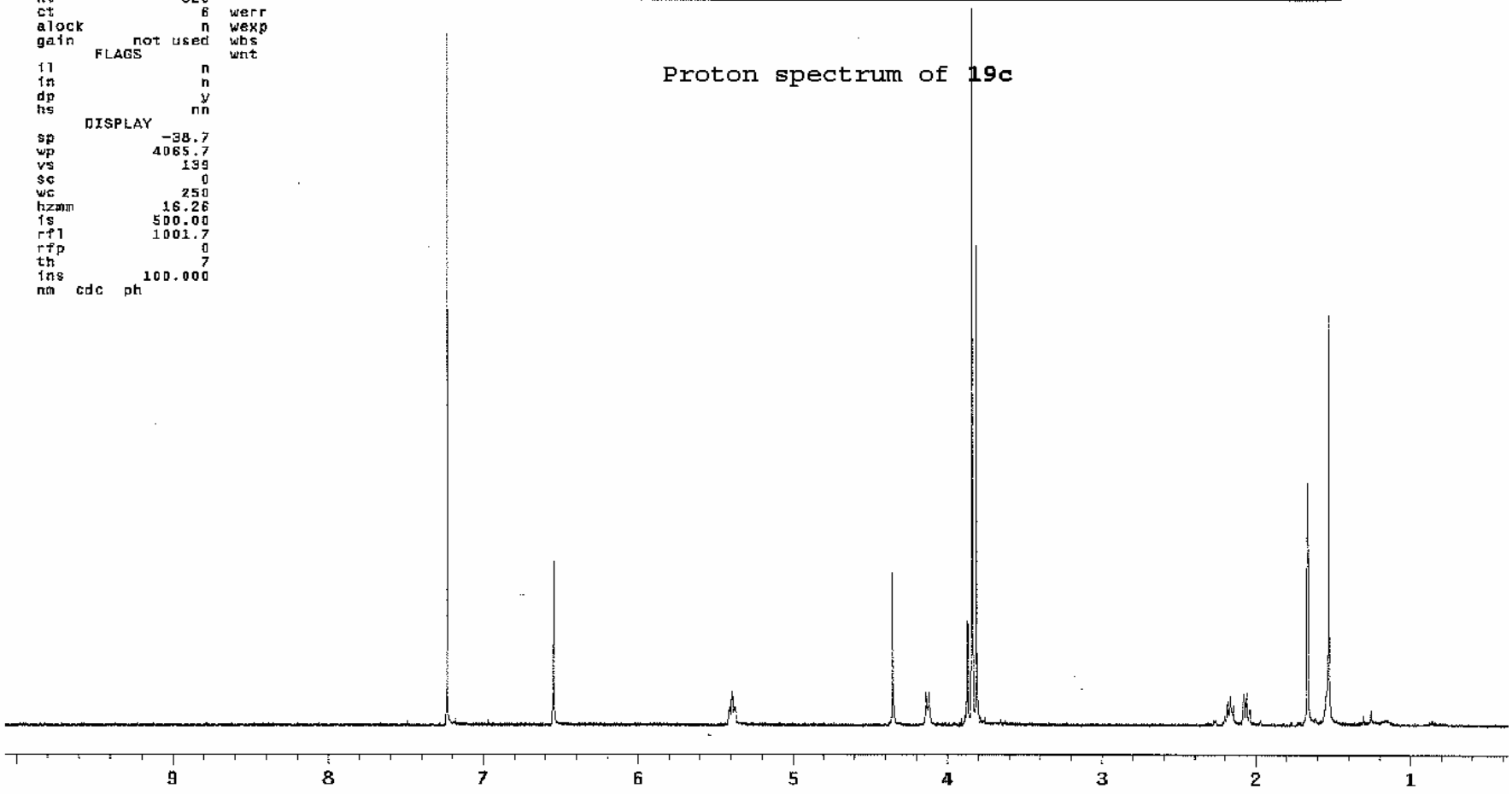
TS-TrimethoxyPGOH

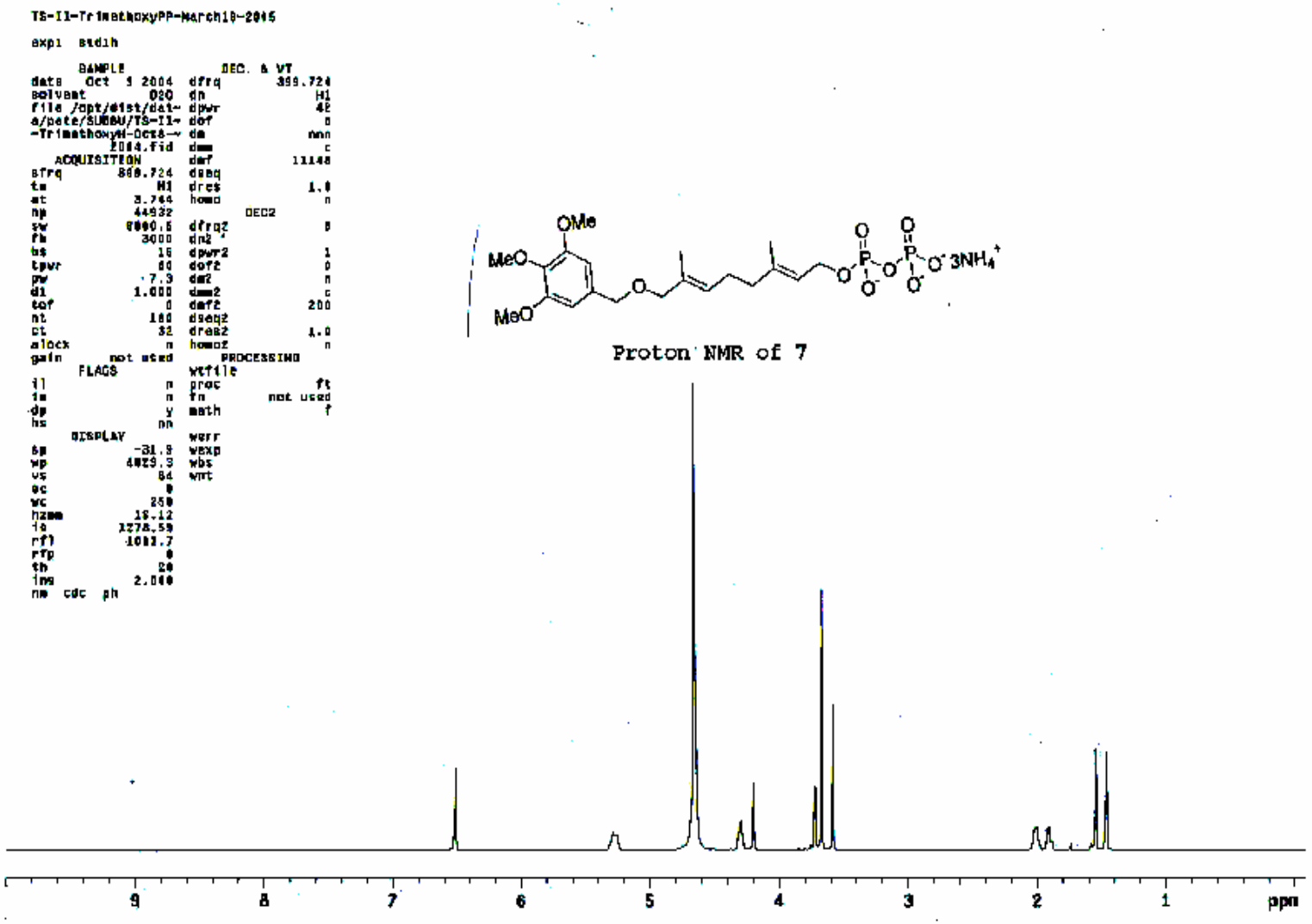
expi stdih

SAMPLE		DEC. & VT	
date	Nov 14 2007	dfrq	399.717
solvent	CDCl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	399.717	dm	nnn
tn	H1	dmm	c
at	3.744	dmf	11148
np	44932	dseq	
sw	6000.6	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	60	wtfile	
pw	7.3	proc	ft
dl	1.000	fn	not used
tof	0	math	f
nt	320		
ct	6	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-38.7		
wp	4065.7		
vs	139		
sc	0		
wc	250		
hzmm	16.26		
is	500.00		
rfl	1001.7		
rfp	0		
th	7		
ins	100.000		
nm cdc ph			



Proton spectrum of 19c



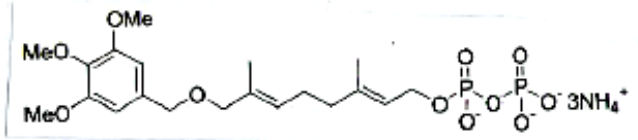

TS-II-TrimethoxyPP-March10-2015
expl stdih
SAMPLE
date Oct 1 2004
Solvent D2O
file /opt/dist/dat~
a/pete/SUBBU/TS-II~
-TrimethoxyH-Oct4-~
2004.fid
ACQUISITION
sfrq 399.724
tn H1
at 3.744
np 44932
sw 6000.5
fb 3000
bs 16
tpwr 60
pw 7.3
d1 1.000
tof 0
nt 160
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -31.8
wp 4829.3
vs 64
sc 0
wc 250
hzmm 19.12
is 1278.59
rfl 1081.7
rfp 0
th 20
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.724
dn H1
dpwr 48
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
OMe
MeO
MeO
3NH4+
Proton NMR of 7
9
8
7
6
5
4
3
2
1
ppm

TS-II-TrimethoxyPP-March19-2005

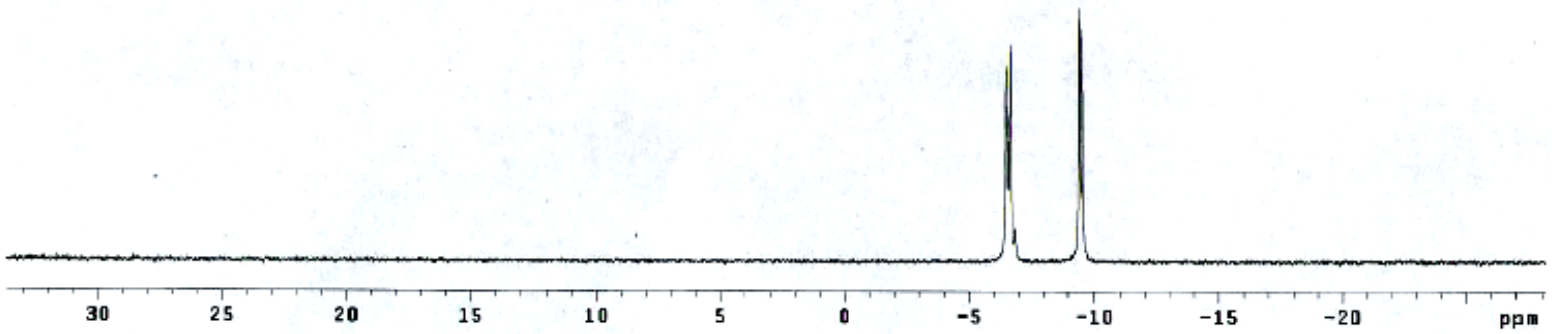
expl stZpul

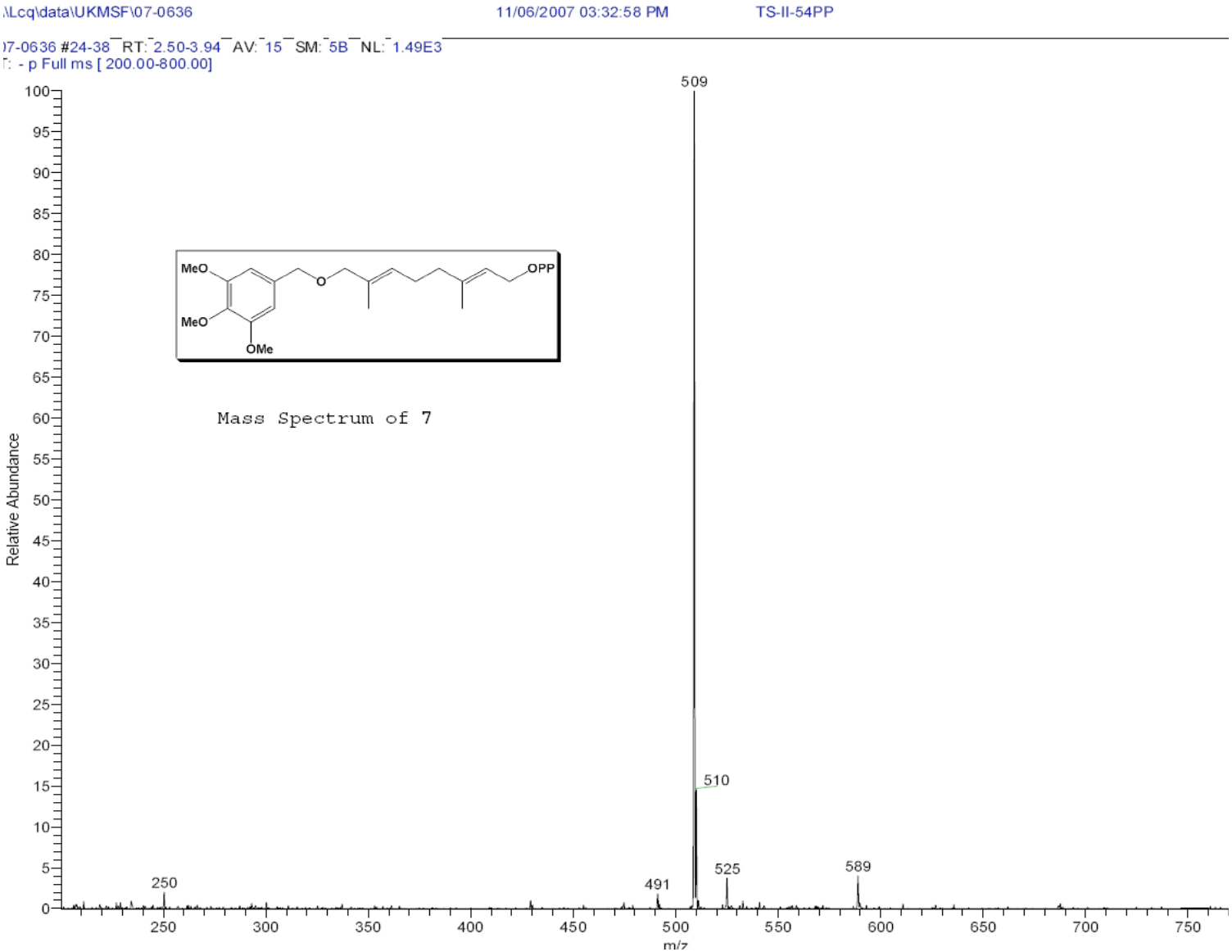
SAMPLE		DEC. & VT	
date	Oct 9 2004	dfrq	399.724
solvent	D2O	dn	H1
file /opt/dist/dat~		dpwr	42
a/pete/SUBBU/TS-II~		dof	0
-Trimethoxy-DPP.fi~		dm	yyy
	d	dmm	w
ACQUISITION		dmf	11148
sfrq	161.811	dseq	
tn	P31	dres	1.0
at	1.000	homo	n
np	16000	DEC2	
sw	10000.0	dfrq2	undefined
fb	6000	dn2	undefined
bs	64	dpwr2	undefined
tpwr	55	dof2	undefined
pw	10.0	dm2	undefined
dl	0	dmm2	undefined
tof	0	dmf2	undefined
nt	160	dseq2	undefined
ct	160	dres2	undefined
alock	n	homo2	undefined
gain	not used	PROCESSING	
FLAGS		lb	1.00
il	n	wtfile	
in	n	proc	ft
dp	y	fn	not used
hs	nn	math	f
DISPLAY			
sp	-4546.0	werr	
wp	9998.8	wexp	
vs	42	wbs	
sc	0	wnt	
wc	250		
hzmm	40.00		
is	4617.09		
rfl	4547.2		
rfp	0		
th	15		
ins	1.000		

nm no ph



Phosphorus NMR of 7



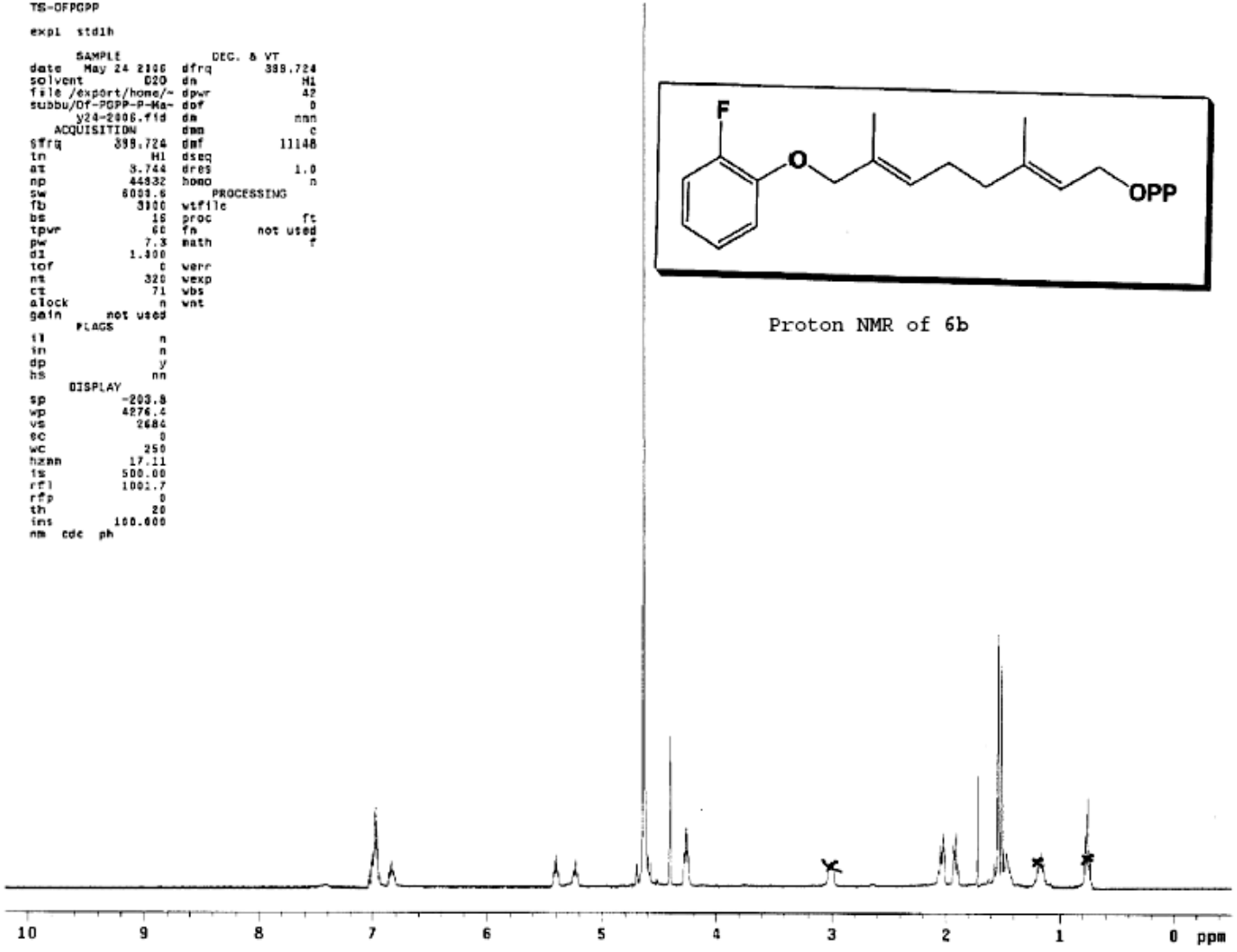

\Lcq\data\UKMSF\07-0636
11/06/2007 03:32:58 PM
TS-II-54PP
07-0636 #24-38 RT: 2.50-3.94 AV: 15 SM: 5B NL: 1.49E3
T: - p Full ms [200.00-800.00]
MeO
MeO
OMe
O
OPP
Mass Spectrum of 7
Relative Abundance
m/z
509
510
525
589
491
250

TS-OFPOPP

expl stdlh

SAMPLE		DEC. & VT	
date	May 24 2006	dfrq	399.724
solvent	D2O	dn	H1
file	/export/home/~	dpwr	42
subbu/Of-POPP-P-Ma-		dof	0
	y24-2006.fid	dm	nnn
ACQUISITION		dmm	c
sfrq	399.724	dmf	11148
tn	H1	dseq	
at	3.744	dres	1.0
np	44832	homo	n
sw	6009.6	PROCESSING	
fb	3100	wtfile	
bs	16	proc	ft
tpwr	60	fn	not used
pw	7.3	math	f
d1	1.300		
tof	0	werr	
nt	320	wexp	
ct	71	wbs	
alock	n	wnt	
gain	not used		
FLAGS			
fl	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-203.8		
wp	4276.4		
vs	2684		
sc	0		
wc	250		
hzmm	17.11		
is	500.00		
rfl	1001.7		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			

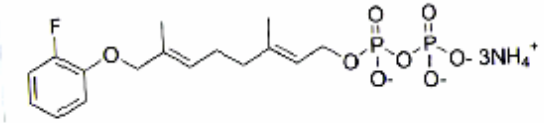
Proton NMR of 6b



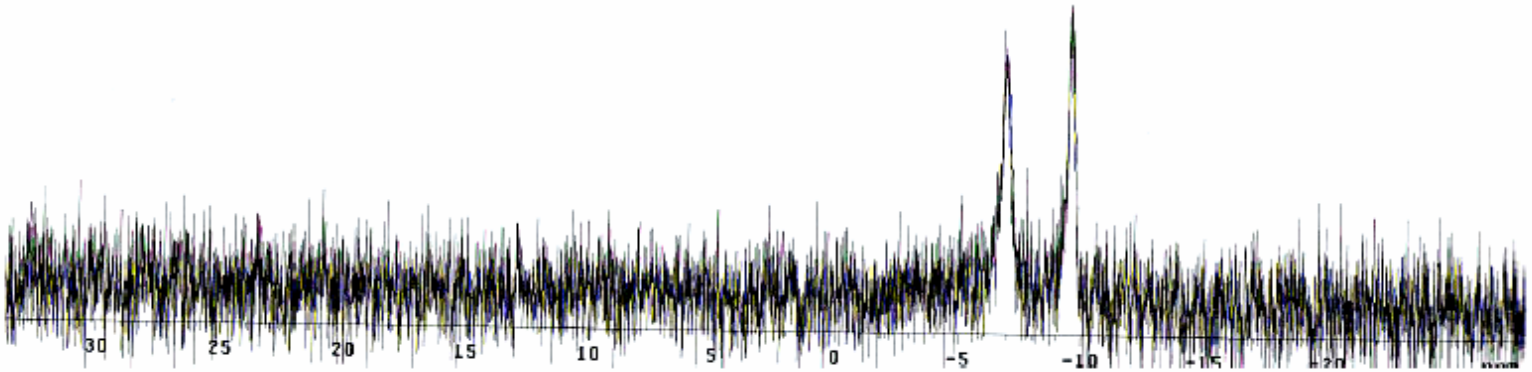
TS-O-FPGPP

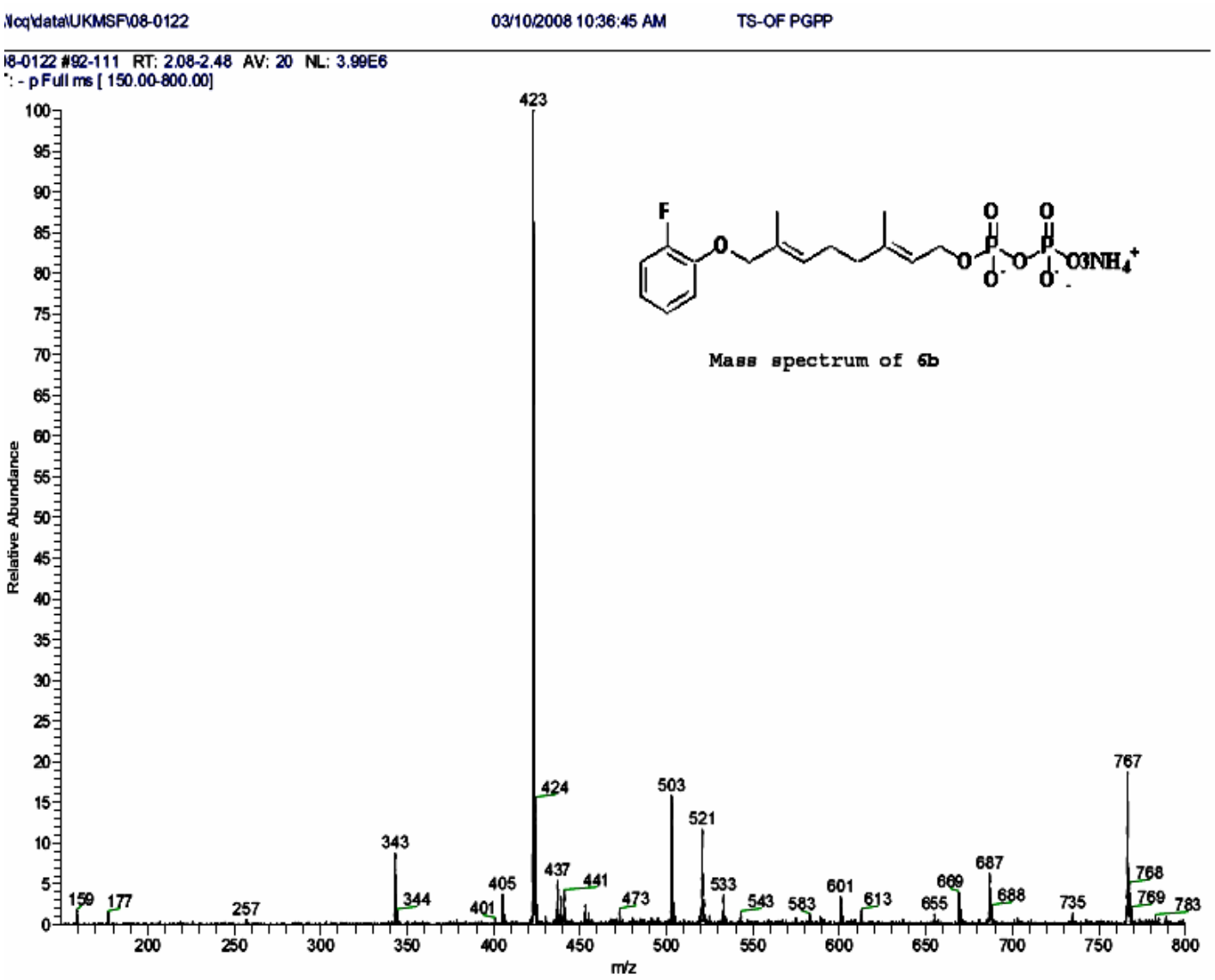
expl s2pul

SAMPLE		DEC. & VT	
date	May 24 2006	dfrq	399.724
solvent	D2O	dn	H1
file	/opt/dlst/dat~	dpwr	42
a/pete/SUBBU/O-F-P~		dof	0
OPP-May24-2006.fid		dm	yyy
ACQUISITION		dmm	w
sfrq	161.811	dmf	11148
tn	P31	dseq	
at	0.680	dres	1.0
np	16080	homo	n
sw	10000.0	DEC2	
fb	6000	dfrq2	undefined
bs	64	dn2	undefined
tpwr	55	dpwr2	undefined
pw	10.4	dof2	undefined
d1	0	dm2	undefined
tof	0	dmm2	undefined
nt	3200	dmf2	undefined
ct	994	dseq2	undefined
alock	n	dres2	undefined
gain	not used	homo2	undefined
FLAGS		PROCESSING	
fl	n	lb	1.00
in	n	wtfile	
dp	y	proc	ft
hs	nn	fn	not used
DISPLAY		math	f
sp	-4546.0	werr	
wp	9998.8	wexp	
vs	50	wbs	
sc	0	wnt	
wc	250		
hzmm	40.00		
is	354.40		
rfl	4547.2		
rfp	0		
th	76		
ins	100.000		
nm no ph			

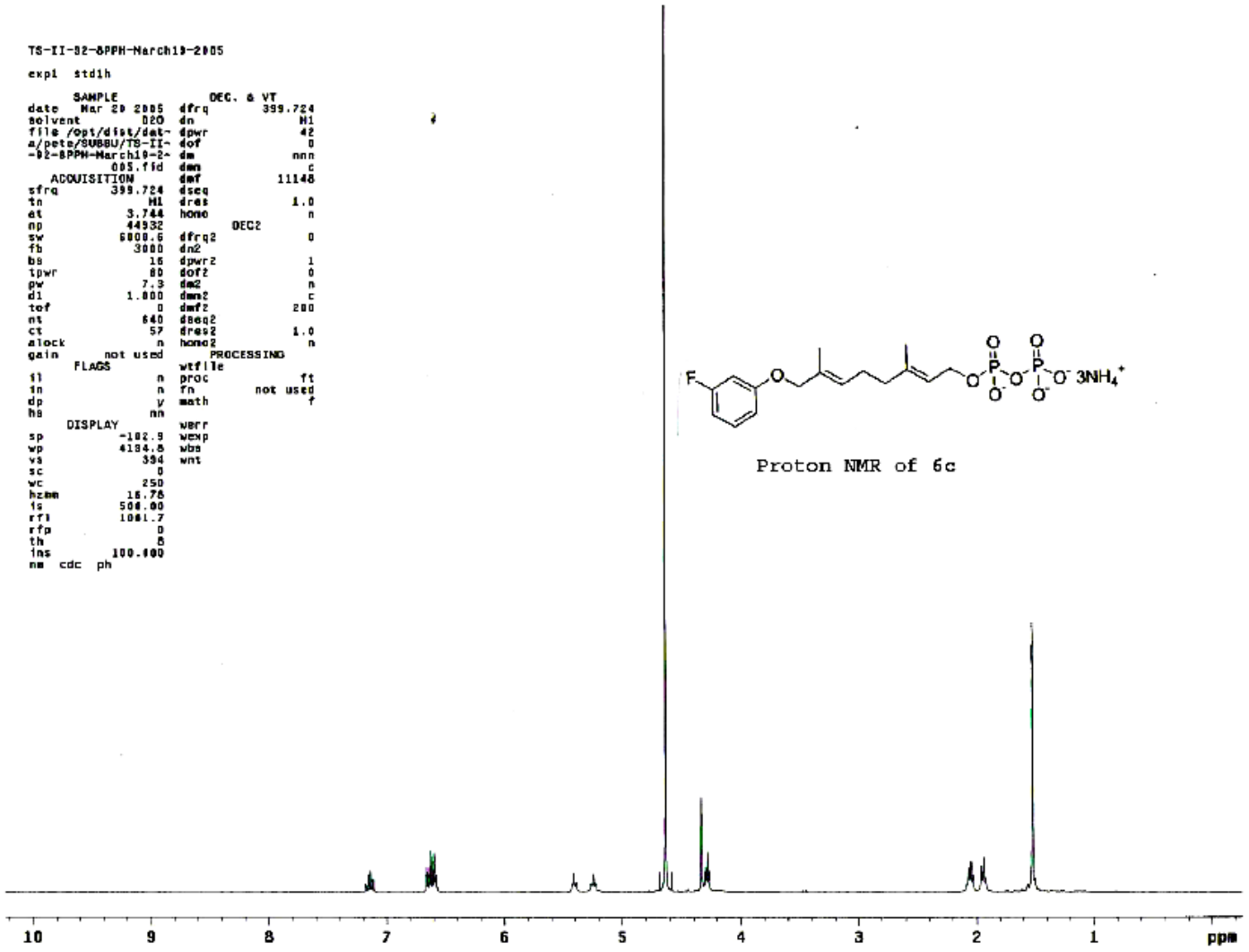


Phosphorus NMR of **6b**





Mass spectrum of 6b

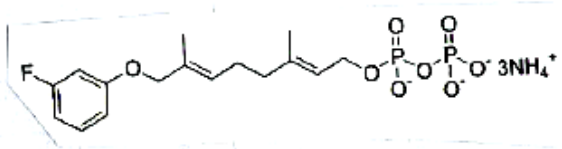


Proton NMR of 6c

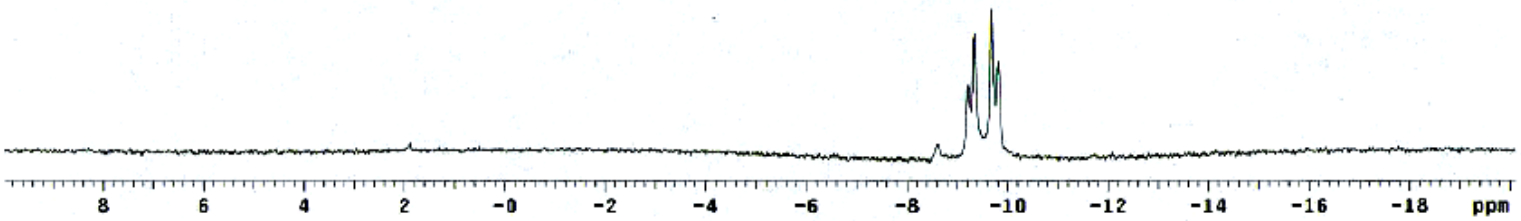
TS-II-82-6PPH-March19-2005

expl s2pul

SAMPLE		DEC. & VT	
date	Mar 20 2005	dfrq	399.724
solvent	D2O	dn	H1
file	/opt/dist/dat~	dpwr	42
a/pete/SUBBU/TS-II~		dof	0
-82-6PP-March19-20~		dm	yyy
	05.fid	dmm	w
ACQUISITION		dmf	11148
sfrq	161.811	dseq	
tn	P31	dres	1.0
at	0.800	homo	n
np	16000	DEC2	
sw	10000.0	dfrq2	undefined
fb	6000	dn2	undefined
bs	64	dpwr2	undefined
tpwr	55	dof2	undefined
pw	10.8	dm2	undefined
d1	0	dmm2	undefined
tof	0	dmf2	undefined
nt	648	dseq2	undefined
ct	234	dres2	undefined
alock	n	homo2	undefined
gain	not used	PROCESSING	
FLAGS		lb	1.00
il	n	wtfile	
in	n	proc	ft
dp	y	fn	not used
hs	nn	math	f
DISPLAY			
sp	-3255.7	werr	
wp	4886.5	wexp	
vs	24	wbs	
sc	0	wnt	
wc	254		
hzmm	18.47		
is	354.4		
rfl	4547.2		
rfp	0		
th	3		
ins	180.000		
nm nc ph			

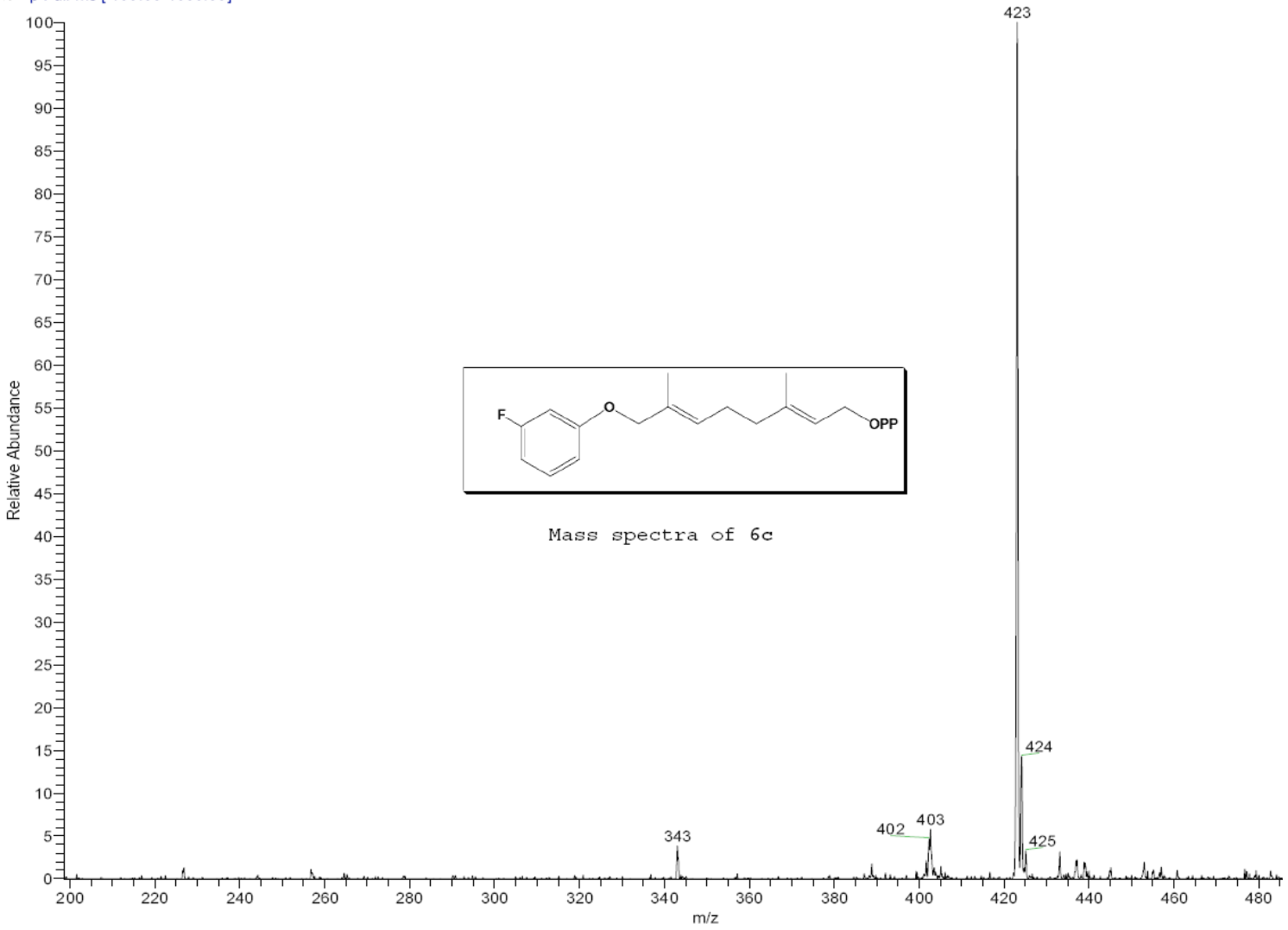


Phosphorus NMR of 6c

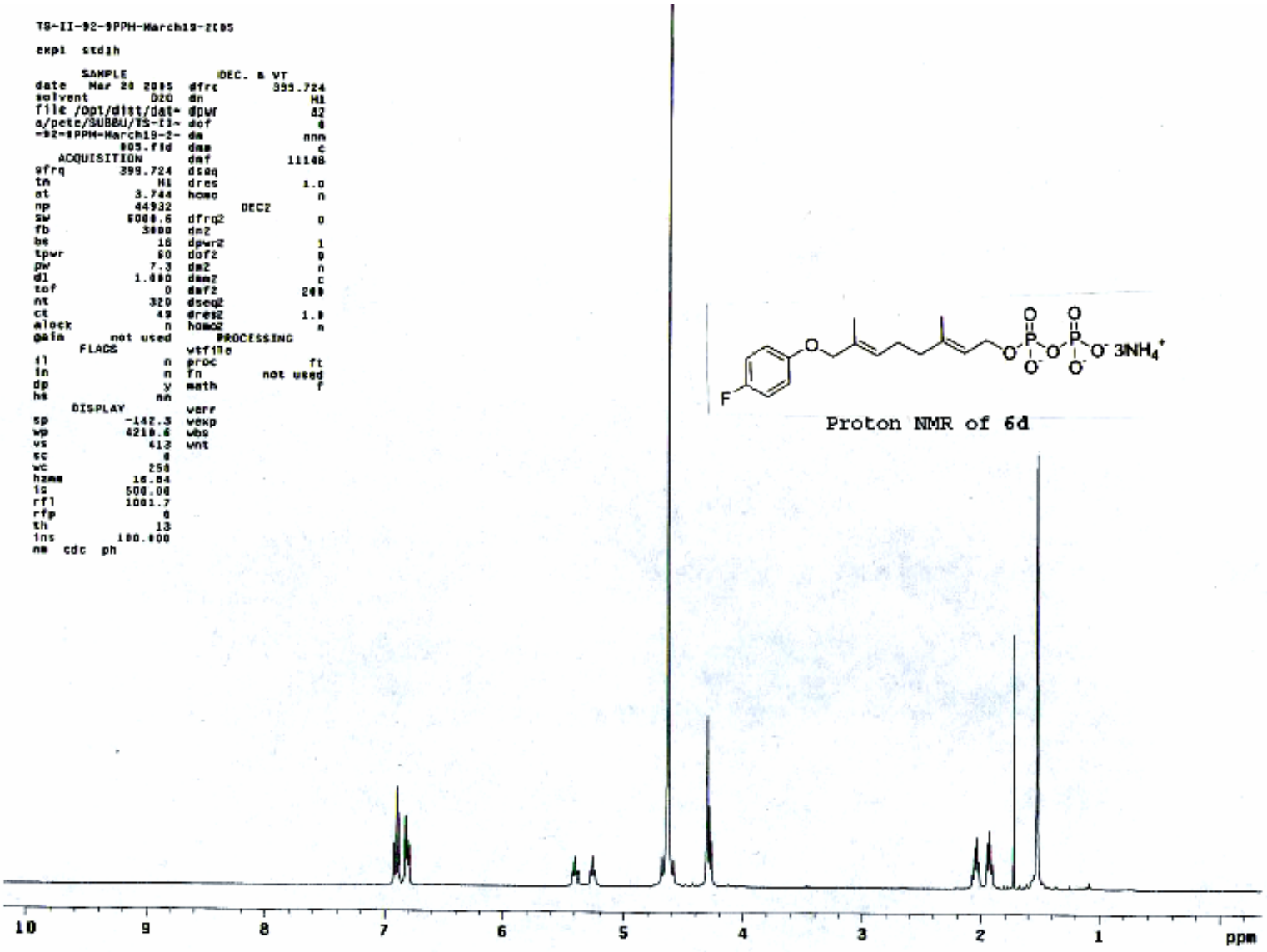


C:\Xcalibur\data\UKMSF\05-0340 03/28/2005 11:22:05 AM TS-II-92-8

05-0340 #23-35 RT: 0.65-1.00 AV: 13 NL: 9.76E4
T: - p Full ms [100.00-1000.00]



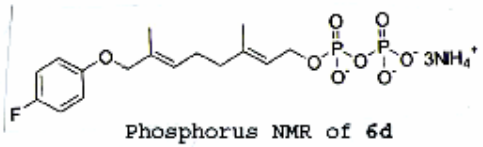
Mass spectra of 6c


TS-II-92-9PPH-March19-2[05
expl stdih
SAMPLE
date Mar 20 2005
solvent D2O
file /Opt/dist/data/pete/SUBBU/TS-II-92-9PPH-March19-2-805.fid
ACQUISITION
sfrq 399.724
tn H1
at 3.744
np 44932
sw 5008.6
fb 3800
bs 16
tpwr 60
pw 7.3
dl 1.000
tof 0
nt 320
ct 48
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -142.3
wp 4210.6
vs 413
sc 0
wc 250
hzmm 16.84
is 500.00
rfl 1001.7
rfp 0
th 13
ins 100.000
nm cdc ph
DEC. & VT
dfrq 399.724
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Proton NMR of 6d
10
9
8
7
6
5
4
3
2
1
ppm

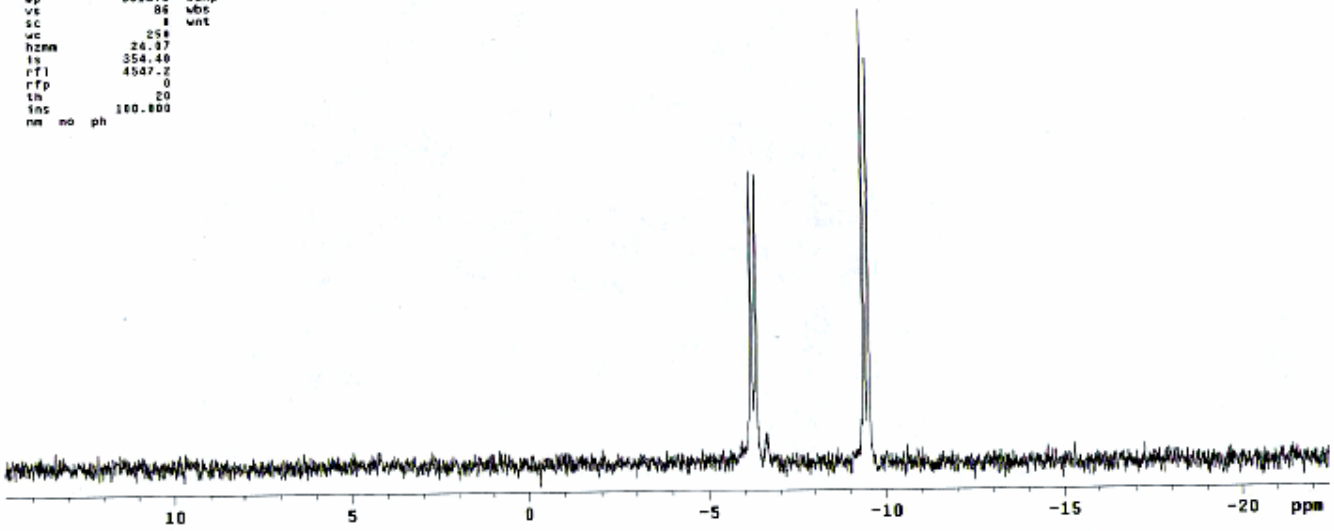
TS-II-92-9PPM-March19-2005

expl s2pul

SAMPLE		DEC. & VT	
date	Mar 21 2005	dfrq	399.724
solvent	D2O	dn	H1
file	/opt/dlsi/dat~	dpwr	42
a/pete/SUBBU/TS-II~		dof	0
-92-9PP-March19-20~		dm	yyy
	05.fid	dmm	w
ACQUISITION		dmf	11145
sfrq	161.811	dseq	
tn	P31	dres	1.0
at	0.808	homo	n
np	16038	DEC2	
sw	10000.0	dfrq2	undefined
fb	6010	dn2	undefined
bs	64	dpwr2	undefined
tpwr	55	dof2	undefined
pw	18.0	dm2	undefined
d1	0	dmm2	undefined
tof	0	dmf2	undefined
nt	640	dseq2	undefined
ct	165	dres2	undefined
alock	n	homo2	undefined
gain	not used	PROCESSING	
FLAGS		lb	1.00
il	n	wtfile	
in	n	proc	ft
dp	y	fn	not used
hs	nn	math	f
DISPLAY		werr	
sp	-3628.1	wexp	
wp	6015.1	wbs	
vs	86	wnt	
sc	0		
wc	250		
hzmm	24.87		
is	354.40		
rfl	4547.2		
rfp	0		
th	20		
ins	100.000		
nm no ph			

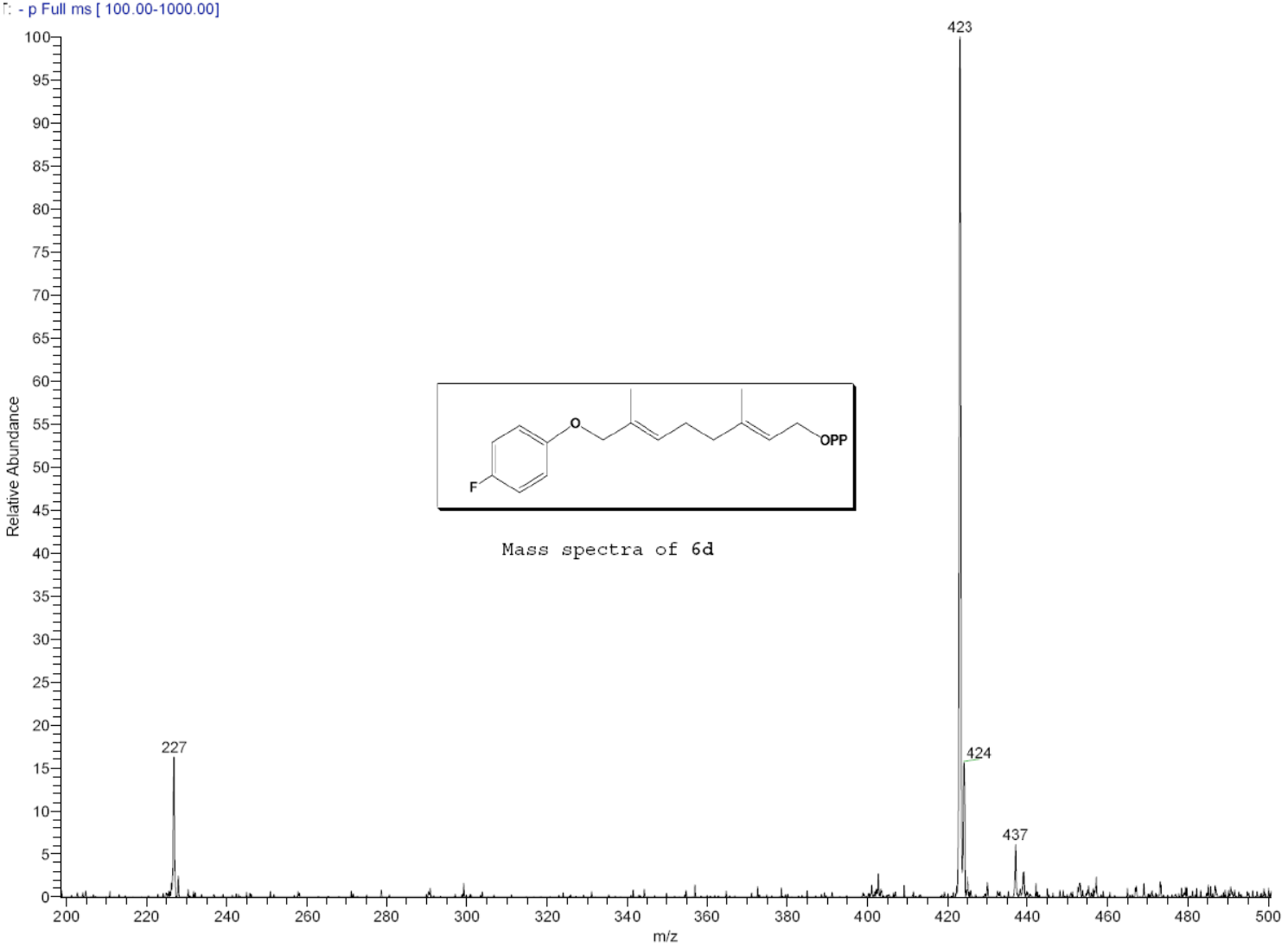


Phosphorus NMR of **6d**

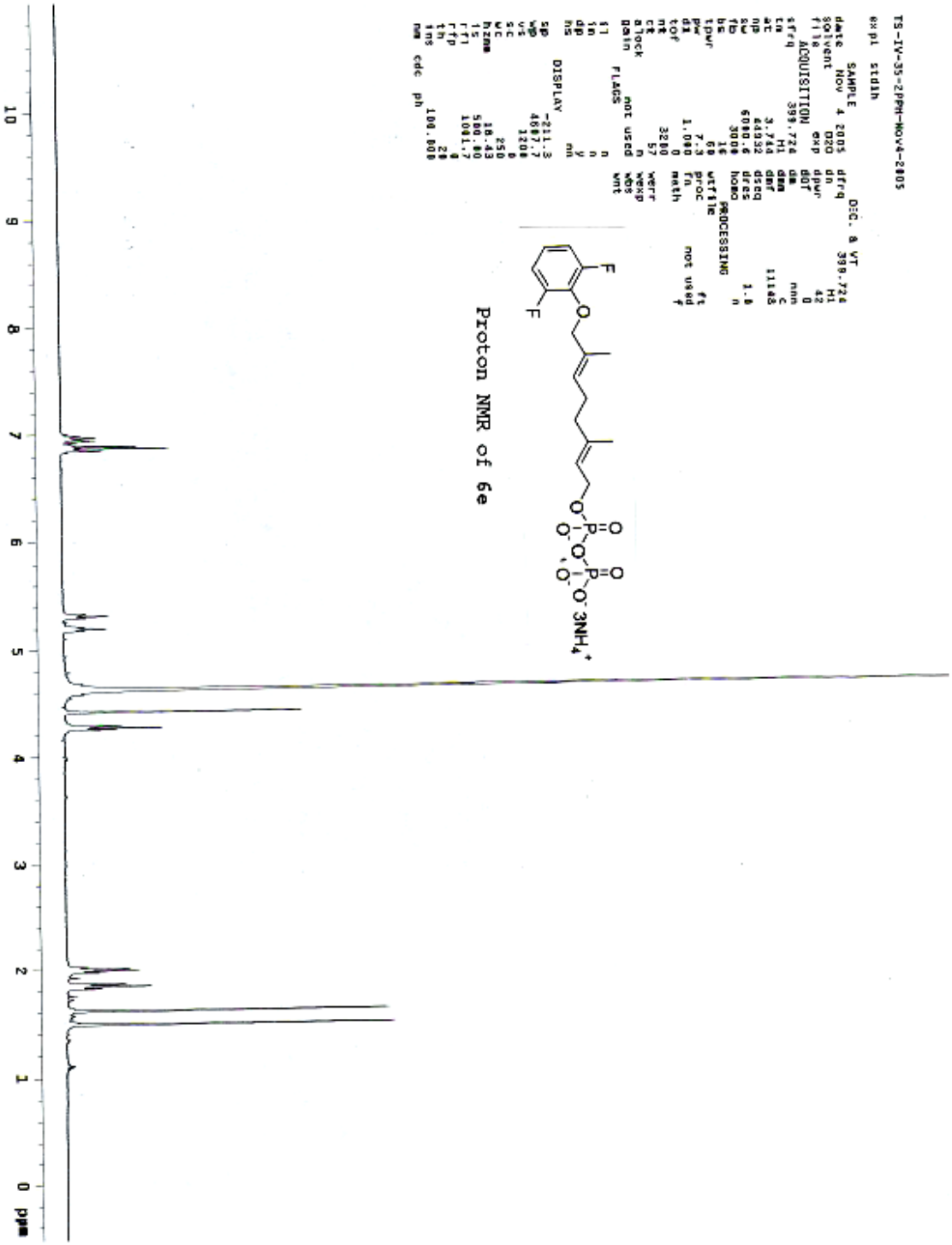


C:\Xcalibur\data\UKMSF\05-0341 03/28/2005 11:33:51 AM TS-II-92-9

05-0341 #25-37 RT: 0.67-1.00 AV: 13 NL: 1.33E5
T: - p Full ms [100.00-1000.00]



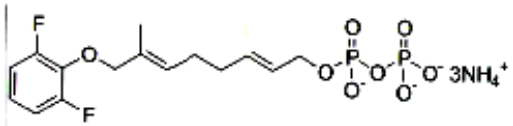
Mass spectra of 6d


TS-IV-35-2PRH-NOV4-2005
Proton NMR of 6e
3NH4+
10
9
8
7
6
5
4
3
2
1
0 ppm

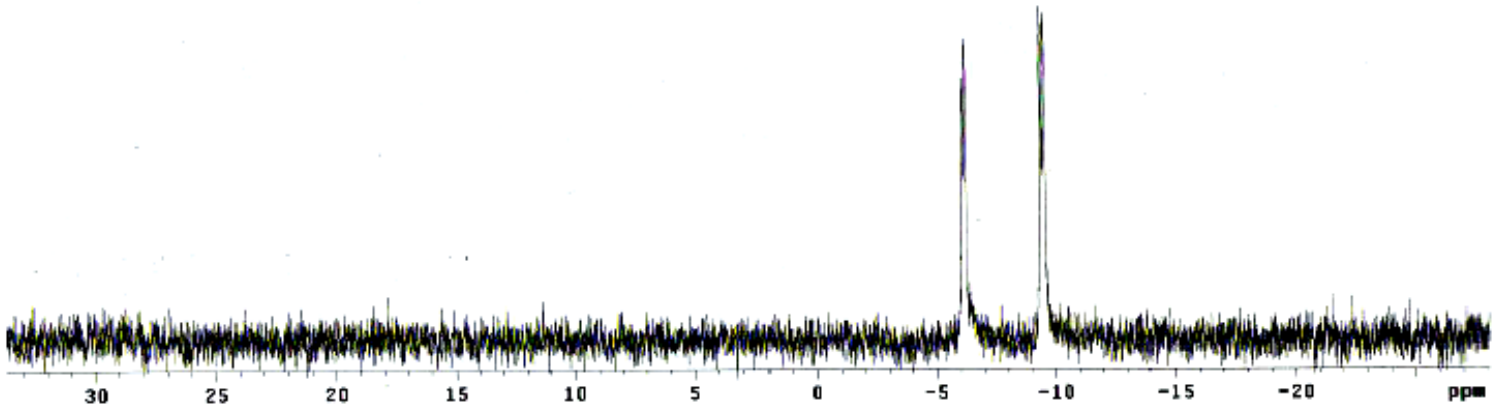
```
TS-IV-35-2PP-Nov4-2005

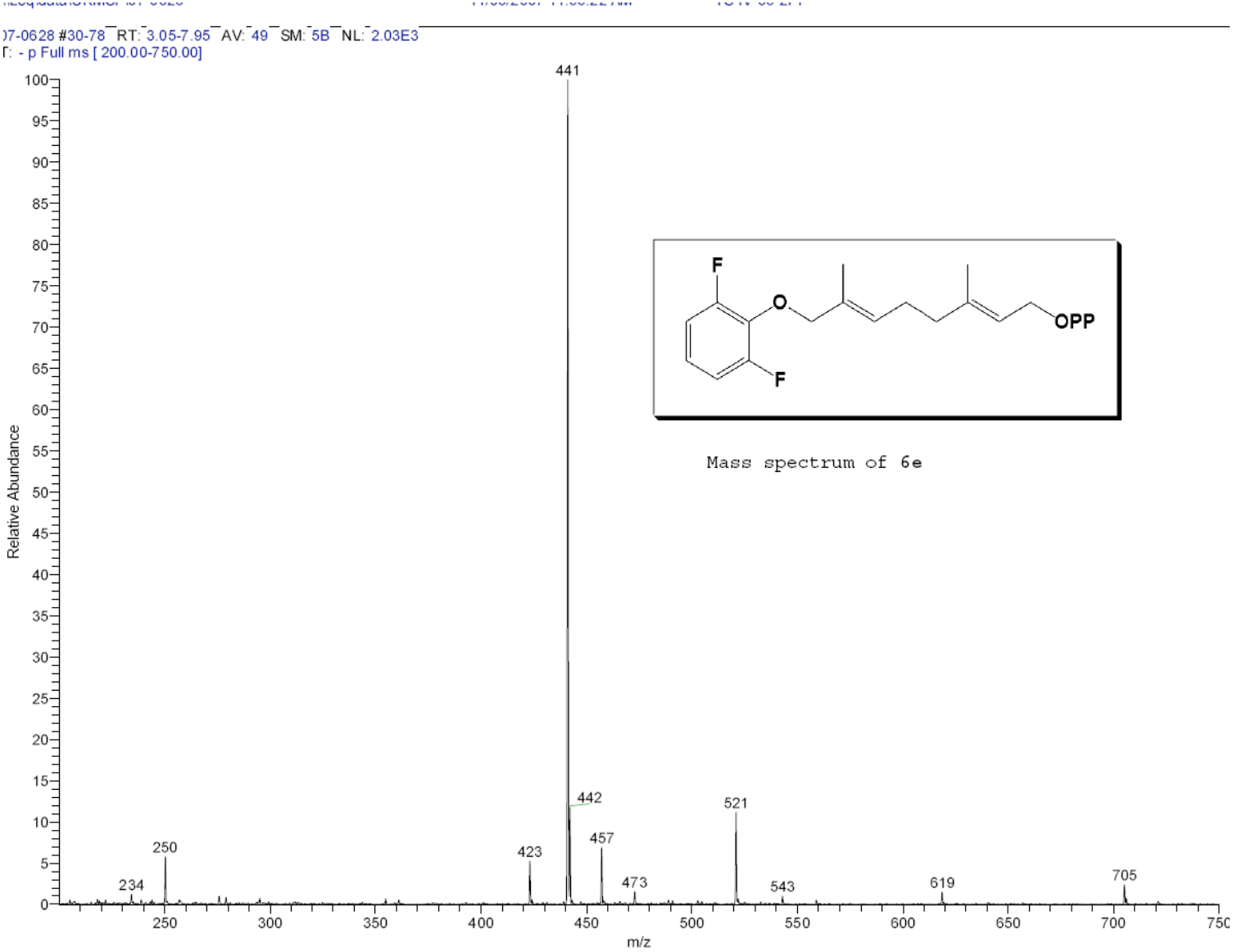
expl  s2pul

      SAMPLE                    DEC. & VT
date   Nov  4 2005  sfrq          399.724
solvent        D2O  dn                H1
file           exp  dpwr              42
   ACQUISITION      dof               0
sfrq        161.811  dm              yyy
tn              P31  dmm               w
at            0.808  dmf           11148
np            16000  dseq
sw         10000.0   drss             1.0
fb             6000  homo               n
bs               64       PROCESSING
tpwr             55  lb              1.00
pw             10.0  wtfile
d1                0  proc              ft
tof               0  fn          not used
nt             3280  math               f
ct              430
alock             n  werr
gain       not used  wexp
      FLAGS          wbt
ti                n  wnt
in                n
dp                y
hs               nn
      DISPLAY
sp           -4567.2
wp           10000.0
vs                56
sc                 0
wc               250
hzcm           40.80
is             354.40
rfl            4547.2
rfp                0
th                20
ins           180.000
nm  ao  ph
```

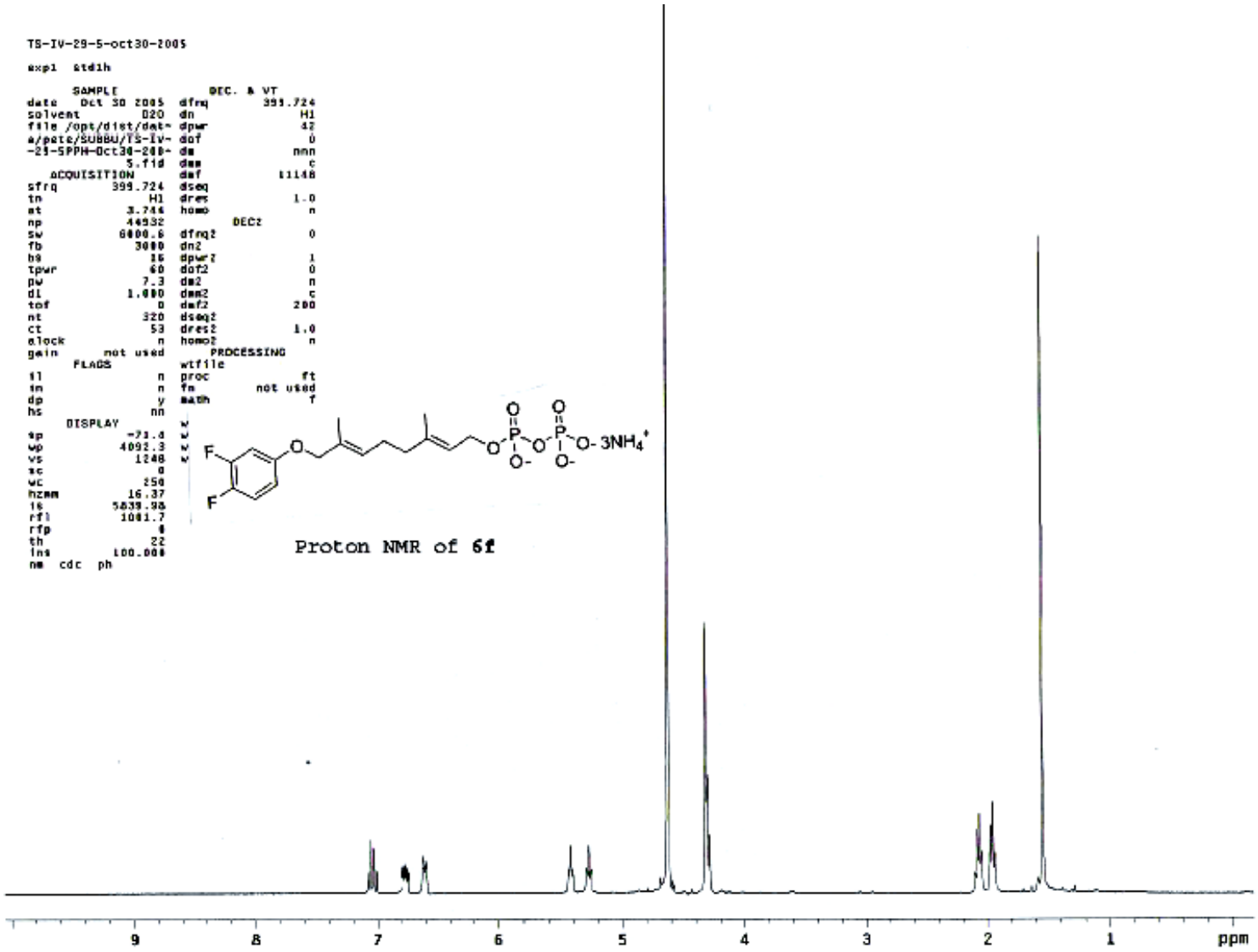


Phosphorus NMR of 6e





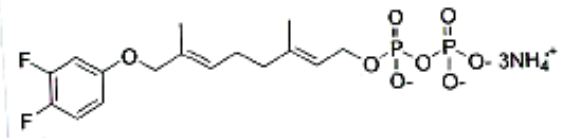
Mass spectrum of 6e


TS-IV-29-5-oct30-2005
exp1 std1h
SAMPLE
date Oct 30 2005
solvent D2O
file /opt/dlst/dat~
a/pete/SUBBU/TS-IV-
-29-5PPM-Oct30-200~
5.fid
ACQUISITION
sfrq 399.724
tn H1
at 3.744
np 44532
sw 6000.6
fb 3000
bs 16
tpwr 60
pw 7.3
dl 1.000
tof 0
nt 320
ct 53
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -73.6
wp 4092.3
vs 1248
sc 0
wc 250
hzmm 16.37
is 5839.98
rfl 1081.7
rfp 0
th 22
ins 100.000
nm cdc ph
DEC. & VT
dfrq 399.724
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn not used
math f
w
w
w
w
F
F
O
O
O
O
O
O
O
O
O
O
P
P
O-
O-
O-
O-
3NH4+
Proton NMR of 6f
9
8
7
6
5
4
3
2
1
ppm

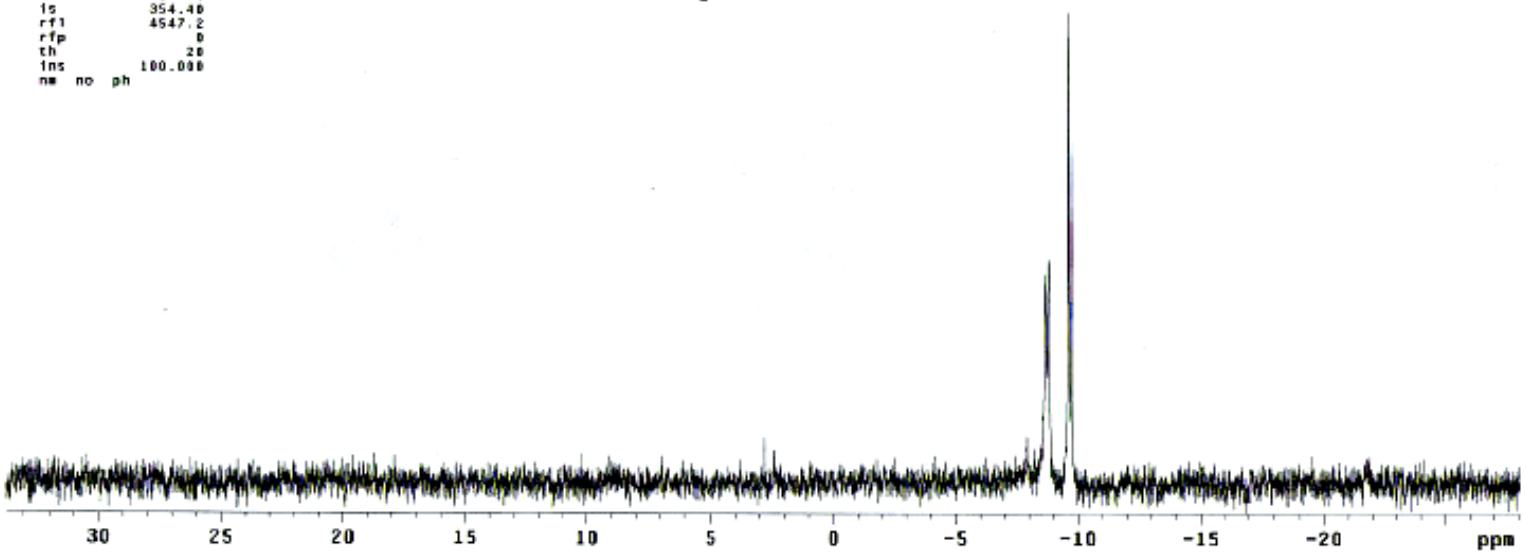
TS-IV-29-5-oct30-2005

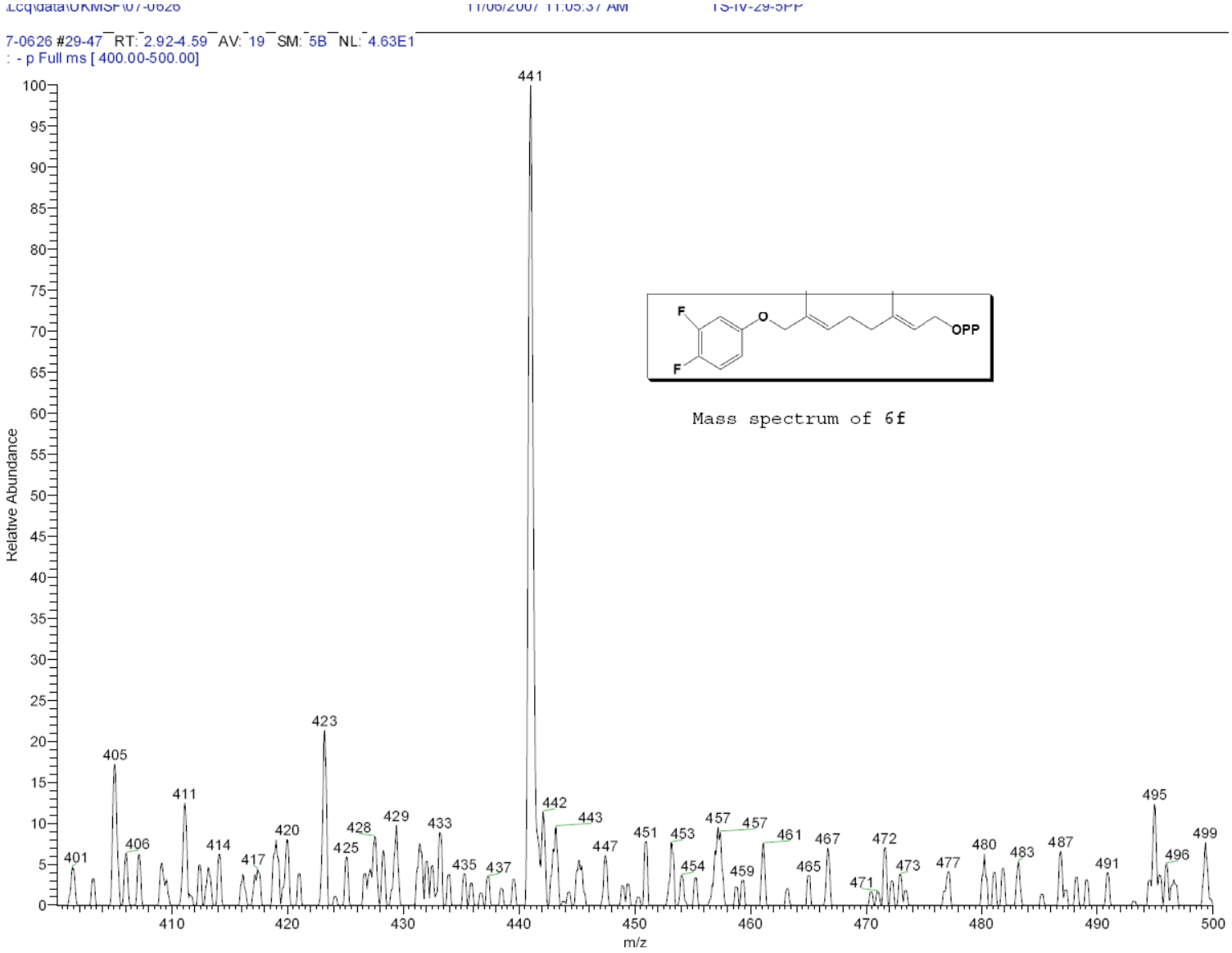
expl sZpul

SAMPLE		DEC. & VT	
date	Oct 30 2005	dfrq	399.724
solvent	DZD	dn	H1
file	/opt/dist/dat~	dpwr	42
a/pate/SUBBU/TS-IV~		dof	0
-29-5-Oct30-2005.f~		dm	yyy
id		dmm	w
ACQUISITION		dmf	11148
sfrq	161.811	dseq	
tn	P31	dres	1.0
at	0.880	homo	n
np	16080	**DEC2**	
sw	10000.0	dfrq2	undefined
fb	6080	dn2	undefined
bs	64	dpwr2	undefined
tpwr	55	dof2	undefined
pw	10.0	dm2	undefined
d1	0	dmm2	undefined
tof	0	dmf2	undefined
nt	3240	dseq2	undefined
ct	416	dres2	undefined
alock	n	homo2	undefined
gain	not used	**PROCESSING**	
FLAGS		lb	1.00
fl	n	wtfile	
in	n	proc	ft
dp	y	fn	not used
hs	nn	math	f
DISPLAY			
sp	-4546.8	werr	
wp	9998.8	wexp	
vs	78	wbs	
sc	0	wnt	
wc	250		
hzmm	28.80		
is	354.40		
rf1	4547.2		
rfp	0		
th	28		
ins	100.000		
ne no ph			

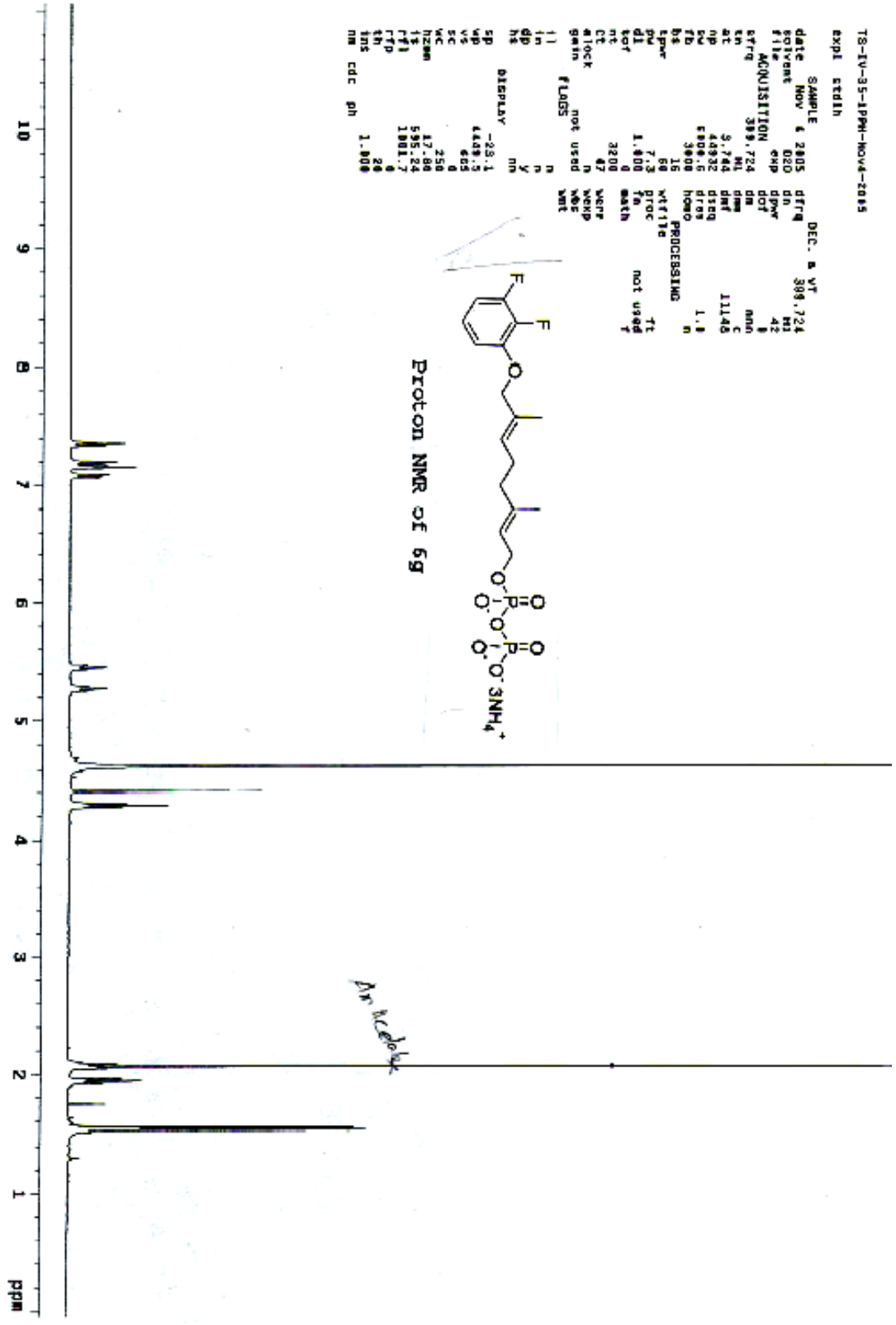


Phosphorus NMR of **6f**





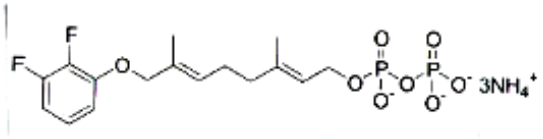
Mass spectrum of **6f**


TS-IV-35-1PM-Nov4-2015
expt stdih
SAMPLE
date Nov 4 2005
solvent D2O
file exp
ACQUISITION
sfrq 399.724 MHz
tn H1
at 3.744
np 44932
fb 6804.6
bs 3908
tpwr 60
pw 7.3
pl 1.000
temp 27.0
nt 3200
ct 47
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -23.1
wp 4449.5
vs 605
sc 0
wc 250
hzcm 17.80
is 595.24
rfl 1881.7
rfp 0
th 28
ins 20
nm cdc ph 1.000
DEC. & VT
dfrq 399.724
dn H1
dpwr 42
dof 0
dm nnn
dmf 11145
dmm c
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Proton NMR of 6g
$3NH_4^+$
Ar methoxy
ppm

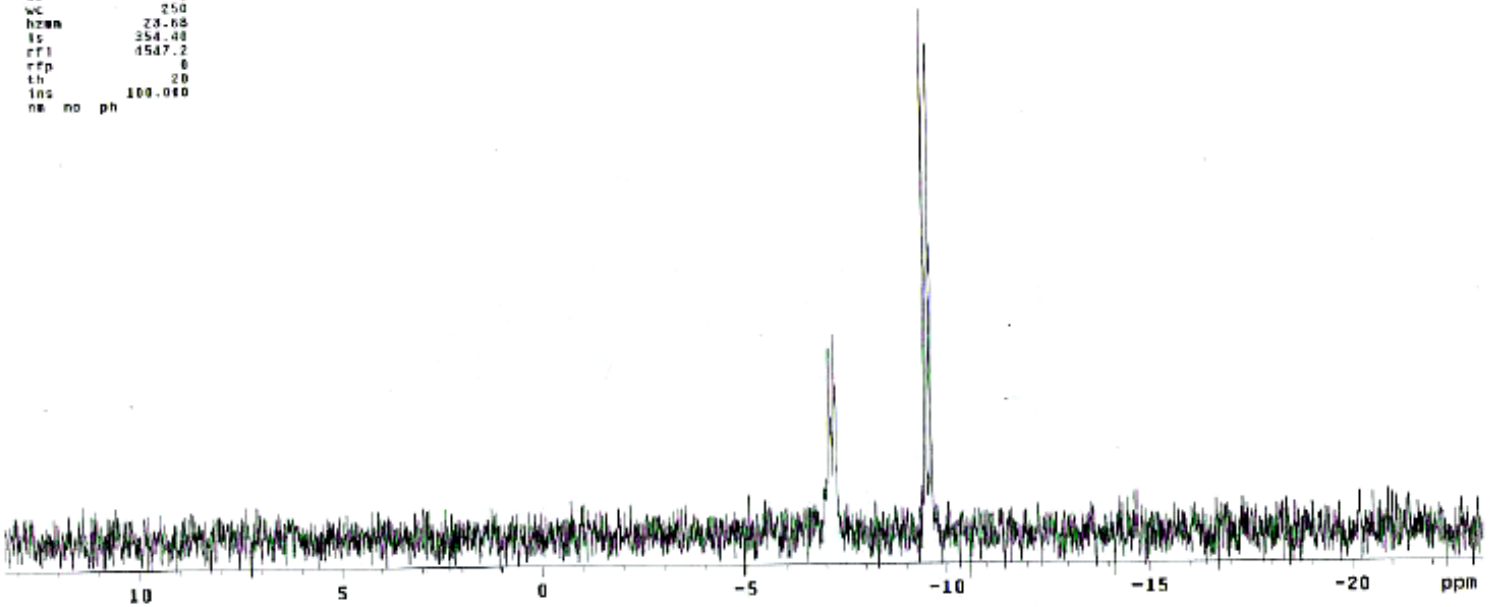
TS-IV-35-1PP-Nov4-2005

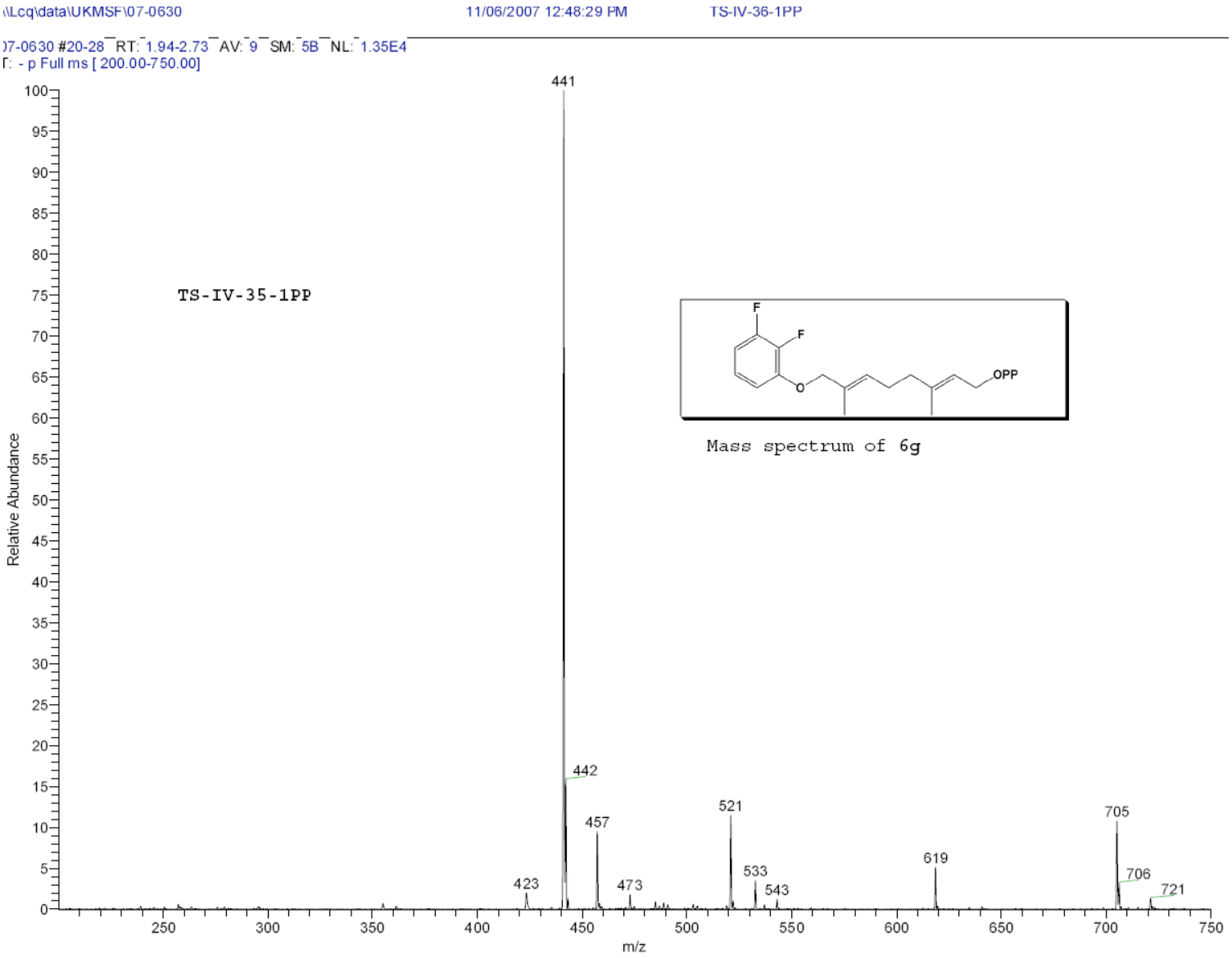
expl s2pul

SAMPLE		DEC. & VT	
date	Nov 4 2005	sfrq	388.724
solvent	D2O	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	161.811	dm	yyy
tn	P31	dmm	w
at	0.880	dmf	11148
np	16000	dseq	
sw	10000.0	dres	1.8
fb	6000	homo	n
bs	64	**PROCESSING**	
lpwr	55	lb	1.00
pw	10.6	wtfile	
d1	8	proc	ft
tof	0	fn	not used
nt	3200	math	f
ct	199		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-3763.5		
wp	5921.1		
vs	89		
sc	0		
wc	250		
hzcm	23.68		
is	354.40		
rfl	4547.2		
rfp	0		
th	20		
ins	100.000		
nm no ph			

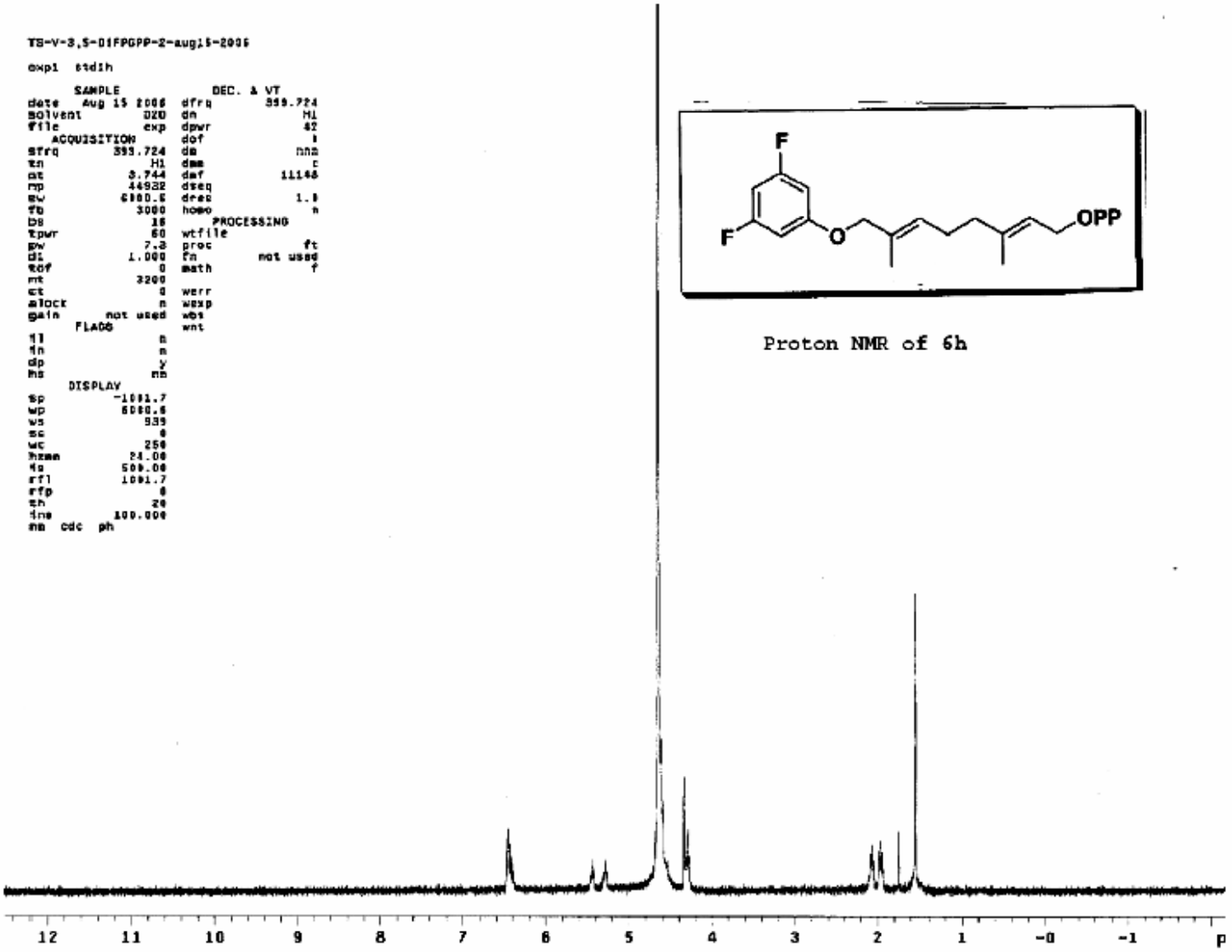


Phosphorus NMR of 6g





Mass spectrum of 6g



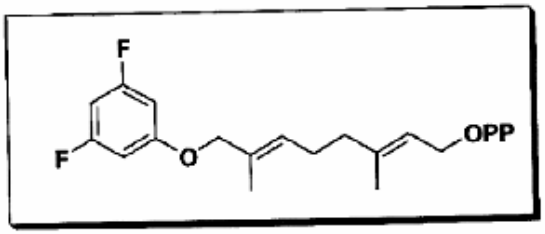
Proton NMR of 6h

TS-V-3,5-DiFPGPP-2-aug15-2006

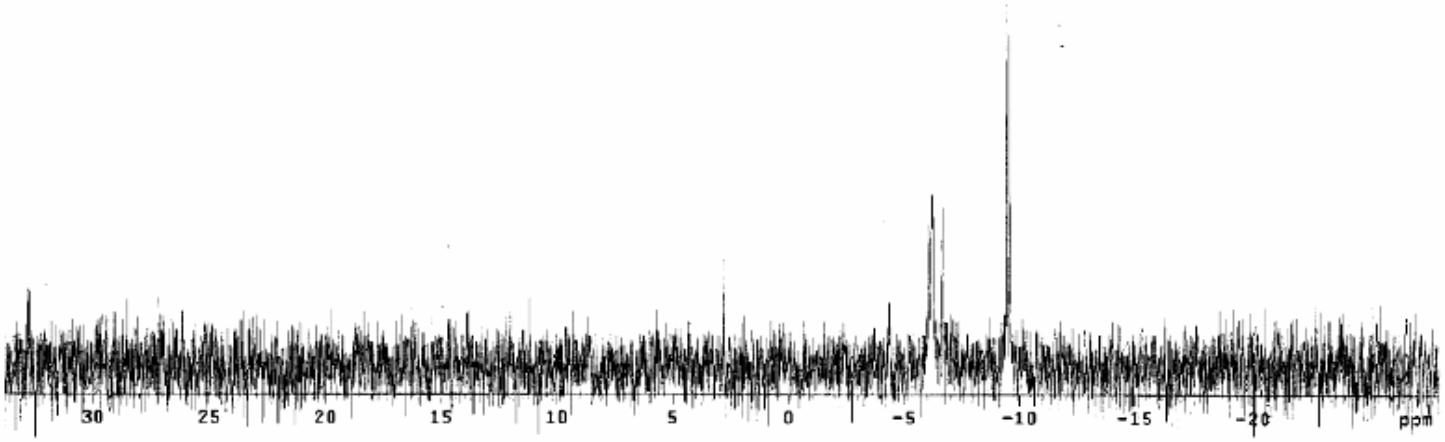
expl s2pul

SAMPLE		DEC. & VT	
date	Aug 15 2006	dfrq	399.724
solvent	D2O	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	161.811	dm	yyy
tn	P31	dmm	w
at	0.800	dmf	11148
np	16000	dseq	
sw	16008.0	dres	1.0
fb	6800	homo	n
bs	64	PROCESSING	
tpwr	55	lb	1.00
pw	10.0	wtfile	
d1	0	proc	ft
tof	0	fn	not used
nt	3200	math	f
ct	0		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-4547.2		
wp	16008.0		
vs	64		
sc	0		
wc	250		
hzmm	40.00		
is	3564.48		
rfl	4547.2		
rfp	0		
th	20		
ins	160.600		

nm no ph

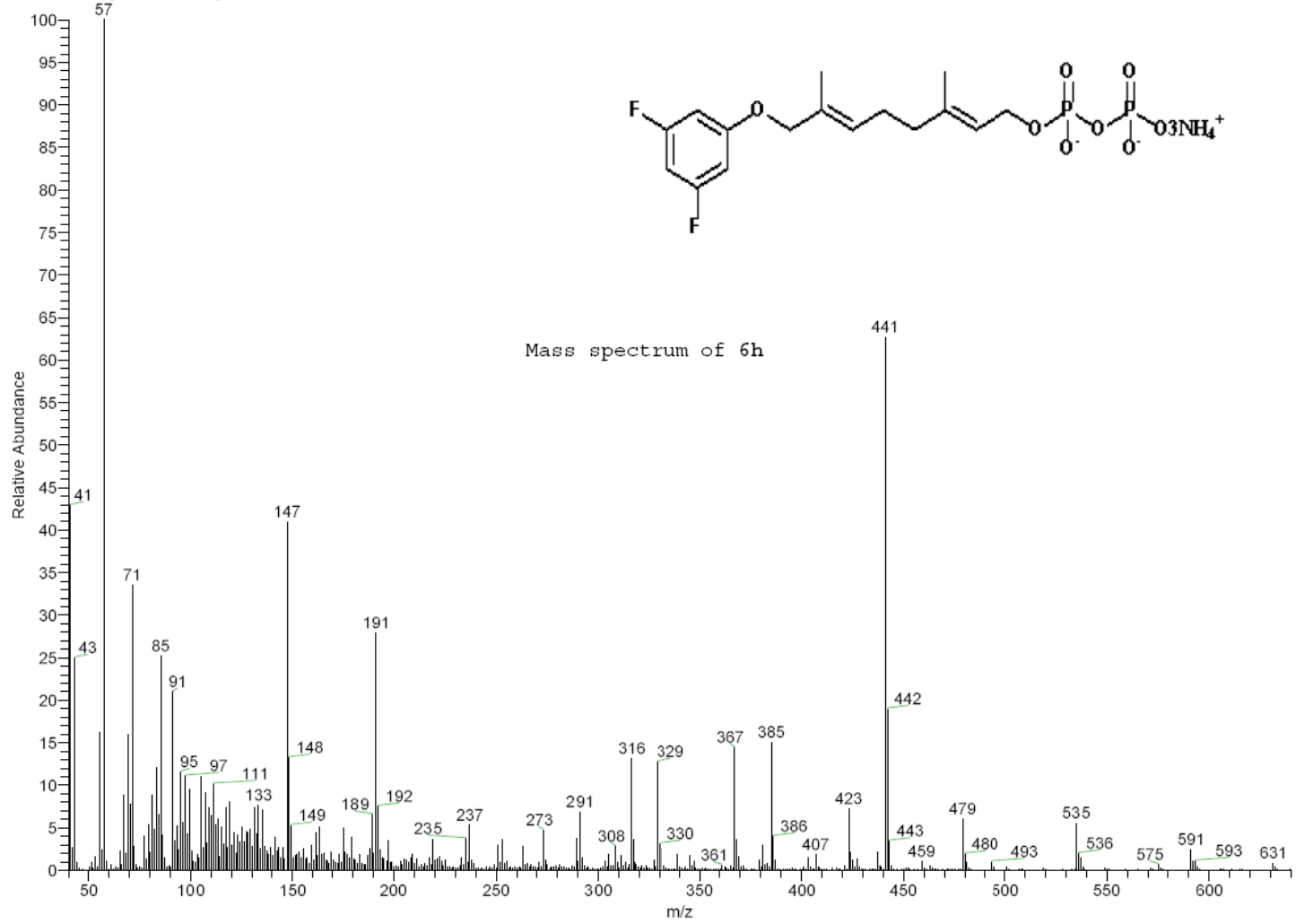


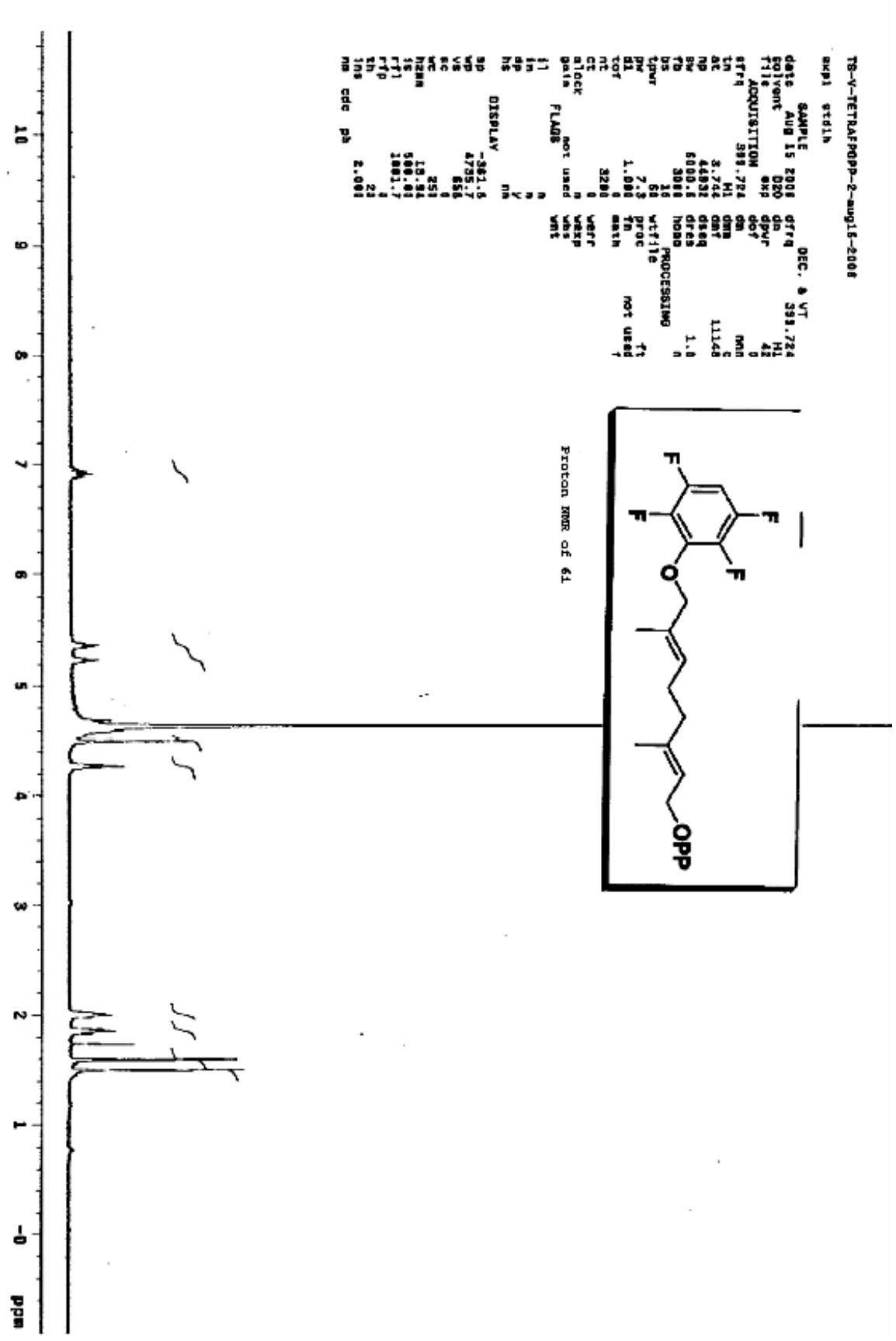
Phosphorus NMR of **6h**



D:\Xcalibur\data\PolarisQ\06-0522 06/22/2006 03:03:10 PM TS-V-56-10

06-0522 #205-224 RT: 3.27-3.60 AV: 20 NL: 6.68E4
T: + c Full ms [40.00-640.00]

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SAMPLE
date Aug 15 2008
solvent d20
file ekp
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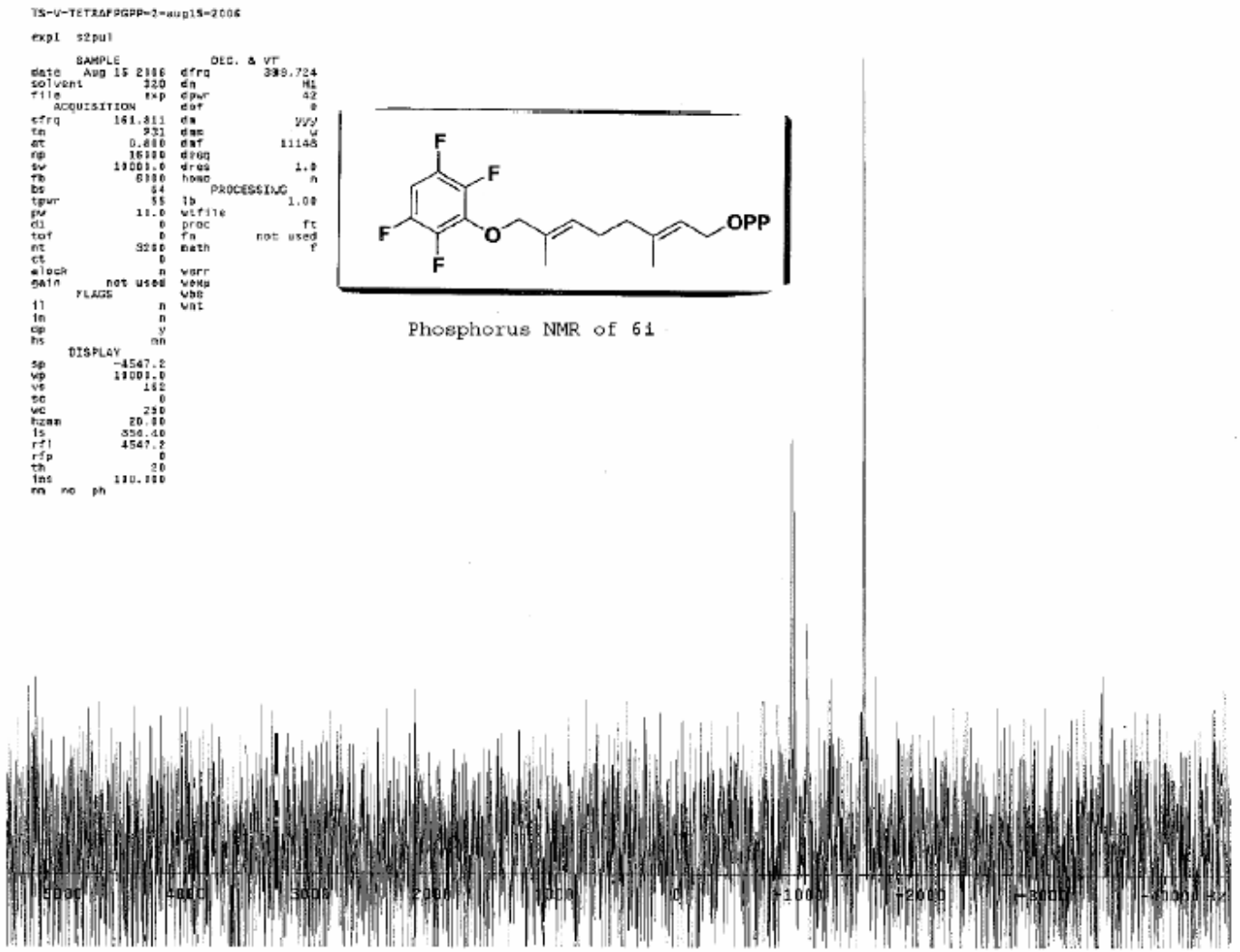
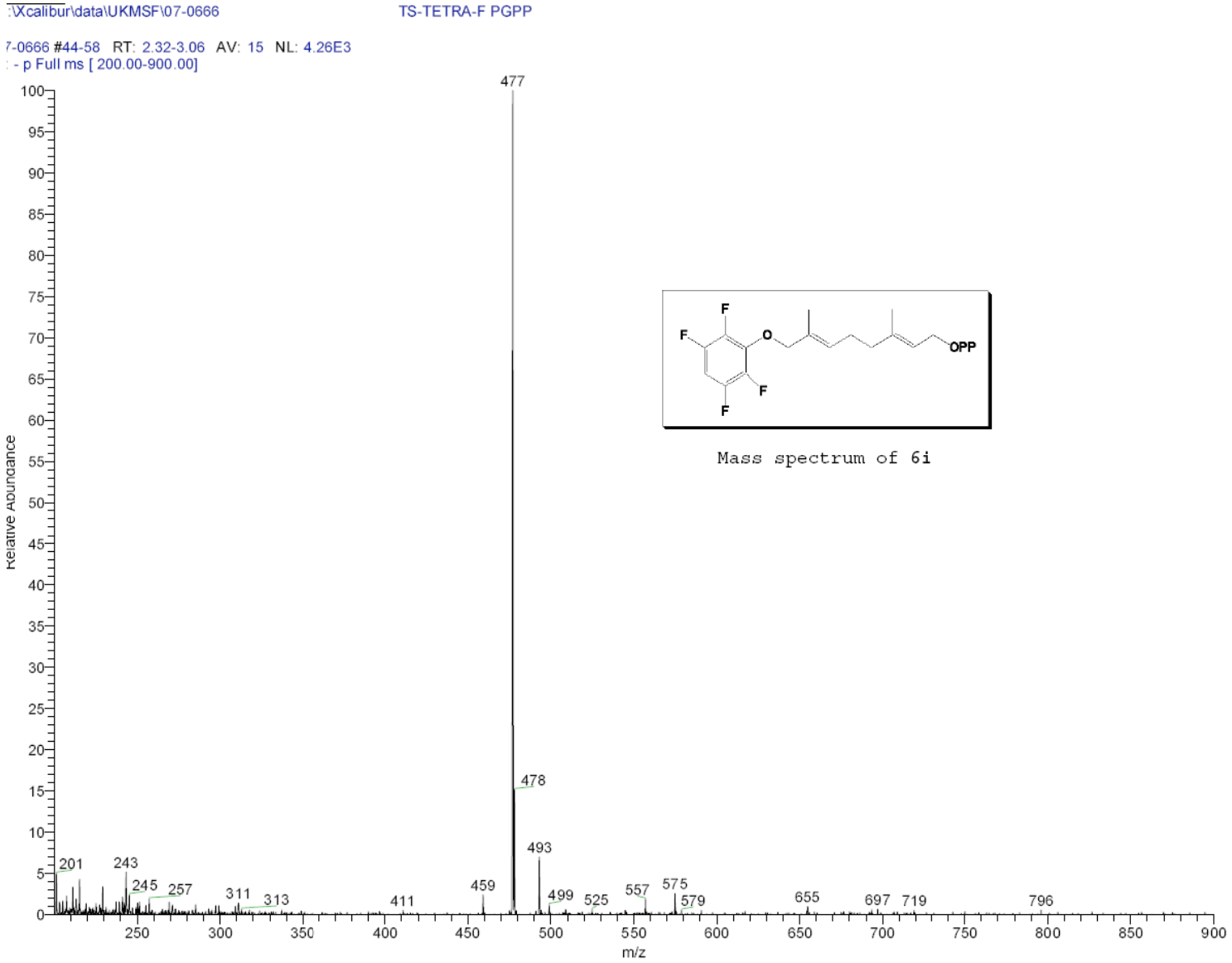
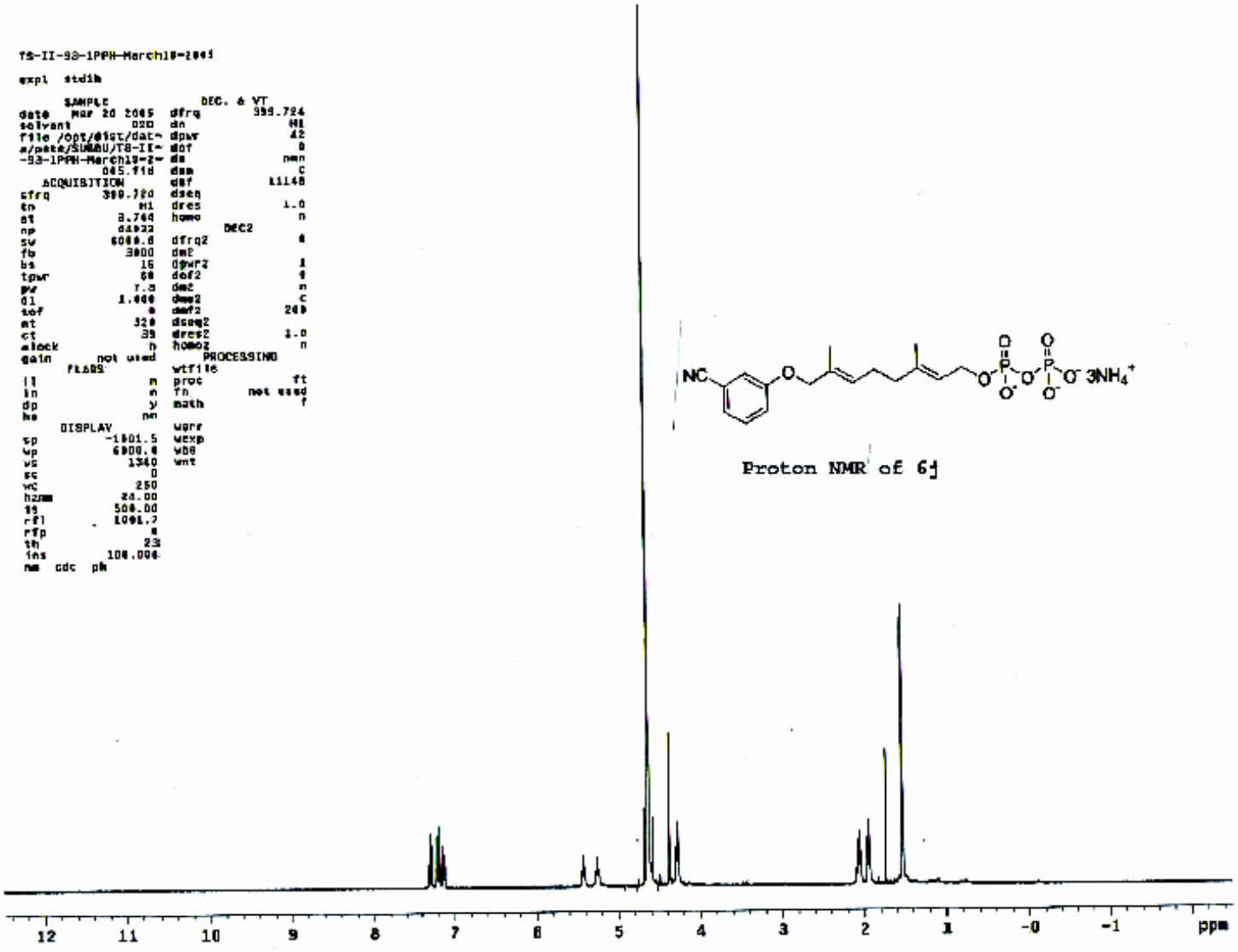
tn H1
at 3.744
np 44932
sw 6000.6
fb 3008
bs 10
tpwr 50
pw 7.3
d1 1.000
tof 3200
nt 8
ct 0
alock n
gain not used
FLAGS
il n
in y
dp n
hs nn
DISPLAY
sp -961.6
wp 4795.7
vs 656
sc 0
wc 253
hzcm 15.94
is 500.01
rfl 1881.7
rfp 1
th 22
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.724
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
wdfr
wexp
was
wnt
F
F
F
O
OPP
Proton NMR of 61
10
9
8
7
6
5
4
3
2
1
-0
ppm



Phosphorus NMR of 61



Mass spectrum of 6i


TS-II-93-1PPH-March19-2005
expl stdih
SAMPLE
date Mar 20 2005
solvent d2O
file /opt/gIGt/data/pake/SURBU/TS-II-93-1PPH-March19-2005.fid
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tn H1
at 3.744
np 44932
sw 6000.6
fb 3800
bs 16
tpwr 58
pw 7.5
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tof 0
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ct 39
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
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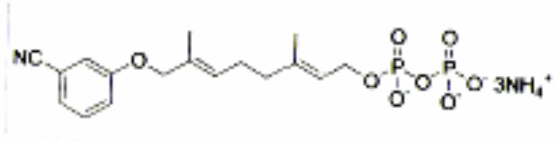
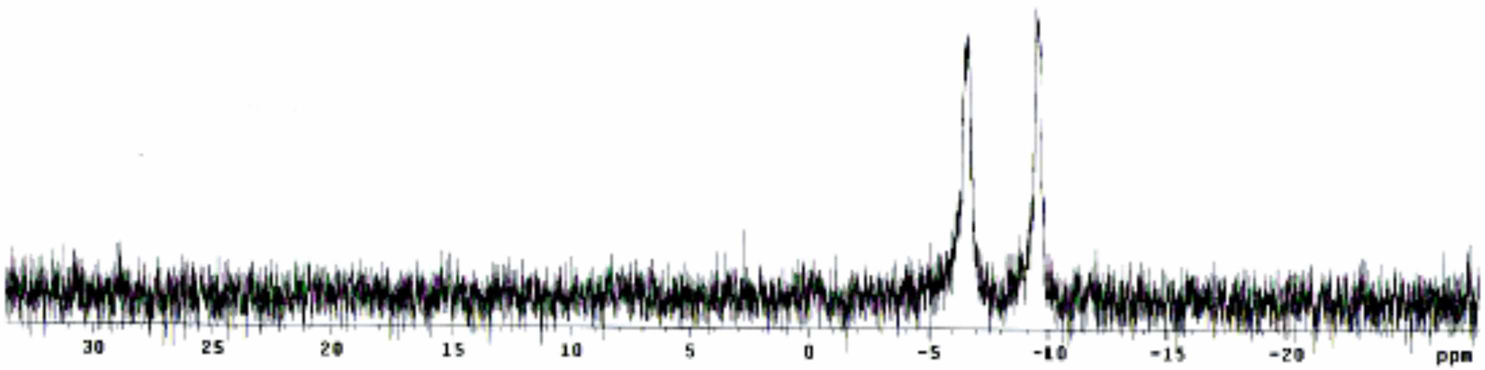
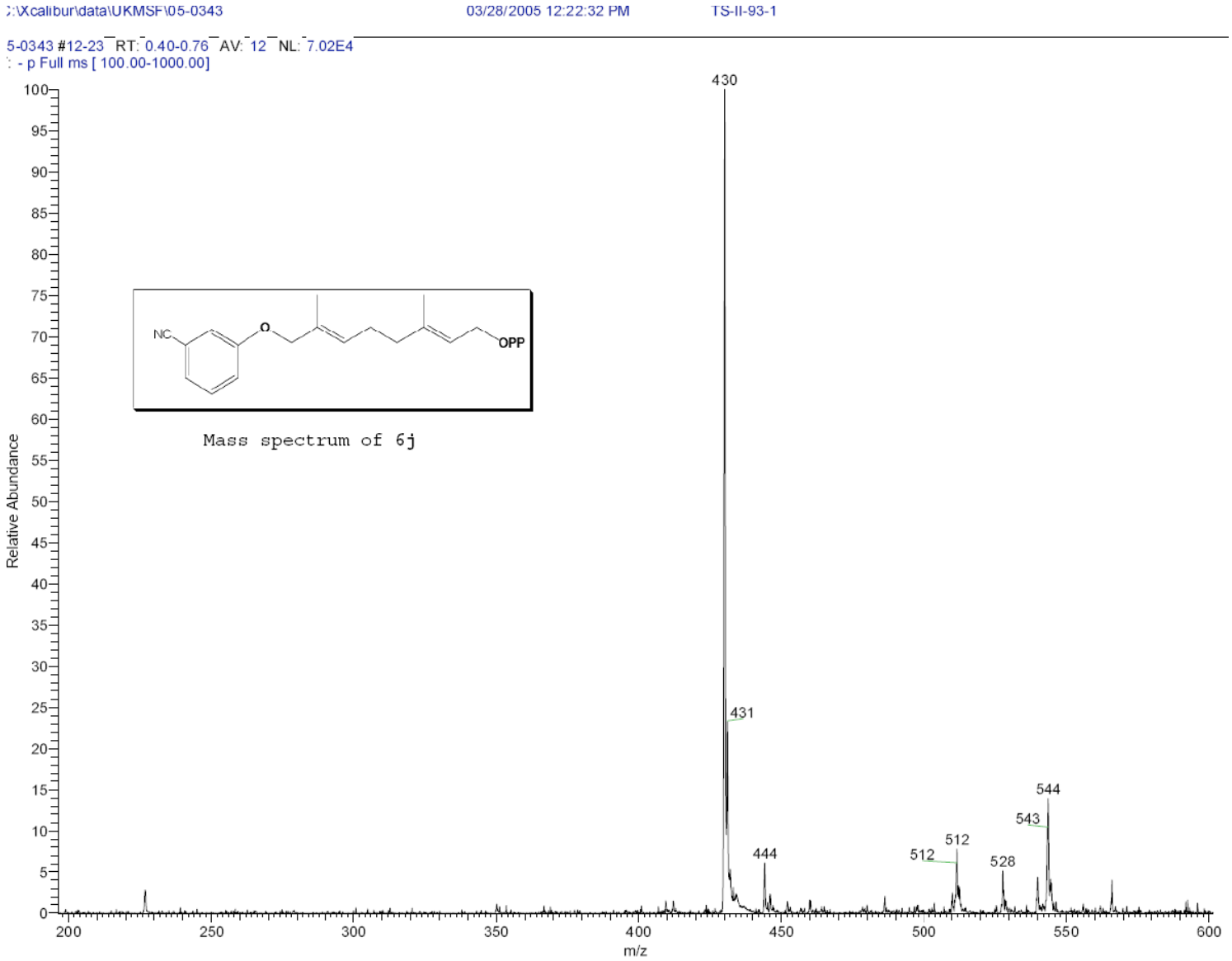
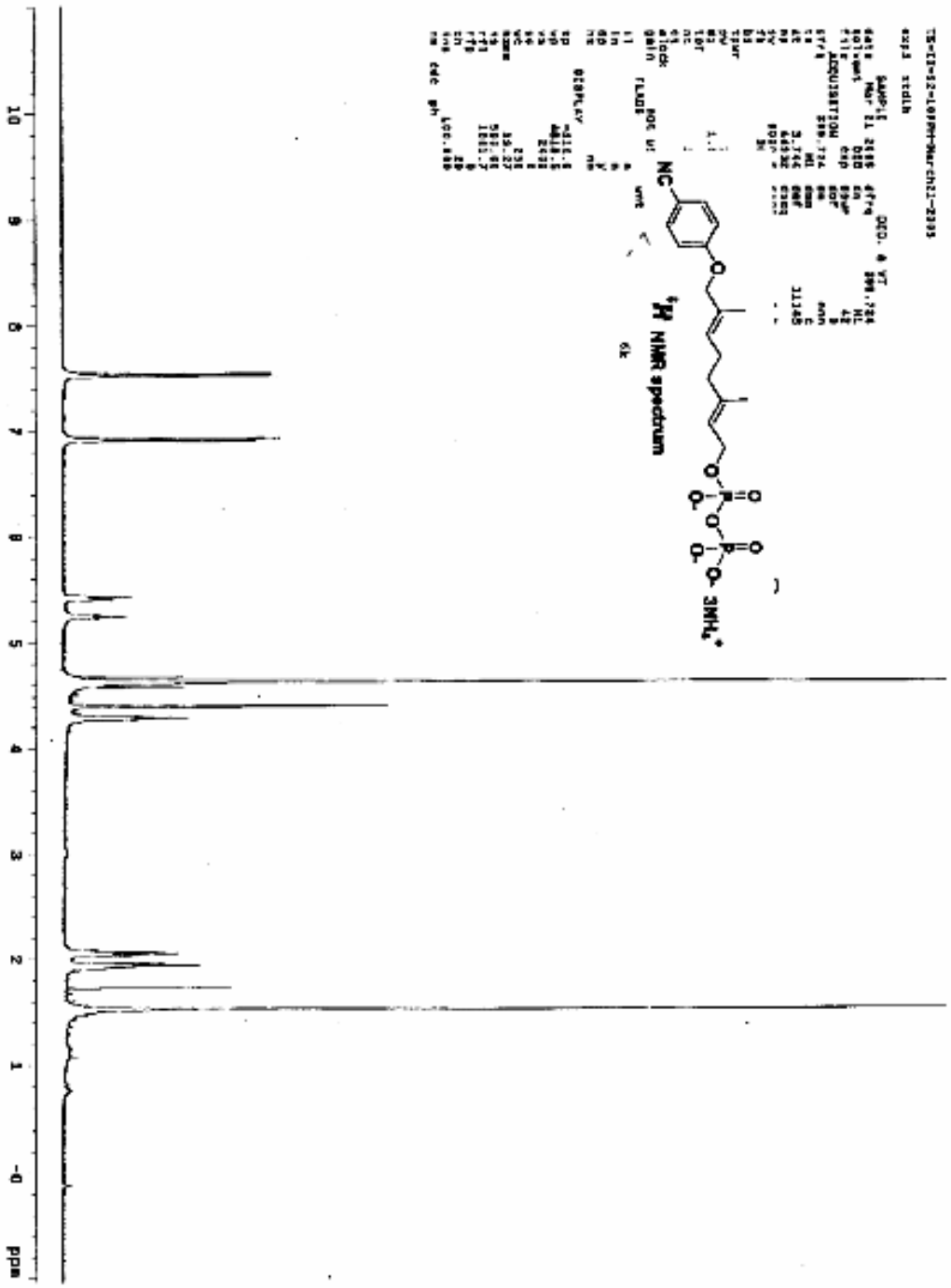
wp 6900.6
vs 1340
sc 0
wc 250
hzcm 24.00
hz 500.00
rfl 1091.7
rfp 0
th 23
ins 100.000
nm cdc pk
DEC. & VT
dfrq 399.724
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 249
dseq2
dres2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn not used
math f
wnr
wexp
wbs
wnt
NC
O
O
O
O
O
O
O
O
O
3NH4+
Proton NMR of 6j
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm



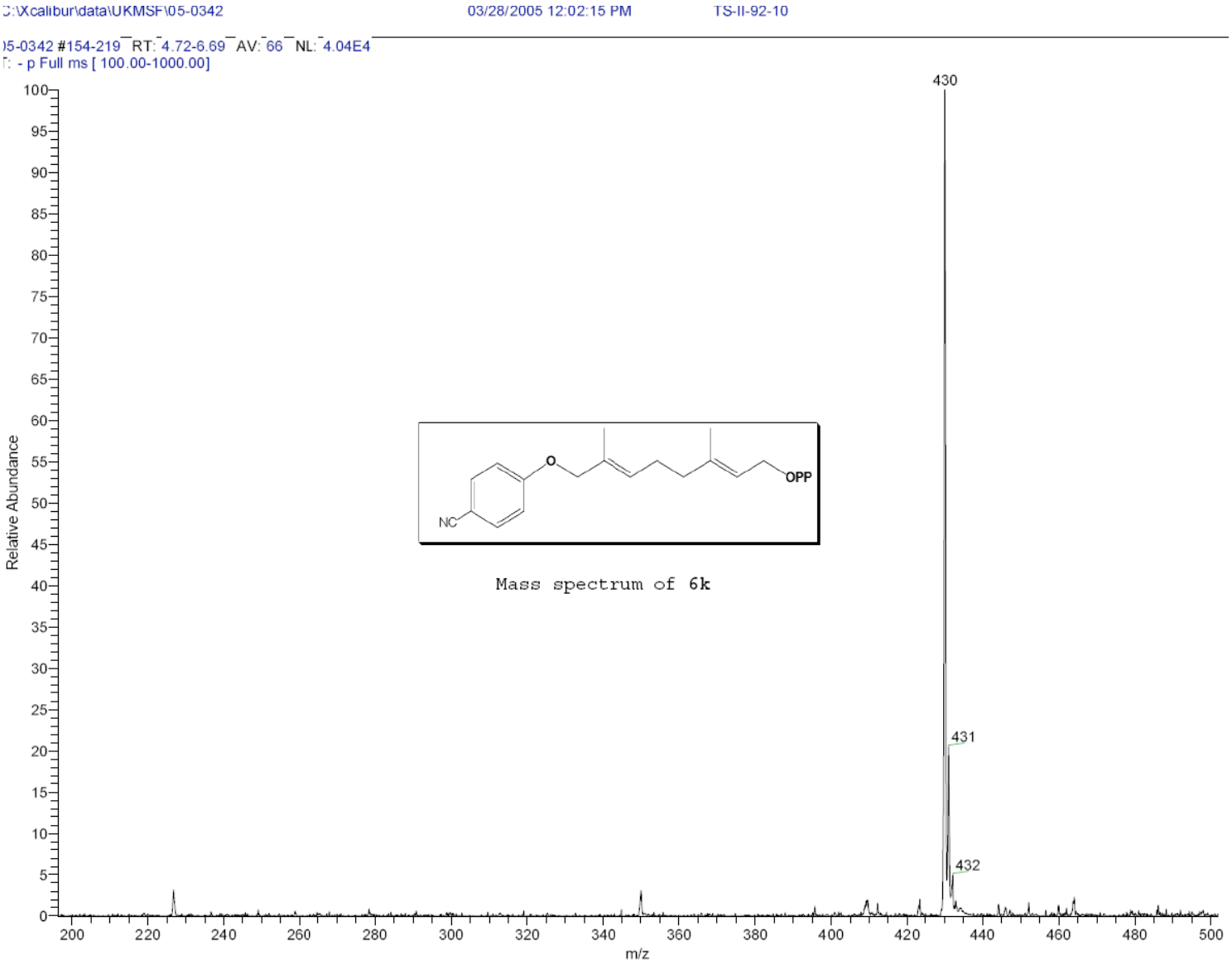
Phosphorus NMR of 6j



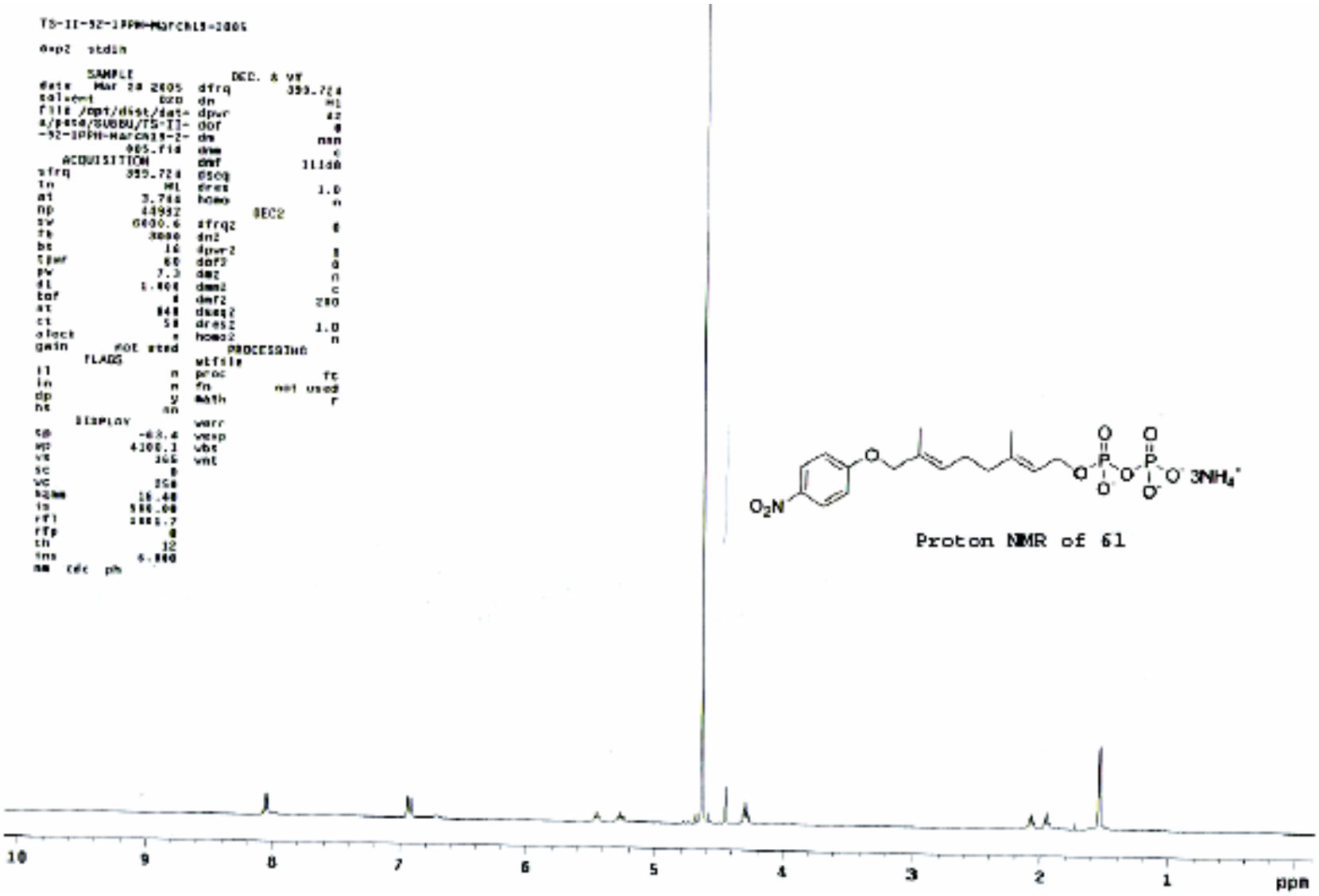

:\Xcalibur\data\UKMSF\05-0343
03/28/2005 12:22:32 PM
TS-II-93-1
5-0343 #12-23 RT: 0.40-0.76 AV: 12 NL: 7.02E4
: - p Full ms [100.00-1000.00]
NC
O
OPP
Mass spectrum of 6j
Relative Abundance
m/z
430
431
444
512
512
528
543
544


^1H NMR spectrum
NC
$3NH_4^+$
10
9
8
7
6
5
4
3
2
1
-0
ppm

NC
O
O
P
O
O
O
P
O
O
O
3NH4+
31P NMR spectrum
6k
30
25
20
15
10
5
0
-5
-10
-15
-20
ppm

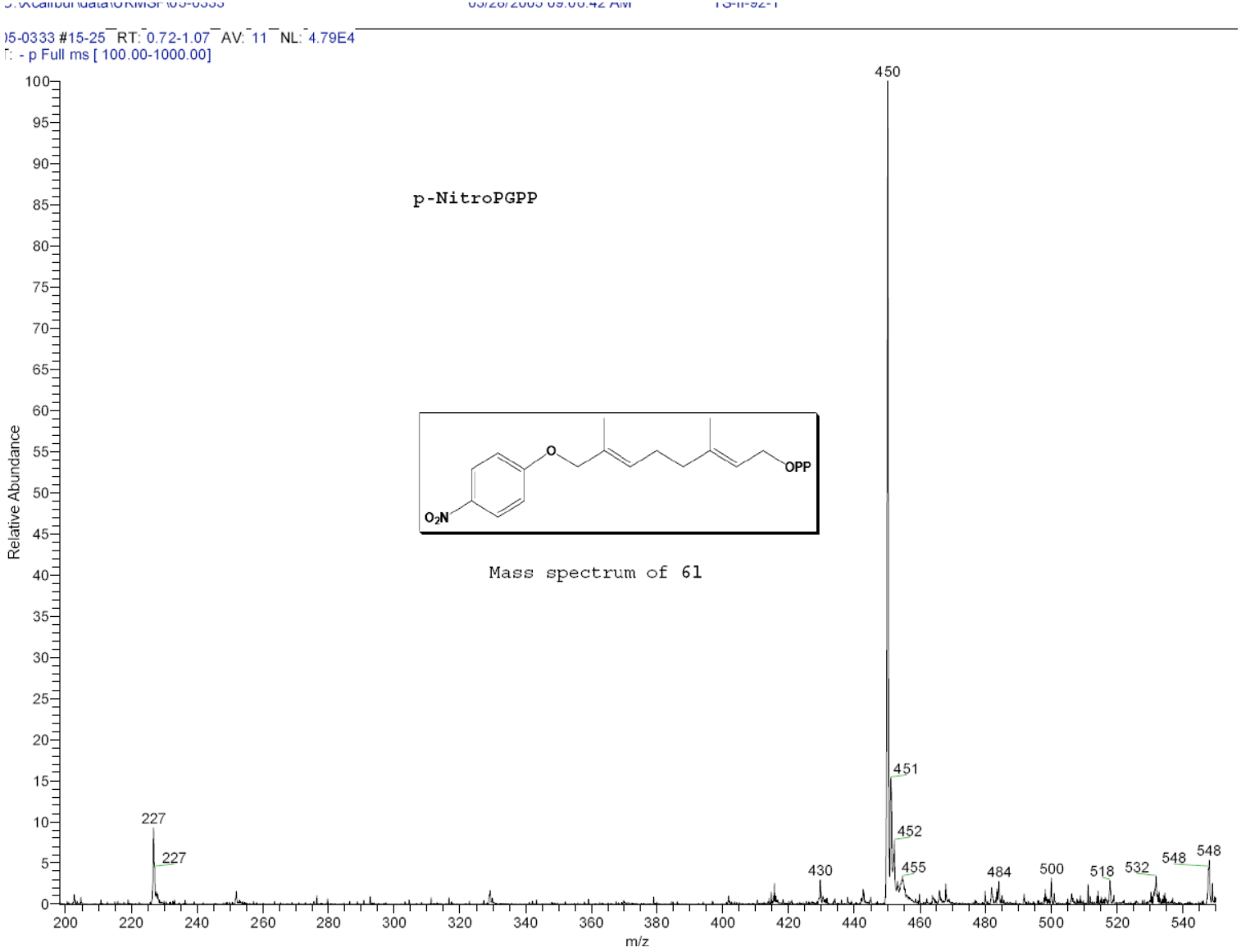


Mass spectrum of **6k**

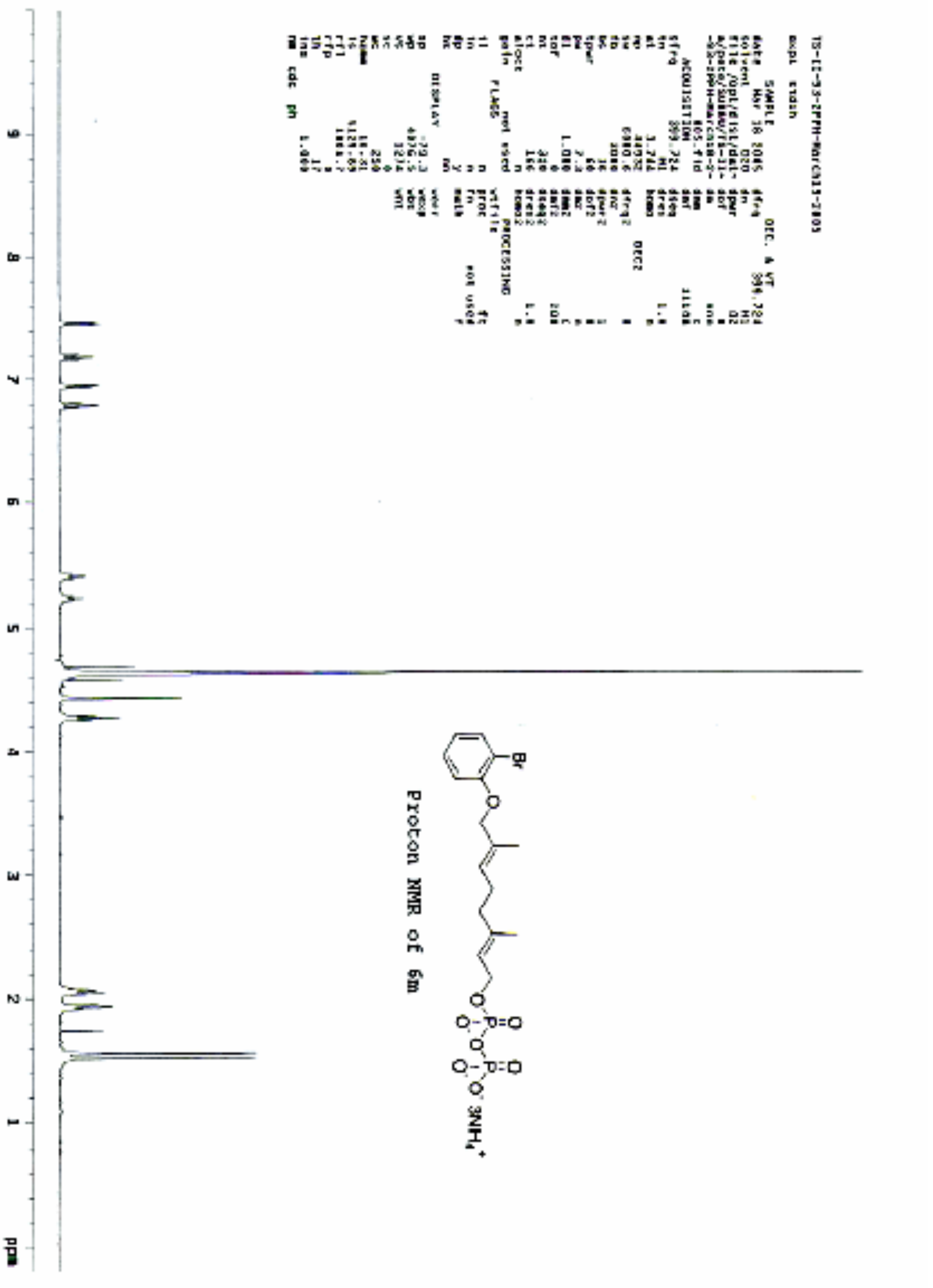


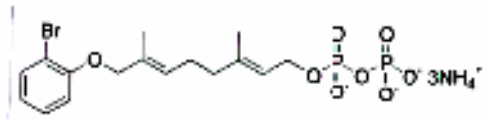
Proton NMR of 61

Phosphorus NMR of 61

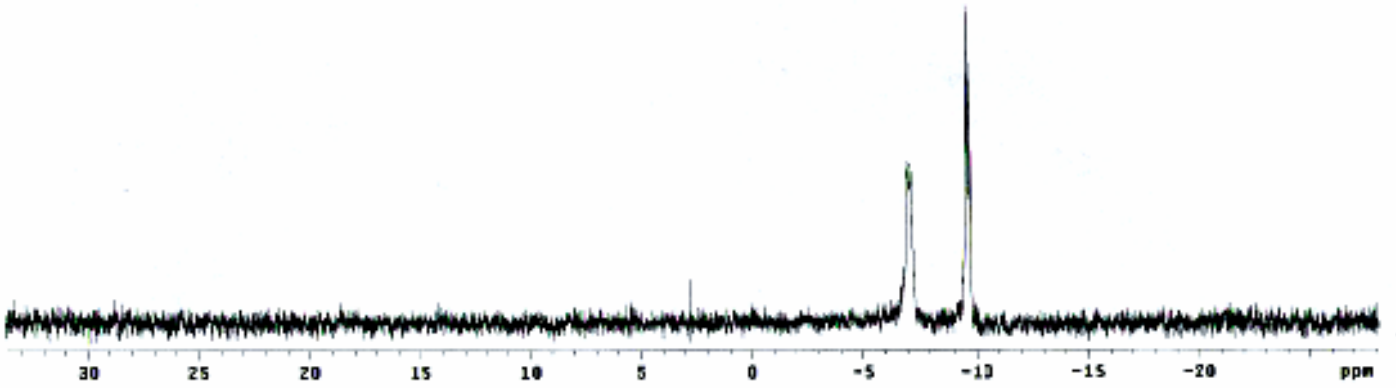


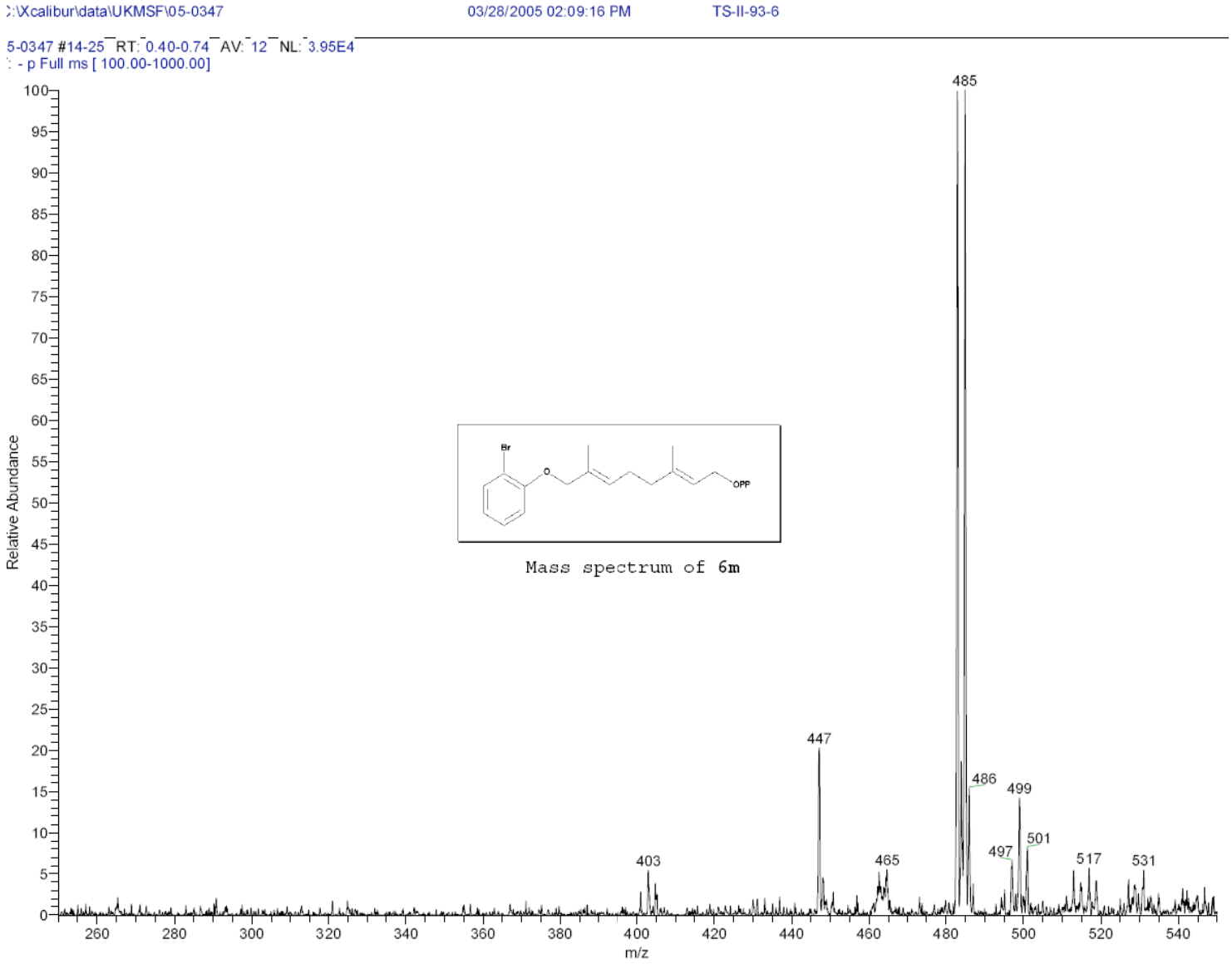
Mass spectrum of 61


Proton NMR of 6m
ppm

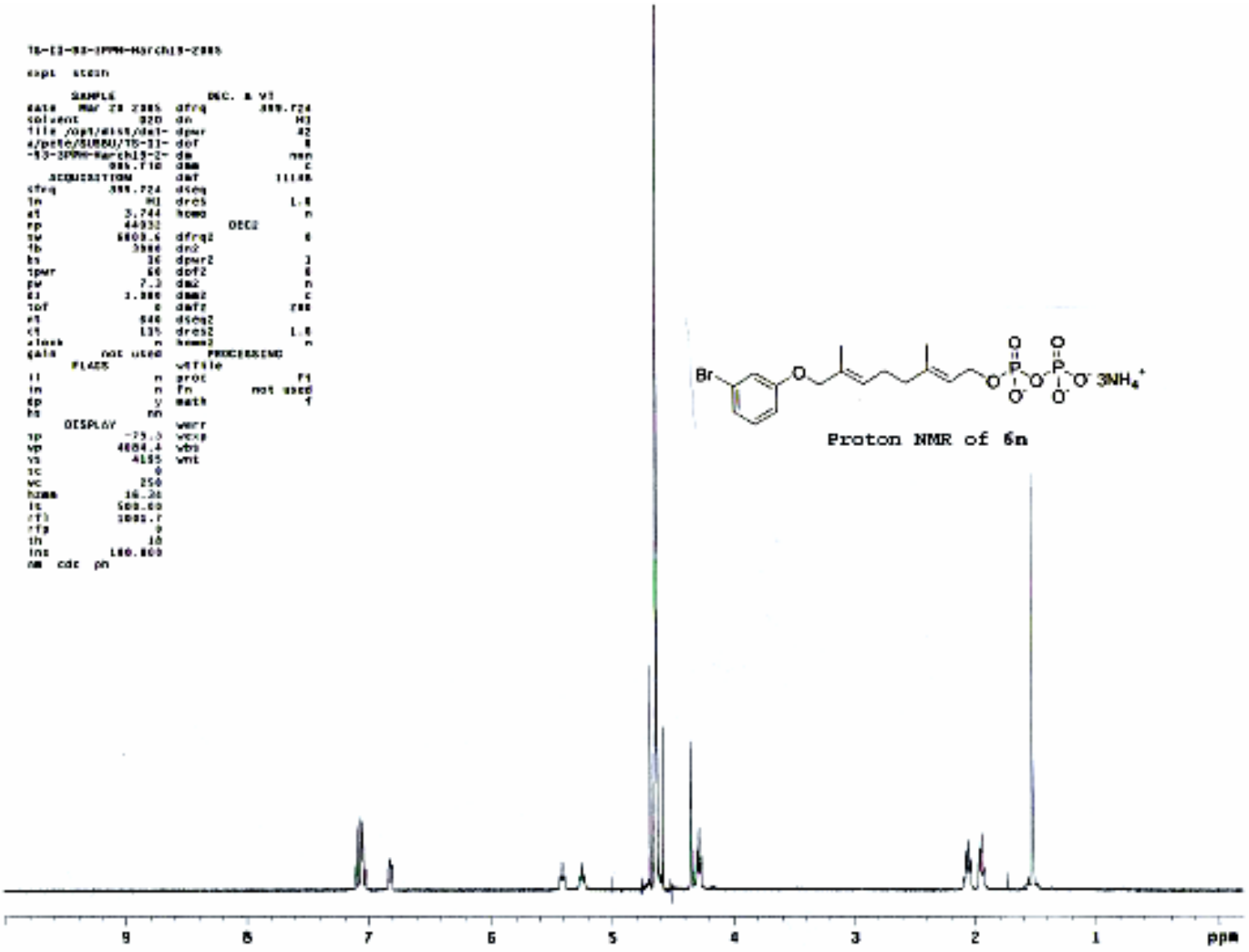


Phosphorus NMR of 6m





Mass spectrum of **6m**

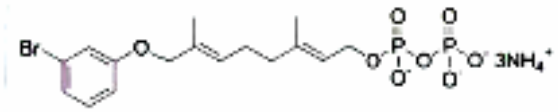


Proton NMR of 6n

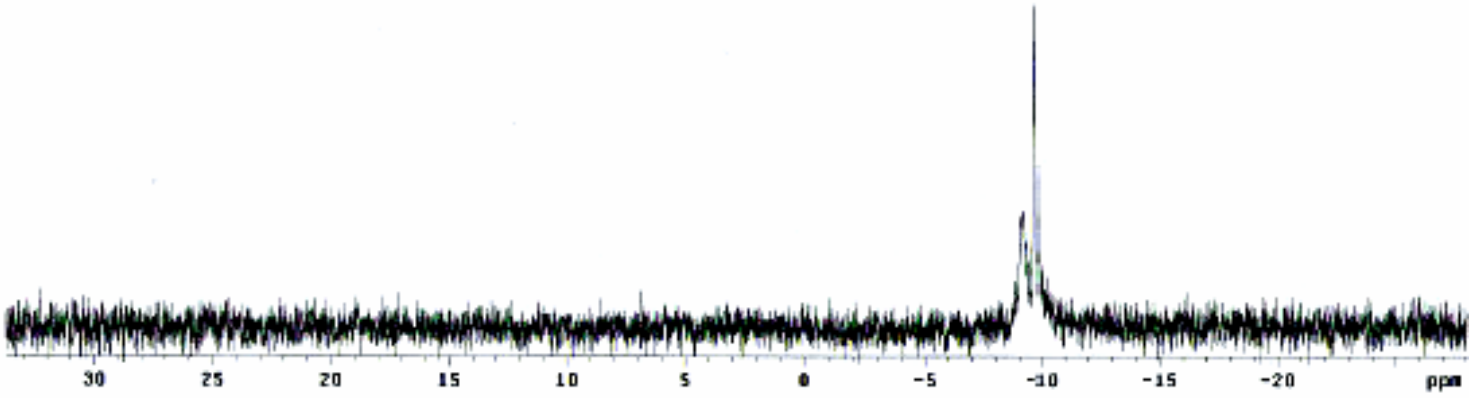
```
TS-II-93-3PP-March19-2005

expt  ccpul

      SAMPLE                  EEC- & WT
date    Mar 19 2005   dfrq          399.724
solvent        D2O    dn                HL
file /opt/dist/dat>   dpwr              42
e/pete/SUBBU/TS-II>   dof                0
-93-3PP-March19-20>   dm                yyy
             85.fid   dmm                 w
   ACQUISITION        dmf             11468
sfrq        161.811   dseq
tn              P31   dres              1.0
at            0.880   homo                n
np            16860         DEC2
sw         14008.0    dfrq2     undefined
fb            6860    dn2       undefined
bs              64    dpwr2     undefined
tpwr            55    dof2      undefined
pw            18.0    dm2       undefined
d1               0    dmm2      undefined
taf              0    dmf2      undefined
nt            3280    dseq2     undefined
ct            2938    dres2     undefined
alock            n    homo2     undefined
gain      not used        PROCESSING
      FLAGS           lb              1.00
il               n    wtfile
in               n    proc              ft
dp               y    TM          not used
hs              nn    math               f
      DISPLAY
sp         -4546.0    werr
wp          9595.0    wexp
vs               56    wbs
sc                0    wnt
wc              250
hzmm          40.80
is           354.40
rfl         4547.2
rfp               0
th               20
ins         186.860
nm  no  ph
```

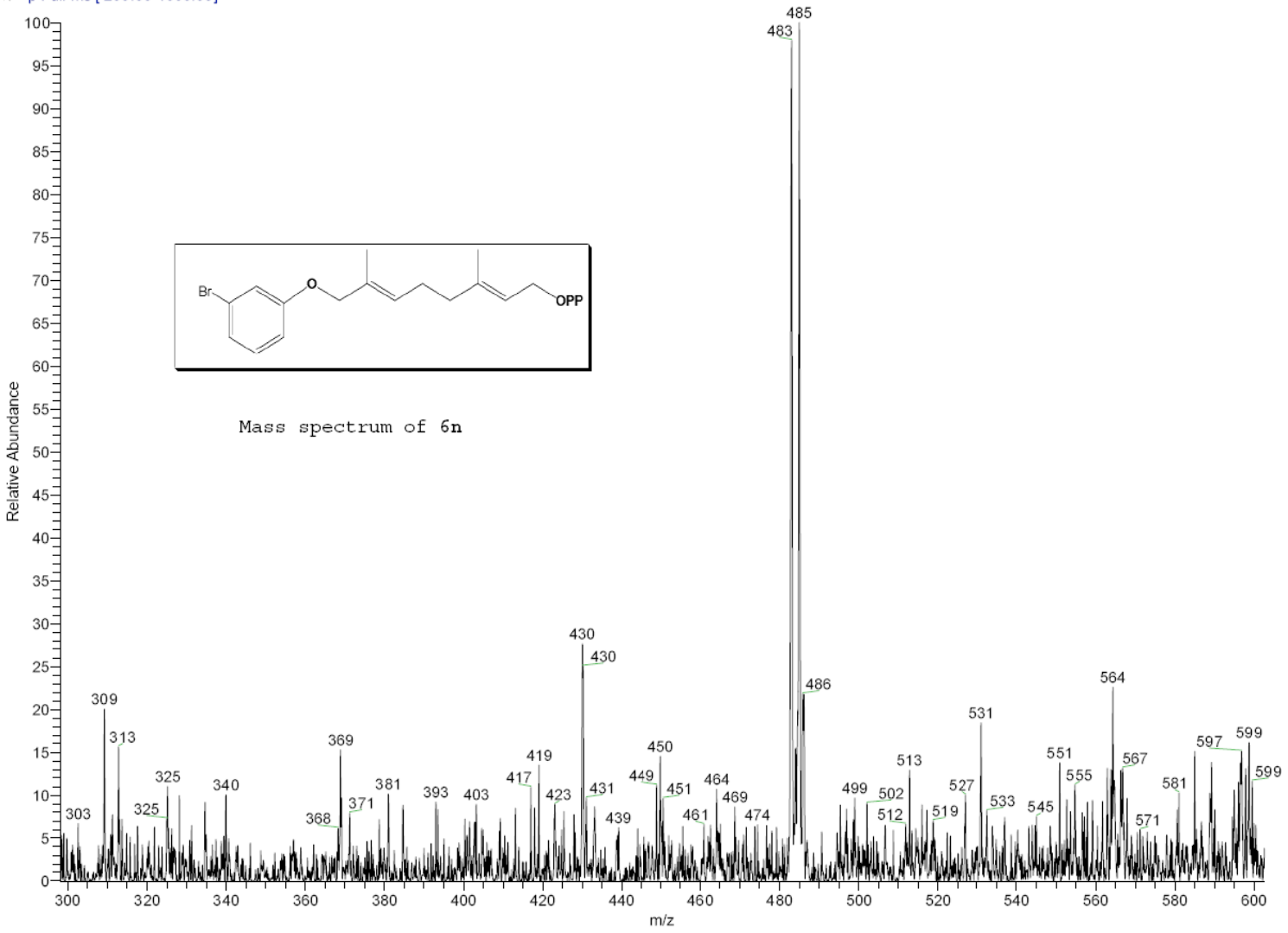


Phosphorus NMR of 6n

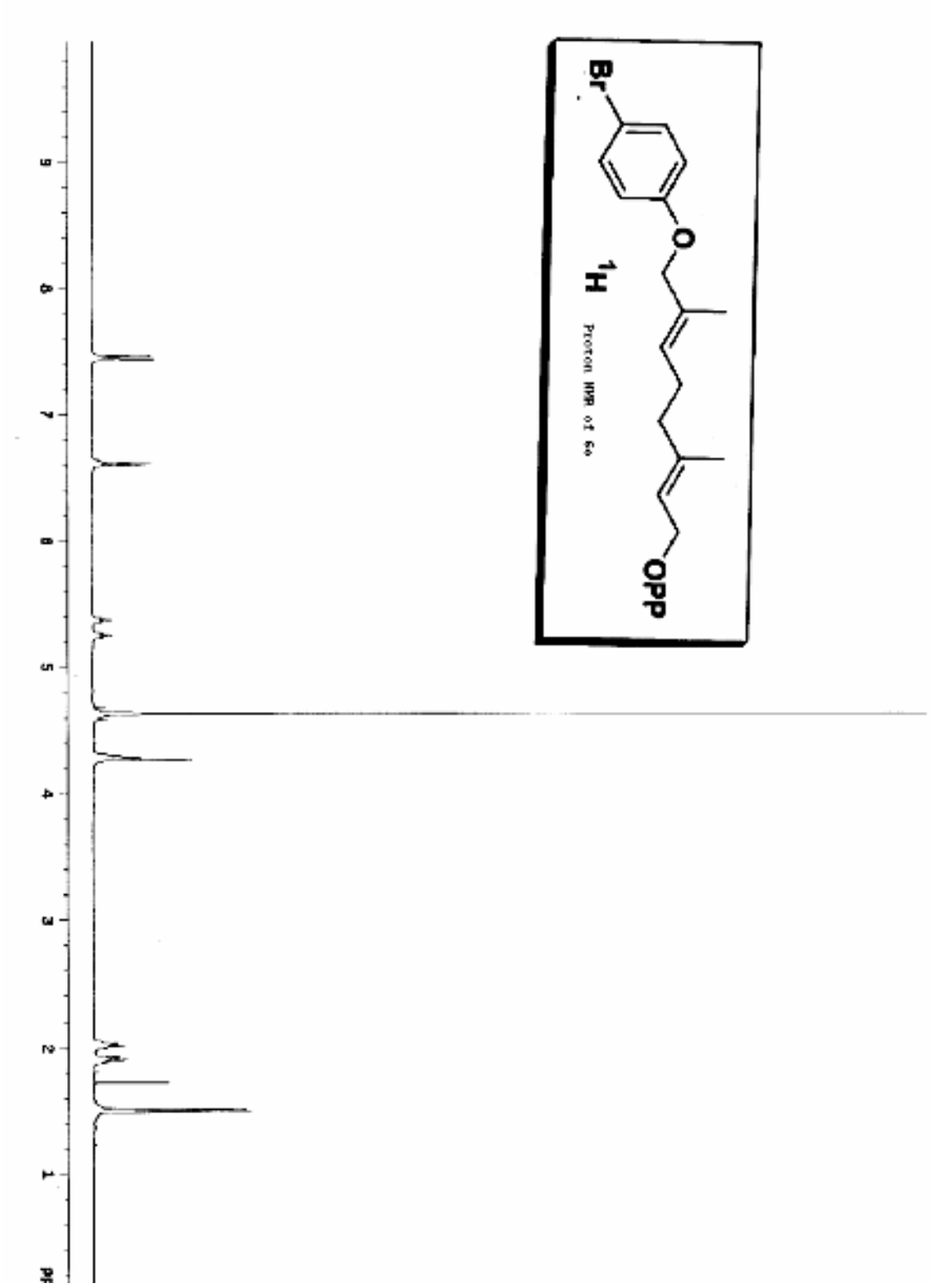


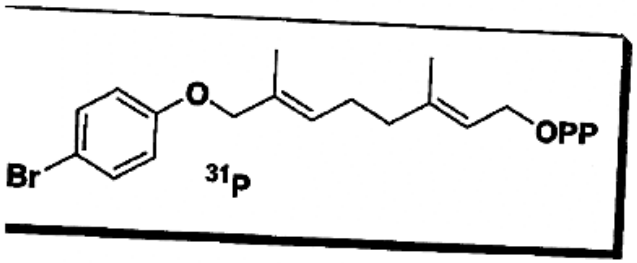
C:\Xcalibur\data\UKMSF\05-0345 03/28/2005 01:41:14 PM TS-II-93-3

05-0345 #50-64 RT: 1.56-2.04 AV: 15 NL: 5.07E3
T: - p Full ms [230.00-1000.00]

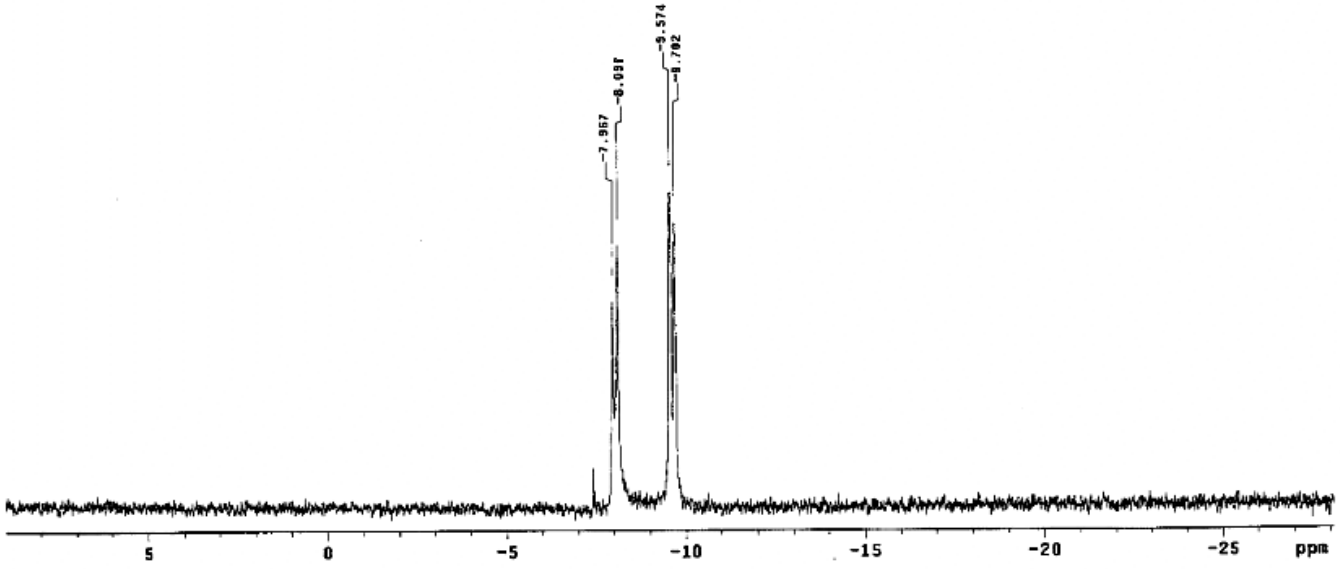


Mass spectrum of 6n


Br
O
OPP
1H
Proton NMR of 6a

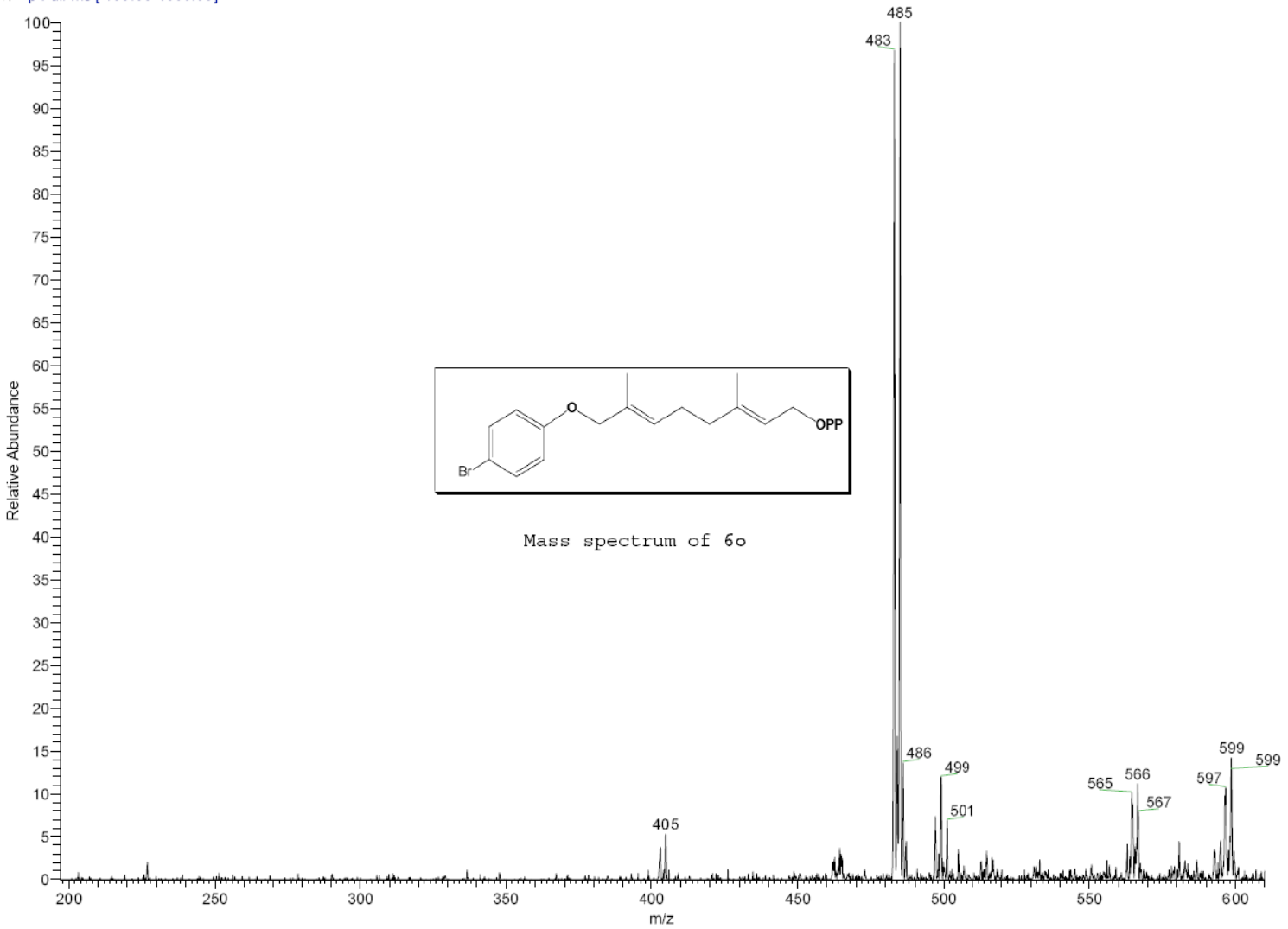


Phosphorus NMR of 6o

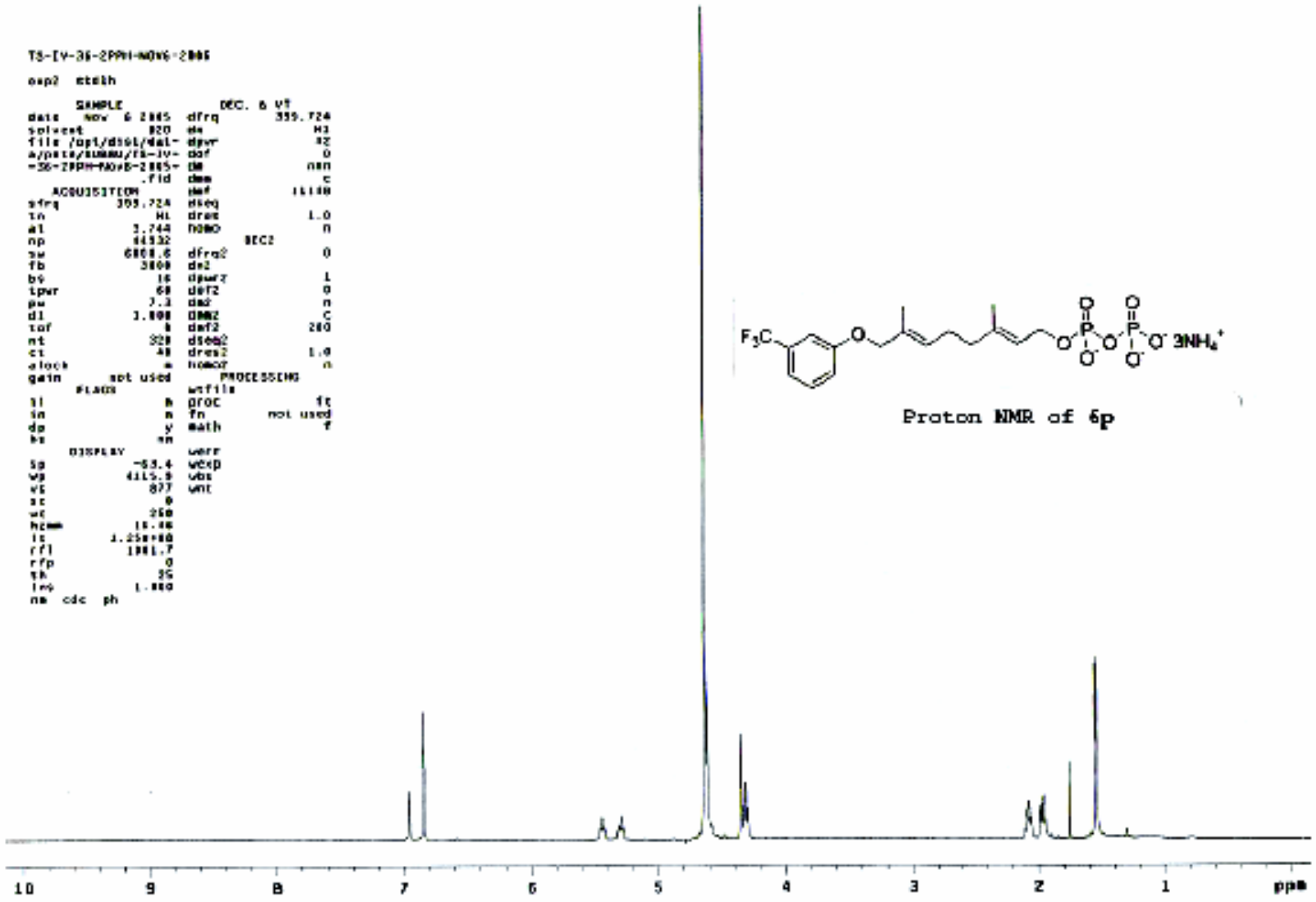


C:\Xcalibur\data\UKMSF\05-0344 03/28/2005 01:18:47 PM TS-II-93-2

05-0344 #13-21 RT: 0.35-0.57 AV: 9 NL: 1.67E5
T: - p Full ms [100.00-1000.00]



Mass spectrum of 6o


TS-IV-36-2PPM-H2O#6-2805
exp2 etalh
SAMPLE
date Nov 6 2005
solvent H2O
file /opt/dtds1/dal-a/pete/SUMMU/ts-iv-36-2PPM-H2O#6-2805-.fid
ACQUISITION
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tn H1
at 3.744
np 44532
sw 6868.6
fb 3804
bs 16
tpwr 68
pw 7.1
d1 1.000
tof 0
nt 320
ct 48
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -53.4
wp 4115.9
vs 877
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is 1.25e+00
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rfp 0
th 35
lvl 1.000
nm cdc ph
DEC. 6 VT
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dn H1
dpwr 42
dof 0
DM nnn
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dmf 15140
dseq
drec 1.0
homo n
DEC2
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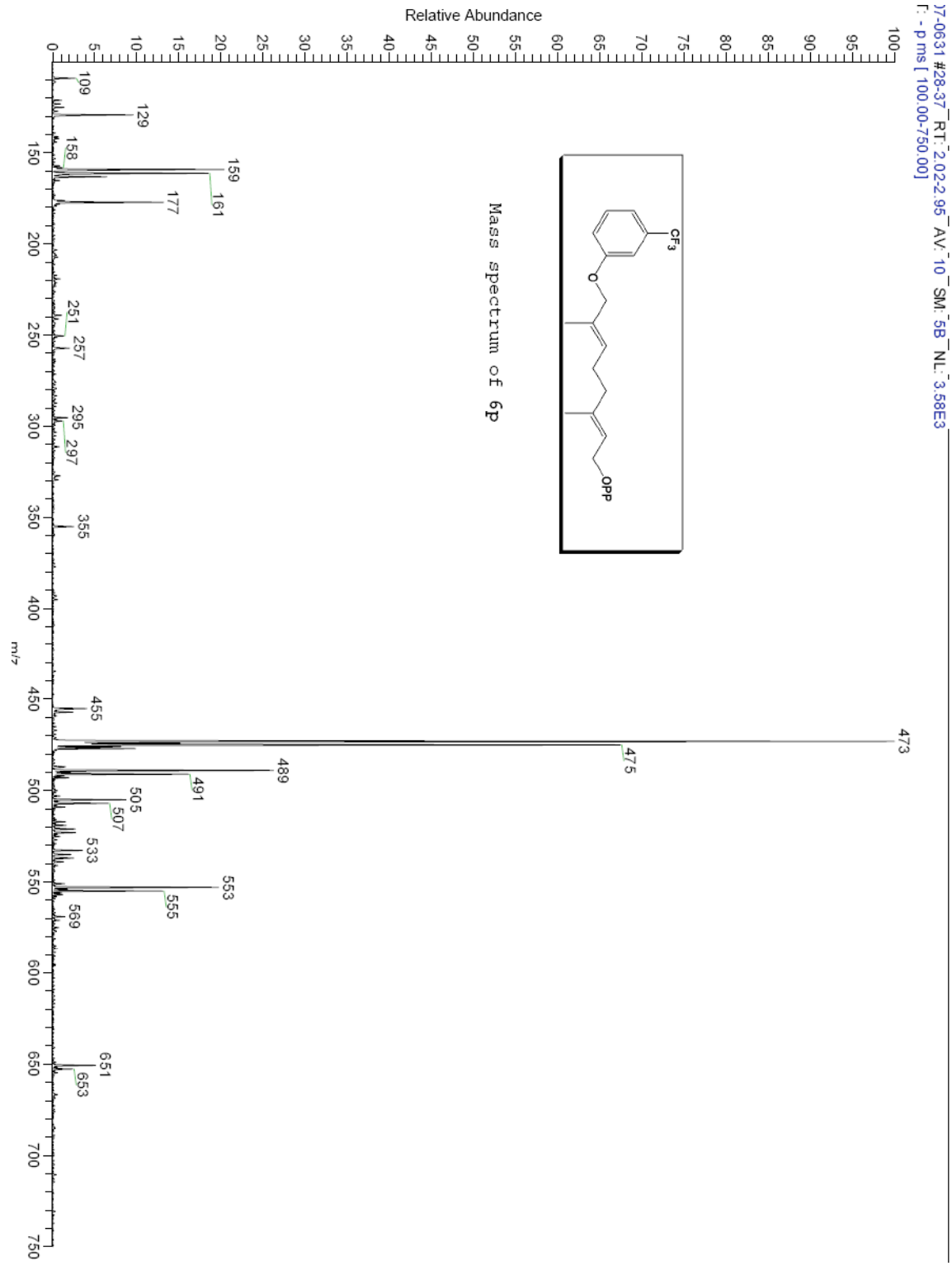
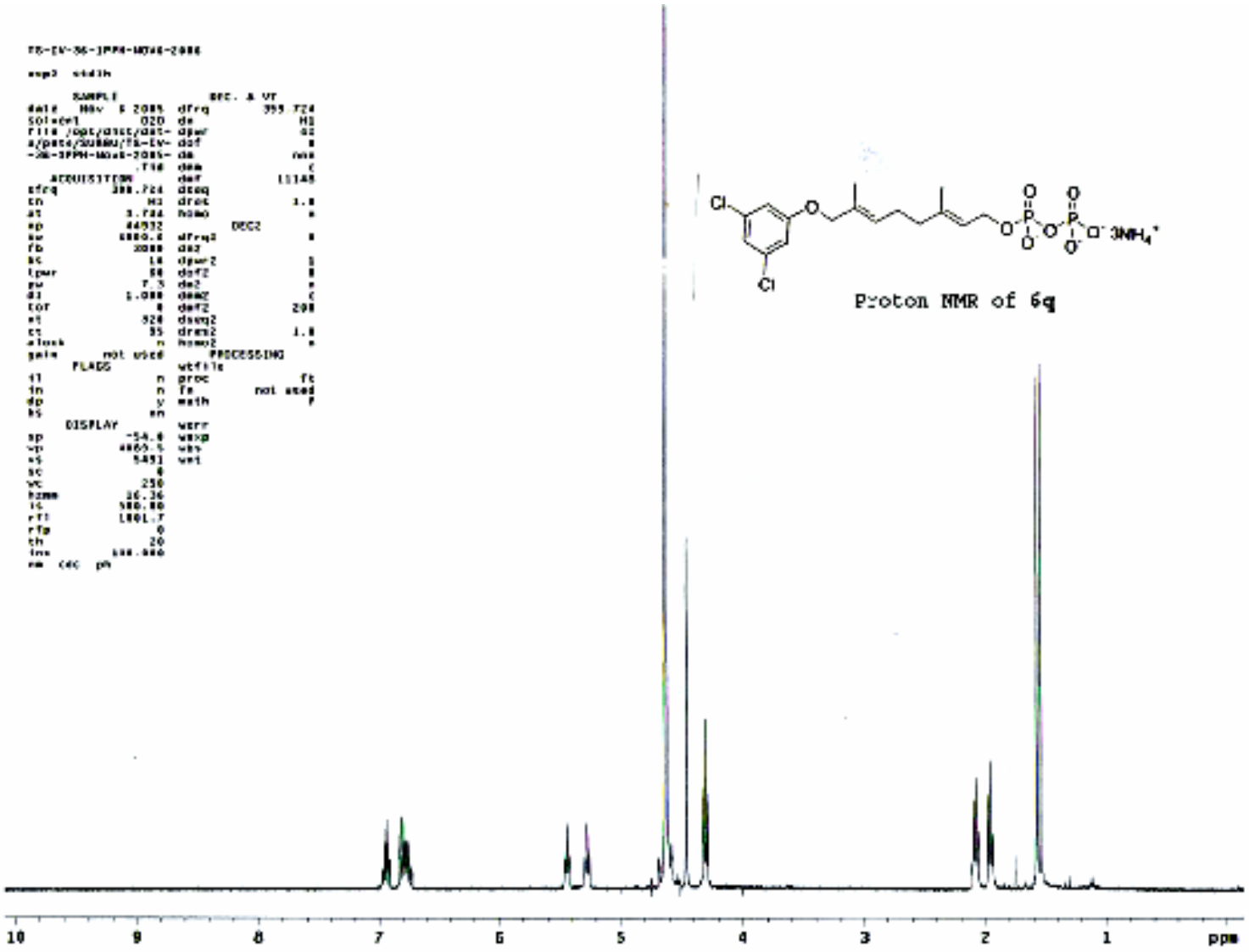
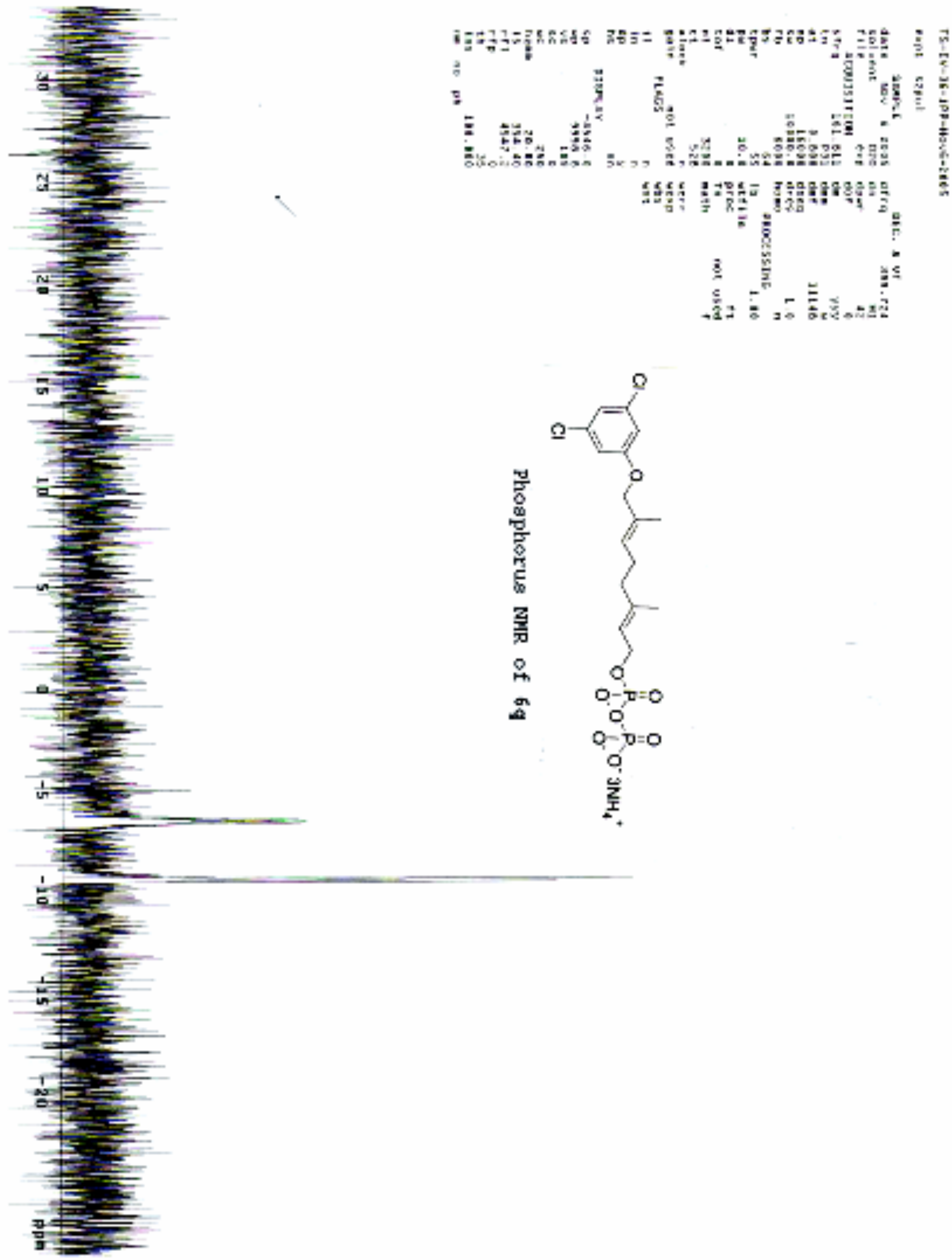
dpwr2 1
dof2 0
dm2 n
DMM2 c
dmf2 200
dseq2
drec2 1.0
homo2 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Proton NMR of 6p
10
9
8
7
6
5
4
3
2
1
ppm

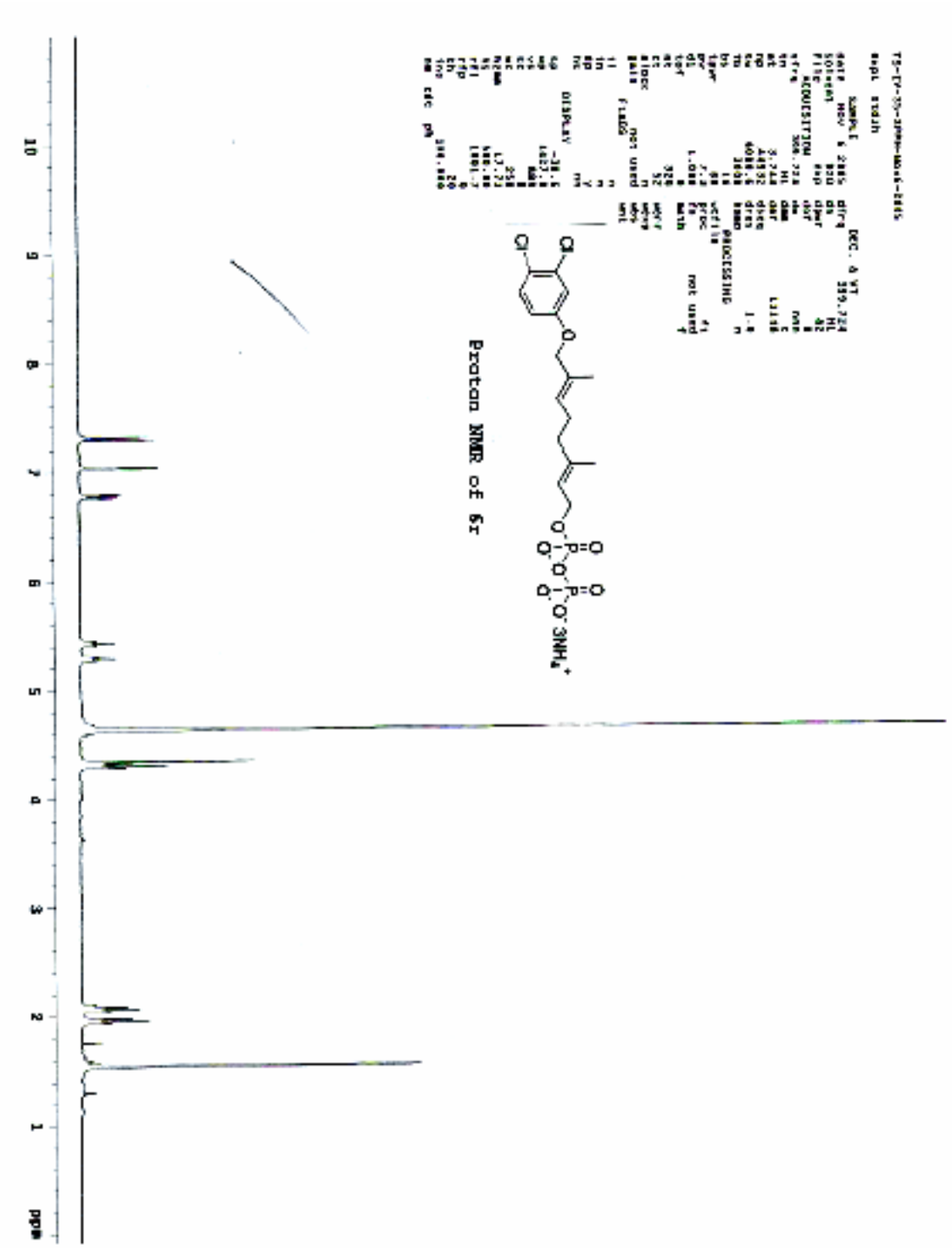
Phosphorus NMR of 6g

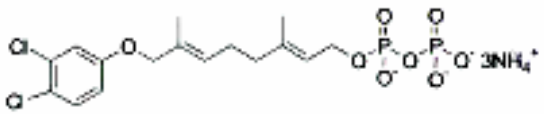

)7-0631 #28-37 RT: 2.02-2.95 AV: 10 SM: 5B NL: 3.58E3
F: - p ms [100.00-750.00]
Relative Abundance
m/z
Mass spectrum of 6p
CF3
O
OPP
109
129
158
159
161
177
251
257
295
297
355
455
473
475
489
491
505
507
533
553
555
569
651
653


FS-EV-36-1PPM-MEAS-2006
exp3 width
SAMPLE
date Nov 5 2005
solvent D2O
file /opt/dlcc/dat-a/pete/DUBBU/FS-EV-36-1PPM-MEAS-2005-.fid
ACQUISITION
sfrq 399.724
tn H1
at 3.724
np 44932
sw 6860.6
fb 2000
bs 16
tpwr 58
pw 7.5
d1 1.000
tof 0
rt 928
ct 55
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -54.8
wp 6869.5
rfl 5451
rc 0
vc 250
hzmm 16.36
is 560.80
rfl 1401.7
rfp 0
th 20
ins 100.000
ns 16 ct 80
dec. & VT
dfrq 399.724
dn H1
dpwr 62
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.8
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 298
dseq2
dres2 1.8
homo2 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Cl
O
O
P
O
O
P
O
O
Cl
3NH4+
Proton NMR of 6q
10
9
8
7
6
5
4
3
2
1
ppm

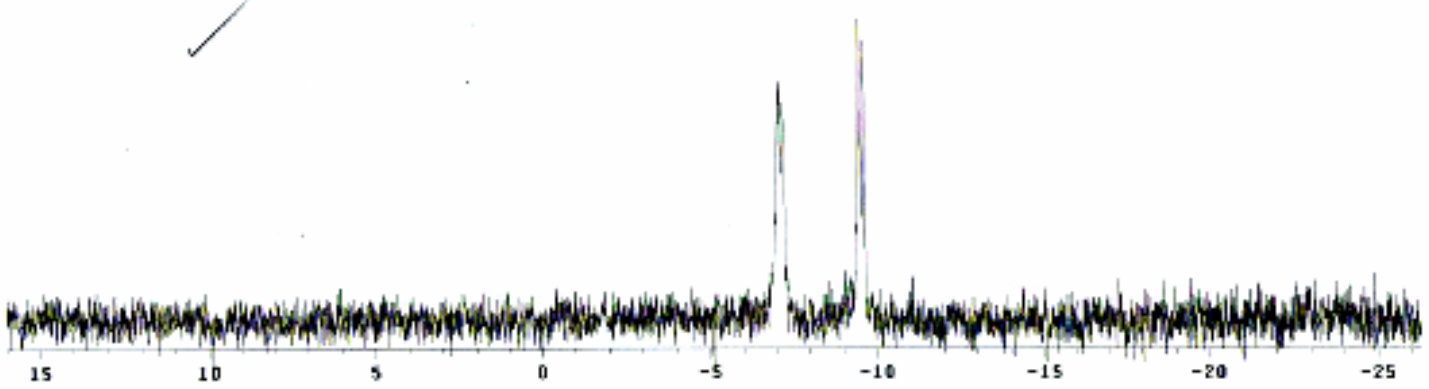


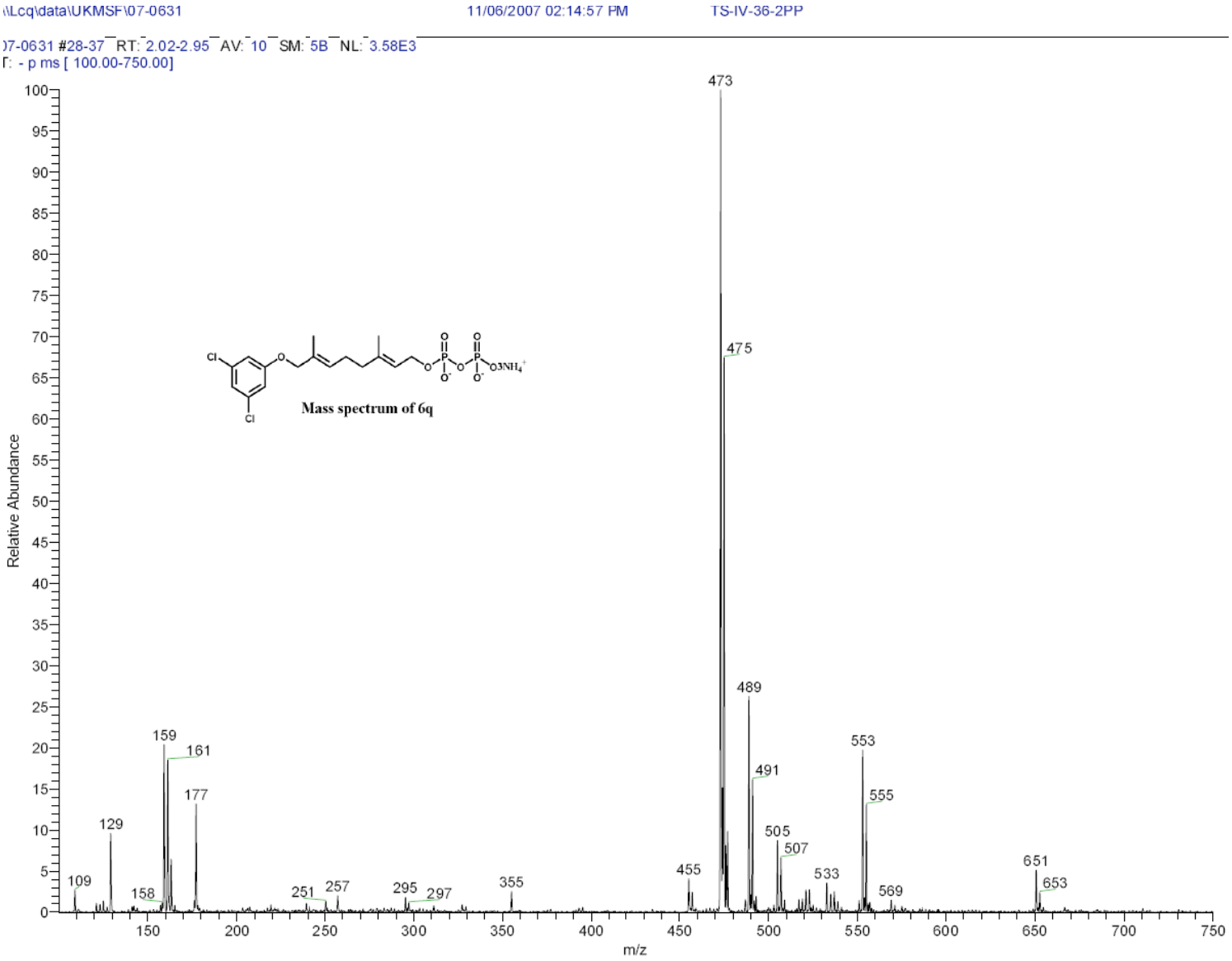
Phosphorus NMR of 6g

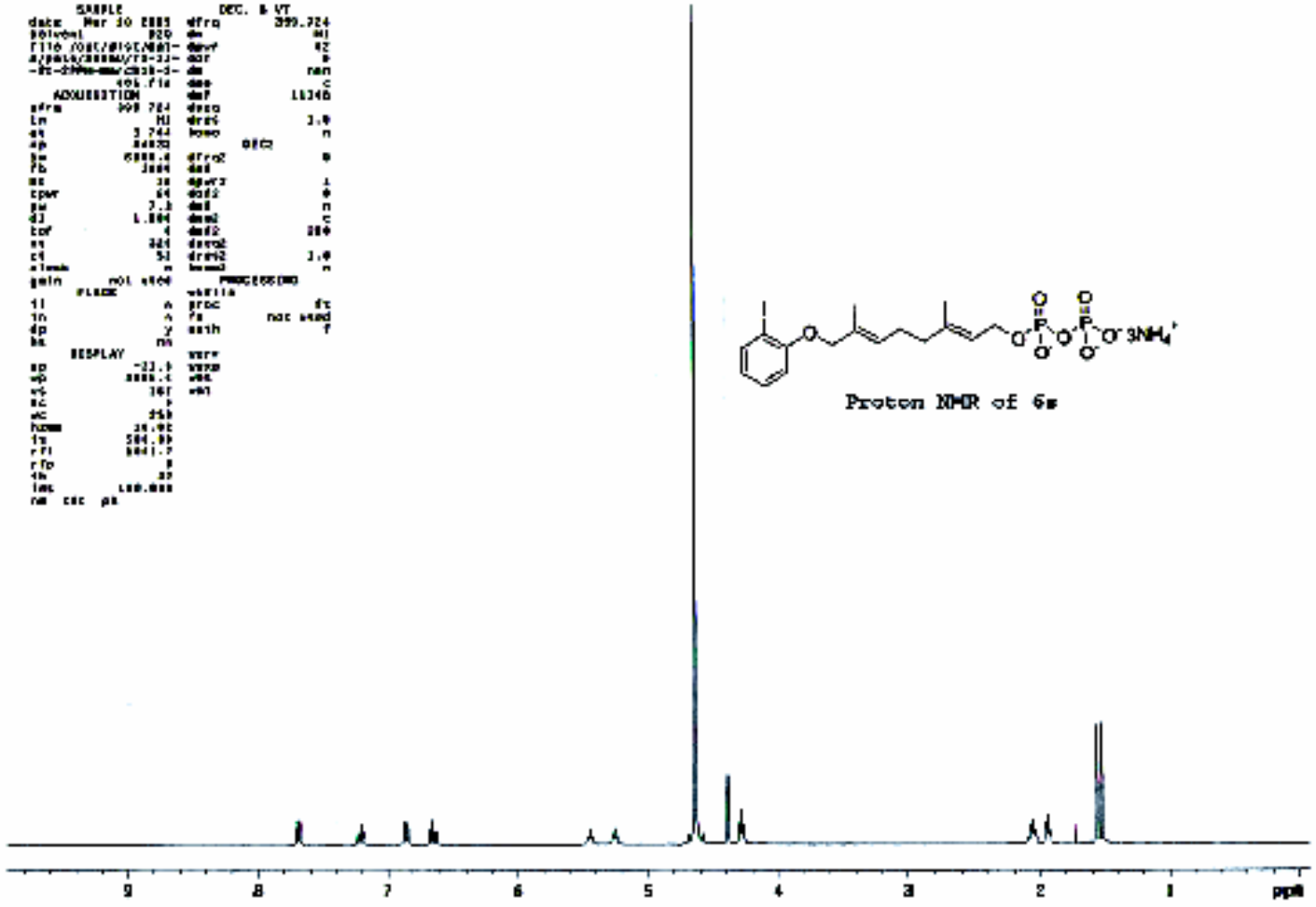

Proton NMR of 6r



Phosphorus NMR of 6r




\Lcq\data\UKMSF\07-0631
11/06/2007 02:14:57 PM
TS-IV-36-2PP
)7-0631 #28-37 RT: 2.02-2.95 AV: 10 SM: 5B NL: 3.58E3
Γ: - p ms [100.00-750.00]
Cl
Cl
O
O
O
O
O
O
O
O3NH4+
Mass spectrum of 6q
Relative Abundance
m/z
473
475
489
491
505
507
533
553
555
569
651
653
455
355
295
297
251
257
159
161
177
129
109
158

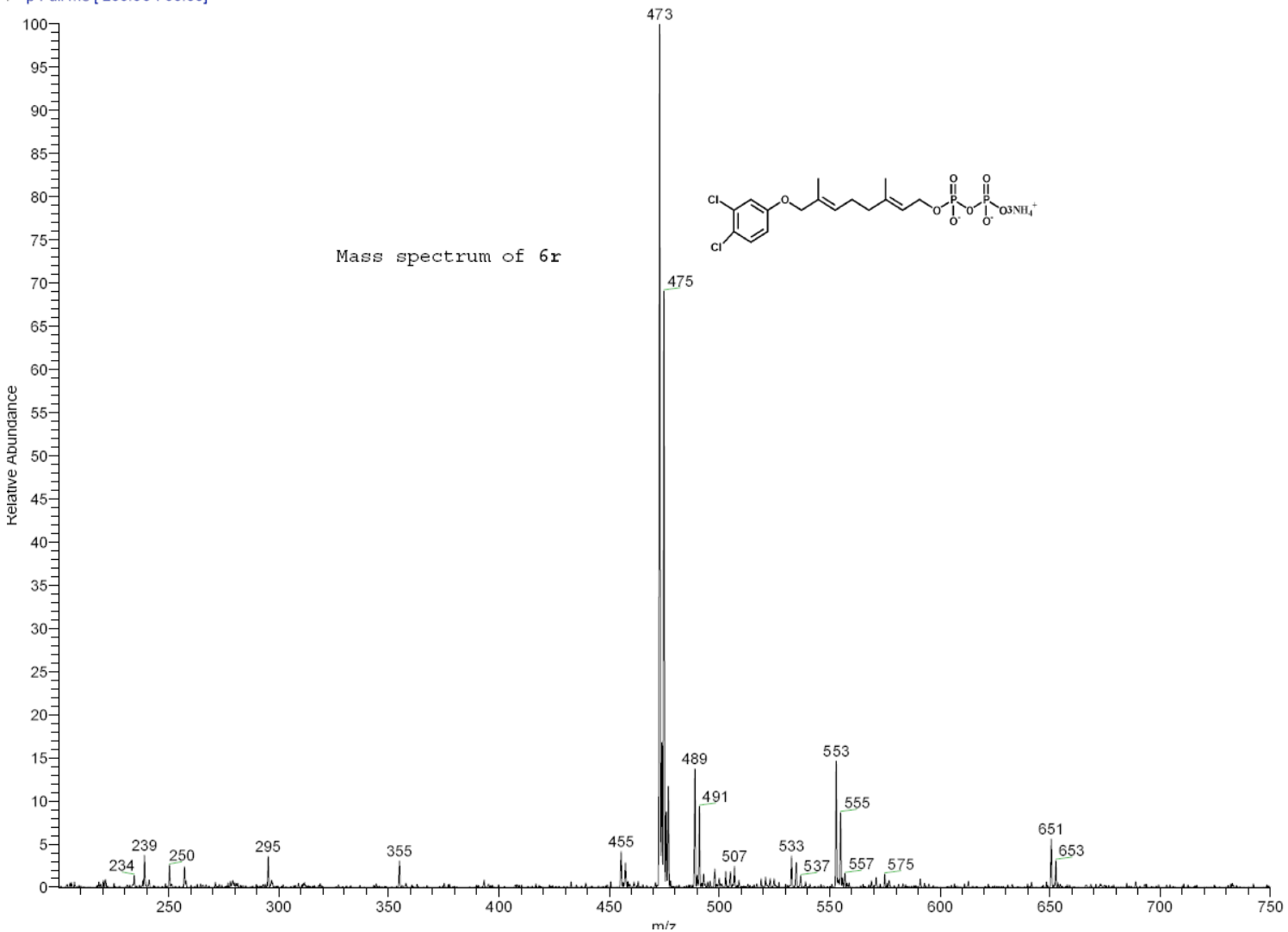


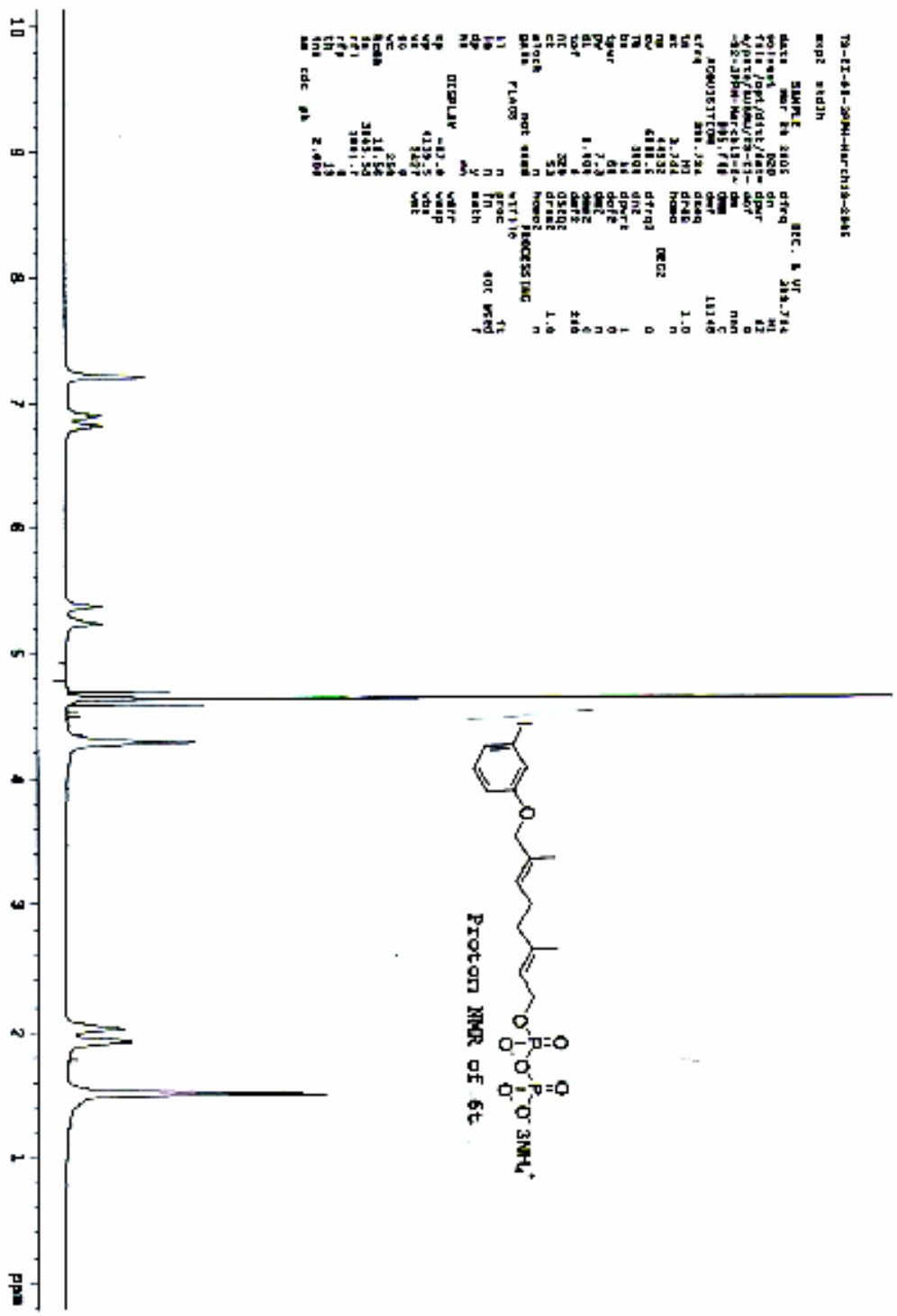
Proton NMR of 6a

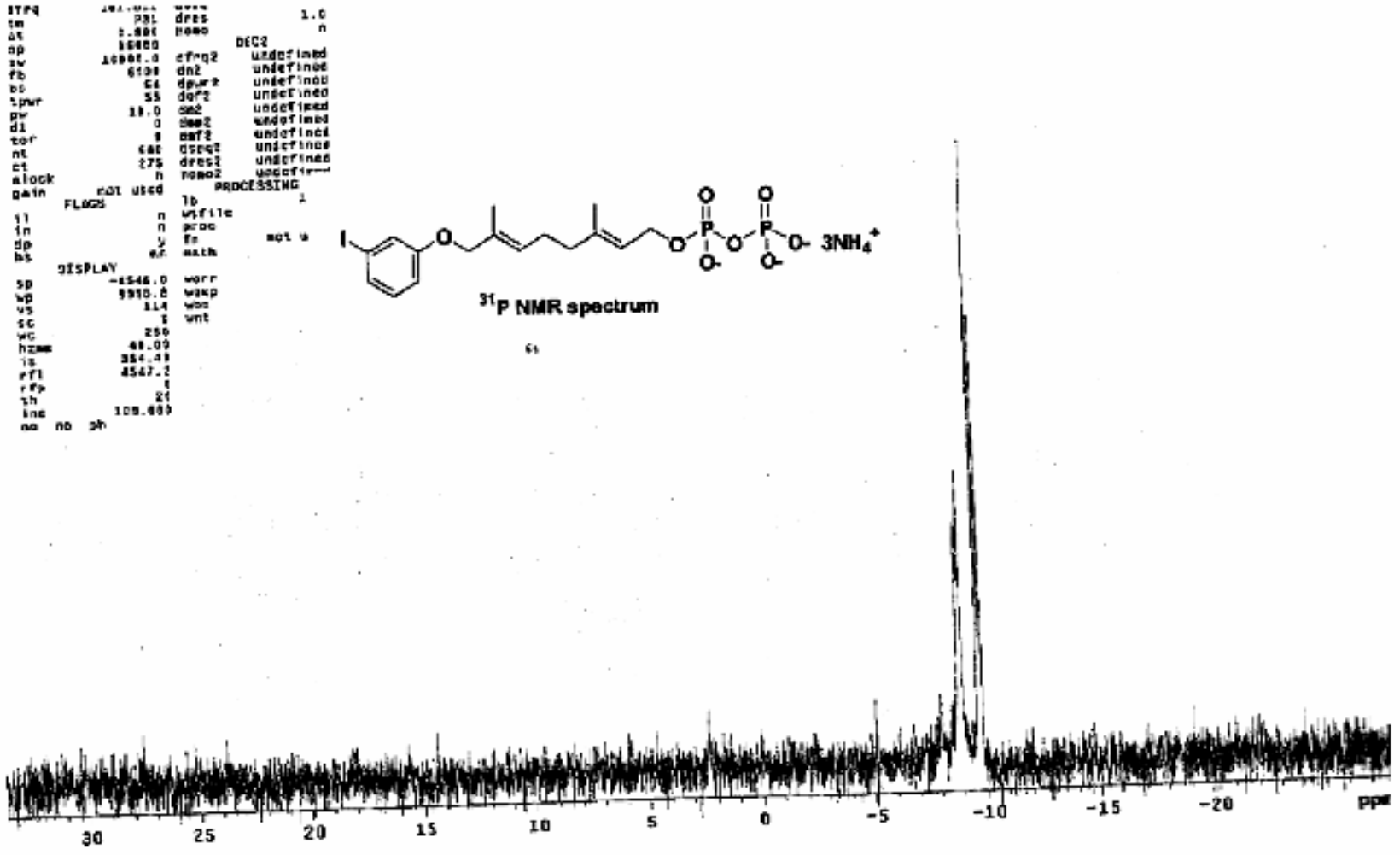
Phosphorus NMR of 6a

Lcq\data\UKMSF\07-0629 11/06/2007 12:14:59 PM TS-IV-35-3PP

7-0629 #17-24 RT: 1.69-2.40 AV: 8 SM: 5B NL: 3.36E3
: - p Full ms [200.00-750.00]

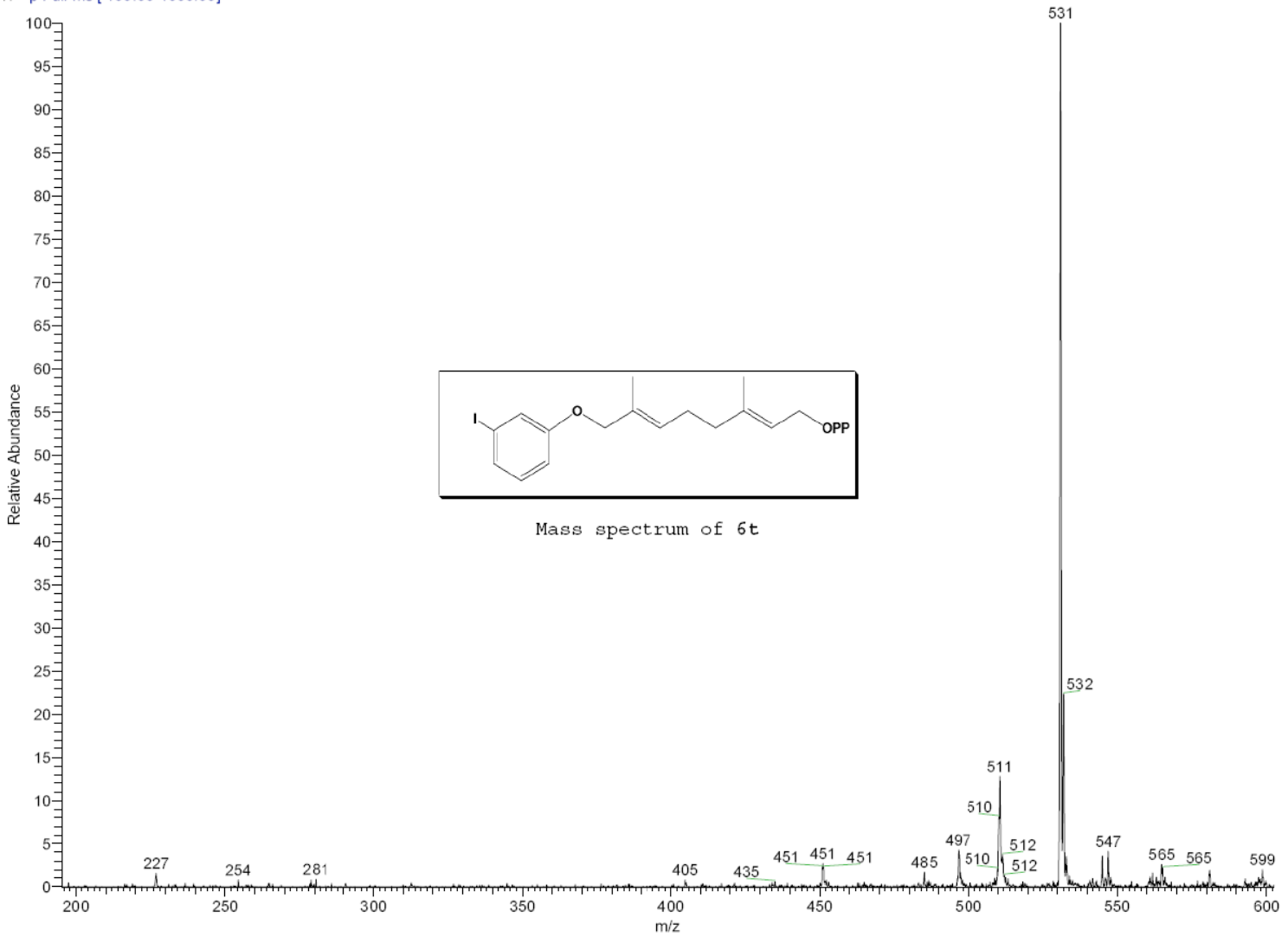



Proton NMR of 6t
10
9
8
7
6
5
4
3
2
1
ppm


^{31}P NMR spectrum
3NH4+
ppm
30
25
20
15
10
5
0
-5
-10
-15
-20

C:\Xcalibur\data\UKMSF\05-0335 03/28/2005 09:45:53 AM TS-II-92-3

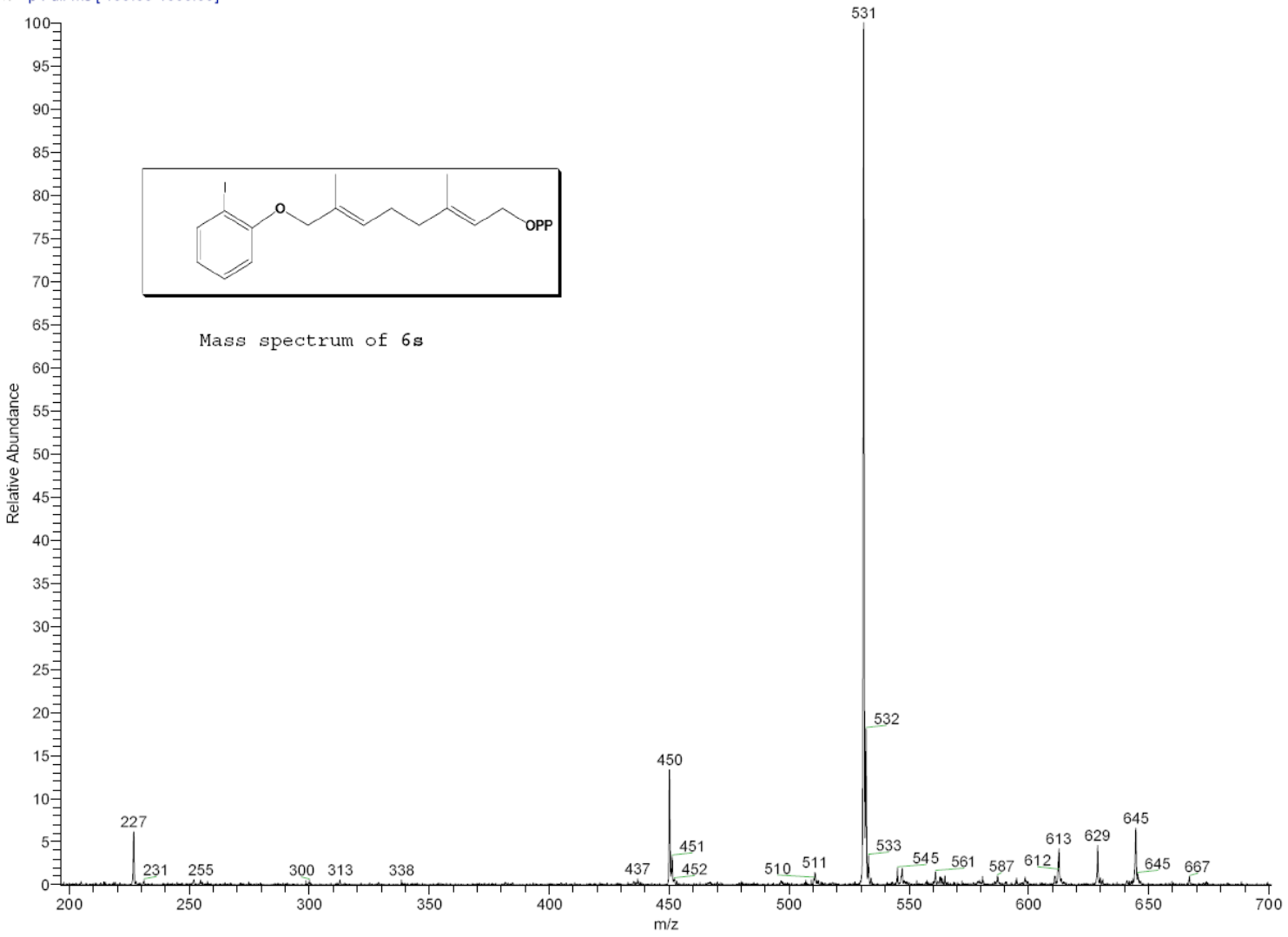
05-0335 #18-25 RT: 0.50-0.72 AV: 8 NL: 8.24E4
T: - p Full ms [100.00-1000.00]

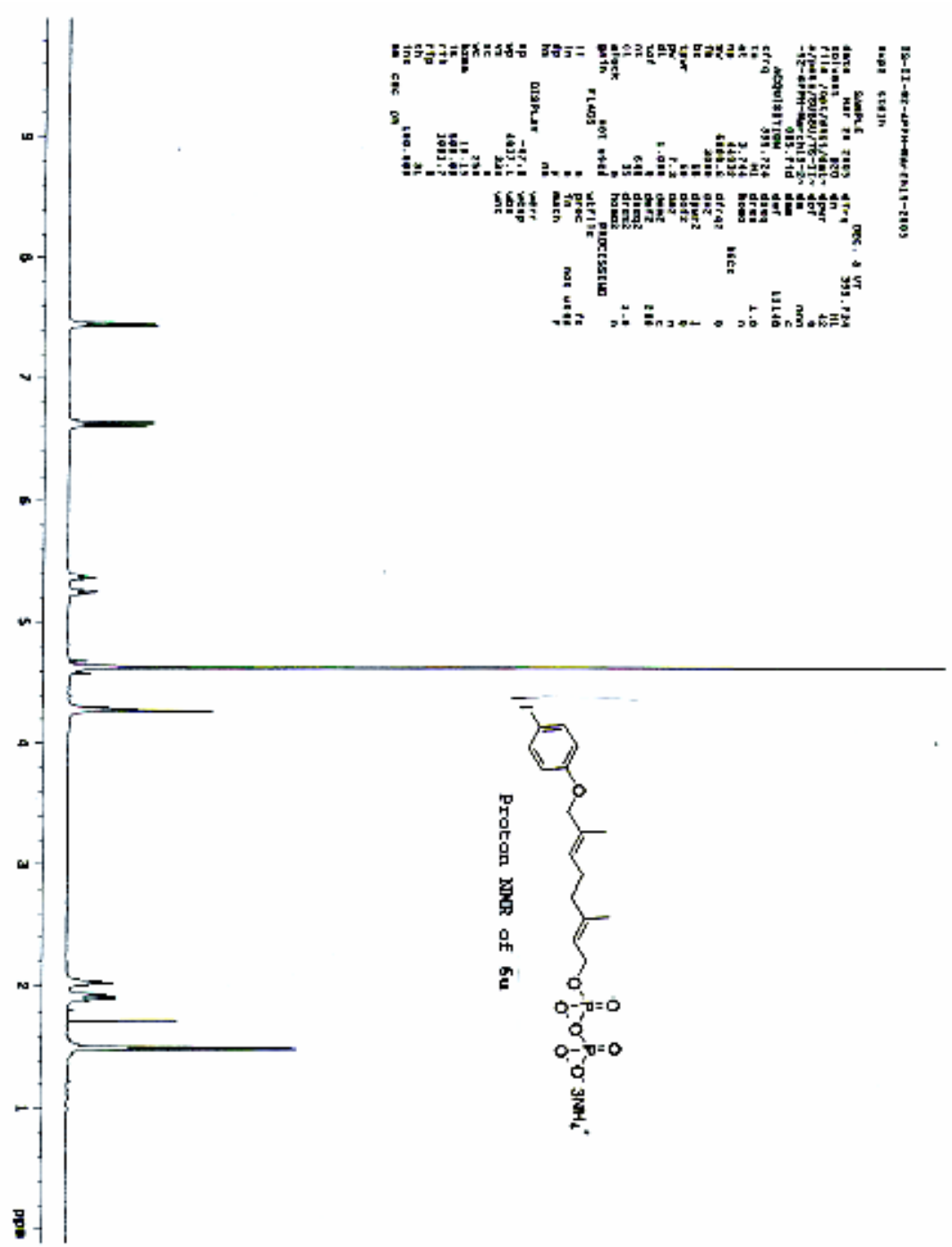


Mass spectrum of 6t

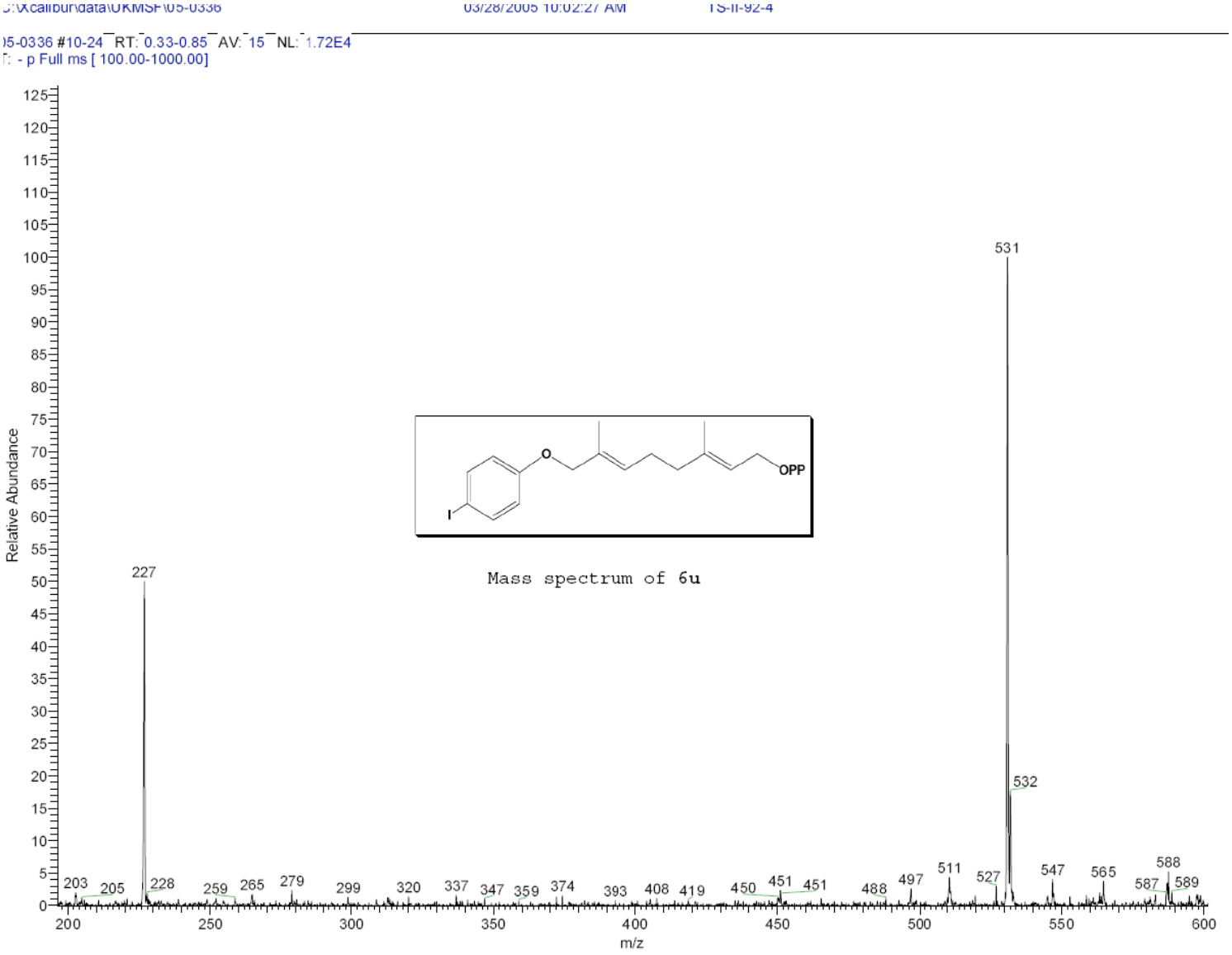
C:\Xcalibur\data\UKMSF\05-0334 03/28/2005 09:18:28 AM TS-II-92-2

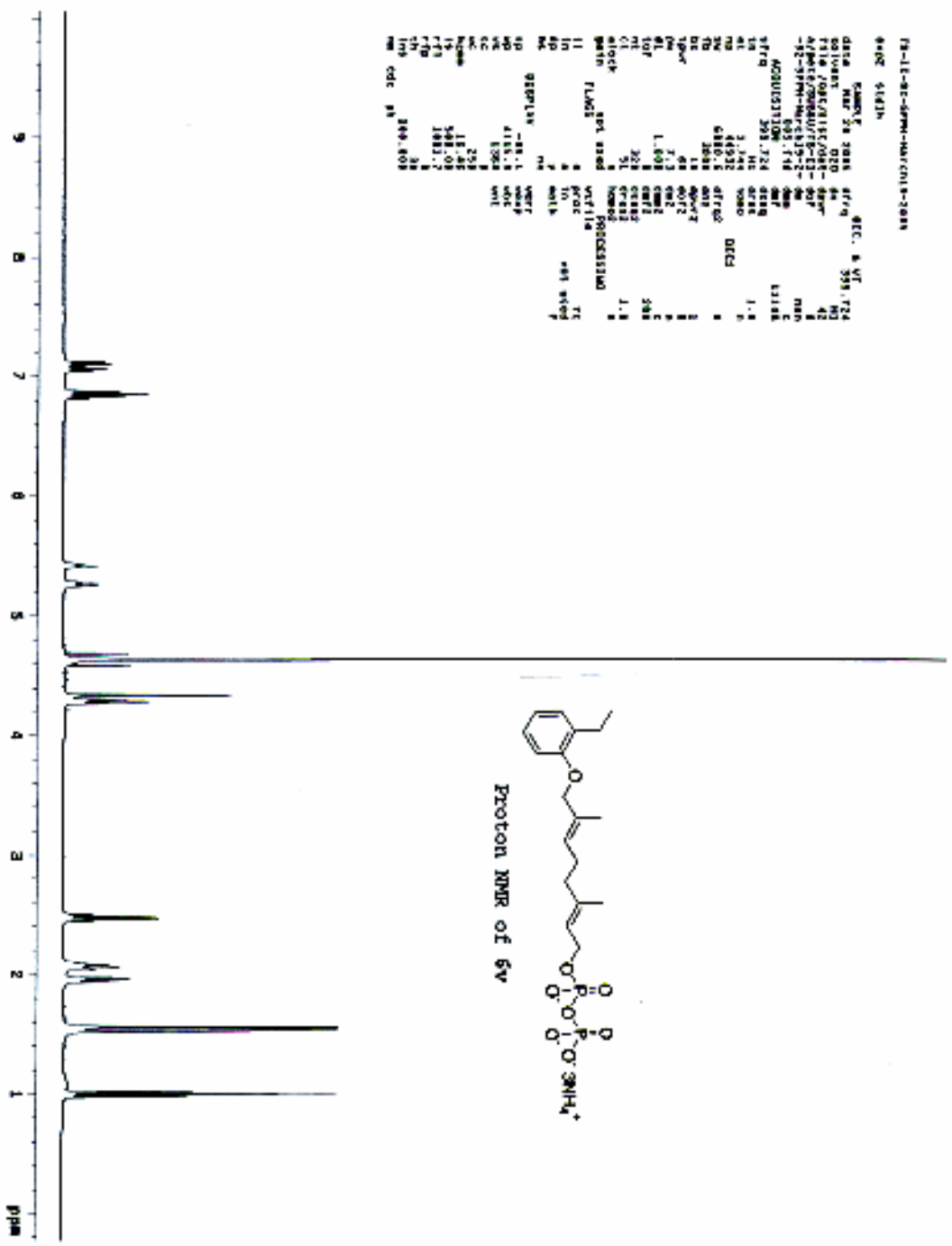
05-0334 #41-62 RT: 1.38-2.23 AV: 22 NL: 4.03E4
T: - p Full ms [100.00-1000.00]



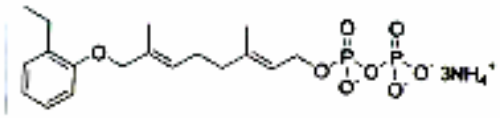

Proton NMR of 6u

Phosphorus NMR of 6a

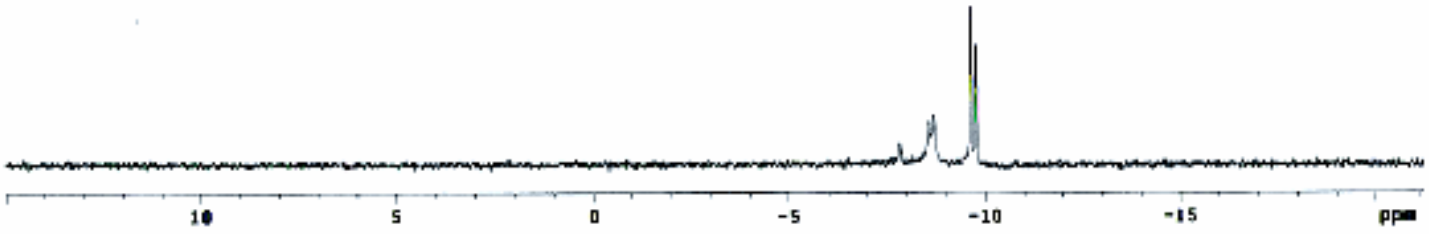

C:\Xcalibur\data\UKMSF\05-0336 03/28/2005 10:02:27 AM TS-II-92-4
05-0336 #10-24 RT: 0.33-0.85 AV: 15 NL: 1.72E4
T: - p Full ms [100.00-1000.00]
Relative Abundance
m/z
O
OPP
I
227
531
532
Mass spectrum of 6u



Proton NMR of 6y

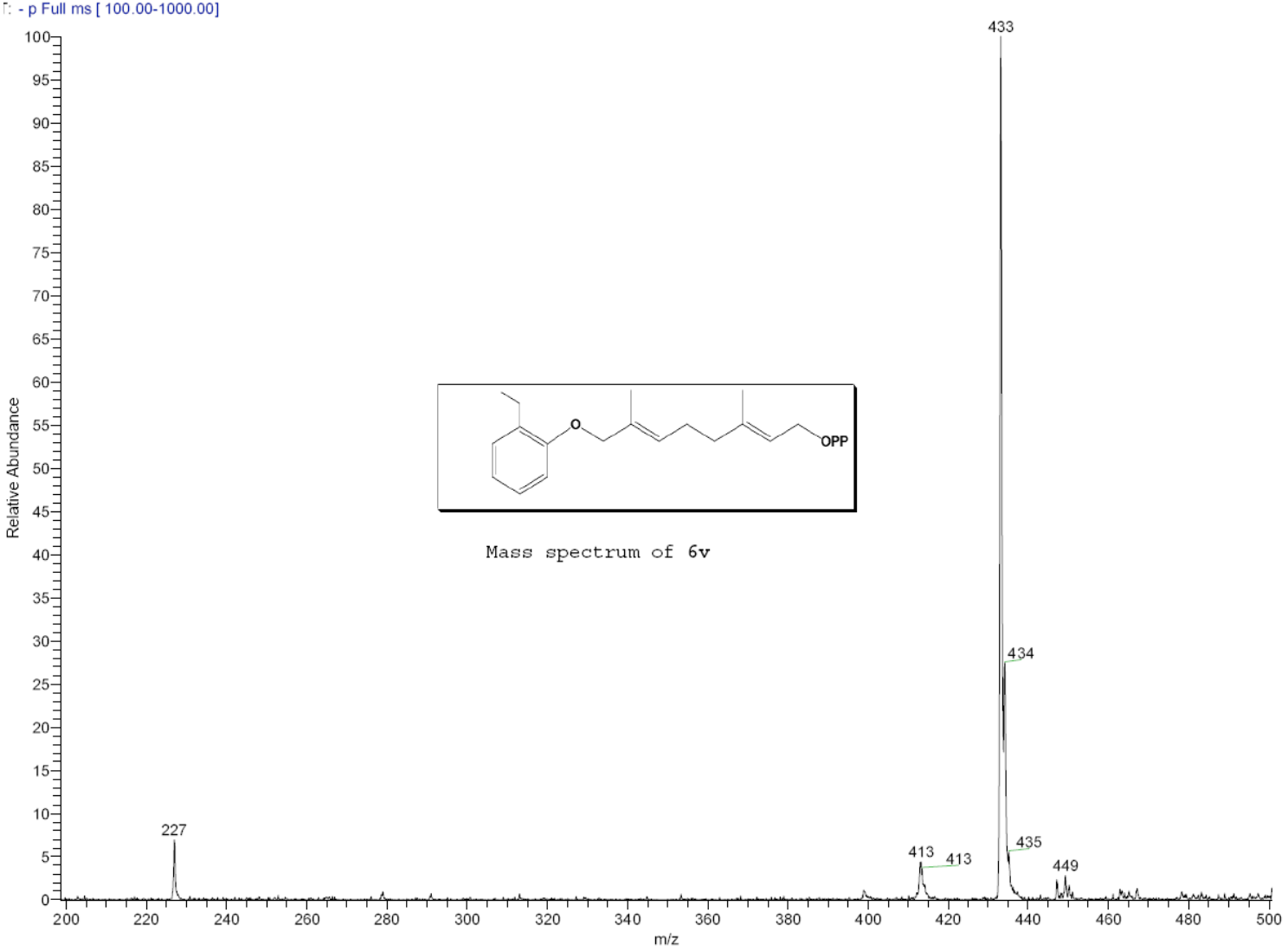


Phosphorus NMR of 6v

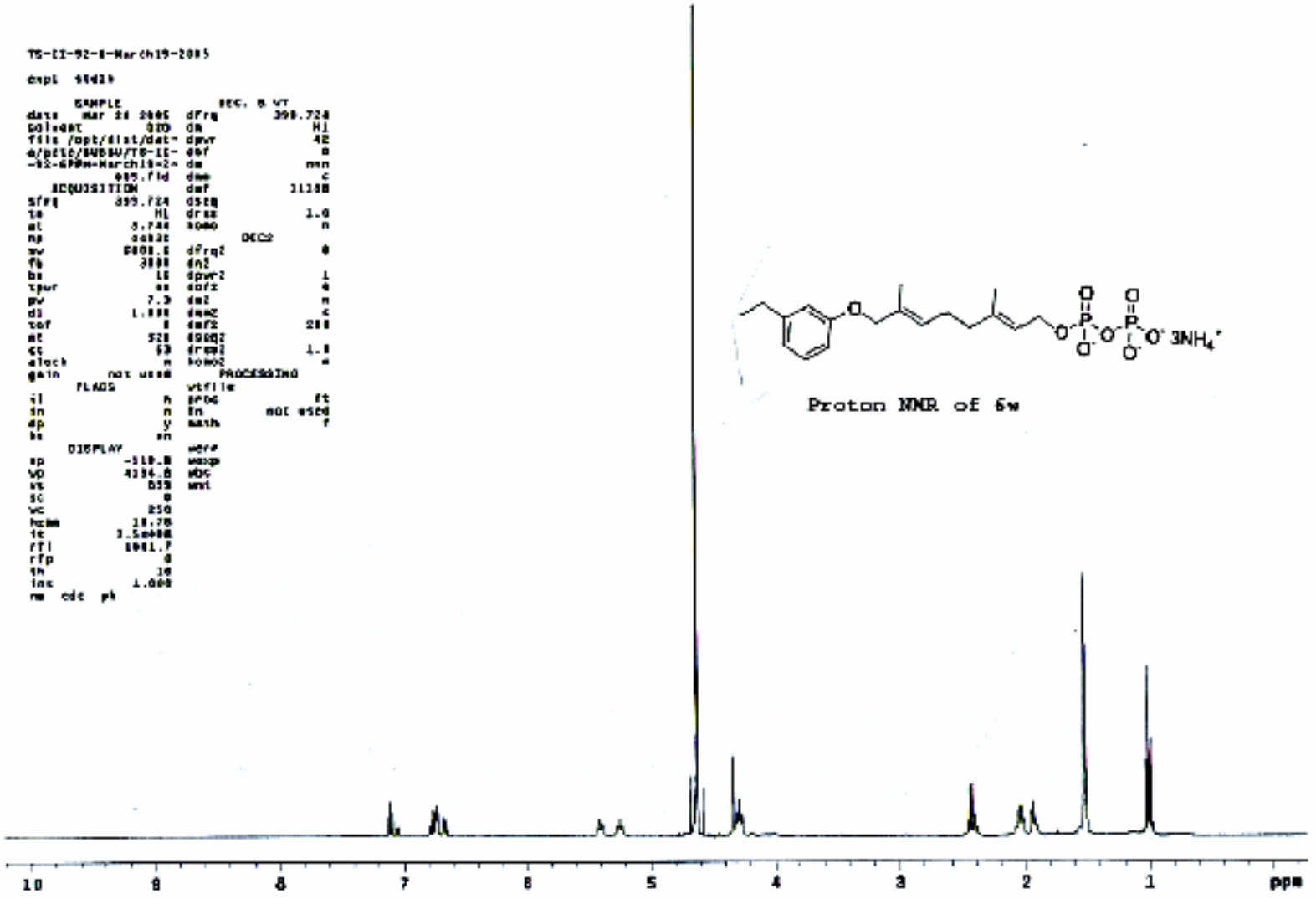


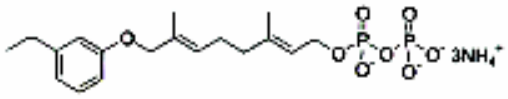
C:\Xcalibur\data\UKMSF\05-0337 03/28/2005 10:20:48 AM TS-II-92-5

05-0337 #13-22 RT: 0.55-0.94 AV: 10 NL: 5.09E4
T: - p Full ms [100.00-1000.00]

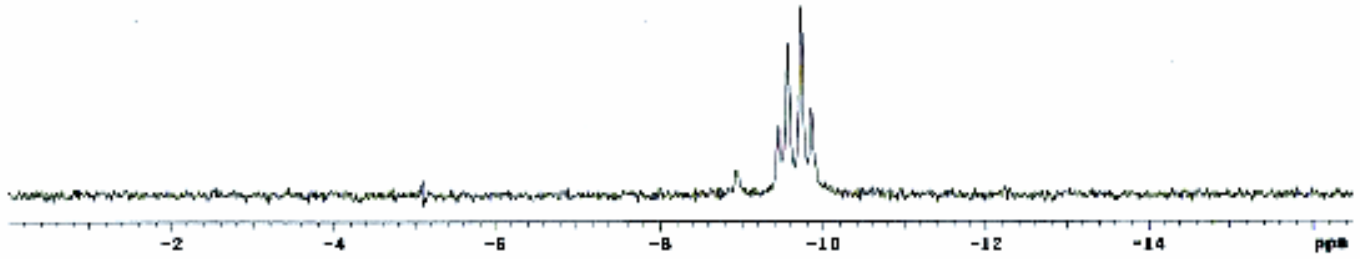


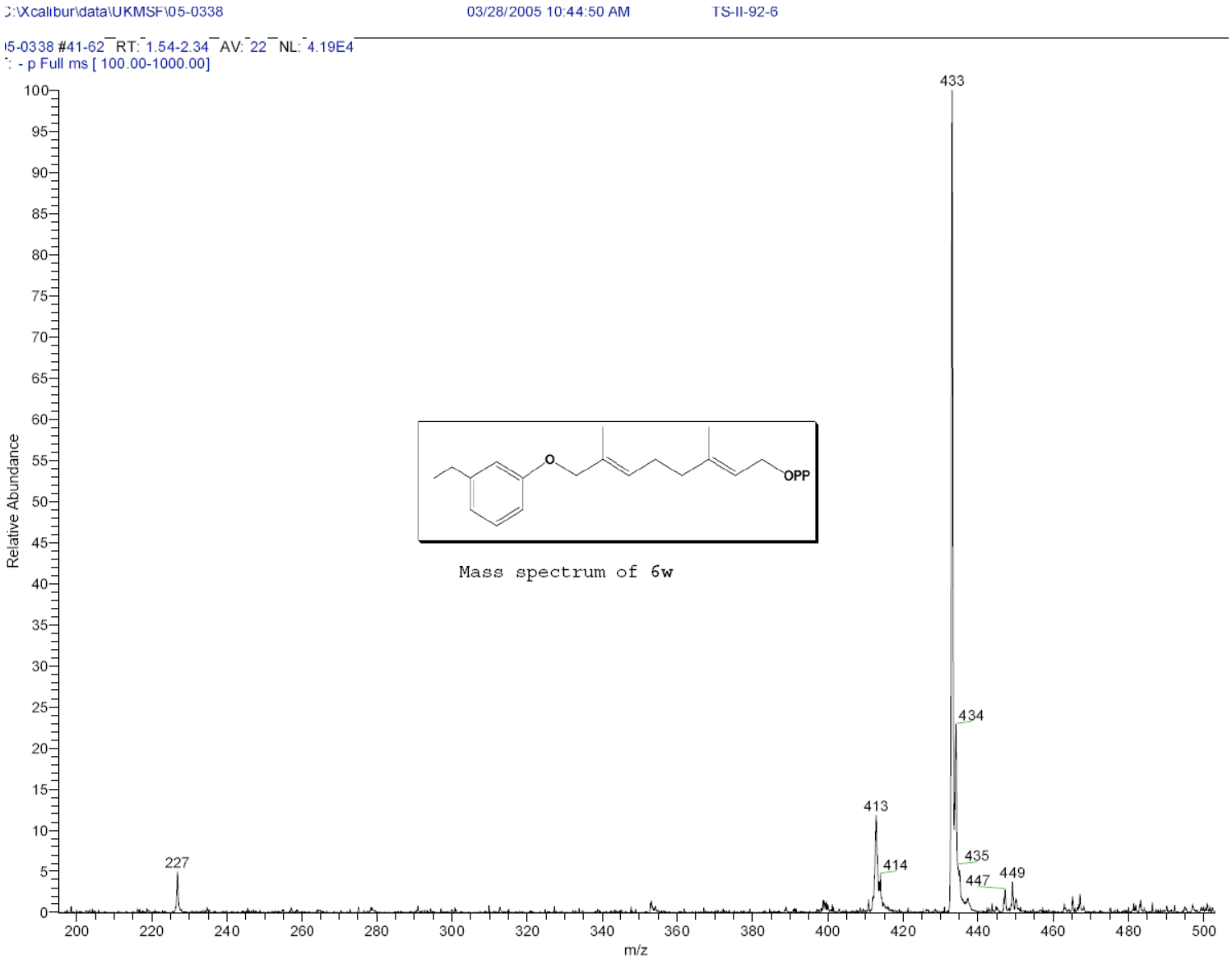
Mass spectrum of 6v


TS-II-92-6-March19-2015
expt 5603.h
SAMPLE
date Mar 19 2015
solvent DMSO
file /opt/dlat/dat-a/pdic/DMSO/TS-II-92-6PPm-March19-2^
005.fid
ACQUISITION
sfrq 399.724
tn H1
at 5.734
np 64132
sw 8008.0
fb 3898
bs 16
tpwr 46
pw 7.3
d1 1.000
tof 0
nt 528
ct 63
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -110.8
wp 4194.8
sc 0
vc 250
hzcm 10.78
fc 1.5e+08
rfl 1981.7
rfp 0
th 10
lnx 1.000
nm cdc pk
DEC. 8 VT
dfrq 398.724
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11188
dseq
drss 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 288
dseq2
drss2 1.8
homo2 n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Proton NMR of 6w
3NH4+
10
9
8
7
6
5
4
3
2
1
ppm

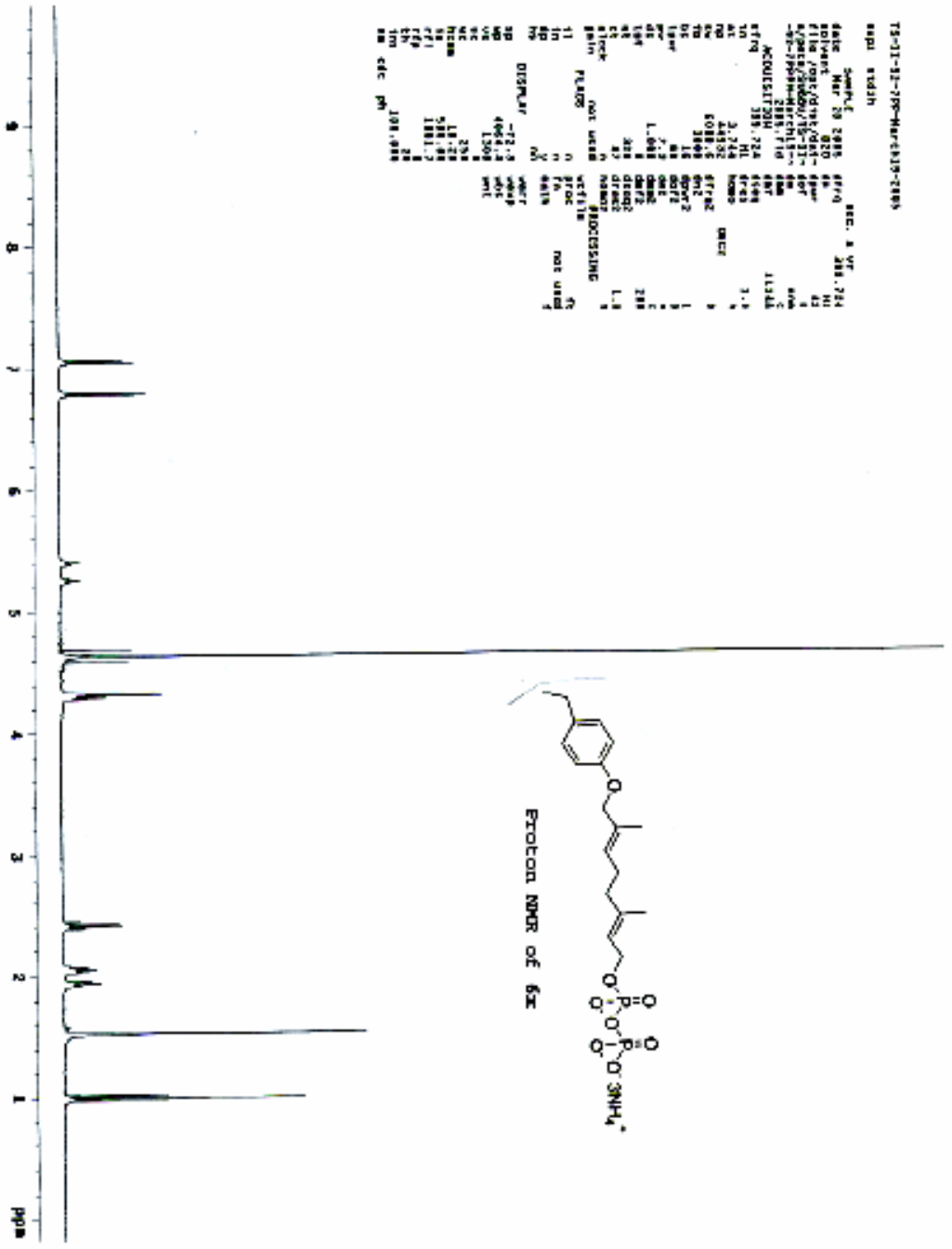


Phosphorus NMR of 6w

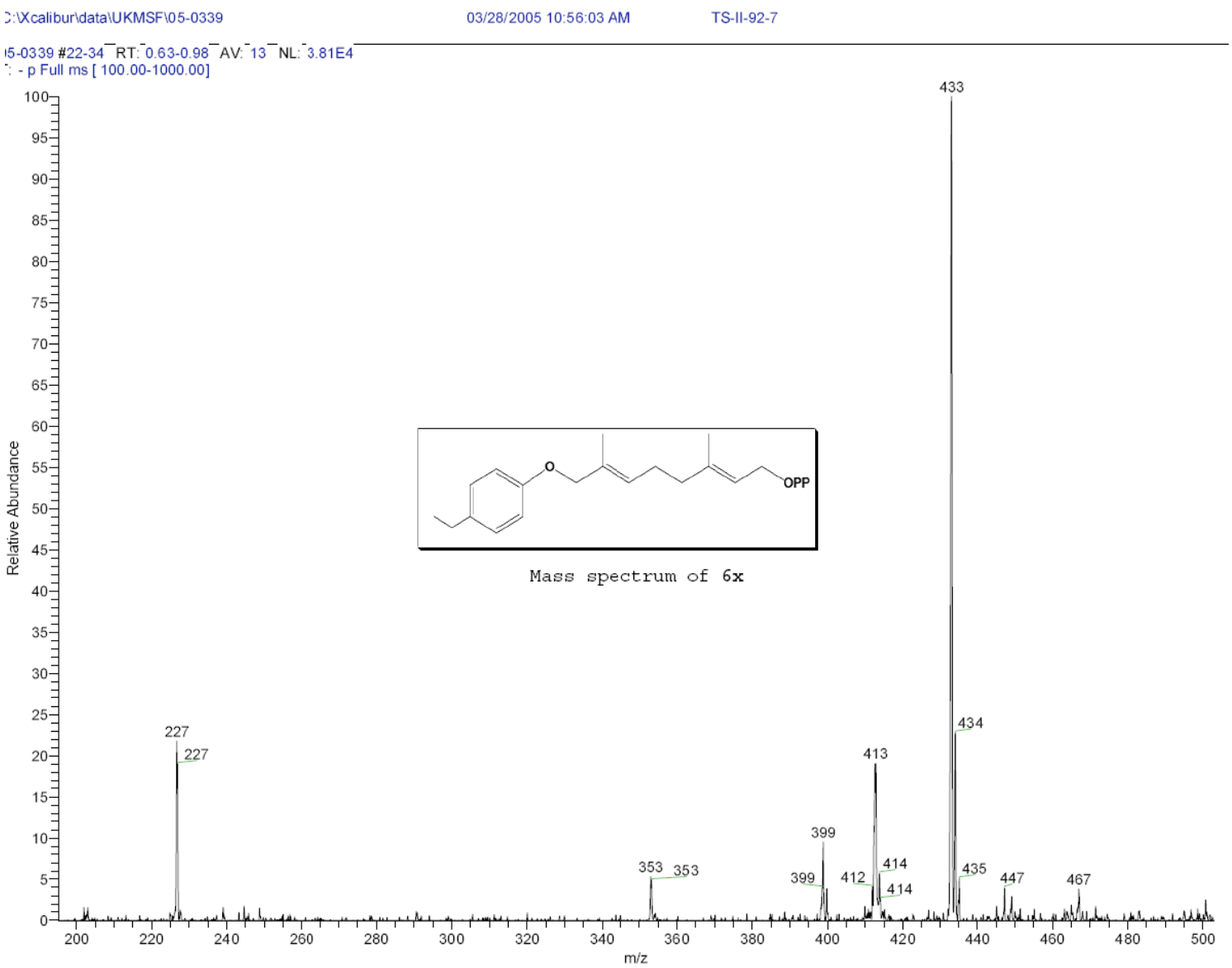




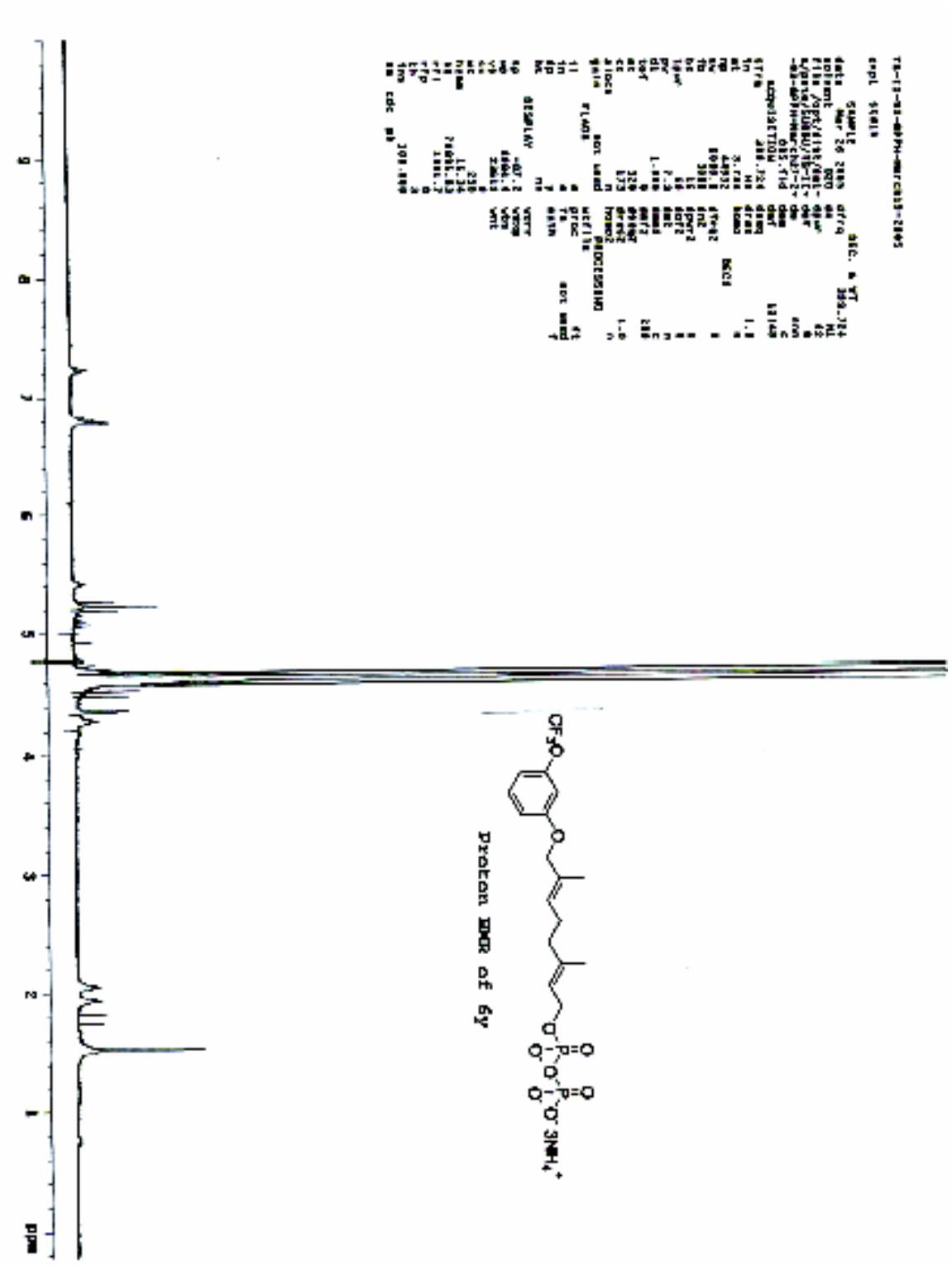
Mass spectrum of 6w

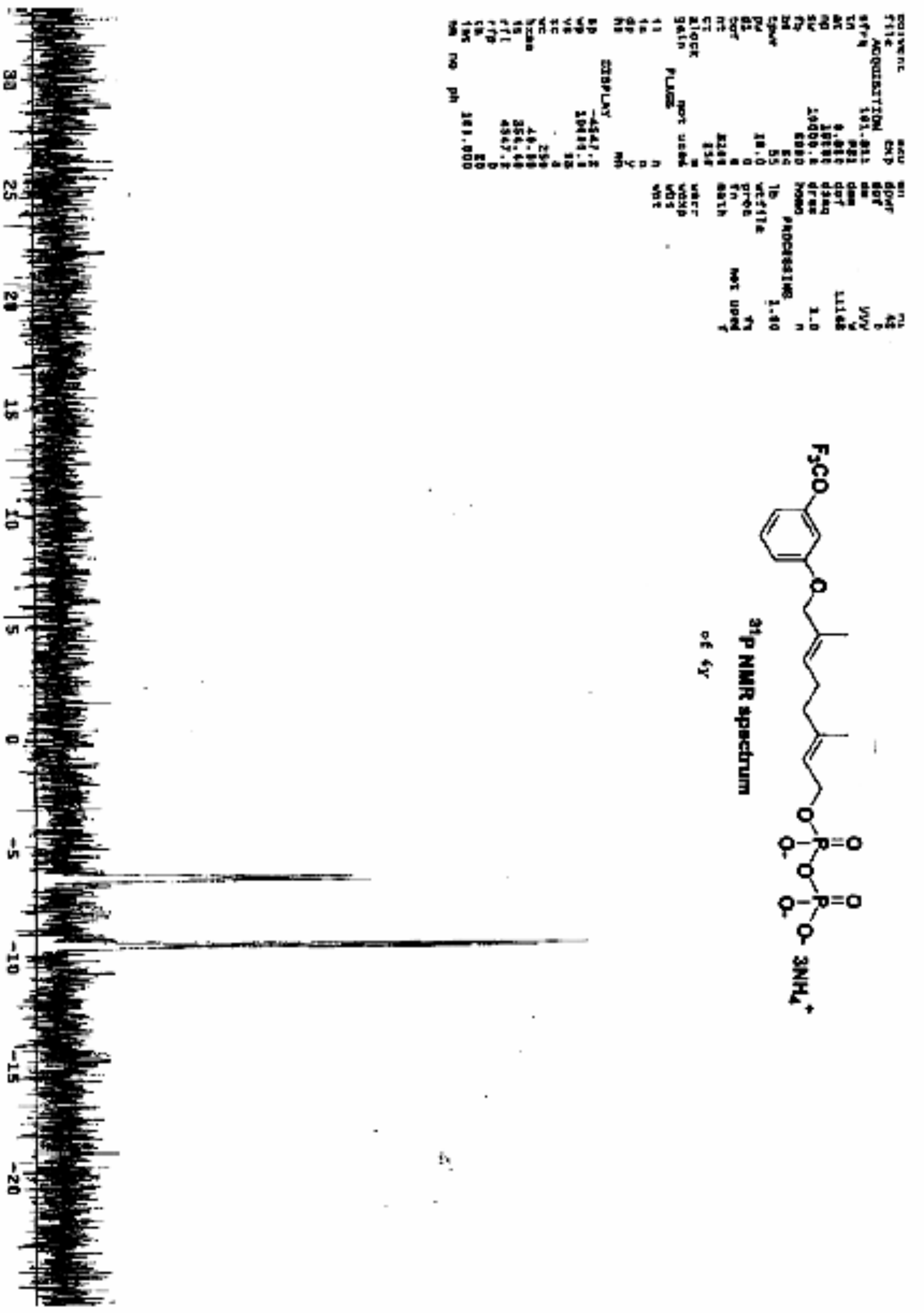

Proton NMR of 6x
NH4+

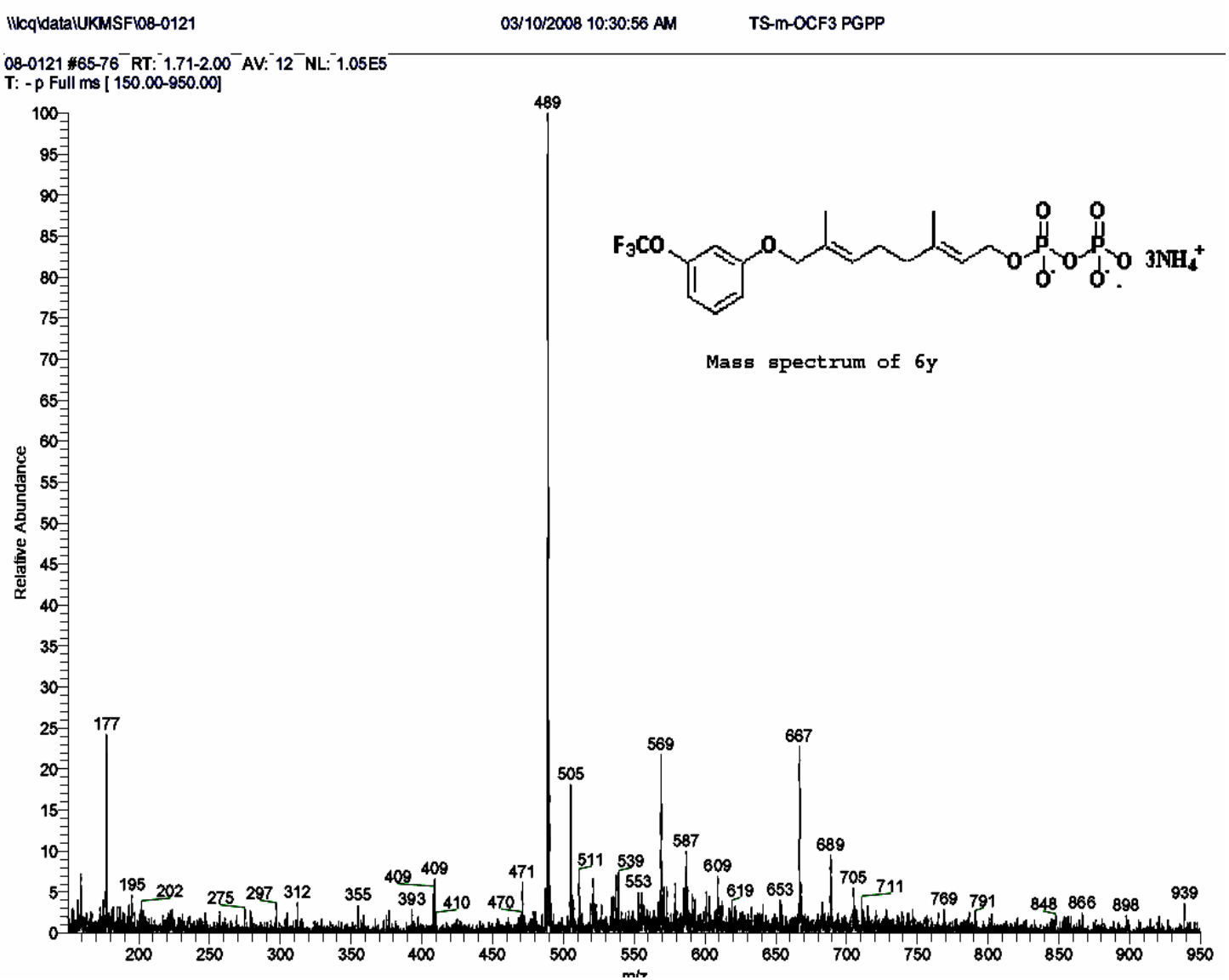
Phosphorus NMR of 6x
3NH4



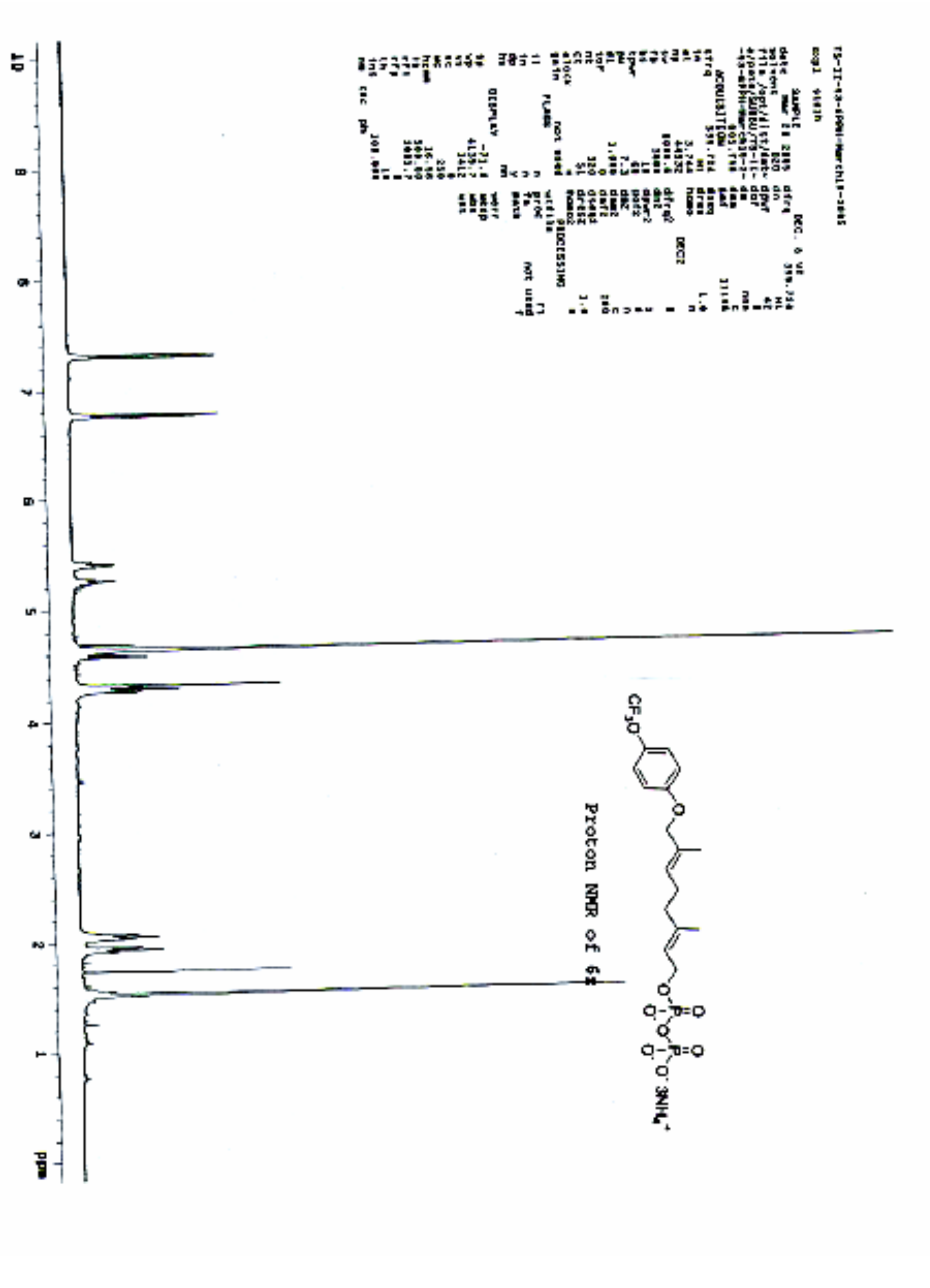
Mass spectrum of **6x**

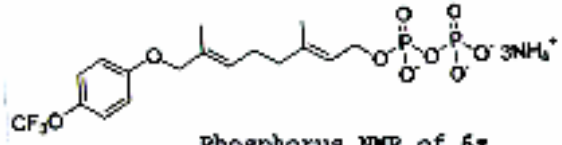

Proton NMR of 6y
CF_3O
$3NH_4^+$


F3CO
3NH4+
31P NMR spectrum
of 4y
30
25
20
15
10
5
0
-5
-10
-15
-20

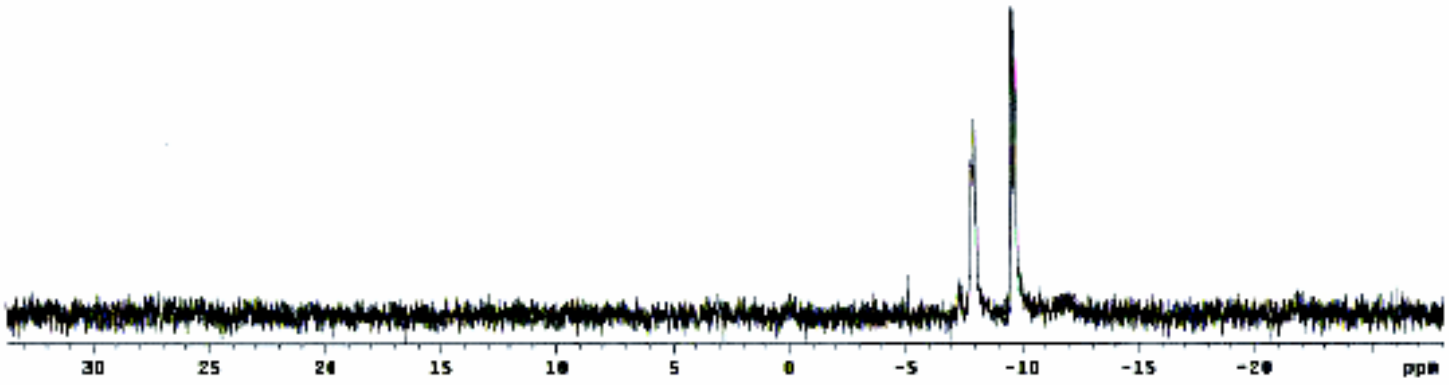


Mass spectrum of 6y


Proton NMR of 62

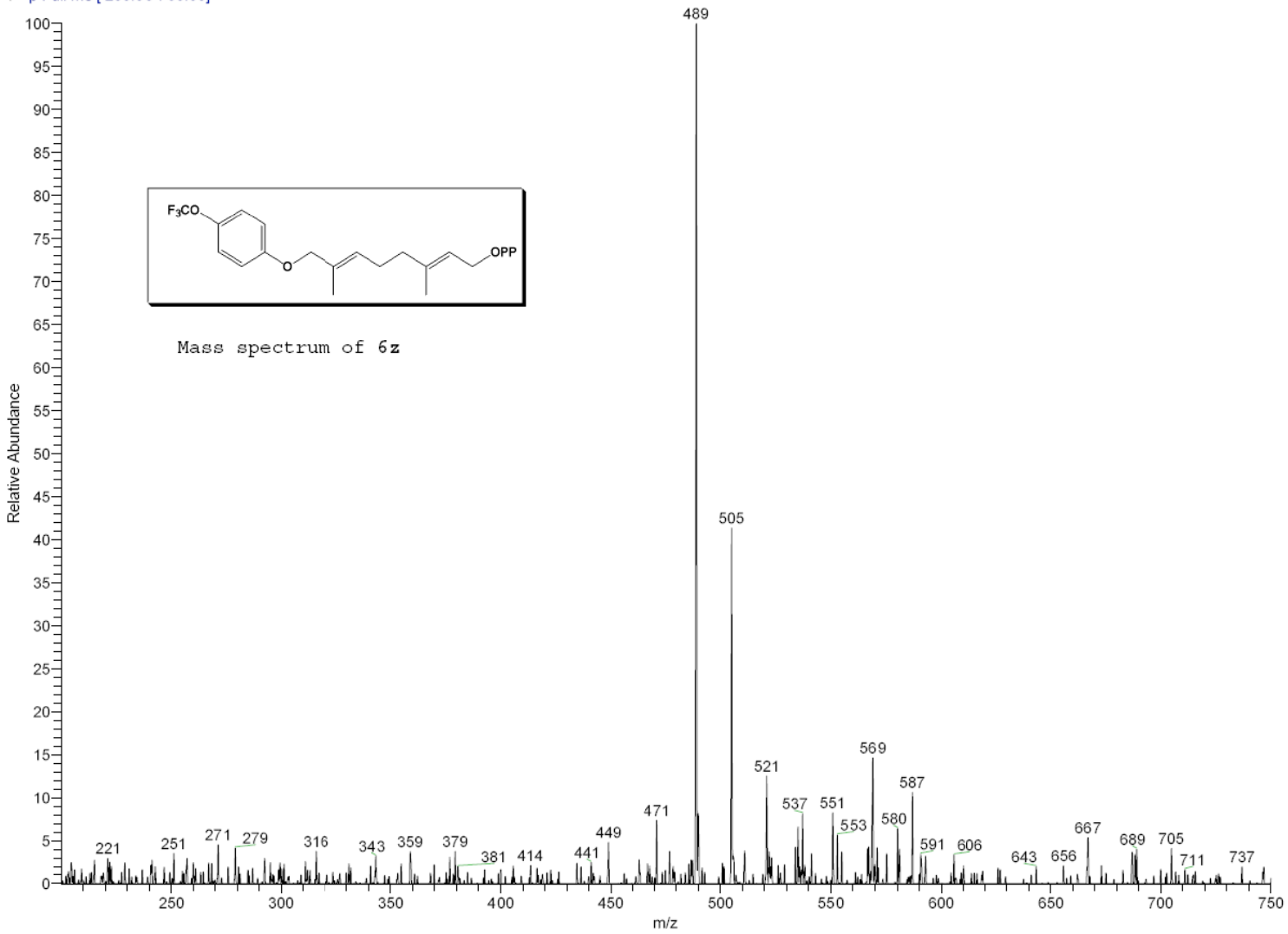


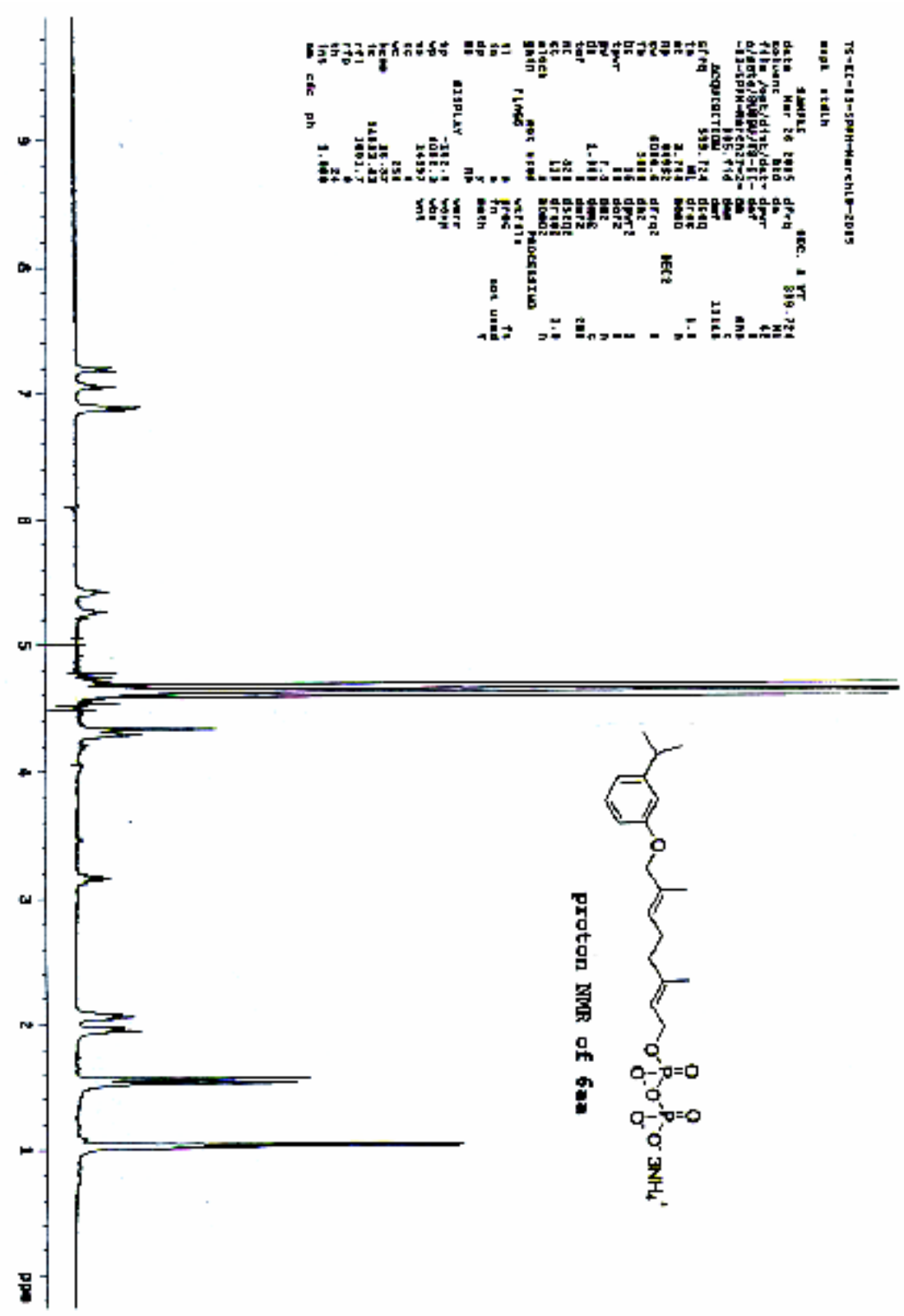
Phosphorus NMR of 6z



\Lcq\data\UKMSF\07-0632 11/06/2007 02:35:26 PM TS-IV-76-2PP

07-0632 #10-18 RT: 0.99-1.81 AV: 9 SM: 5B NL: 1.30E2
T: - p Full ms [200.00-750.00]

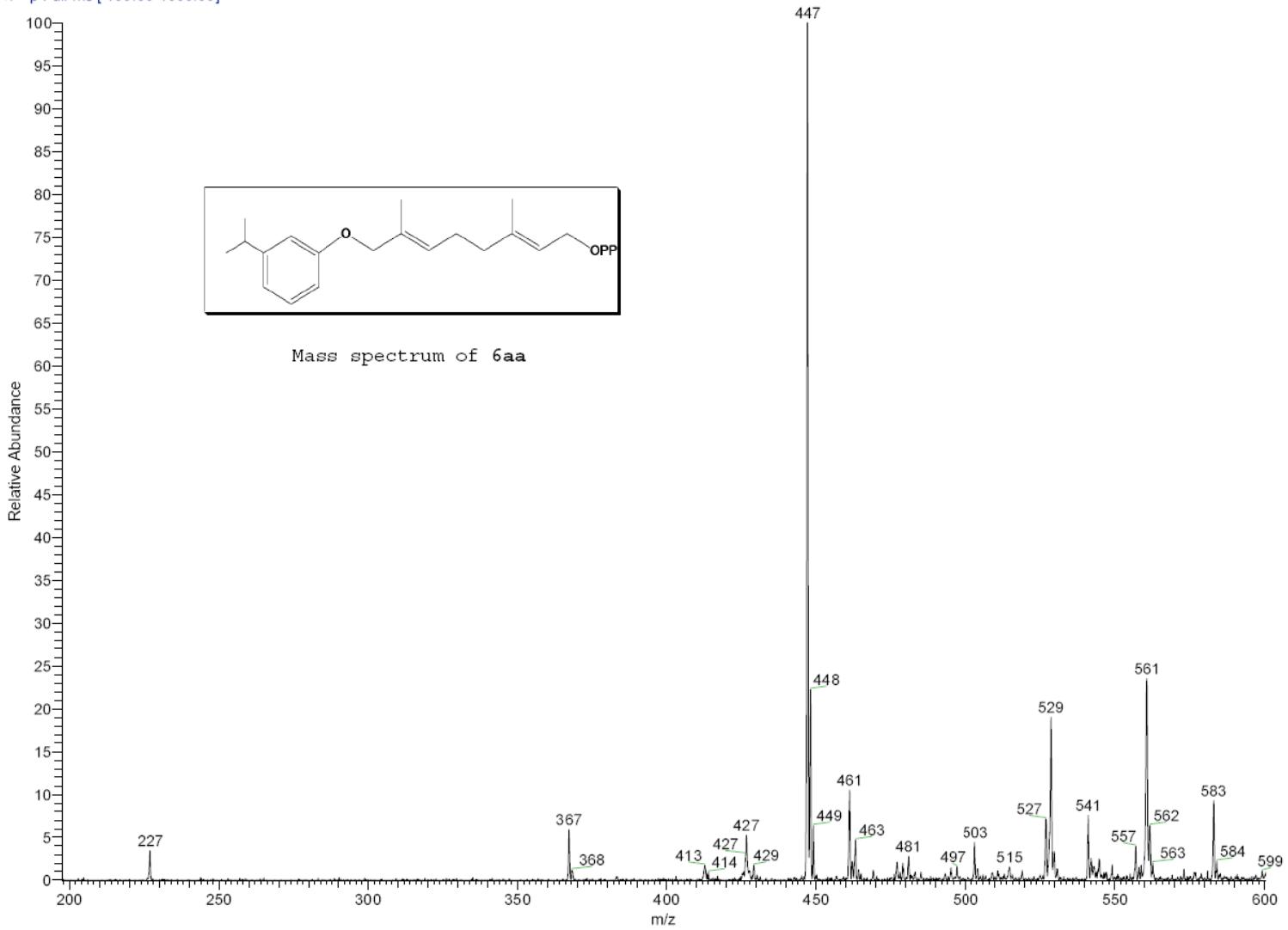



proton NMR of 6aa

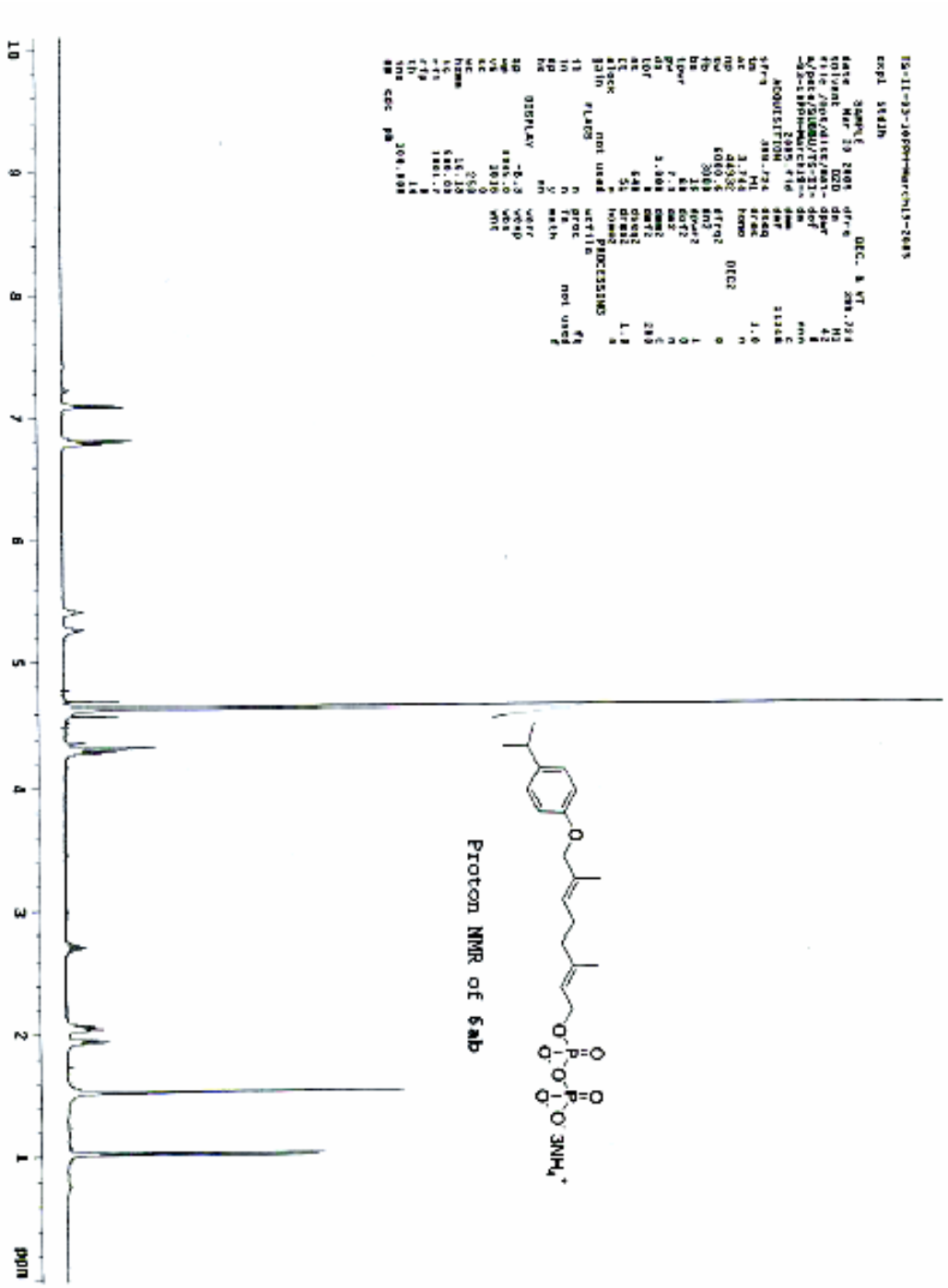
Phosphorus NMR of 6aa
3NH4+
30
25
20
15
10
5
0
-5
-10
-15
-20
ppm

C:\Xcalibur\data\UKMSF\05-0346 03/28/2005 01:53:52 PM TS-II-93-4

05-0346 #17-27 RT: 0.49-0.79 AV: 11 NL: 1.69E5
T: - p Full ms [100.00-1000.00]



Mass spectrum of **6aa**

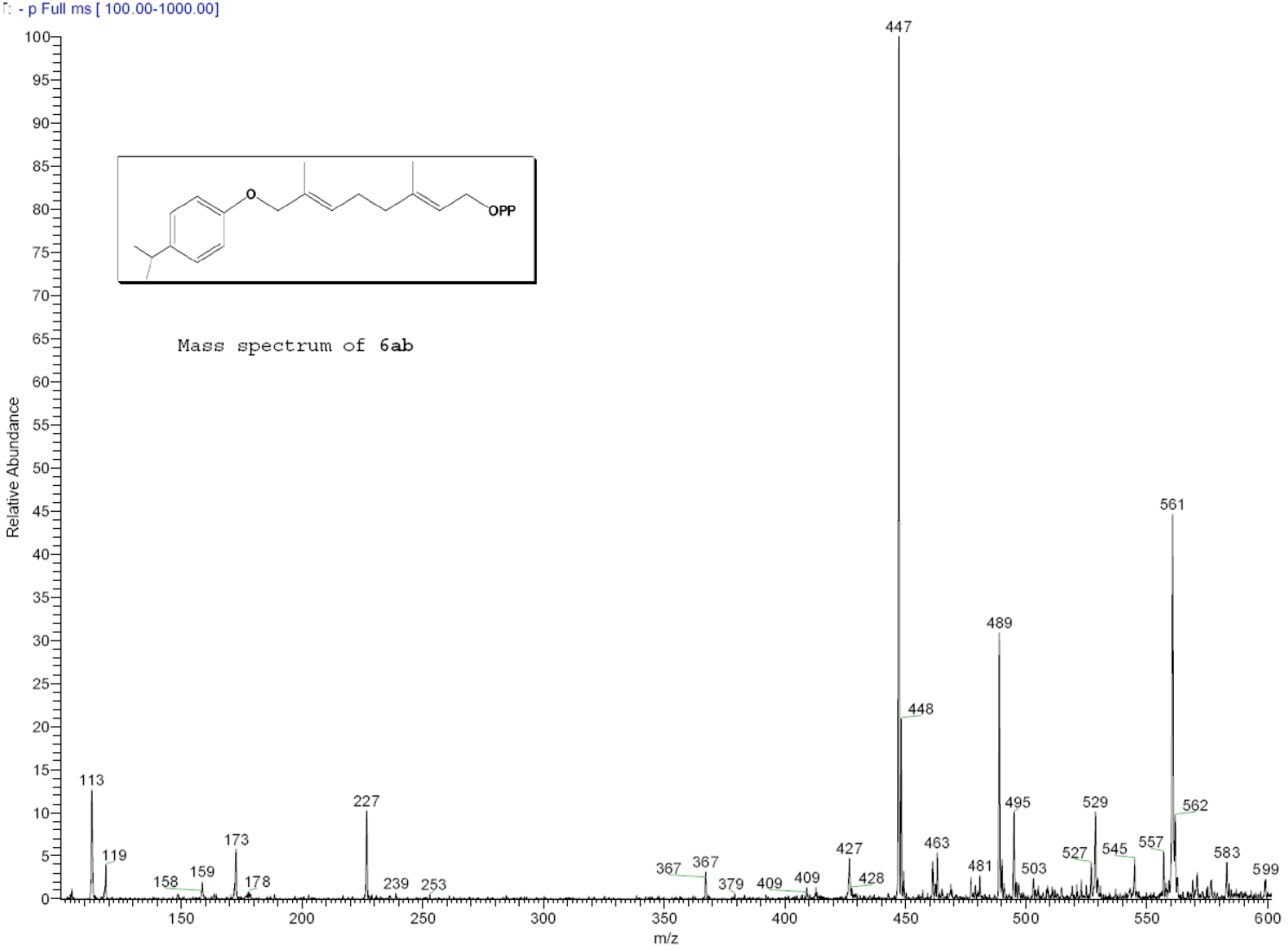

Proton NMR of 6ab
ppm

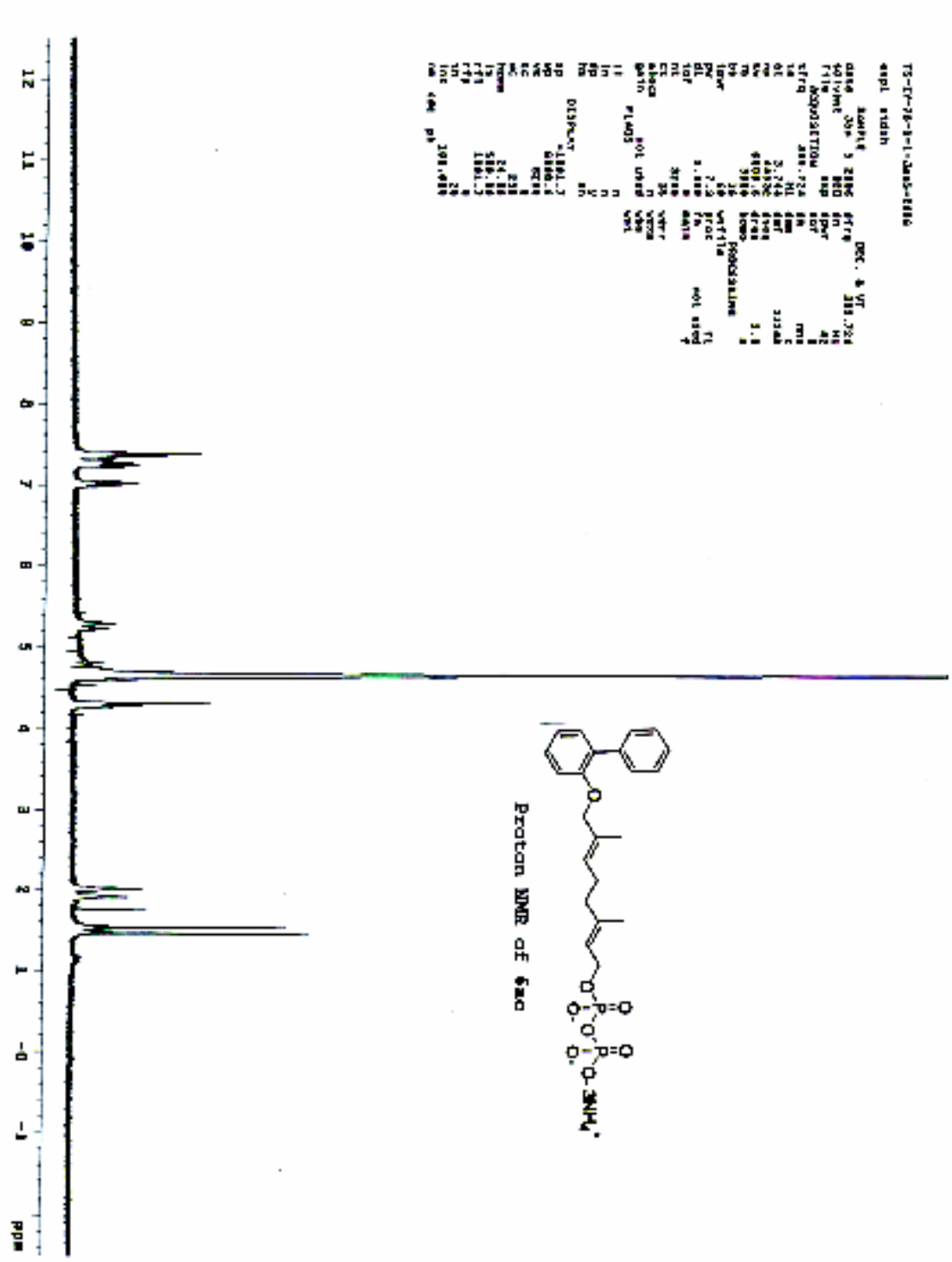
Phosphorus NMR of 6ab

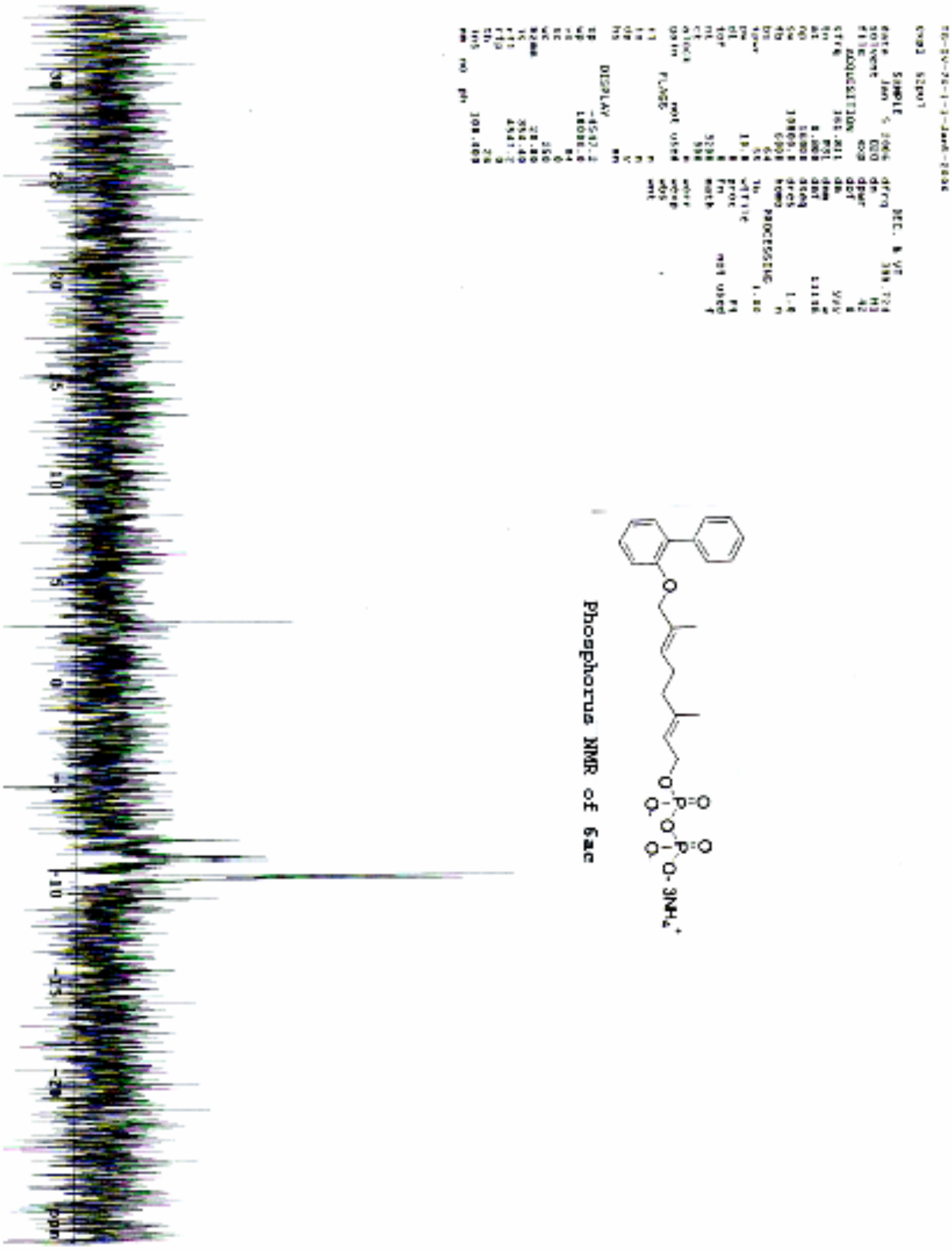
C:\Xcalibur\data\UKMSF\05-0349 03/28/2005 02:38:42 PM TS-II-93-10

05-0349 #14-22 RT: 0.39-0.63 AV: 9 NL: 1.54E5
T: - p Full ms [100.00-1000.00]

Mass spectrum of 6ab



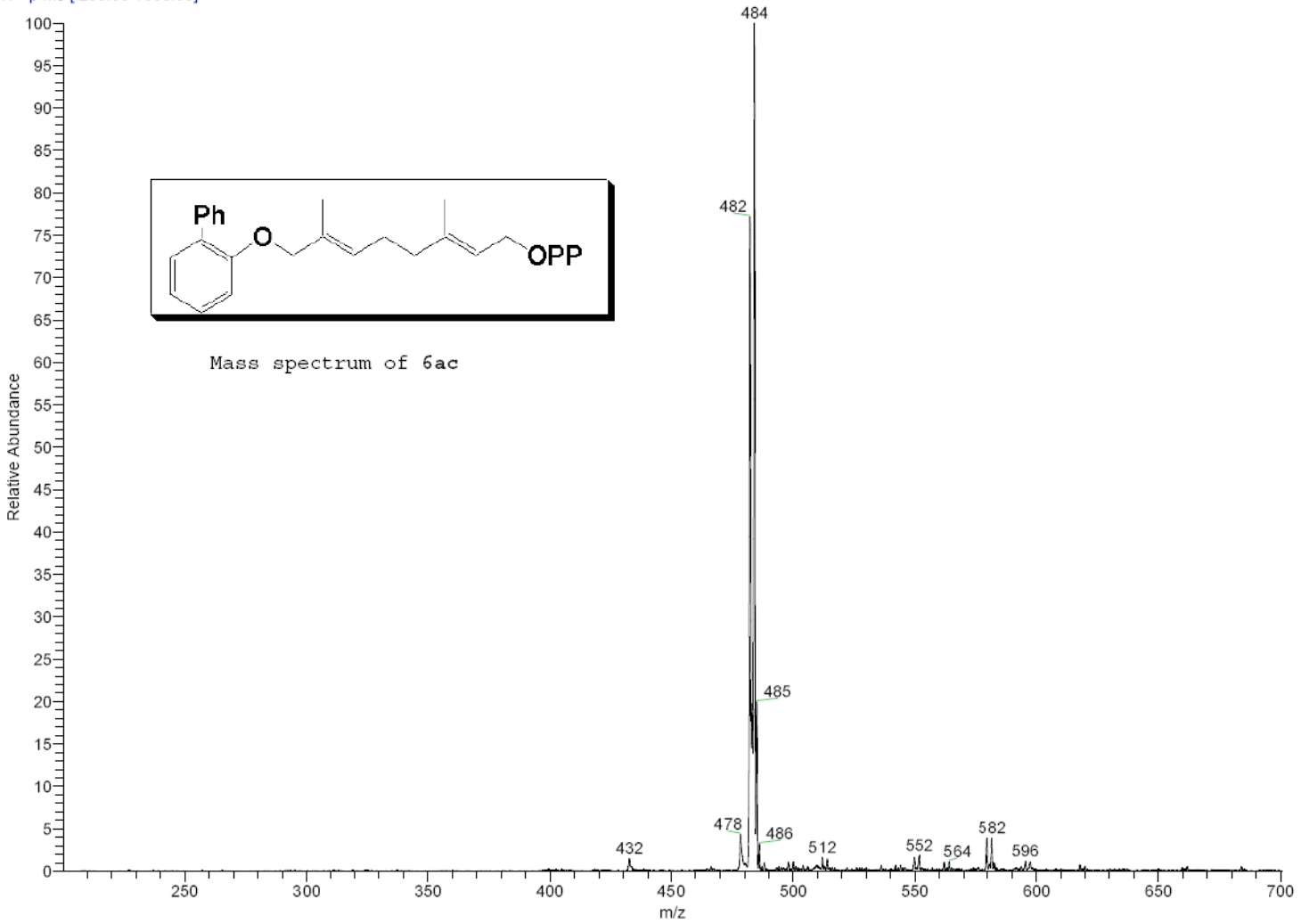

Proton NMR of 6aa



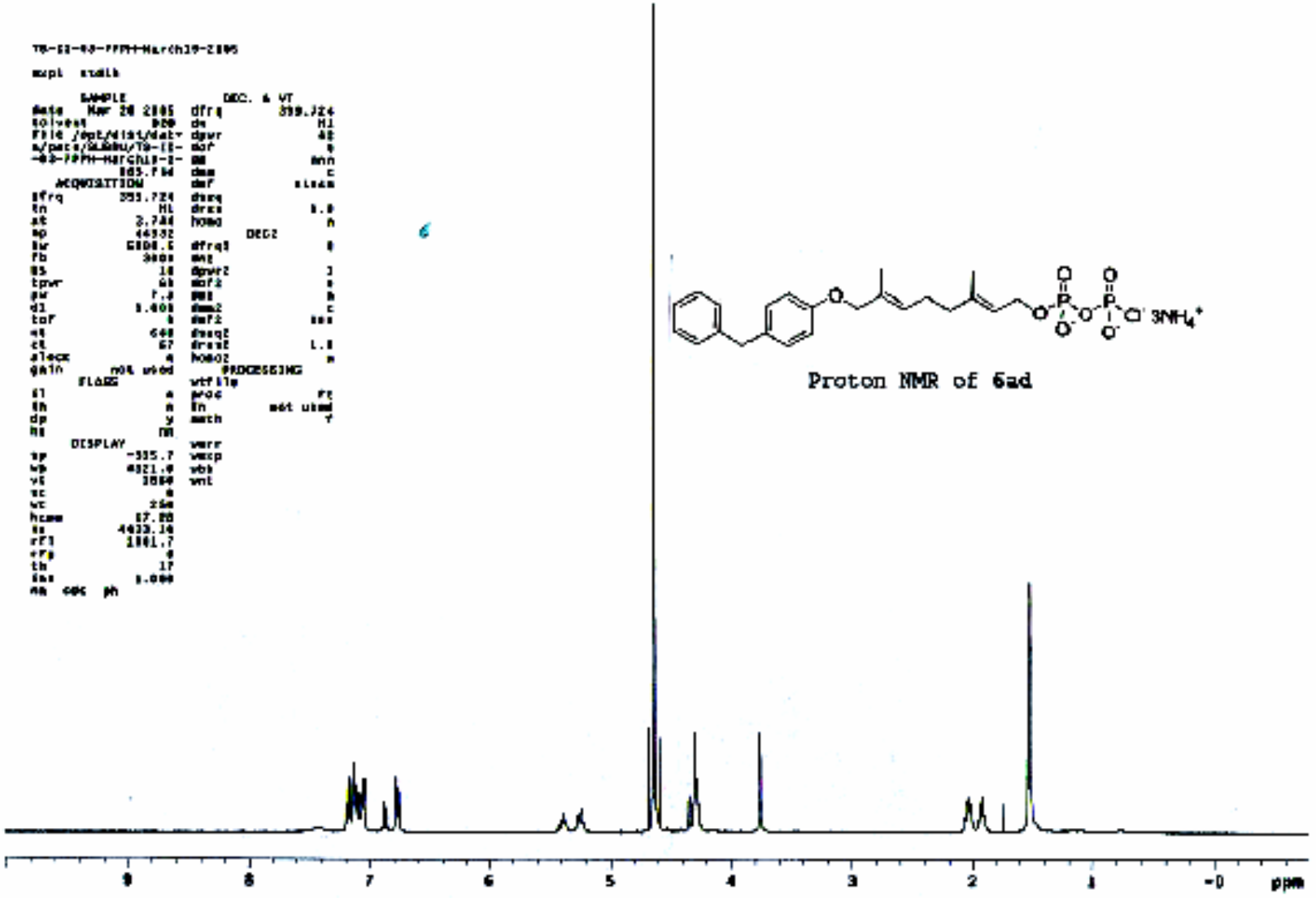
Phosphorus NMR of 6ac

\\Lcq\data\UKMSF\05-0208 02/04/2005 11:18:46 AM TS-II-90-6

05-0208 #4-16 RT: 0.10-0.46 AV: 13 SM: 5B NL: 2.57E5
T: - p ms [200.00-1000.00]



Mass spectrum of 6ac

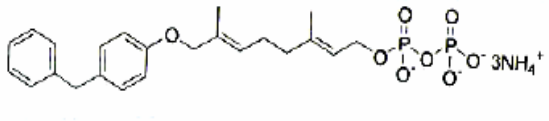

3NH4+
Proton NMR of 6ad
9
8
7
6
5
4
3
2
1
-0
ppm

```
TS-II-93-7PP-March19-2005

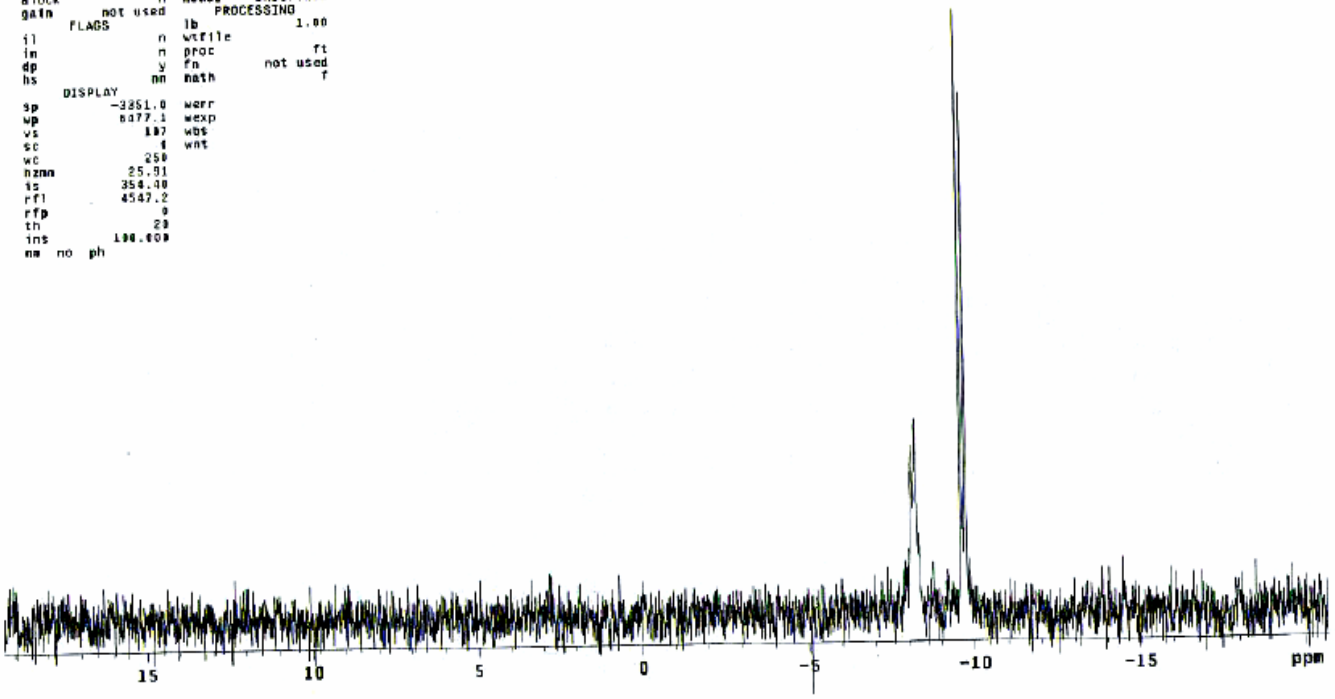
expt  s2pul

      SAMPLE
date   Mar 20 2005
solvent        D2O
file /opt/dist/dat~
e/pete/SUBBU/TS-II~
-93-7PP-March19-20~
              05.fid
    ACQUISITION
sfrq        161.811
tn              P31
at            0.809
np            16000
sw          10000.0
fb             6809
bs               64
tpwr             55
pw             10.0
di                0
tof               0
nt              640
ct              365
alock             n
gain        not used
       FLAGS
il                n
in                n
dp                y
hs               nn
      DISPLAY
sp          -3351.0
wp           6477.1
vs              197
sc                1
wc              250
nzmm          25.91
is           354.48
rfl          4547.2
rfp               0
th               28
ins          100.000
nm  no  ph

       DEC. & VT
dfrq         399.724
dn                H1
dpwr              42
dof                0
dm               yyy
dmm                w
dmf            11148
dseq
dres             1.0
homo               n
       DEC2
dfrq2      undefined
dn2        undefined
dpwr2      undefined
dof2       undefined
dm2        undefined
dmm2       undefined
dmf2       undefined
dseq2      undefined
dres2      undefined
homo2      undefined
     PROCESSING
lb               1.00
wtfile
proc               ft
fn          not used
math                f
werr
wexp
wbs
wnt
```

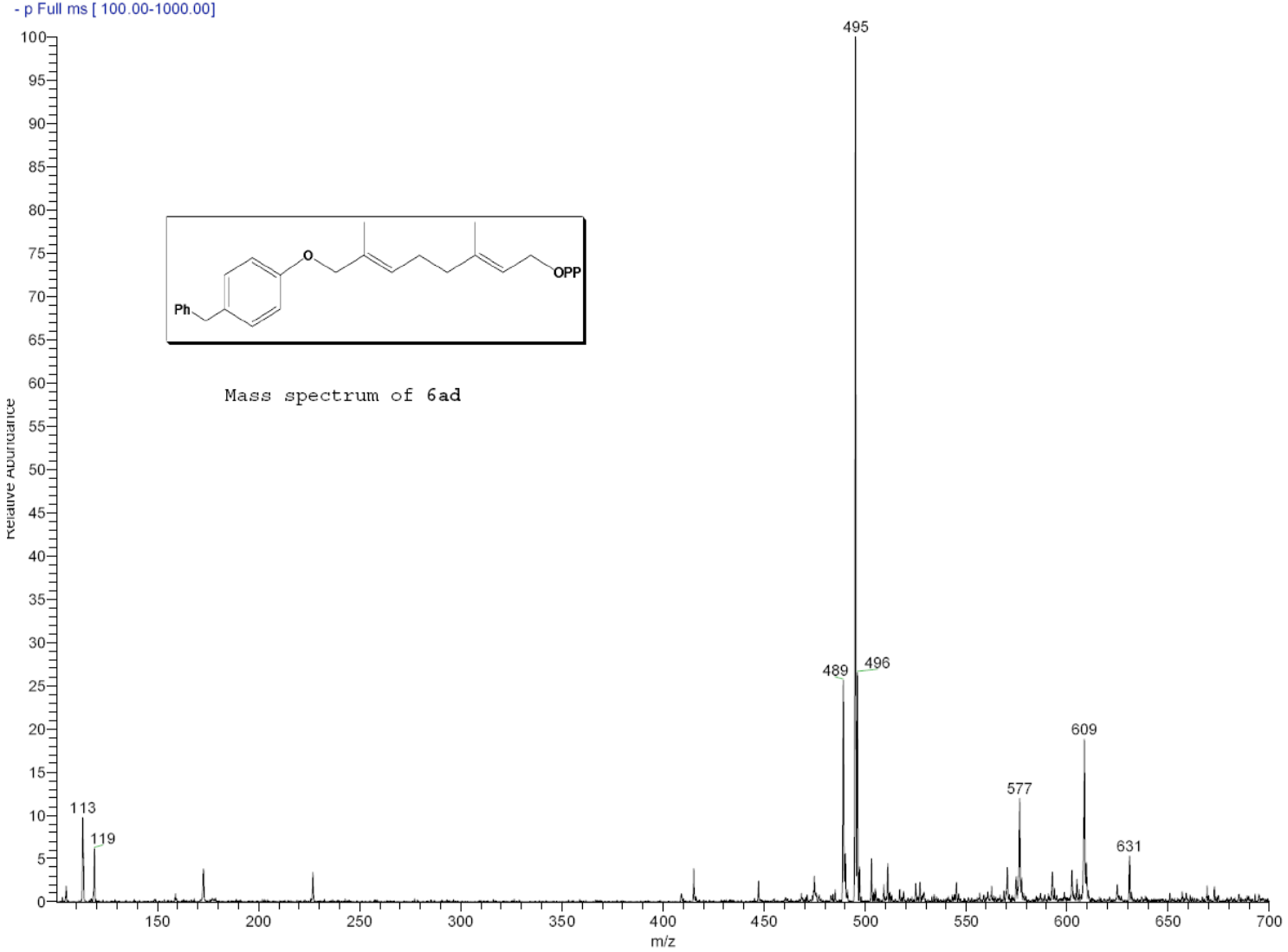


Phosphorus NMR of 6ad



:\Xcalibur\data\UKMSF\05-0348 03/28/2005 02:28:56 PM TS-II-93-7

5-0348 #29-46 RT: 0.79-1.36 AV: 18 NL: 1.37E5
- p Full ms [100.00-1000.00]



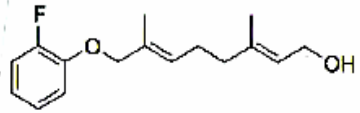
Mass spectrum of **6ad**

```
expl  std1h

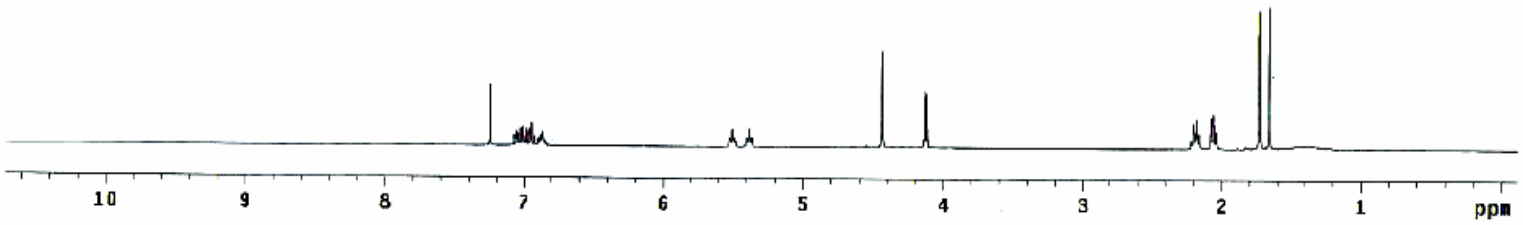
      SAMPLE
date    May 19 2006
solvent       CDCl3
file /opt/dist/dat~
a/pete/SUBBU/TS-V--~
SG-12-May19-2006.f~
                  id
   ACQUISITION
sfrq         399.723
tn                H1
at            3.744
np            44932
sw           6000.6
fb             3000
bs               16
tpwr             60
pw              7.3
d1            1.000
tof               0
nt              320
ct               33
alock             n
gain       not used
      FLAGS
fl                n
in                n
dp                y
hs               nn
     DISPLAY
sp            -50.4
wp           4834.5
vs              23
sc               0
wc             250
hzmm         17.34
is           500.00
rfl          1001.7
rfp               0
th               20
ins         100.000
nm  cdc  ph

      DEC. & vT
dfrq         399.723
dn                H1
dpwr              42
dof               0
dm              nnn
dmm               c
dmf           11148
dseq
dres            1.0
homo              n
      DEC2
dfrq2             0
dn2
dpwr2             1
dof2              0
dm2               n
dmm2              c
dmf2            200
dseq2
dres2           1.0
homo2             n
     PROCESSING
wtfile
proc             ft
fn         not used
math              f

werr
wexp
wbs
wnt
```

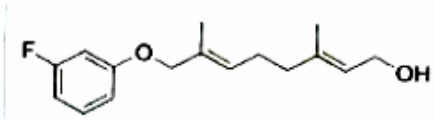


24b

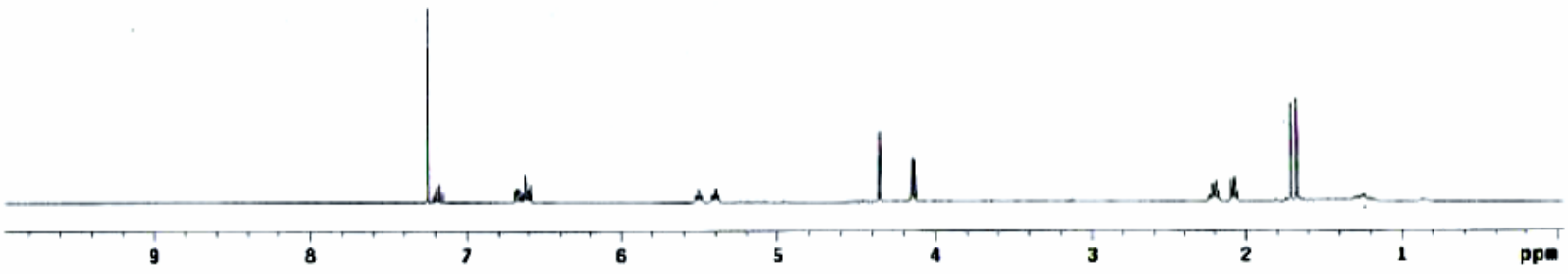


```
expl  stdih

      SAMPLE                DEC. & VT
date     Jan  3 2006  dfrq         399.723
solvent       CDCl3   dn               H1
file /opt/dist/dat-   dpwr             42
a/pete/SUBBU/TS-IV-   dof               0
-38-2-Jan3-2005.fi-   dm              nnn
                  d   dmm               c
   ACQUISITION        dmf           11148
sfrq         399.723   dseq
tn                H1   dres            1.8
at             3.744   homo              n
np             44932        DEC2
sw            6000.6   dfrq2             0
fb               3000   dn2
bs                 16   dpwr2             1
tpwr               60   dof2              0
pw                7.3   dm2               n
d1              1.000   dmm2              c
tof                0   dmf2            200
nt               320   dseq2
ct                81   dres2           1.8
alock              n   homo2             n
gain       not used        PROCESSING
      FLAGS            wtfile
il                 n   proc              ft
in                 n   fn         not used
dp                 y   math              f
hs                nn
      DISPLAY          werr
sp             -16.1   wexp
wp            3997.6   wbs
vs                33   wnt
sc                 0
wc               250
hzmm           15.99
is             171.27
rf1            1001.7
rfp                0
th                 9
ins           100.000
nm  cdc  ph
```

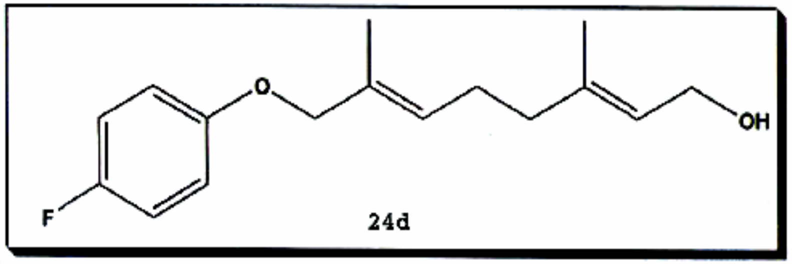


24c

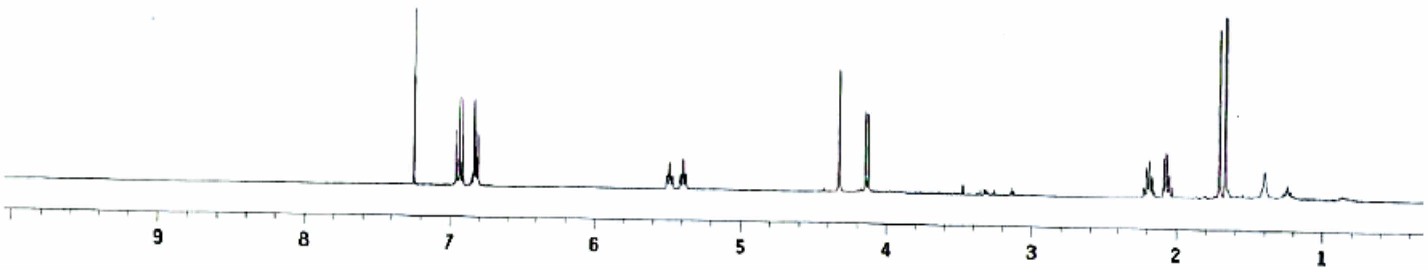


expl stain

```
        SAMPLE                   DEC. & VT
date   Oct  6 2005   dfrq         399.723
solvent       CDCl3   dn               H1
file            exp   dpwr             42
     ACQUISITION      dof               0
sfrq        399.723   dm              nnn
tn               H1   dmm               c
at            3.744   dmf           11148
np            44932   dseq
sw           6000.6   dres            1.0
fb             3000   homo              n
bs               16        PROCESSING
tpwr             60   wtfile
pw              7.3   proc             ft
d1            1.000   fn         not used
tof               0   math              f
nt               32
ct               23   werr
alock             n   wexp
gain       not used   wbs
        FLAGS         wnt
il                n
in                n
dp                y
hs               nn
       DISPLAY
sp             -2.6
wp           4020.4
vs               29
sc                0
wc              250
hzmm          16.08
is           500.00
rfl          1001.7
rfp               0
th               20
ins         100.000
nm  cdc  ph
```



24d

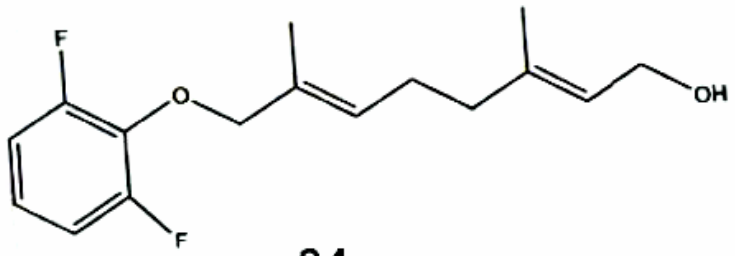


TS-1V-38-9-Oct9-2005

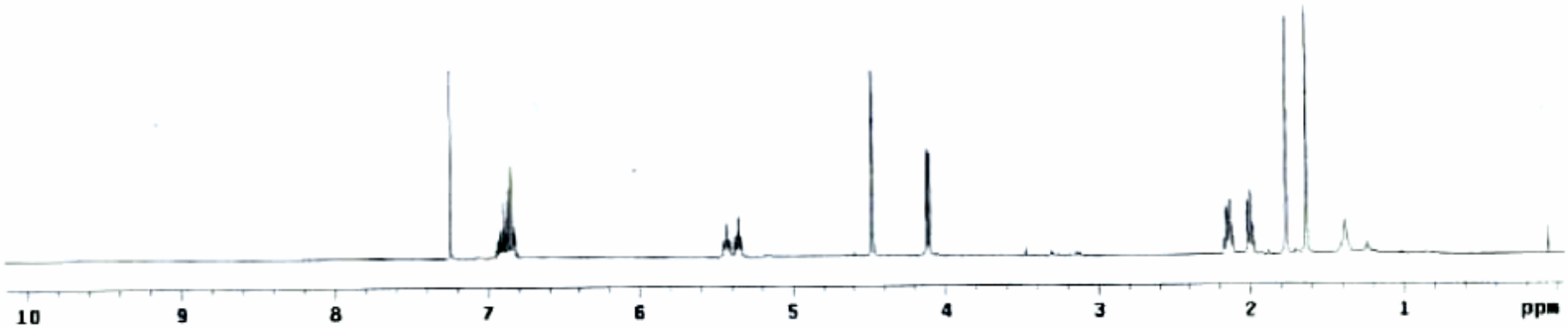
expl stdin

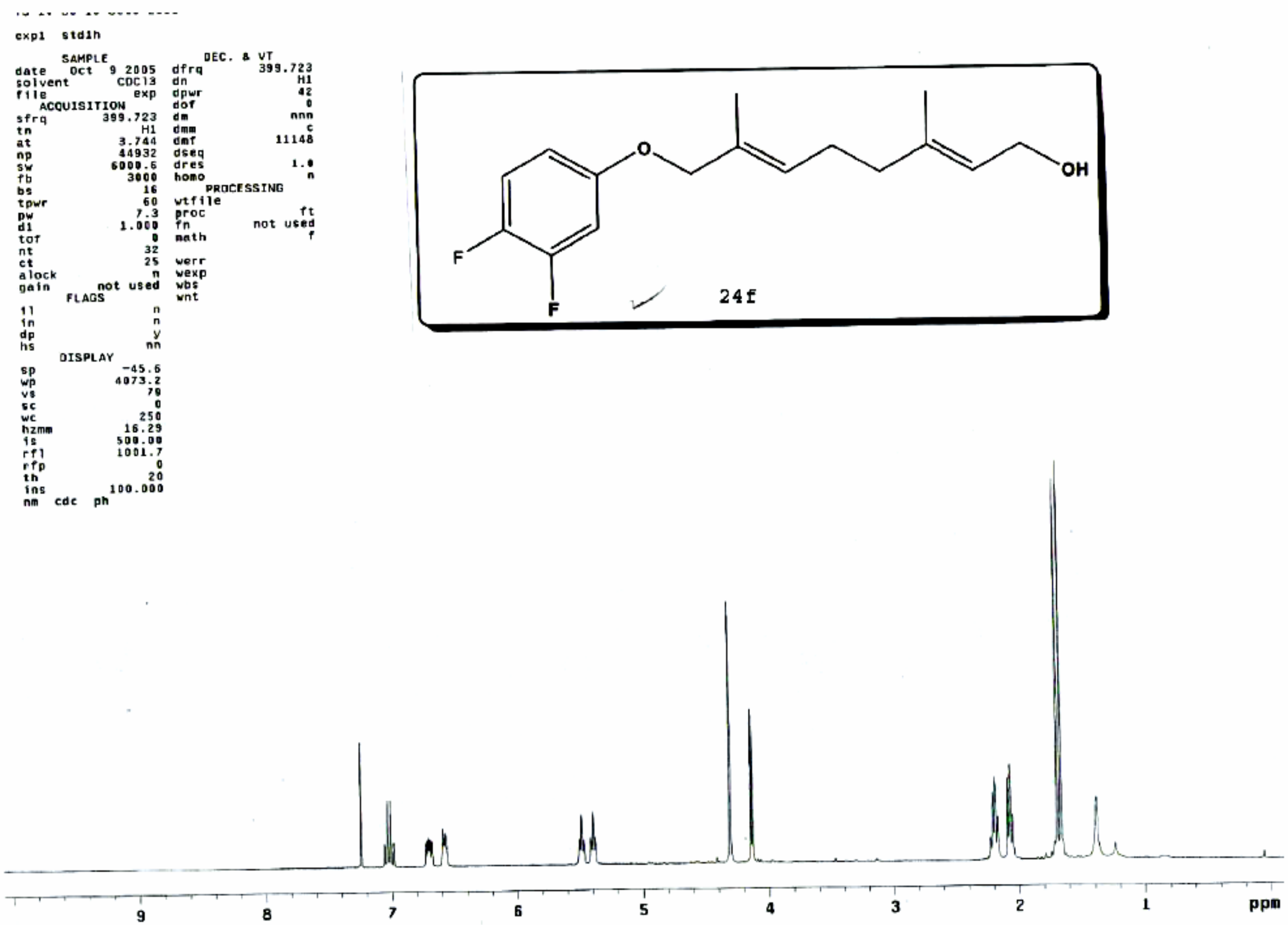
SAMPLE		DEC. & VT	
date	Oct 9 2005	dfrq	399.723
solvent	CDCl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	399.723	dm	nnn
tn	H1	dmm	c
at	3.744	dmf	11148
np	44932	dseq	
sw	6000.6	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	60	wtfile	
pw	7.3	proc	ft
d1	1.000	fn	not used
tof	0	math	f
nt	32		
ct	19	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-23.1		
wp	4080.7		
vs	41		
sc	0		
wc	250		
hzmm	16.32		
is	500.00		
rfl	1001.7		
rfp	0		
th	20		
ins	100.000		

nm cdc ph

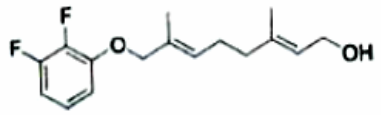


24e

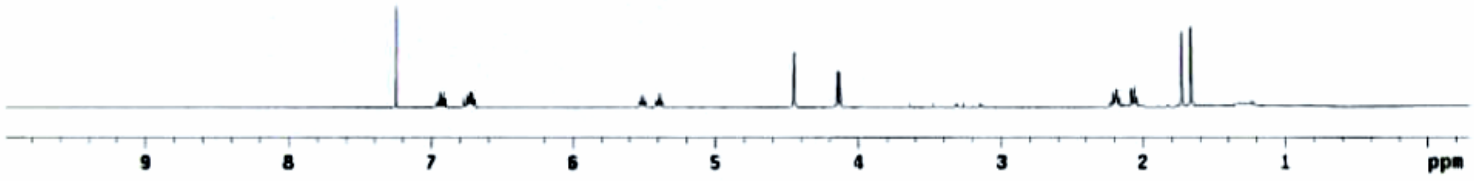


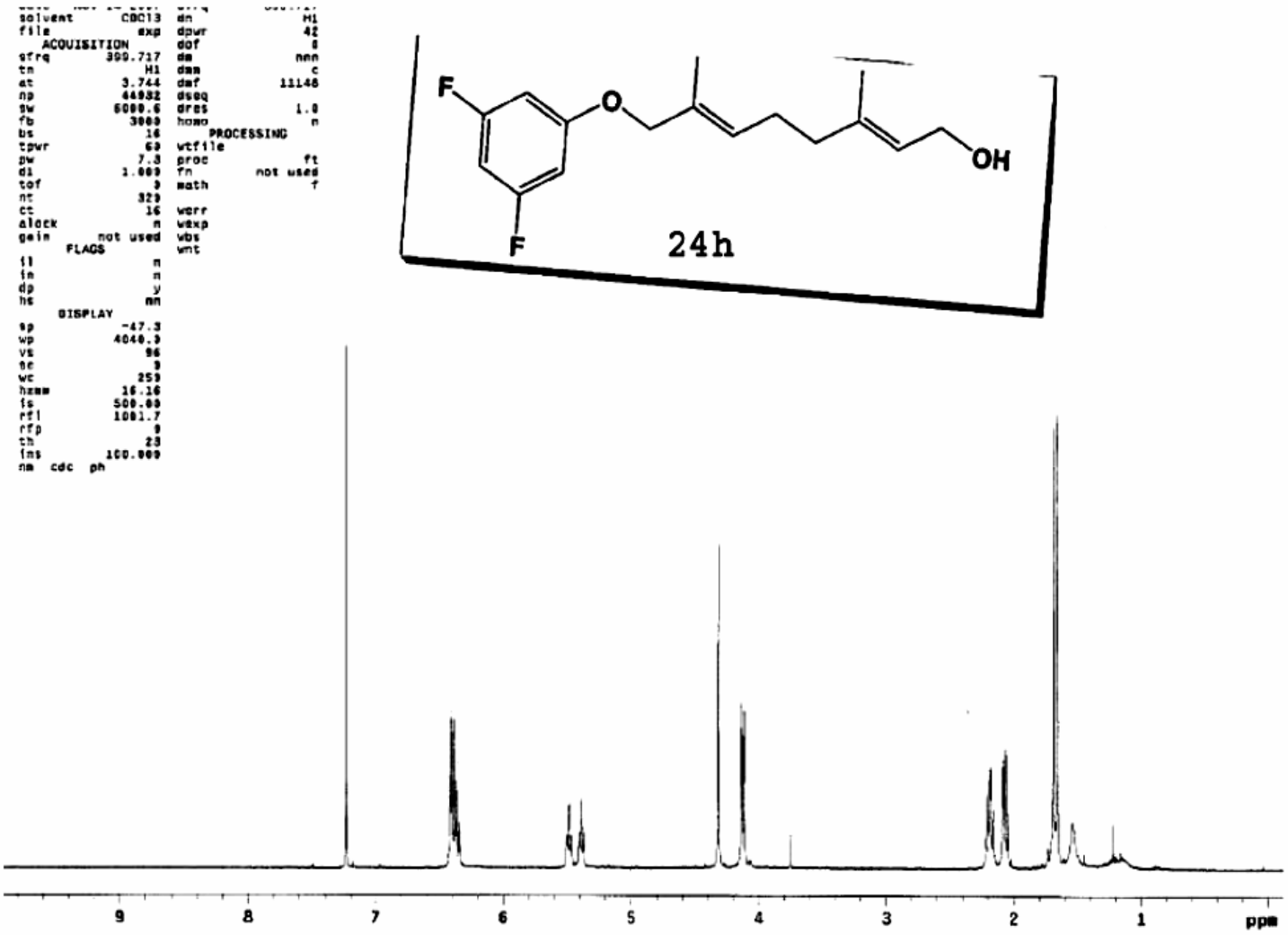

expl stdlh
SAMPLE
date Oct 9 2005
solvent CDCl3
file exp
ACQUISITION
sfrq 399.723
tn H1
at 3.744
np 44932
sw 6000.6
fb 3000
bs 16
tpwr 60
pw 7.3
d1 1.000
tof 0
nt 32
ct 25
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -45.6
wp 4073.2
vs 79
sc 0
wc 250
hzmm 16.29
is 500.00
rfl 1001.7
rfp 0
th 20
ins 100.000
nm cdc ph
DEC. & VT
dfrq 399.723
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
O
OH
F
F
24f
9
8
7
6
5
4
3
2
1
ppm

SAMPLE		DEC. & VT	
date	Dec 28 2005	dfrq	399.723
solvent	CDCl3	dn	H1
file	/opt/dist/dat-a/pete/SUBBU/TS-IV--38-4-dec28-2005.f-	dpwr	42
		dof	0
		dm	nnn
	1d	dmm	c
ACQUISITION		dmf	11148
sfrq	399.723	dseq	
tn	H1	dres	1.0
at	3.744	homo	n
np	44932	DEC2	
sw	6008.6	dfrq2	0
fb	3880	dn2	
bs	16	dpwr2	1
tpwr	60	dof2	0
pw	7.3	dm2	n
d1	1.000	dmm2	c
tof	0	dmf2	200
nt	320	dseq2	
ct	48	dres2	1.0
alock	n	homo2	n
gain	not used	PROCESSING	
FLAGS		wtfile	
il	n	proc	ft
in	n	fn	not used
dp	y	math	f
hs	nn		
DISPLAY		werr	
sp	-118.7	wexp	
wp	4188.0	wbs	
vs	16	wnt	
sc	0		
wc	250		
hzmm	16.43		
is	1230.86		
rfl	1001.7		
rfp	0		
th	24		
ins	100.000		
nm cdc ph			



24g



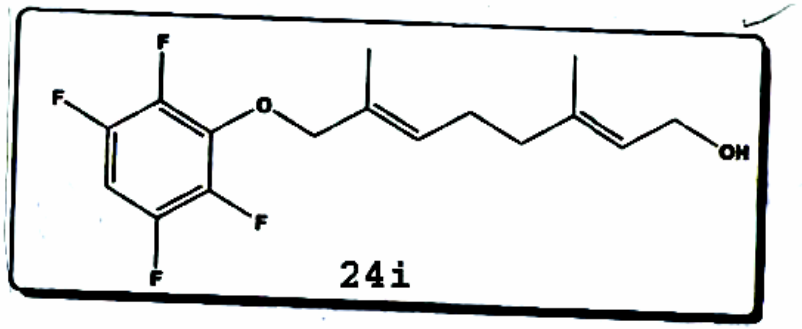

solvent CDCl3
file exp
ACQUISITION
sfrq 399.717
tn H1
at 3.744
np 44932
sw 6000.6
fb 3000
bs 16
tpwr 69
pw 7.3
d1 1.000
tof 0
nt 320
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -47.3
wp 4040.9
vs 96
sc 0
wc 253
hzmm 16.16
is 500.00
rfl 1081.7
rfp 0
th 23
ins 100.000
nm cdc ph
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
F
O
OH
F
24h
9
8
7
6
5
4
3
2
1
ppm

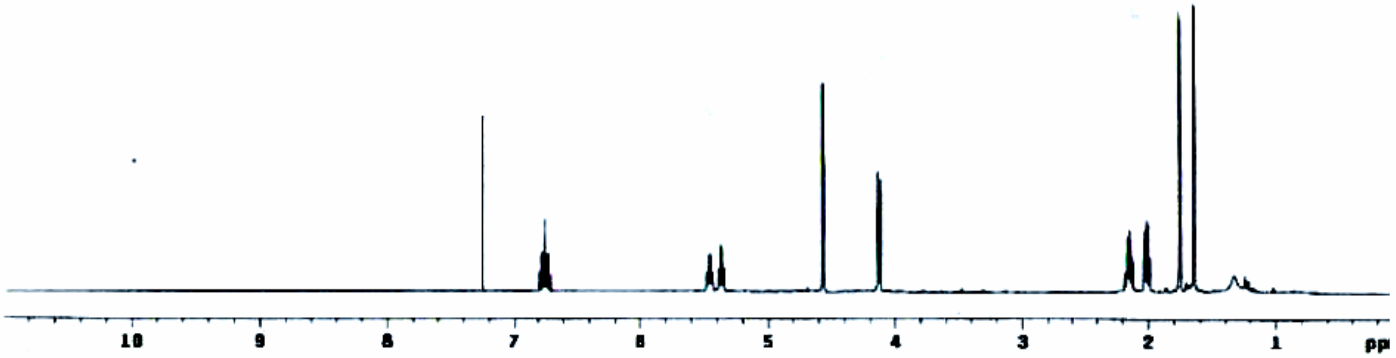
```
expl  stdin

      SAMPLE
date    Oct  2 2005
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        399.723
tn               H1
at              3.744
np             44932
sw             6000.6
fb              3000
bs                16
tpwr              60
pw               7.3
pl             1.000
tof                0
nt                32
ct                24
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
    DISPLAY
sp              -8.1
wp            4484.4
vs                51
sc                 0
wc               250
hzmm           17.62
is             62.50
rfl          1001.7
rfp                0
th                28
ins          100.000
ap  cdc  ph

      DEC. & VT
dfrq        399.723
dn               H1
dpwr             42
dof               0
dm              nnn
dmm               c
dmf           11148
dseq
dres            1.0
homo              n
    PROCESSING
wtfile
proc             ft
fn         not used
math              f

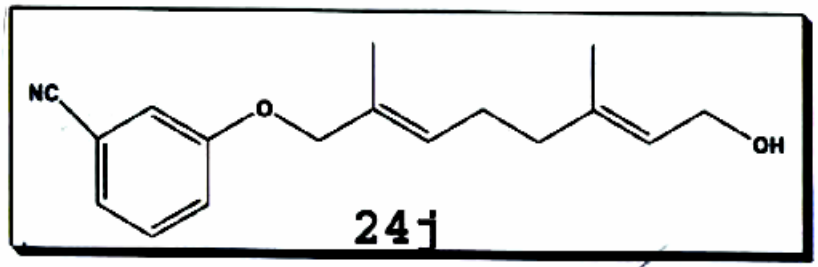
werr
wexp
wbs
wnt
```



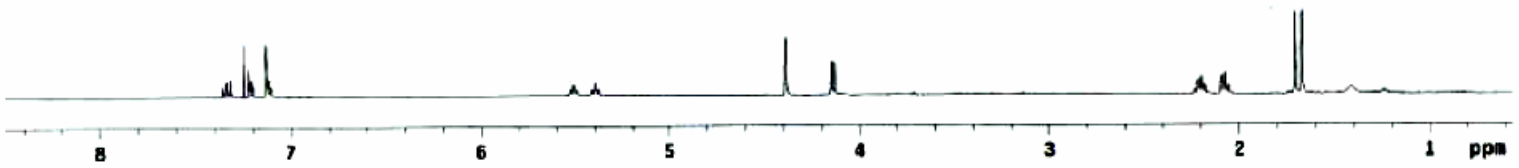


expl stdih

SAMPLE		DEC. & VT	
date	Oct 2 2005	dfrq	399.723
solvent	CDCl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	399.723	dm	nnn
tn	H1	dmm	c
at	3.744	dmf	11148
np	44932	dseq	
sw	6000.6	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	60	wtfile	
pw	7.3	proc	ft
dl	1.000	fn	not used
tof	0	math	f
nt	32		
ct	20	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		vnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	225.4		
wp	3169.6		
vs	14		
sc	0		
wc	250		
hzmm	12.68		
is	31.25		
rfl	1001.7		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			



241

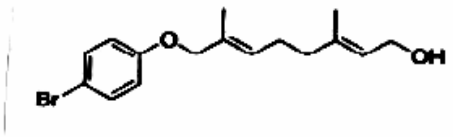


24k
NC
O
OH

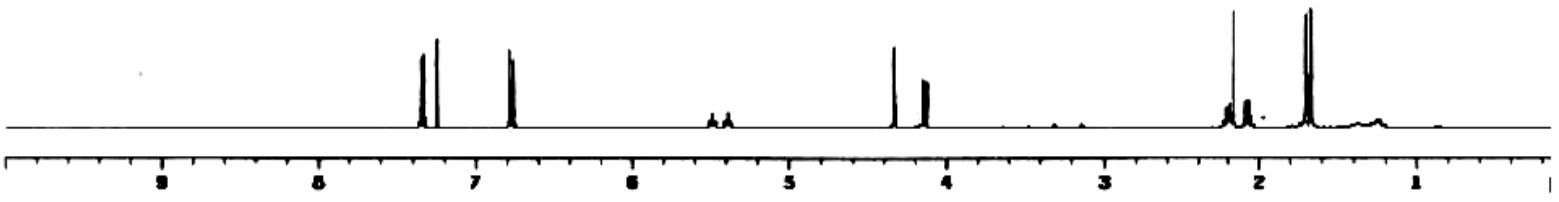
```
TS-IV-58-3-Jan3-2005

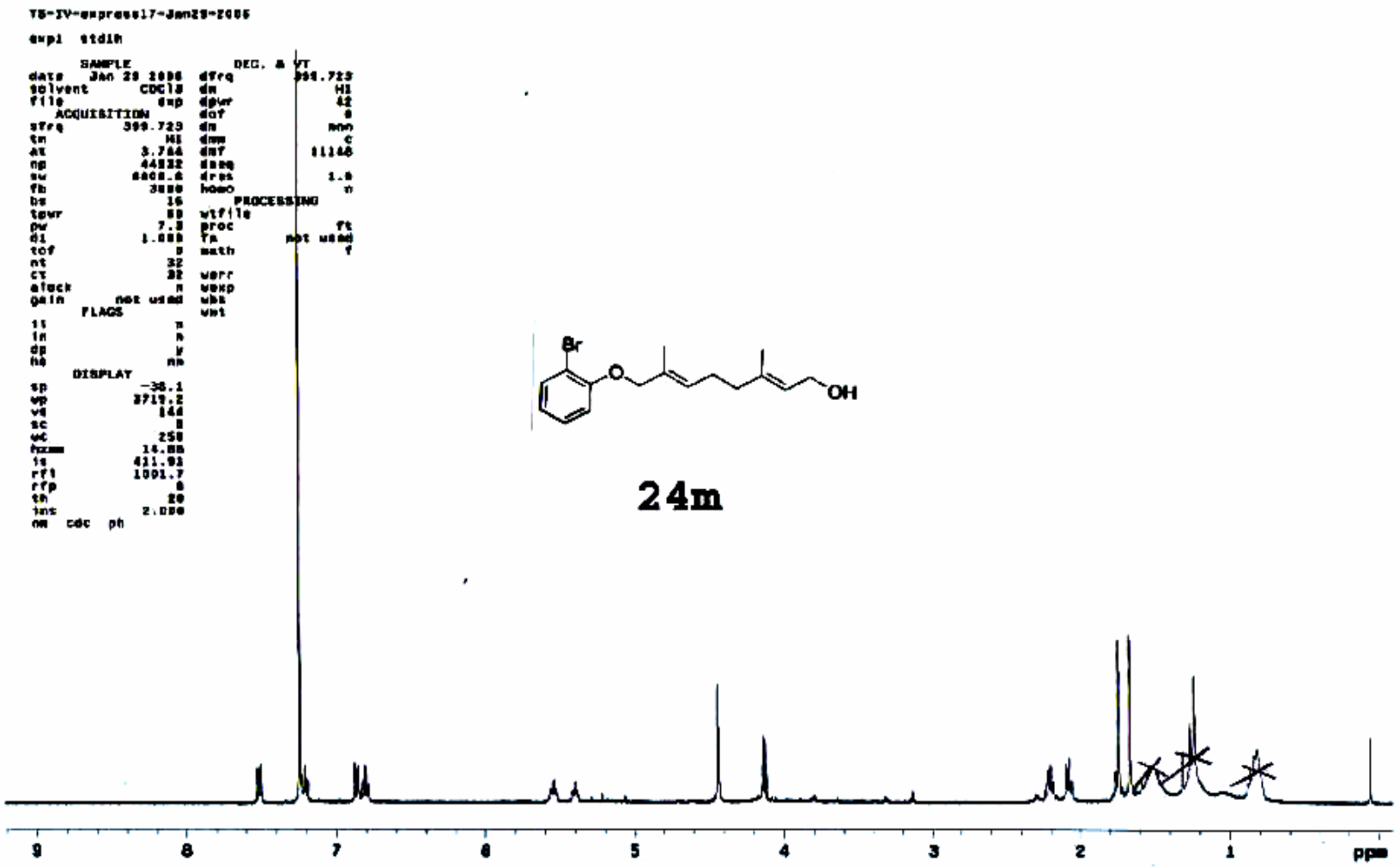
expl  std1h

      SAMPLE                  SEC. A VT
date    Dec 28 2005  dfrq          399.723
solvent       CDCl3  dn               H1
file /opt/dist/dat-  dpwr             42
a/pete/SUBDU/TS-IV-  dof               0
-58-3-dec28-2005.f-  dm              nnn
              hd     dmm               c
   ACQUISITION       dmf           11148
sfrq          399.723 dseq
tn                H1  dres            1.0
at              3.744 homo             n
np             44852          SEC2
sw             5494.8 dfrq2           0
fb             3698   dn2
bs               16   dpwr2             1
tpwr             66   dof2              0
pw              7.3   dm2               n
dl              1.698  dmm2              c
tof                0   dmf2          200
nt               320   dseq2
ct               37    dres2           1.0
alock              n   homo2              n
gain        not used       PROCESSING
      FLAGS             wtfile
il                 n   proc              ft
in                 n   fn          not used
dp                 y   math               f
hs                nn
    DISPLAY            wtr
sp             -55.5   wexp
wp           4452.3    wbs
vs                20   ssb
sc                 0   lb
wc               250   gf
hzpm           16.21   gfs
ft             259.88  gfs
rfl           1881.7   awc
rfp                0   lbc
th                14   phc0
int          198.688   phc1
nm  cdc  ph
```



241



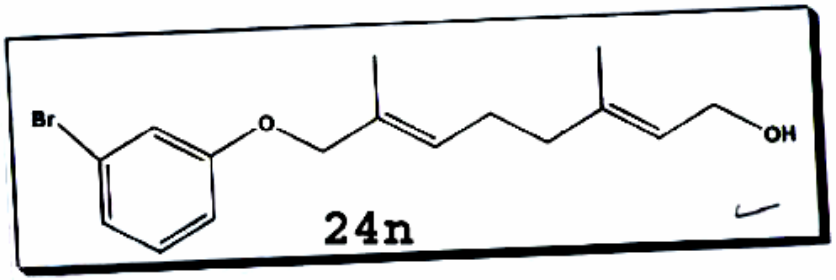

YB-IV-express17-Jan29-2006
expl stdin
SAMPLE
date Jan 29 2006
solvent CDCl3
file exp
ACQUISITION
sfrq 399.723
tn H1
at 3.784
np 44832
sw 8468.8
fb 3880
bs 16
tdav 80
pw 7.8
d1 1.000
tof 0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -38.1
wp 3719.2
vs 144
sc 0
wc 258
hzcm 16.88
is 411.91
rfl 1091.7
rfp 0
th 20
ins 2.000
nm cdc ph
DEC. & VT
dfrq 399.723
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.8
homo n
PROCESSING
wtfile
proc ft
fa not used
math f
werr
wexp
wbs
wnt
Br
O
OH
24m
9
8
7
6
5
4
3
2
1
ppm

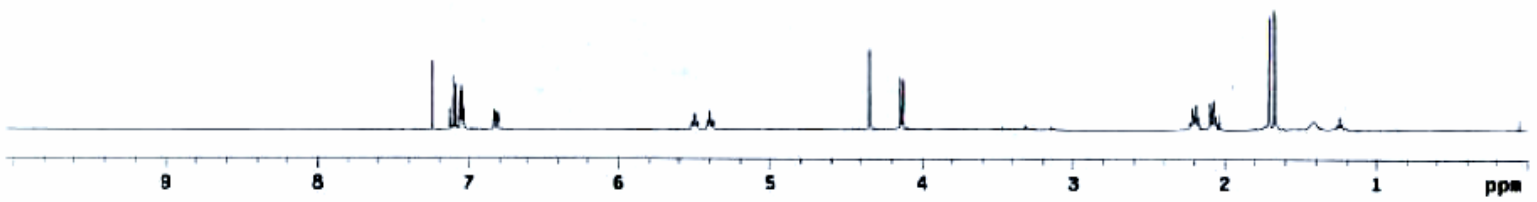
TS-IV-38-3-oct7-2005

expl stdih

SAMPLE		DEC. & VT	
date	Oct 6 2005	dfrq	399.723
solvent	CDCl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	399.723	dm	nnn
tn	H1	dmm	c
at	3.744	dmf	11148
np	44932	dseq	
sw	6000.6	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	60	wtfile	
pw	7.3	proc	ft
d1	1.000	fn	not used
tof	0	math	f
nt	32		
ct	32	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2.6		
wp	4029.4		
vs	21		
sc	0		
wc	250		
hzmm	16.08		
is	500.00		
rf1	1601.7		
rfp	0		
th	20		
ins	100.000		

nm cdc ph

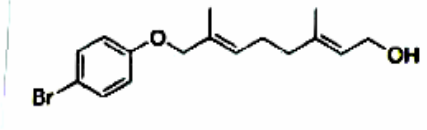




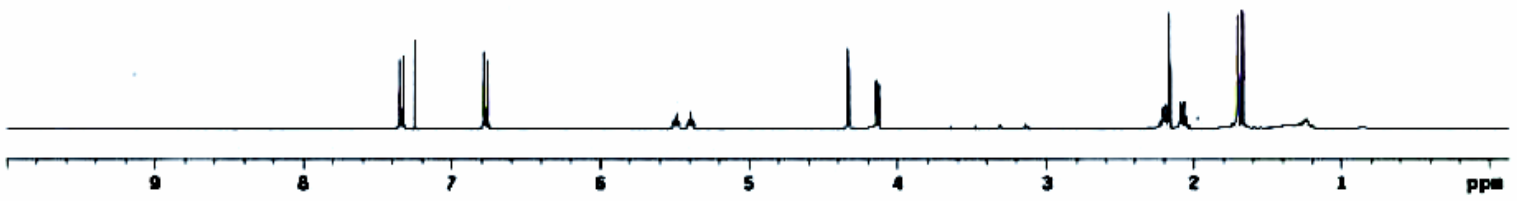
TS-IV-38-3-Jan3-2005

expl stdih

SAMPLE		DEC. & VT	
date	Dec 28 2005	dfrq	399.723
solvent	CDCl3	dn	H1
file	/opt/dist/dat~	dpwr	42
a/pete/SHARU/TS-IV~		dof	0
~38-3-dec28-2005.f~		dm	nnn
	id	dmm	c
ACQUISITION		dmf	11148
sfrq	399.723	dseq	
tn	H1	dres	1.0
at	3.744	homo	n
np	44932	DEC2	
sw	6990.6	dfrq2	0
fb	3068	dn2	
bs	16	dpwr2	1
tpwr	68	dof2	0
pw	7.3	dm2	n
d1	1.000	dmm2	c
tof	0	dmf2	200
nt	328	dseq2	
ct	37	dres2	1.0
alock	n	homo2	n
gain	not used	PROCESSING	
FLAGS		wtfile	
il	n	proc	ft
in	n	fn	not used
dp	y	math	f
hs	nn		
DISPLAY		werr	
sp	-55.5	wexp	
wp	4852.8	wbs	
vs	28	wnt	
sc	0		
wc	250		
hzcm	16.21		
is	250.00		
rfl	1001.7		
rfp	0		
th	16		
ins	100.000		
nm cdc ph			



24o

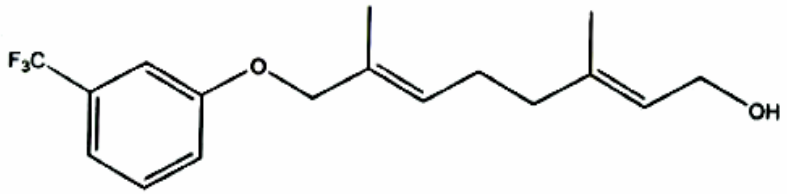


```
TS-IV-30-8-oct7-2005

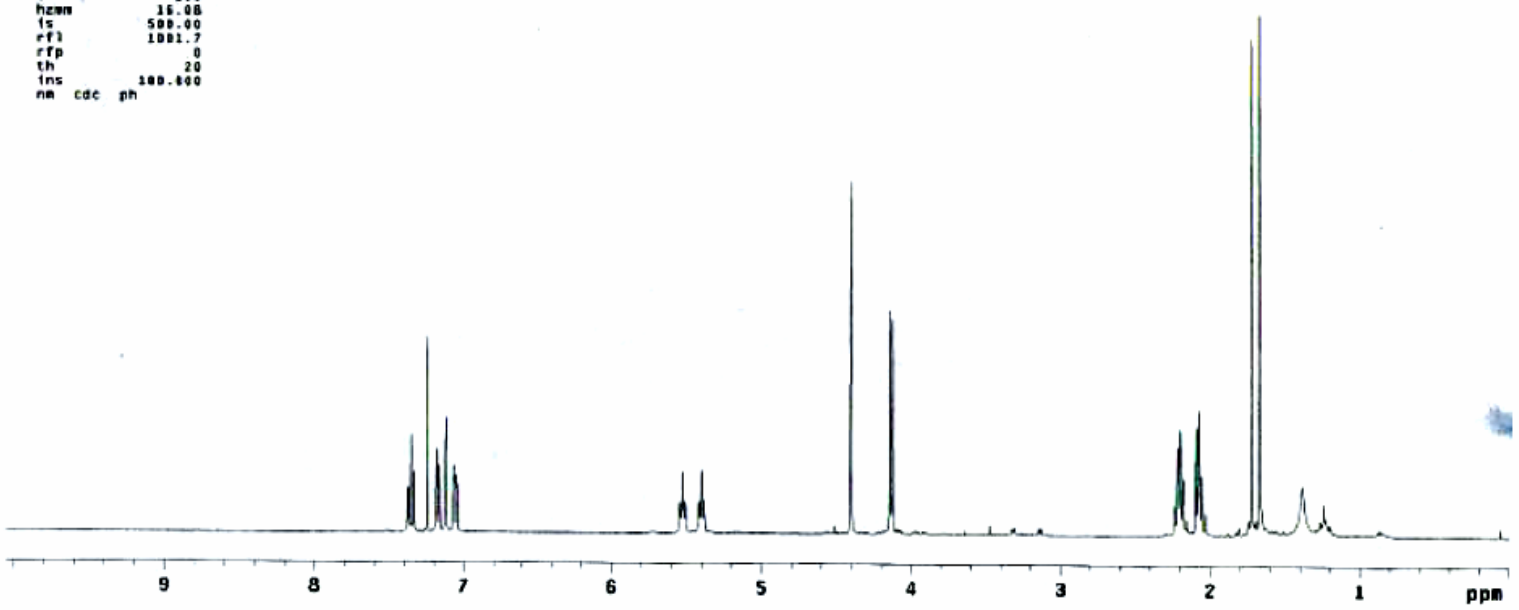
expl  stdlh

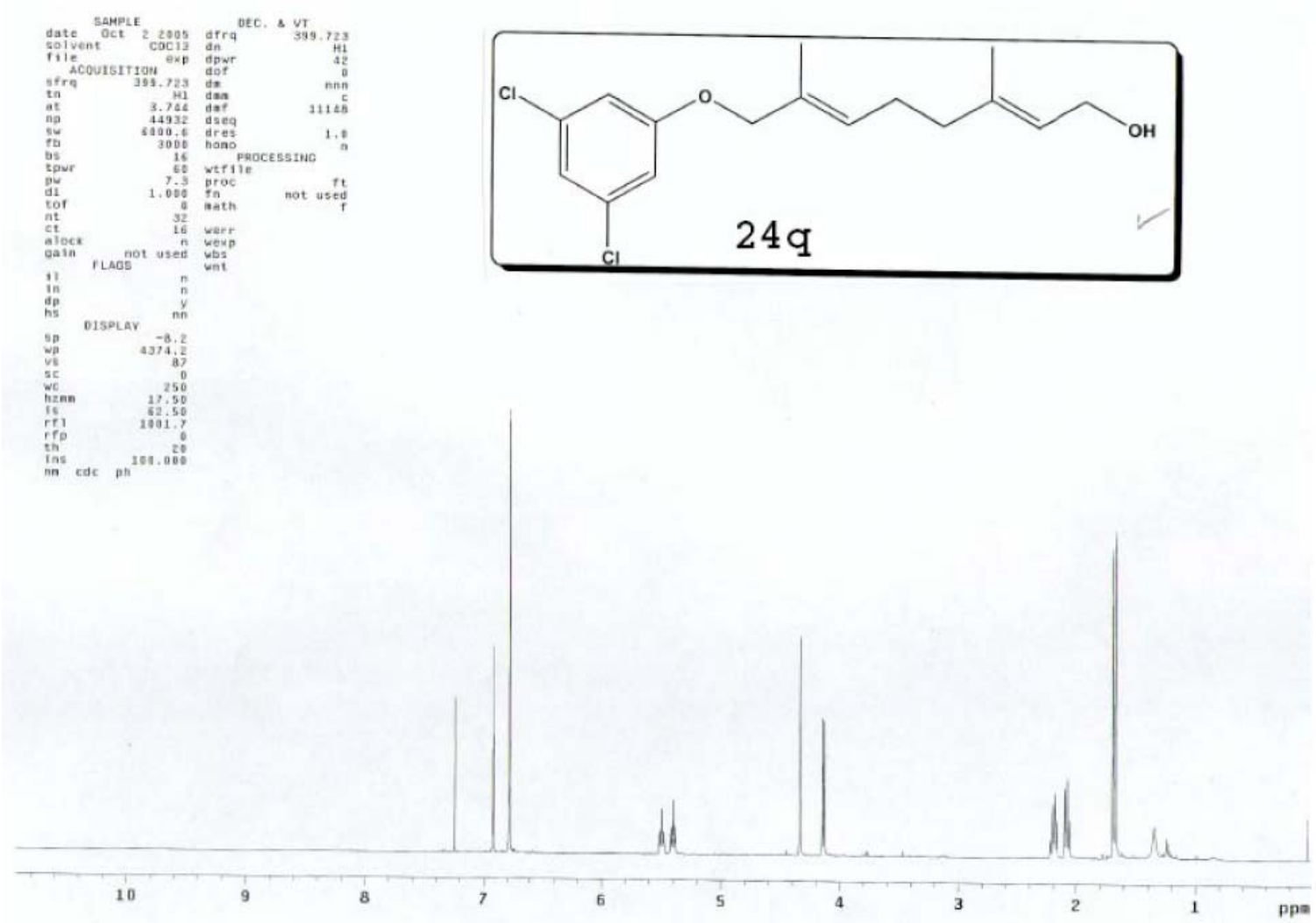
      SAMPLE
date    Oct  6 2005
solvent      CDCl3
file            exp
   ACQUISITION
sfrq        399.723
tn               H1
at              3.744
np              44932
sw             6000.6
fb              3000
bs                16
tpwr              60
pw               7.3
d1             1.000
tof                0
nt                32
ct                16
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp              -2.6
wp           4020.4
vs                88
sc                 0
wc               250
hzmm           15.08
is           500.00
rfl          1881.7
rfp                0
th                20
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq        399.723
dn               H1
dpwr             42
dof               0
dm              nnn
dmm               c
dmf           11148
dseq
dres           1.0
homo             n
    PROCESSING
wtfile
proc             ft
fn         not used
math              f
werr
wexp
wbs
wnt
```



24p



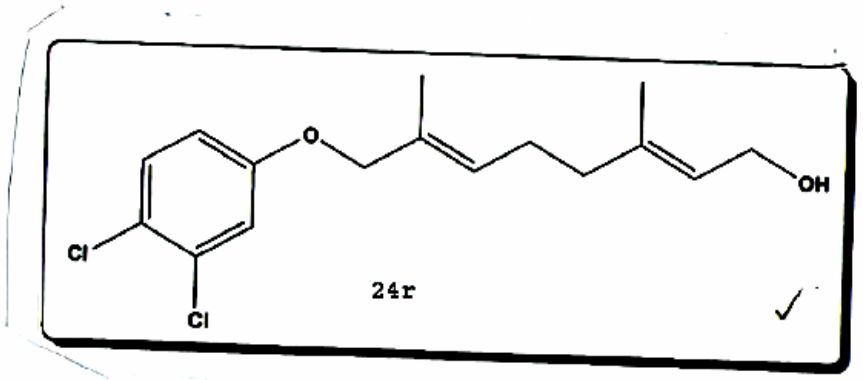

SAMPLE
date Oct 2 2005
solvent CDCl3
file exp
ACQUISITION
sfrq 399.723
tn H1
at 3.744
np 44932
sw 6800.6
fb 3000
bs 16
tpwr 60
pw 7.3
d1 1.000
tof 0
nt 32
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -8.2
wp 4374.2
vs 87
sc 0
wc 250
hzmm 17.50
is 62.50
rf1 1801.7
rfp 0
th 20
ins 100.000
nm cdc ph
DEC. & VT
dfrq 399.723
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11148
dseq
dres 1.8
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Cl
O
OH
Cl
24q
10
9
8
7
6
5
4
3
2
1
ppm

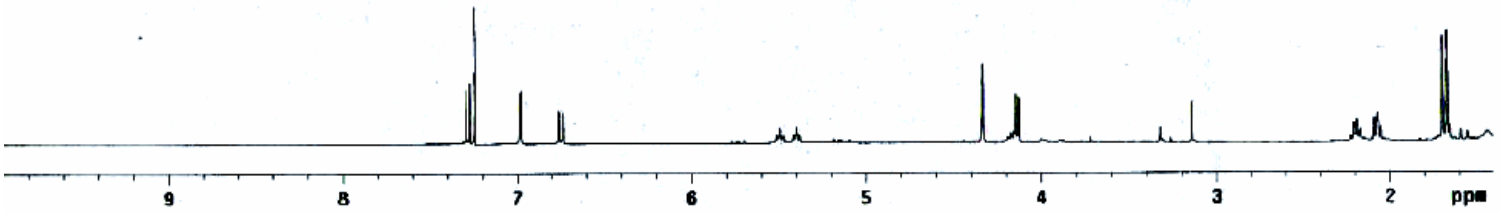
```
exp2  std1h

      SAMPLE
date    Oct  2 2005
solvent        CDCl3
file /opt/dist/dat~
e/petc/SUBBU/TS-II~
-15-13-oct2-2005.f~
                 id
   ACQUISITION
sfrq        399.723
tn               H1
at             3.744
np             44932
sw            6098.6
fb             3000
bs               16
tpwr             60
pw              7.3
d1            1.000
tof                0
nt                32
ct                26
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp             569.7
wp            3437.1
vs                42
sc                 0
wc               250
hzmm           13.75
is             62.50
rfl           1001.7
rfp                0
th                20
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq        399.723
dn               H1
dpwr             42
dof               0
dm              nnn
dmm               c
dmf           11148
dseq
dres            1.0
homo              n
     DEC2
dfrq2             0
dn2
dpwr2             1
dof2              0
dm2               n
dmm2              c
dmf2            200
dseq2
dres2           1.0
homo2             n
     PROCESSING
wtfile
proc             ft
fn         not used
math              f

werr
wexp
wbs
wnt
```

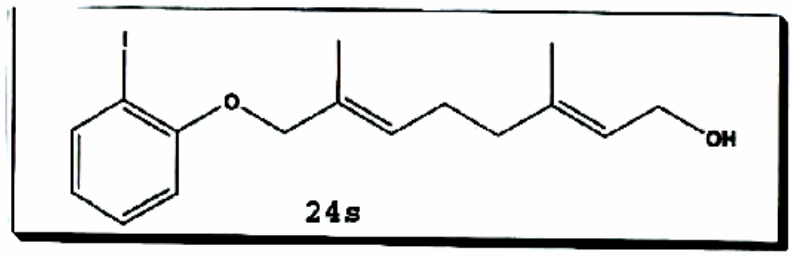




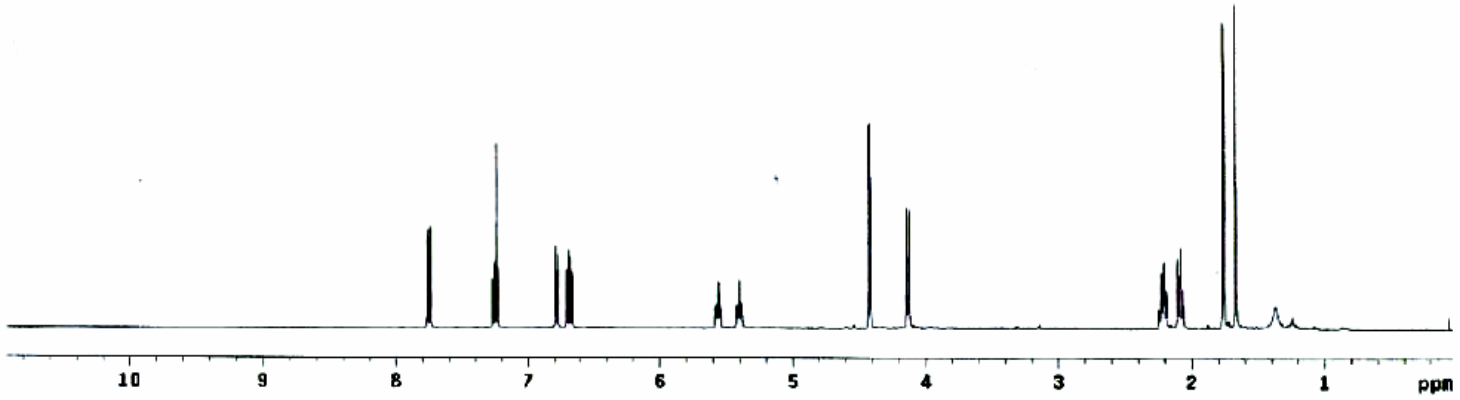
```
      SAMPLE
date   Oct  2 2005
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        399.723
tn              H1
at            3.744
np            44932
sw          5000.6
fb             3000
bs               16
tpwr             60
pw              7.3
d1            1.000
tof               0
nt               32
ct               25
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp             -8.2
wp           4374.2
vs               56
sc                0
wc              250
hzmm          17.50
is            62.50
rf1         1061.7
rfp               0
th               28
ins          100.000
nm  cdc  ph
```

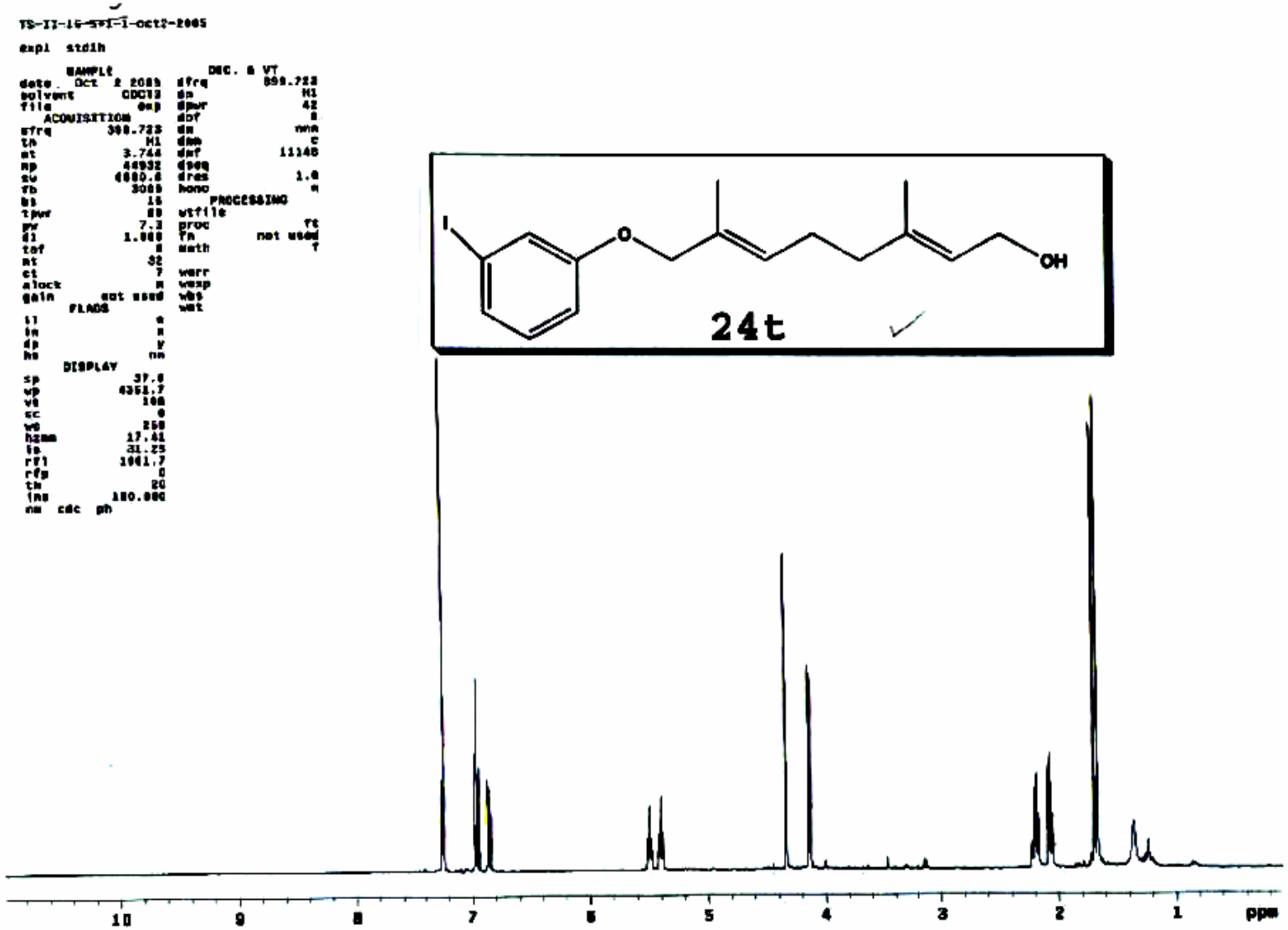
```
      DEC. & VT
dfrq        399.723
dn               H1
dpwr             42
dof               0
dn              nnn
dmm               c
dmf           11146
dseq
dres            1.0
homo              n
     PROCESSING
wtfile
proc             ft
fn        not used
math              f

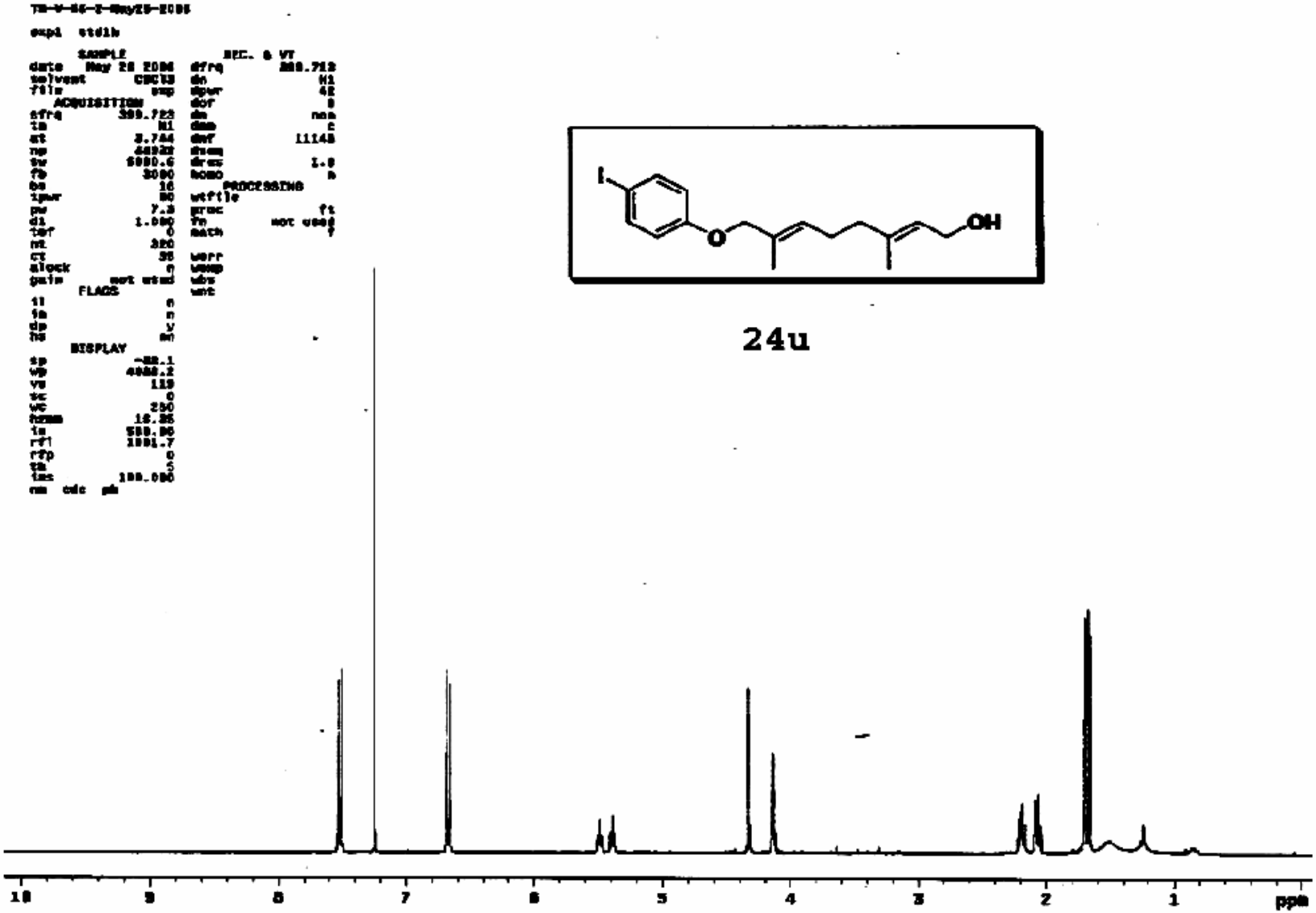
werr
wexp
wbs
wnt
```

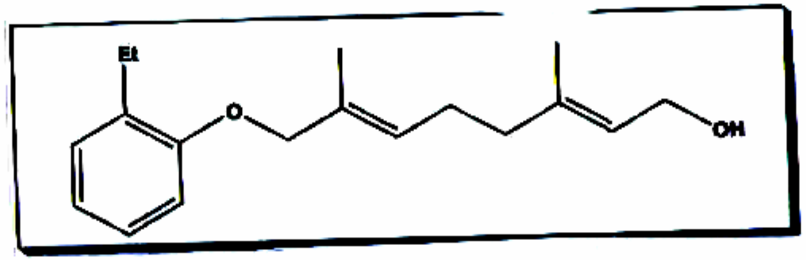


24s




TS-II-16-SPI-1-oct2-2005
expl stdin
SAMPLE
date Oct 2 2005
solvent CDCl3
file exp
ACQUISITION
sfrq 300.725
tn H1
at 3.744
np 44932
sw 6880.6
fb 3085
bs 16
tpwr 59
pw 7.3
d1 1.000
tof 0
nt 32
ct 7
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 37.6
wp 4351.7
vs 168
sc 0
wc 250
hzmm 17.41
is 31.25
rfl 1961.7
rfp 0
th 20
ins 180.000
nm cdc ph
DEC. & VT
dfrq 899.723
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11145
dmfq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
I
O
OH
24t
10
9
8
7
6
5
4
3
2
1
ppm


24u
OH
O
I
ppm



24v

TS-IV-30-6-oct7-2005

expl stdlh

SAMPLE
date Oct 6 2005
solvent CDCl3
file exp

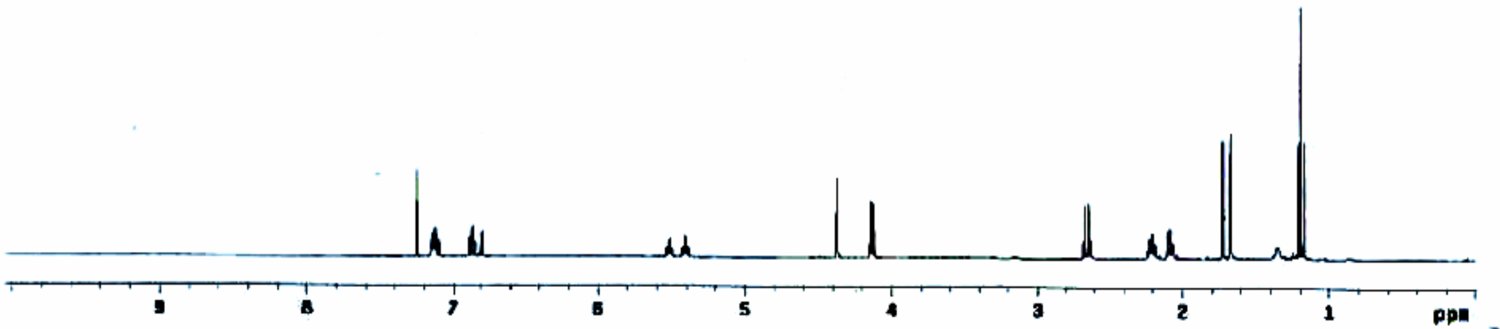
ACQUISITION
sfrq 399.723
tn H1

DEC. & VT
dfrq 399.723
dn H1
dpwr 42

PROCESSING
proc ft
fn not used

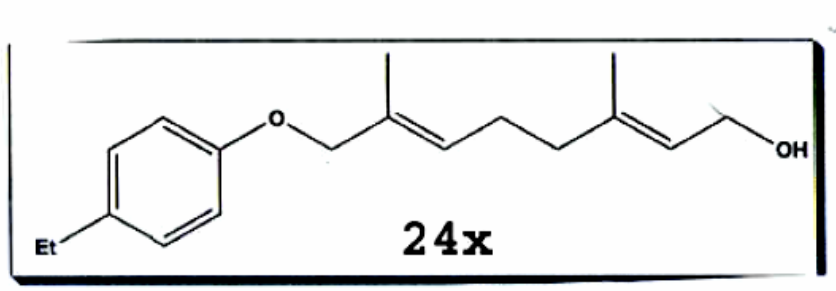
FLAGS

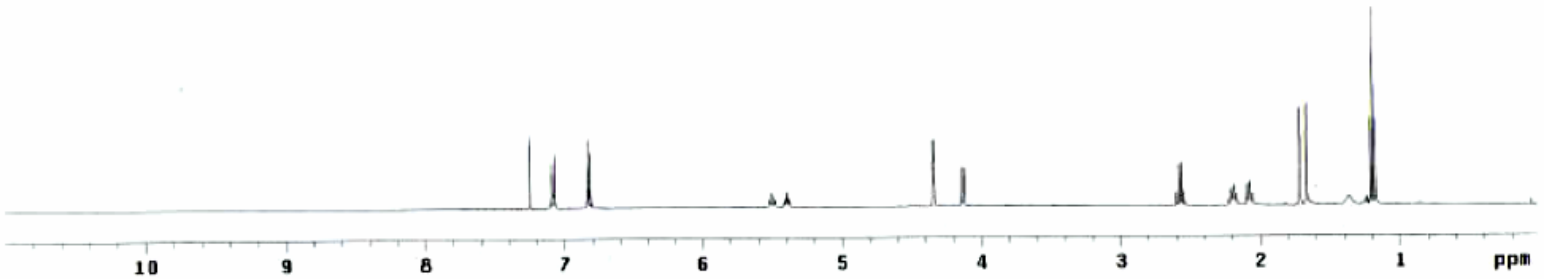
DISPLAY

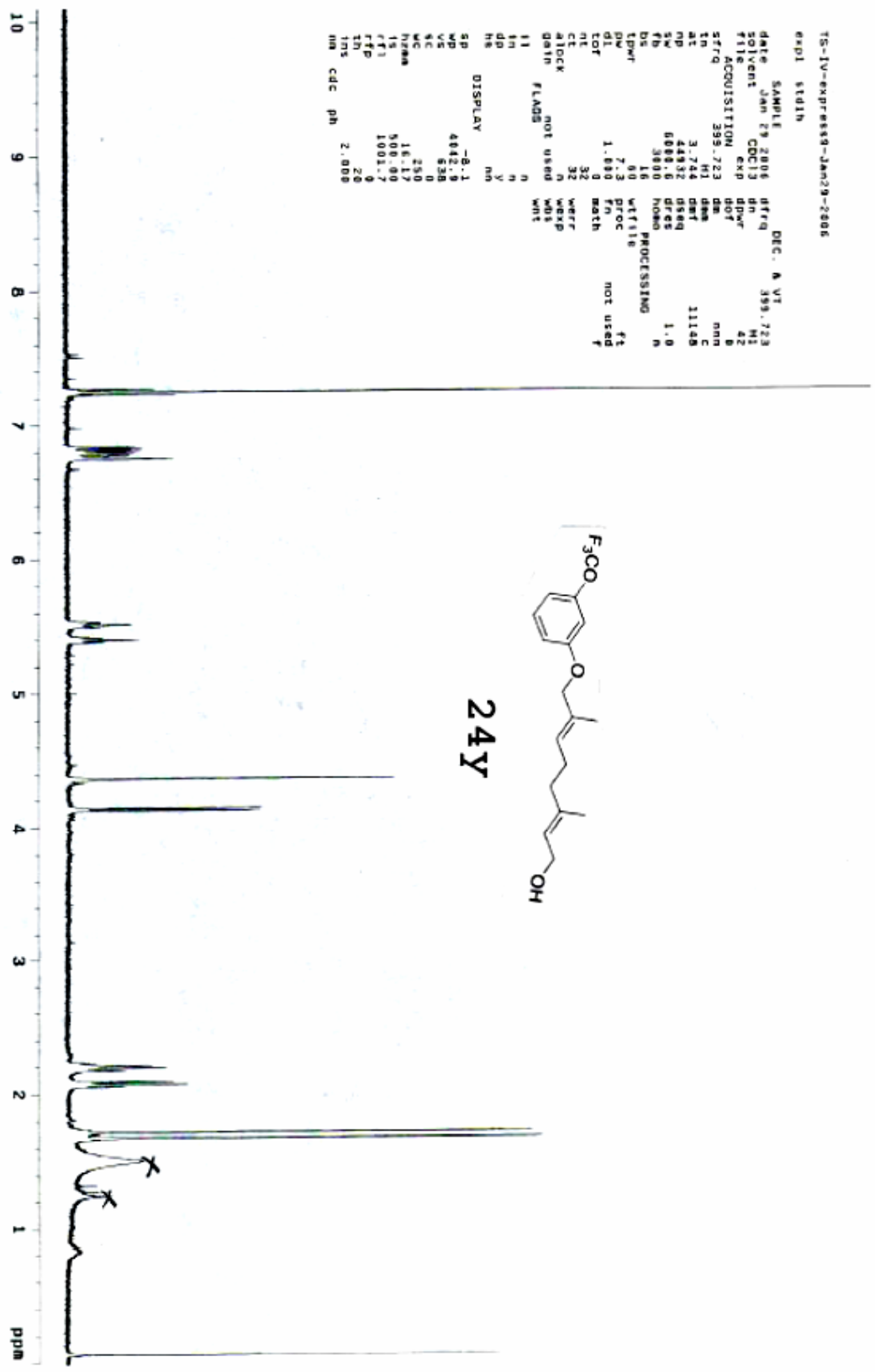


```
exp1  stdih

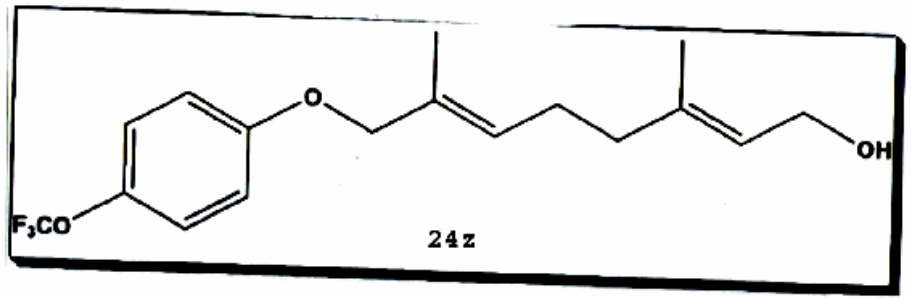
      SAMPLE                    DEC. & VT
date    Oct  2 2005  dfrq          399.723
solvent       CDCl3  dn               H1
file            exp  dpwr             42
   ACQUISITION       dof               0
sfrq          399.723  dm              nnn
tn               H1  dmm               c
at             3.744  dmf           11148
np            44932  dseq
sw           6000.6  dres             1.0
fb             8000  homo              n
bs               16       PROCESSING
tpwr             60  wtfile
pw              7.3  proc              ft
d1            1.000  fn          not used
tof               0  math              f
nt               32
ct               25  werr
alock             n  wexp
gain      not used  wbs
      FLAGS         wnt
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp             7.1
wp          4389.2
vs              32
sc               0
wc             250
hzmm         17.56
is           500.00
rfl          1001.7
rfp              0
th              20
ins          100.000
nm  cdc  ph
```



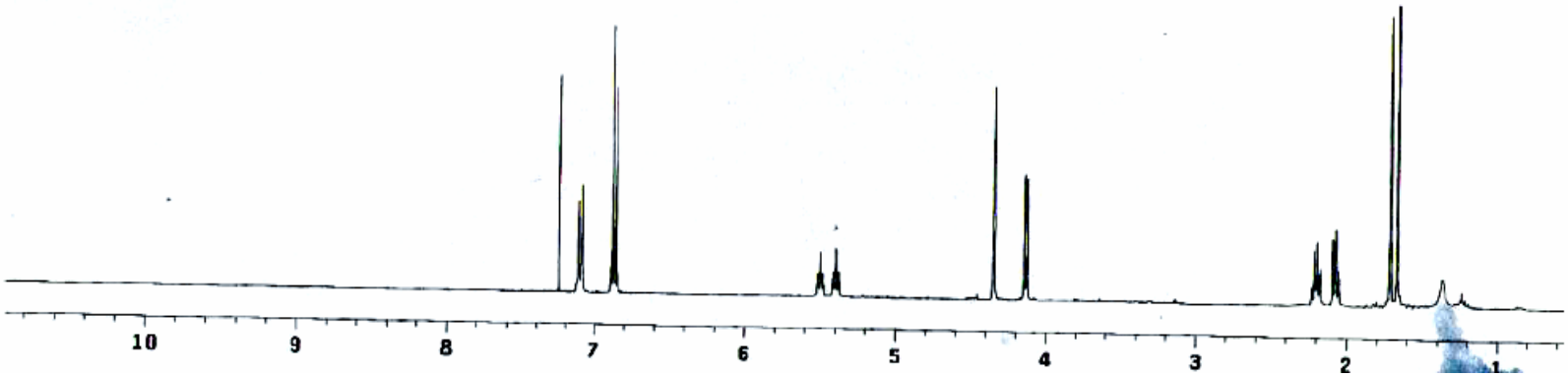



ts-iv-express3-Jan29-2006
expl stdin
SAMPLE
date Jan 29 2006
solvent CDCl3
file
ACQUISITION
sfrq 399.723
tn
at
np
sw
fb
bs
tpwr
pw
dl
tof
nt
ct
alock
gain
FLAGS
il
in
dp
hs
DISPLAY
sp -8.1
wp 4042.9
vs 638
sc 0
wc 250
hzmm 16.17
is 500.00
rfl 1001.7
rfp 0
th 20
ins 2.000
nm cdc ph
DEC. A VT
dfrq 399.723
dfrq
dmr
dmf
dm
dmm
dpwr
dpwf
spare
not used
PROCESSING
wtfile
proc
ft
fn
not used
f
math
werr
wexp
wbs
wnt
n
n
y
nn
24y
F3CO
O
OH
10
9
8
7
6
5
4
3
2
1
ppm

date	Oct 2 2005	dfrq	399.723
solvent	CDCl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	399.723	dm	nnn
tn	H1	dmm	c
at	3.744	dmf	11148
np	44932	dseq	
sw	6000.6	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	68	wtfile	
pw	7.3	proc	ft
d1	1.000	fn	not used
tof	0	math	f
nt	32		
ct	16	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-8.2		
wp	4374.2		
vs	47		
sc	0		
wc	250		
hzmm	17.58		
is	62.50		
rfl	1001.7		
rfp	0		
th	20		
ins	100.000		
nm cdc ph			



24z

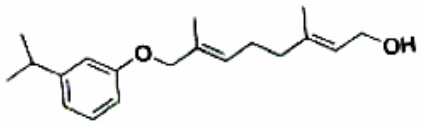


TS-IV-38-7-OCT7-2005

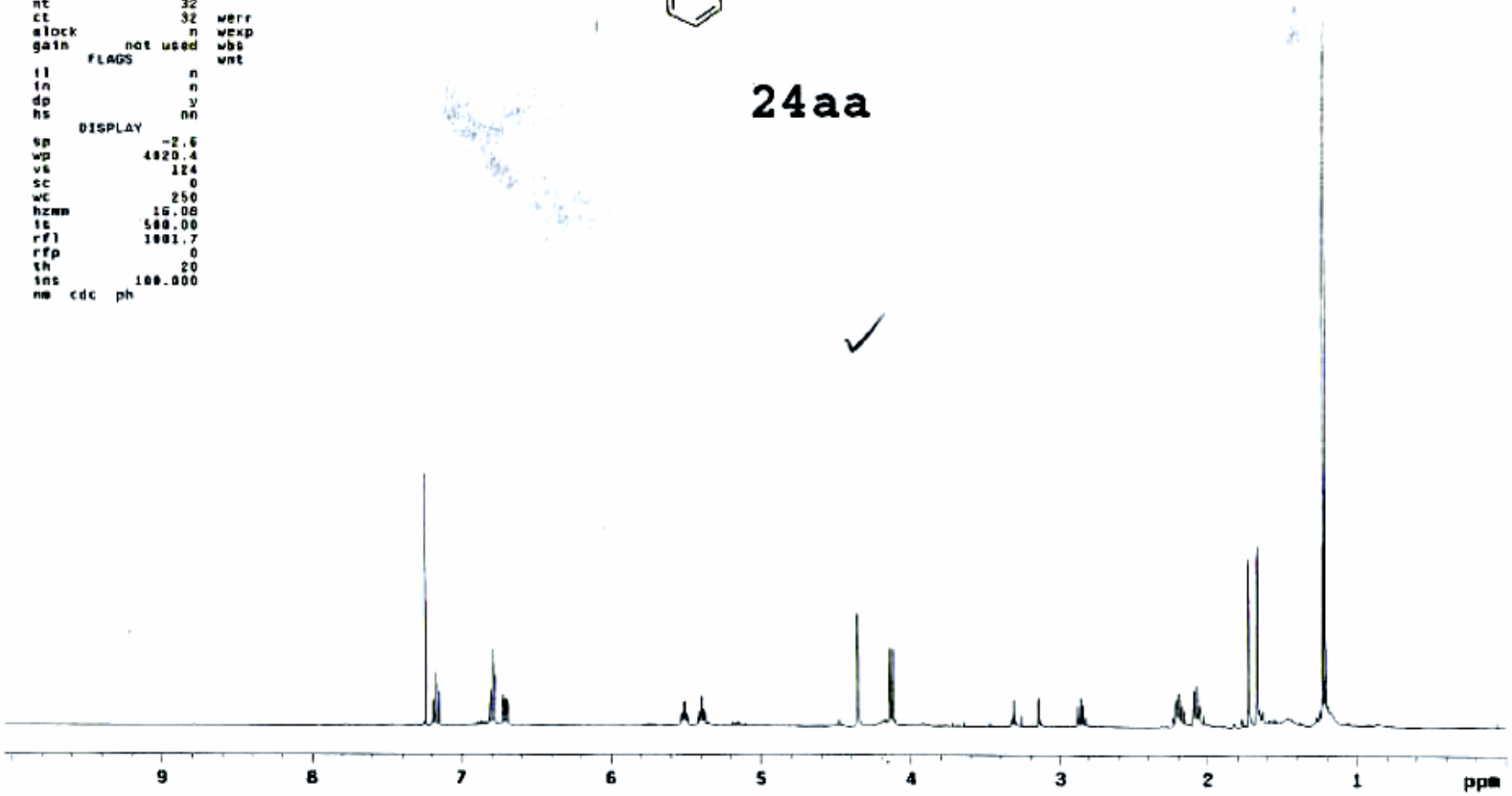
expl stdih

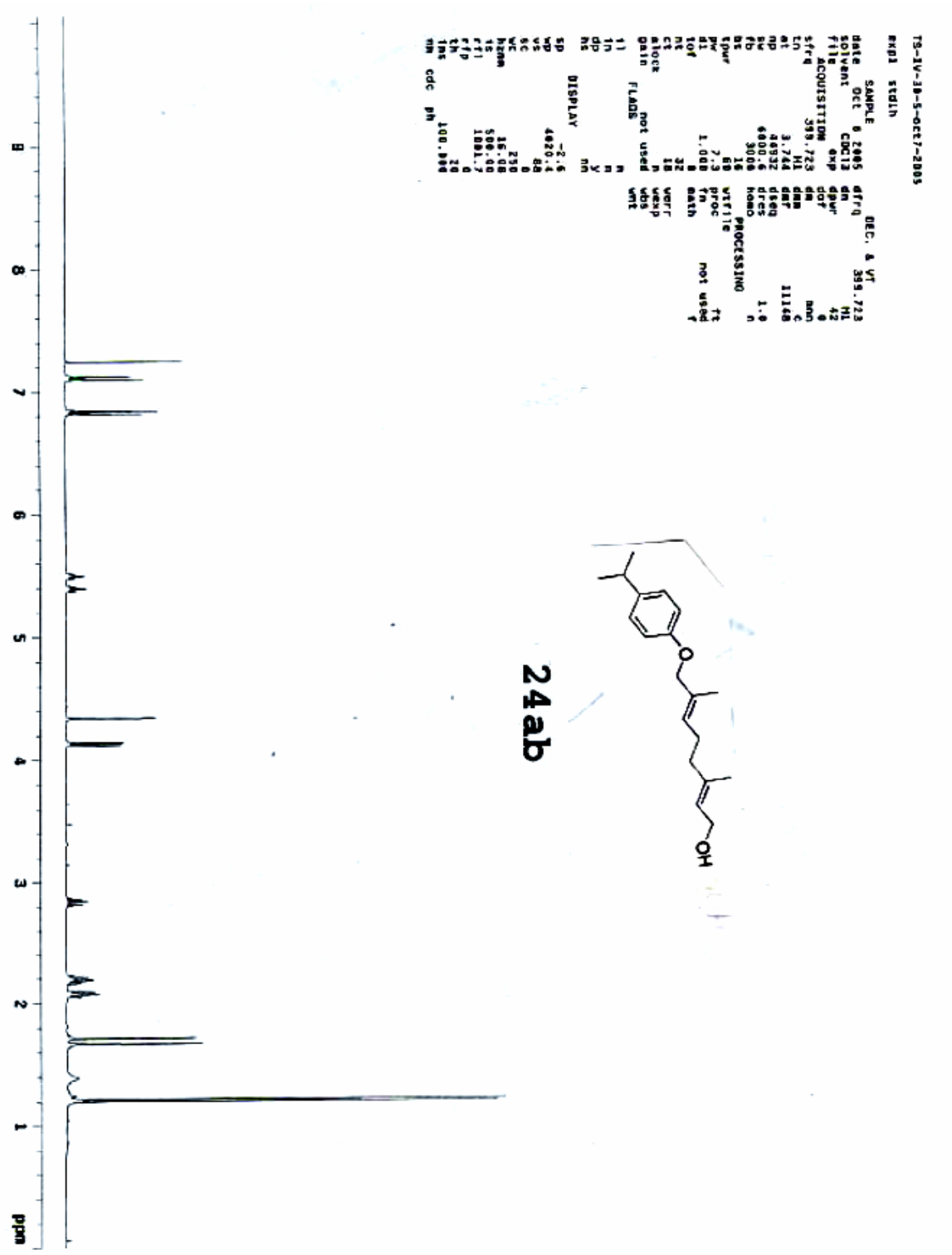
SAMPLE		DEC. & VT	
date	Oct 6 2005	dfrq	399.723
solvent	CDCl3	dn	H1
file	rxp	dpwr	42
ACQUISITION		dof	0
sfrq	399.723	dm	nnn
tn	H1	dmm	c
at	3.744	dmf	11148
np	44932	dseq	
sw	6000.6	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	60	wtfile	
pw	7.3	proc	ft
d1	1.000	fn	not used
tof	0	math	f
nt	32		
ct	32	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2.6		
wp	4920.4		
vs	124		
sc	0		
wc	250		
hzmm	16.08		
is	500.00		
rfl	1001.7		
rfp	0		
th	20		
ins	100.000		

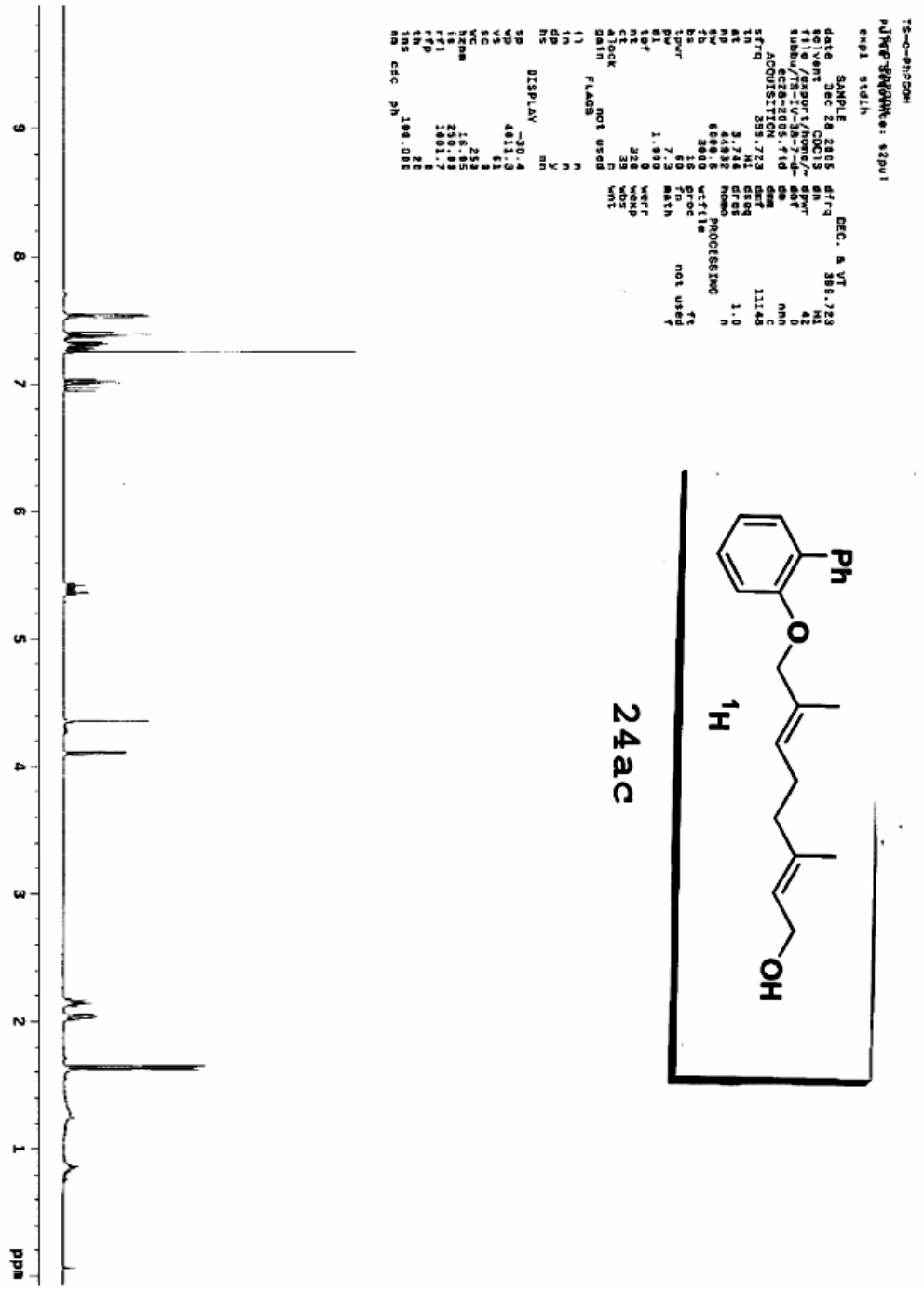
nm cdc ph



24aa



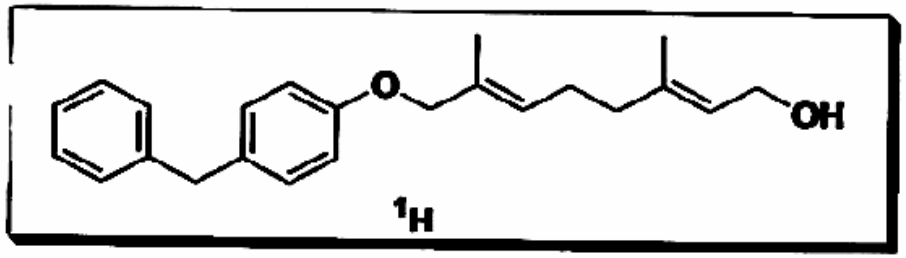

TS-IV-38-5-oct7-2005
expl stqlh
SAMPLE
date Oct 8 2005
solvent CDCl3
file
ACQUISITION
sfrq 399.723
tn H1
at 3.744
np 46932
sw 6000.6
fb 3000
bs 16
tpwr 60
pw 2.3
d1 1.000
tof 0
nt 32
ct 16
alock n
gain n
FLAGS not used
l1 n
in n
dp y
hs nn
DISPLAY
sp -2.5
wp 4020.4
vs 88
sc 0
wc 250
hzmm 16.08
is 500.00
rfl 1881.7
rfp 0
th 20
lns 100.000
nm cdc pn
DEC. & VT
dfrq 399.723
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11140
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
24ab
OH
8
7
6
5
4
3
2
1
ppm


24ac
^1H
Ph
O
OH

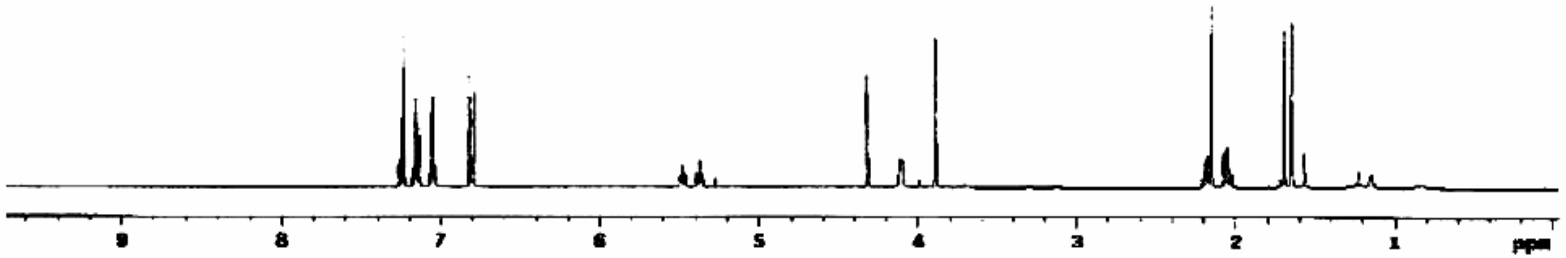
```
S-VIII-67-1-1-Nov28-2007

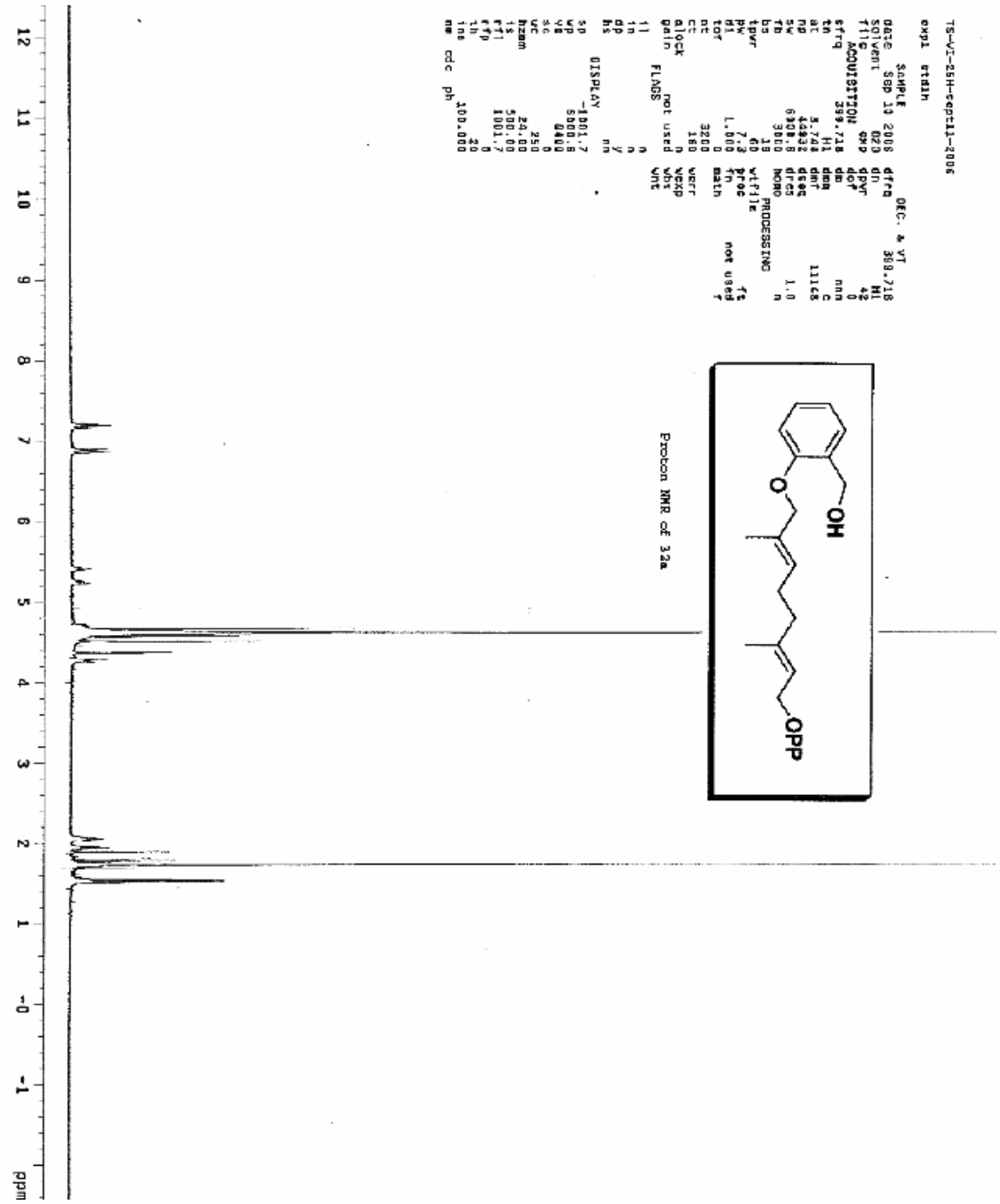
npt  std1h

      SAMPLE                  DEC. & VT
stu   Nov 28 2007  dfrq          399.717
olvent       CDCl3  dn               H1
file           exp  dpwr              42
  ACQUISITION       dof               0
frq          399.717  dm              nnn
o              H1     dmm               c
t            2.744  dmf           11148
p           44632  dseq
w         8888.8  dres             1.0
a            8888  homo               n
s              16      PROCESSING
pwr            60  wtfile
w             7.3  proc              ft
l          1.000  fn          not used
of             0  math              f
t            ste
t              6  vorr
lock           n  vexp
sin      not used  wbs
      FLAGS        wnt
i                0
n                n
p                y
m               nn
      DISPLAY
p            -13.6
sp         4888.8
s              31
c               4
wp           250
wnn         18.63
w          500.00
ft         1001.7
fp               0
h               20
ns         100.000
e  cdc  ph
```



24ad

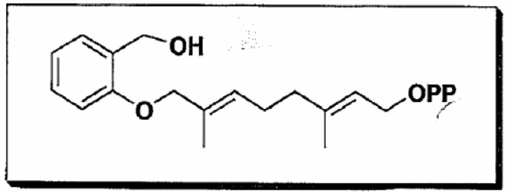



TS-VI-25H-sept13-2006
expt etmh
SAMPLE
Date Sep 13 2006
Solvent d2o
File ACQUISITION
sfrq 399.718
tn H1
at 5.744
np 44932
sw 6908.8
fb 3000
bs 16
tpwr 60
pw 7.3
pl 1.000
tof 0.0
nt 3200
cn 160
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1001.7
wp 5000.6
vs 6400
sc 0
wc 250
hzcm 24.00
is 500.00
rfl 1001.7
rfp 0
th 20
ins 100.000
nm cdc ph
OEC. & VT
dfrq 399.718
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11145
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math
werr
wexp
wbs
wnt
Proton NMR of 32a
OH
O
OPP
ppm

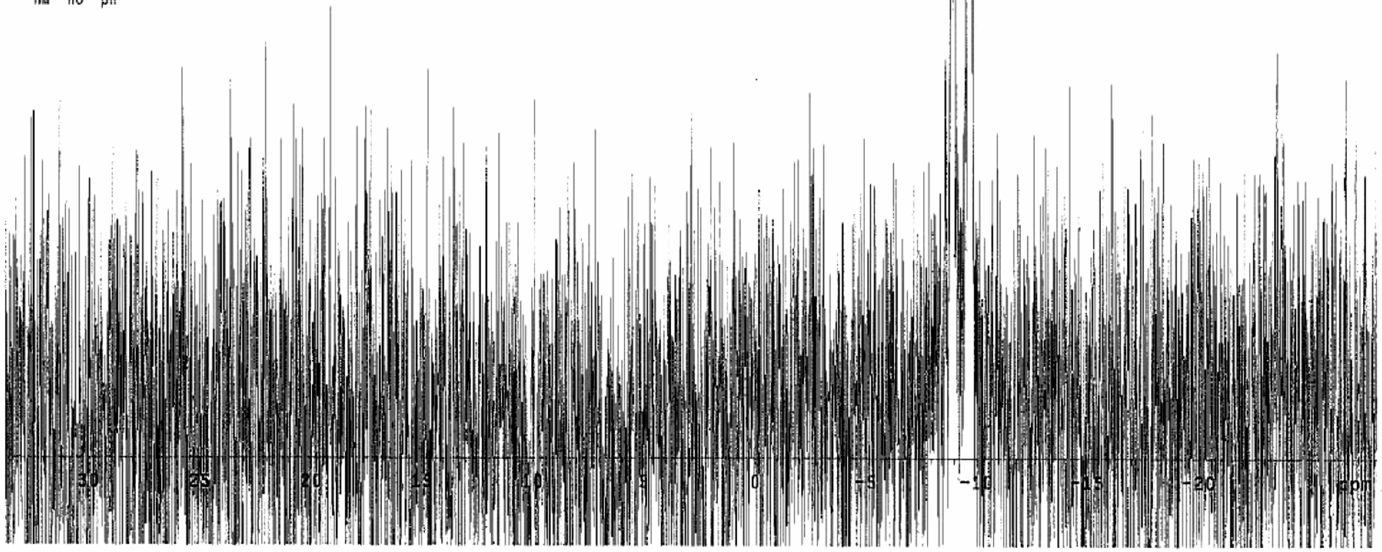
```
TS-VI-25-sept11-2006

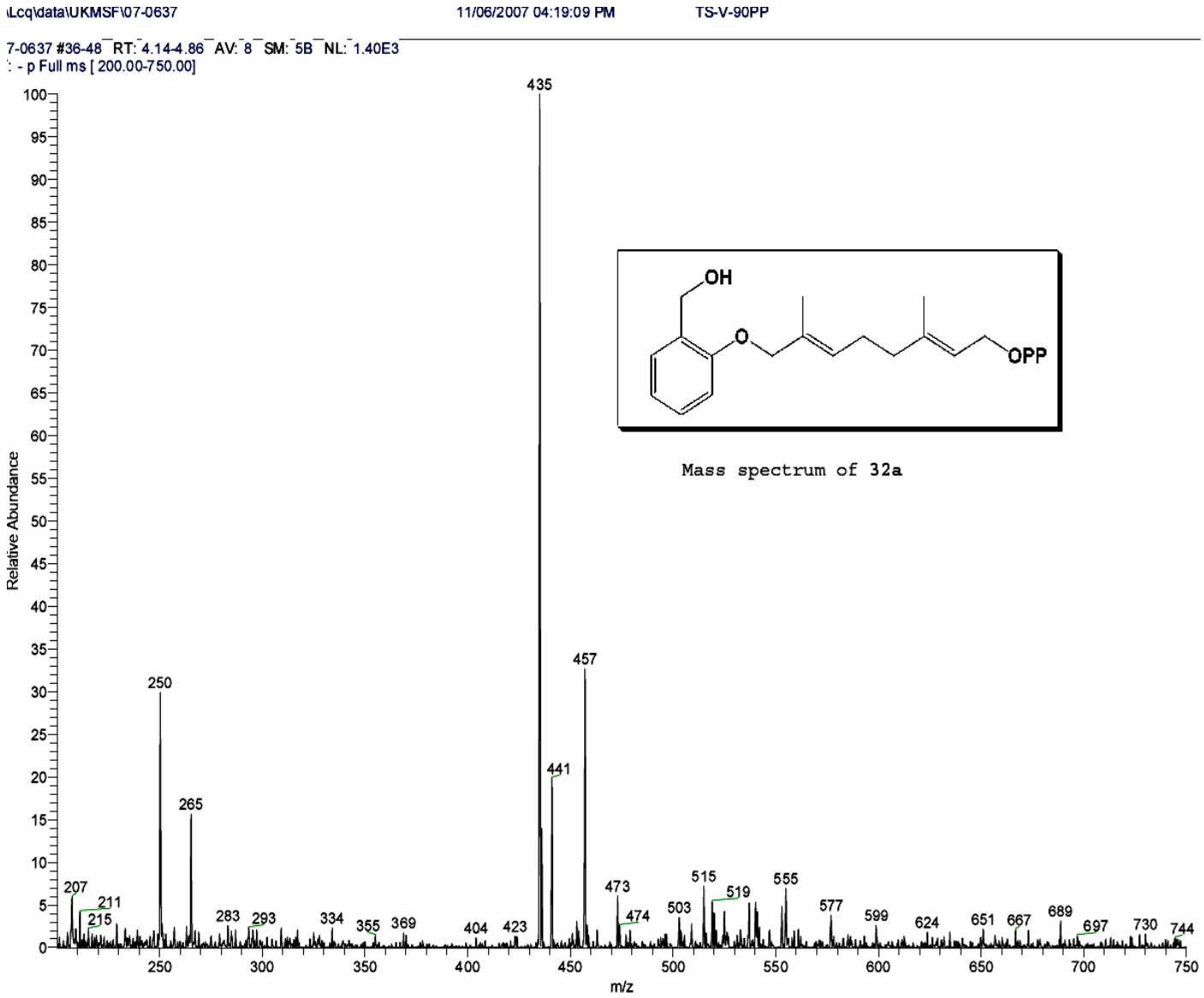
exp1  s2pul

      SAMPLE              DEC. & VT
date    Sep 10 2006   dfrq        399.718
solvent         D2O   dn               H1
file            exp   dpwr             42
   ACQUISITION        dof               0
sfrq        161.808   dm              yyy
tn              P31   dmm               w
at            0.800   dmf           11148
np            16000   dseq
sw          10000.0   dres            1.0
fb             6000   homo              n
bs               64       PROCESSING
tpwr             55   lb             1.00
pw             10.0   wtfile
d1                0   proc             ft
tof               0   fn         not used
nt             3200   math              f
ct             1856
alock             n   werr
gain       not used   wexp
      FLAGS           wbs
il                n   wnt
in                n
dp                y
hs               nn
     DISPLAY
sp          -4547.3
wp          10000.0
vs              154
sc                0
wc              250
hzmm          20.00
is           354.40
rfl          4547.3
rfp               0
th               20
ins         100.000
nm  no  ph
```

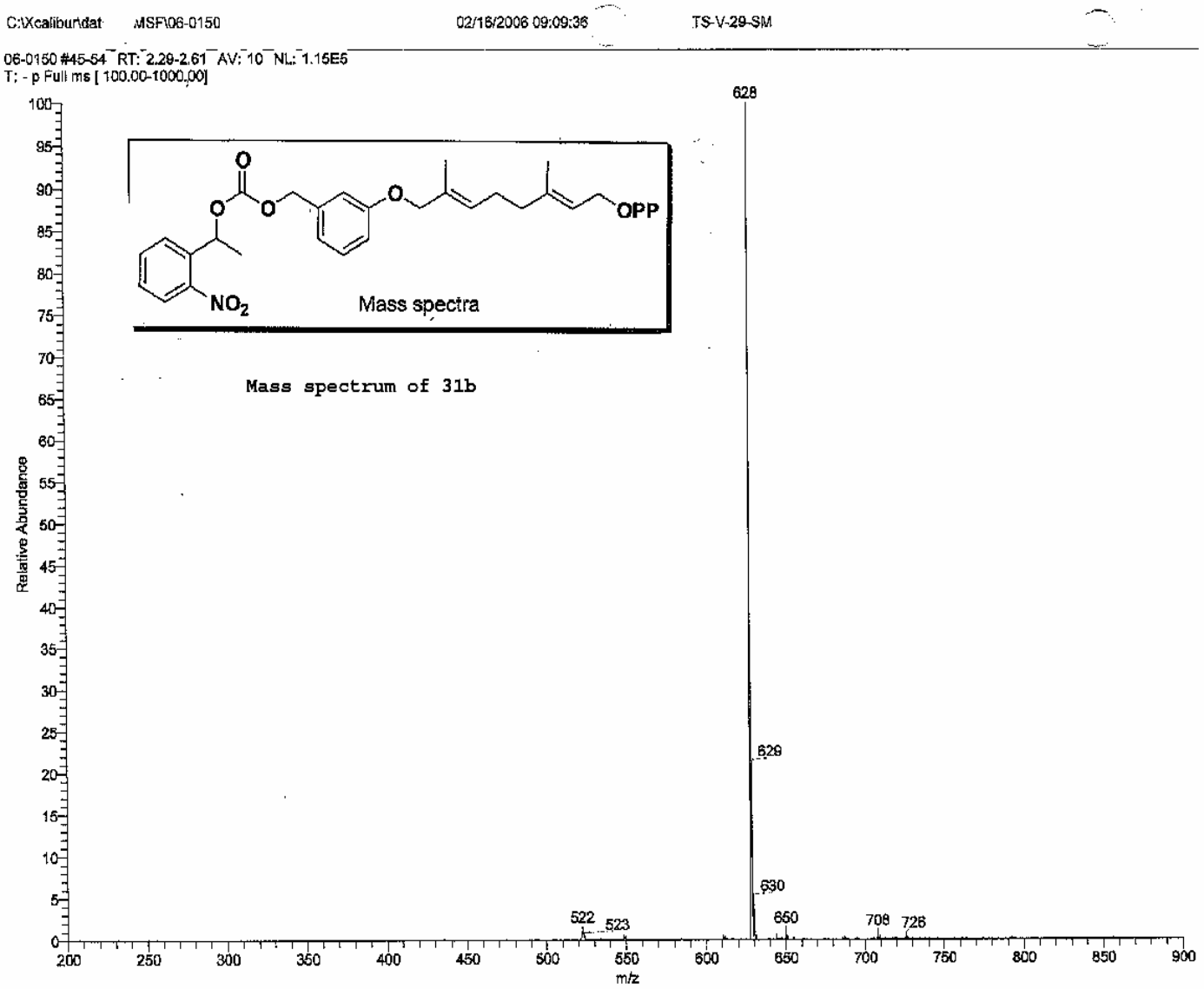


Phosphorus NMR of **32a**





Mass spectrum of **32a**

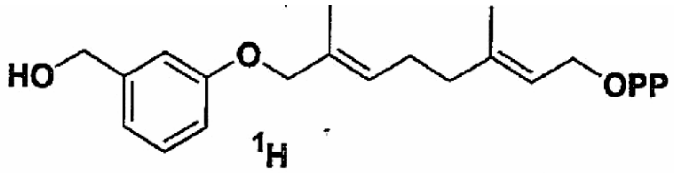

C:\Xcalibur\dat MSF\06-0150 02/16/2006 09:09:36 TS-V-29-SM
06-0150 #45-54 RT: 2.29-2.61 AV: 10 NL: 1.15E5
T: - p Full ms [100.00-1000.00]
O
O
O
O
OPP
NO2
Mass spectra
Mass spectrum of 31b
Relative Abundance
m/z
628
629
630
522
523
650
708
726

TS-V-29-irradiated-Feb22-2006

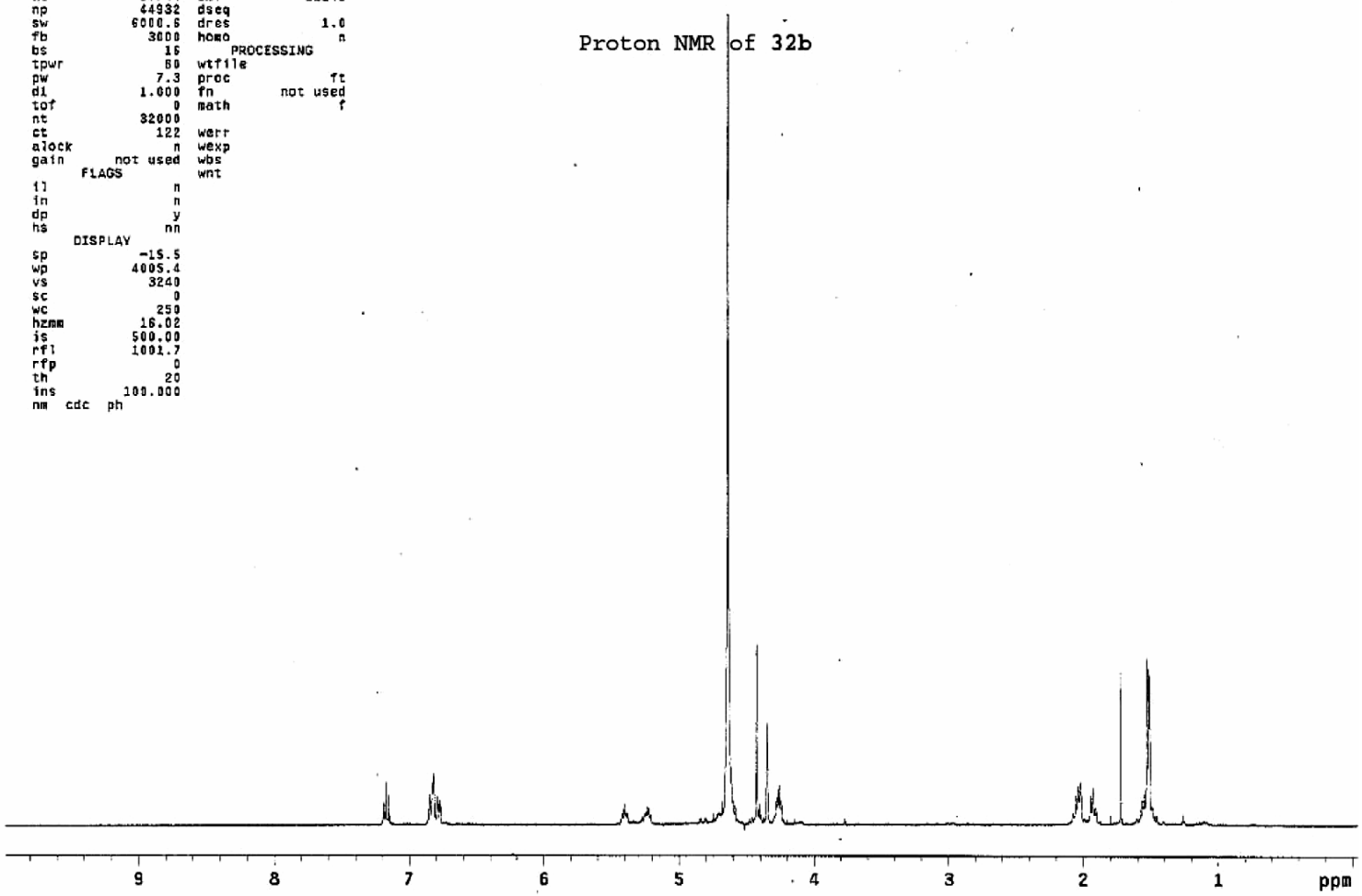
expl stdlh

SAMPLE		DEC. & VT	
date	Feb 22 2006	dfrq	399.724
solvent	D2O	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	0
sfrq	399.724	dm	nnn
tn	H1	dmm	c
at	3.744	dmf	11148
np	44932	dseq	
sw	6000.6	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	60	wtfile	
pw	7.3	proc	ft
d1	1.000	fn	not used
tof	0	math	f
nt	32000		
ct	122	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-15.5		
wp	4005.4		
vs	3240		
sc	0		
wc	250		
hzmm	16.02		
is	500.00		
rfl	1001.7		
rfp	0		
th	20		
ins	100.000		

nm cdc ph



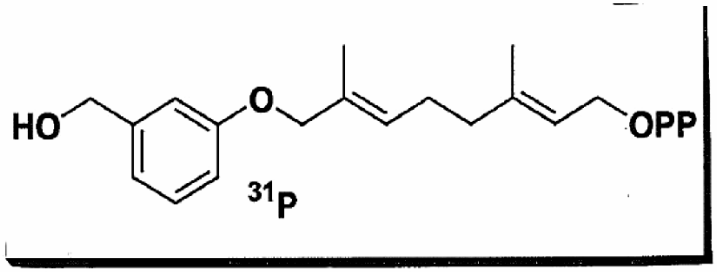
Proton NMR of 32b



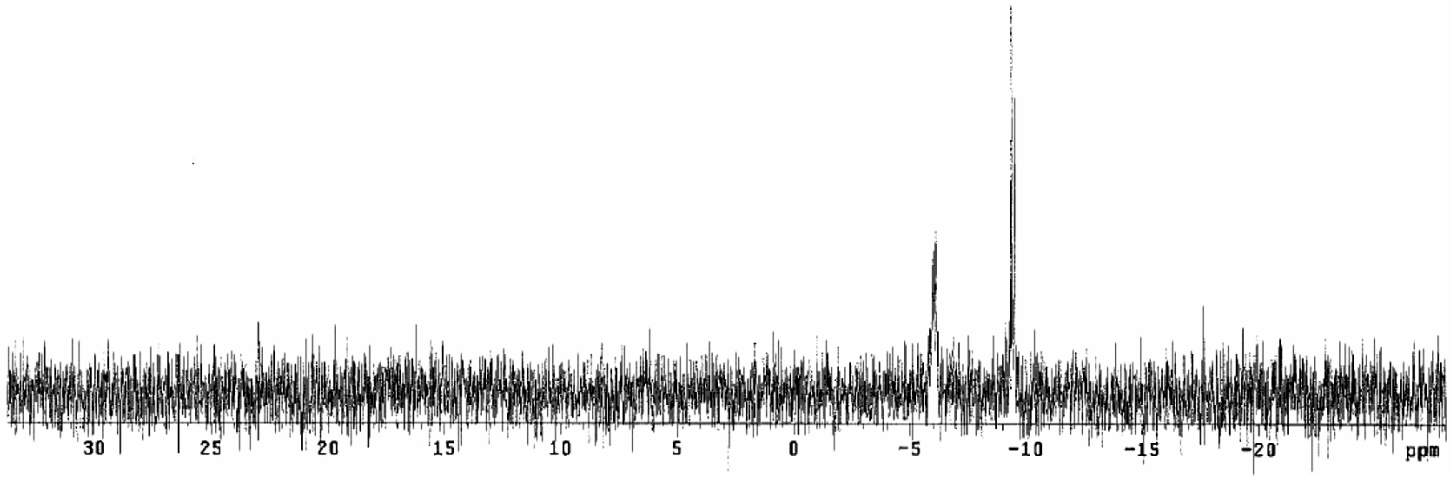
```
TS-IrradiatedPP

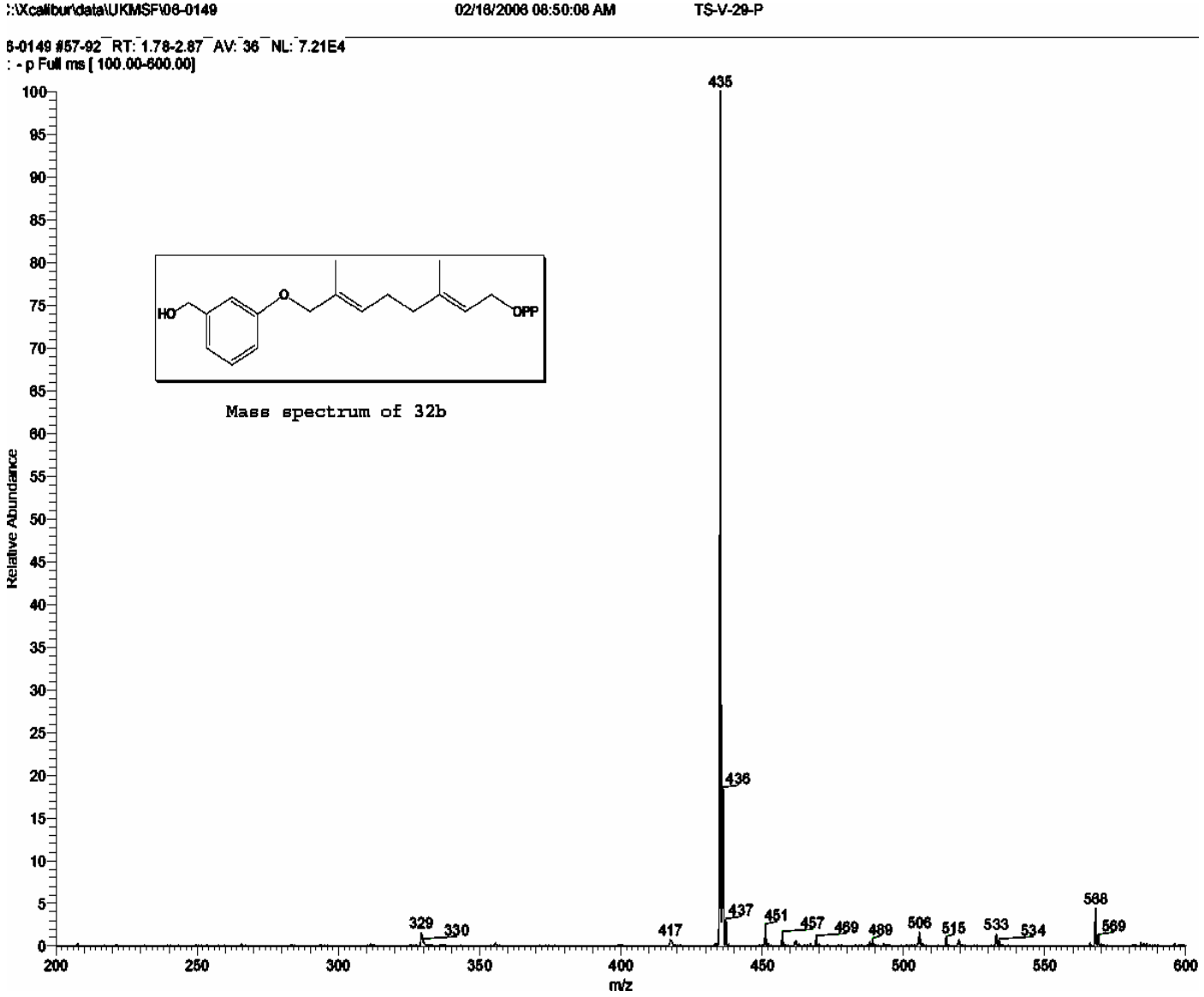
expl  s2pul

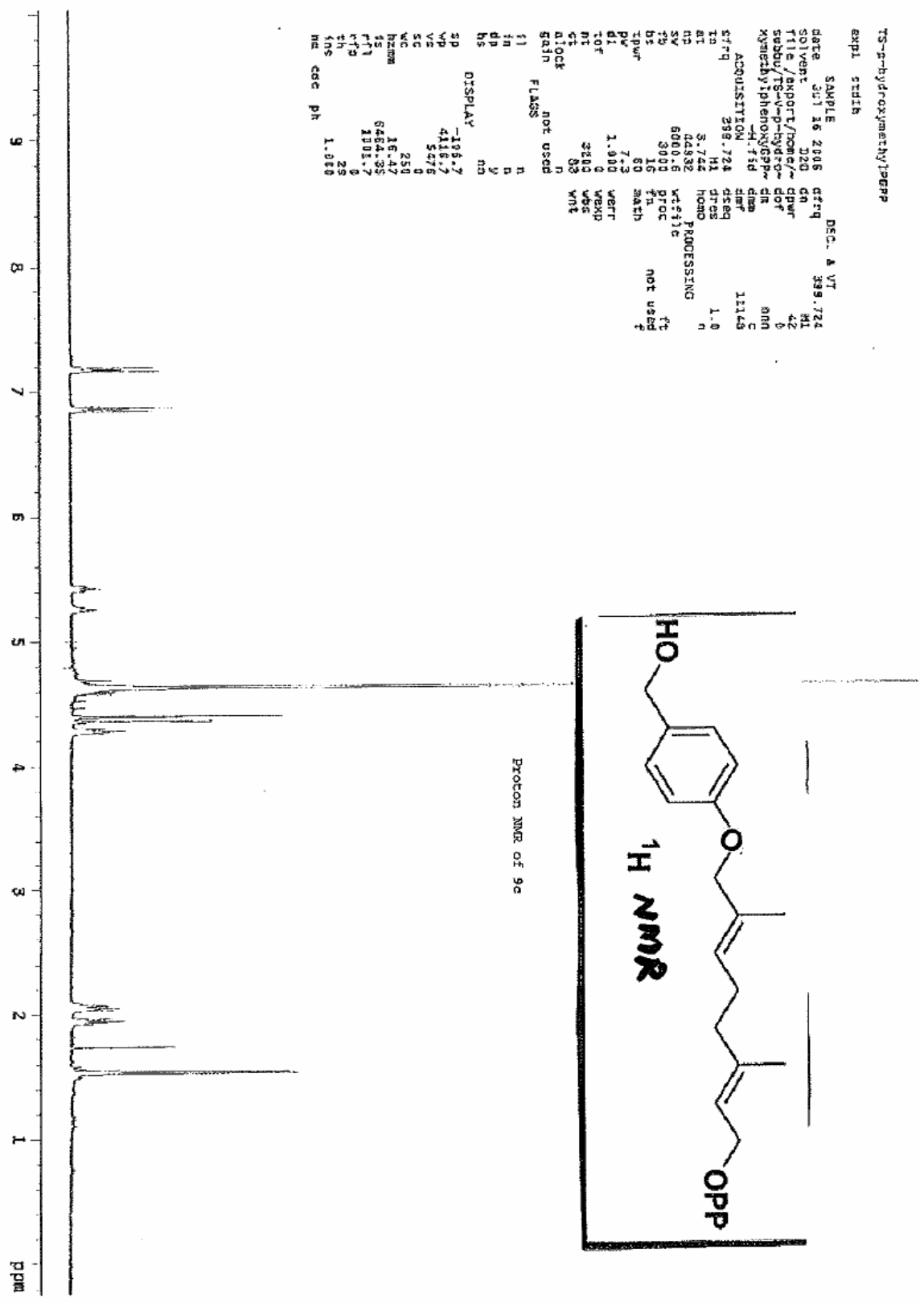
      SAMPLE            DEC. & VT
date   Feb 14 2006  dfrq       399.724
solvent       D2O  dn              H1
file /export/home/~ dpwr            42
subbu/TS-V-29PIR-F~ dof              0
     eb14-2006.fid  dm              yyy
   ACQUISITION      dmm             w
sfrq       161.811  dmf          11148
tn             P31  dseq
at           0.800  dres          1.0
np           16000  homo            n
sw         10000.0      PROCESSING
fb            6000  lb            1.00
bs              64  wtfile
tpwr            55  proc            ft
pw            10.0  fn        not used
d1               0  math             f
tof              0
nt           32000  werr
ct             323  wexp
alock            n  wbs
gain      not used  wnt
      FLAGS
il               n
in               n
dp               y
hs              nn
     DISPLAY
sp         -4547.2
wp         10000.0
vs              63
sc               0
wc             250
hzmm         20.00
is          354.40
rfl         4547.2
rfp              0
th              17
ins         100.000
nm  no  ph
```



Phosphorus NMR of **32b**




:\Xcalibur\data\UKMSF\06-0149
02/16/2006 08:50:08 AM
TS-V-29-P
6-0149 #57-92 RT: 1.78-2.87 AV: 36 NL: 7.21E4
: - p Full ms [100.00-600.00]
HO
O
OPP
Mass spectrum of 32b
Relative Abundance
m/z
435
436
437
329
330
417
451
457
469
489
506
515
533
534
568
569


TS-p-hydroxymethylPEPP
expl study
SAMPLE
date Jul 16 2006
solvent D2O
file /export/home/~ cpwr
sebbu/TS-v-p-hydro~ dof
xylmethylphenoxyPP~ dm
ACQUISITION
sfrq 399.724
tn H1
at 3.744
np 64832
sw 6000.6
fb 3000
bs 16
tpwr 60
pw 7.3
d1 1.000
tof 0
nt 2500
ct 93
alock n
gain not used
FLAGS
fl n
in n
dp y
hs nn
DISPLAY
sp -196.7
wp 4116.7
vs 5476
sc 0
wc 250
hzcm 16.42
sp 6464.93
fb 1101.7
rfl 0
rfp 25
th 1.000
ns
ne cec ph
DEC. & VT
dfrq 399.724
dn H1
dof 42
dm 0
dmm c
dmf 11143
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
ph
not used f
wexp
wbs
wnt
Proton NMR of 9c
HO
O
OPP
1H NMR
ppm
9
8
7
6
5
4
3
2
1

```
TS-p-hydroxymethylPGPP

exp1  s2pul

      SAMPLE              DEC. & VT
date    Jul 16 2006  dfrq        399.724
solvent        D2O   dn              H1
file /export/home/~  dpwr            42
subbu/TS-V-p-hydro~  dof              0
xymethylphenoxyGPP~  dm             yyy
                .fid  dmm              w
   ACQUISITION       dmf          11148
sfrq       161.811   dseq
tn             P31   dres           1.0
at           0.800   homo             n
np           16000       PROCESSING
sw         10000.0   lb             1.00
fb            6000   wtfile
bs              64   proc             ft
tpwr            55   fn         not used
pw            10.0   math              f
d1               0
tof              0   werr
nt            3200   wexp
ct             991   wbs
alock            n   wnt
gain       not used
      FLAGS
il               n
in               n
dp               y
hs              nn
     DISPLAY
sp         -4547.2
wp         10000.0
vs             101
sc               0
wc             250
hzmm         40.00
is          354.40
rfl         4547.2
rfp              0
th              40
ins         100.000
nm  no  ph
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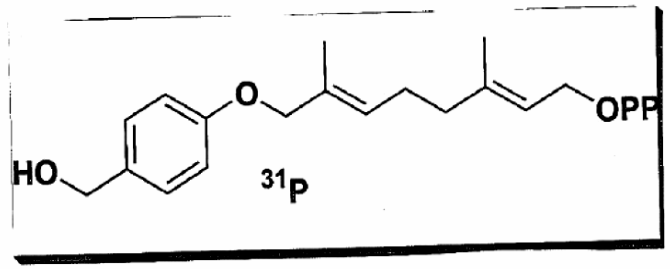
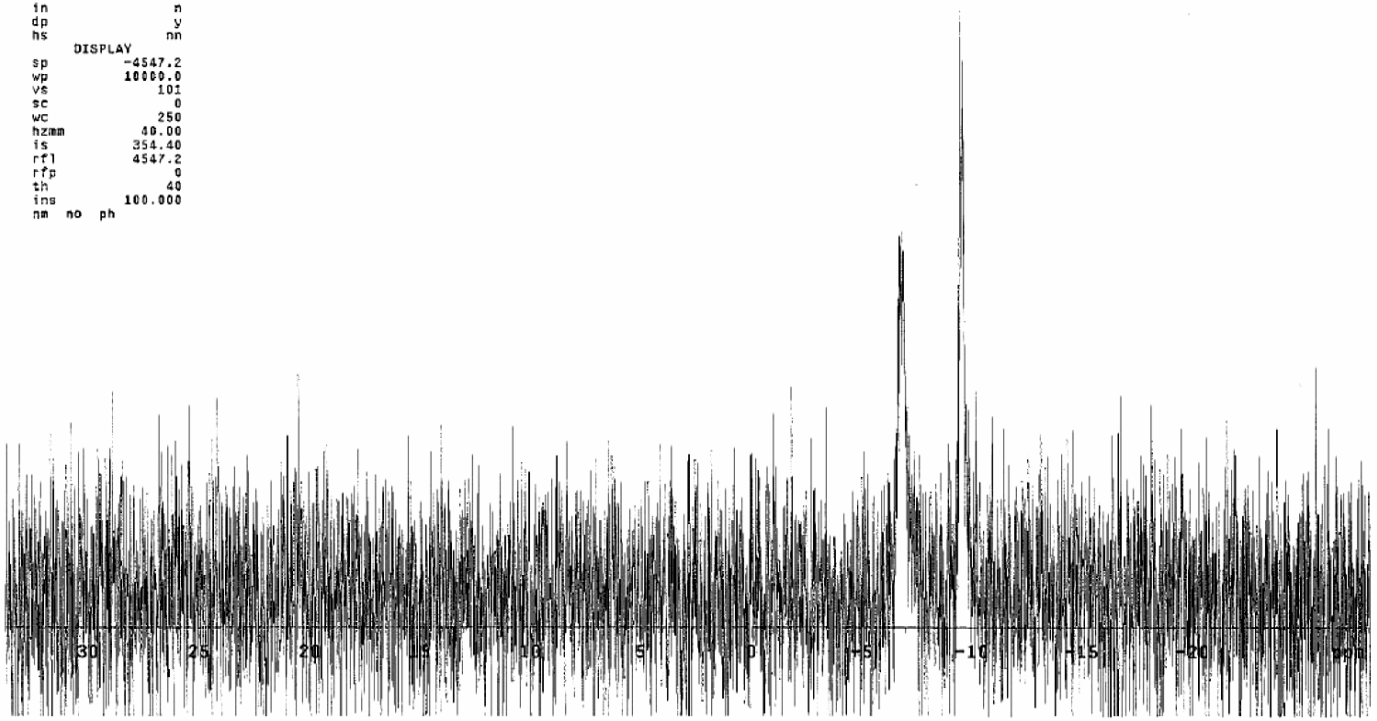
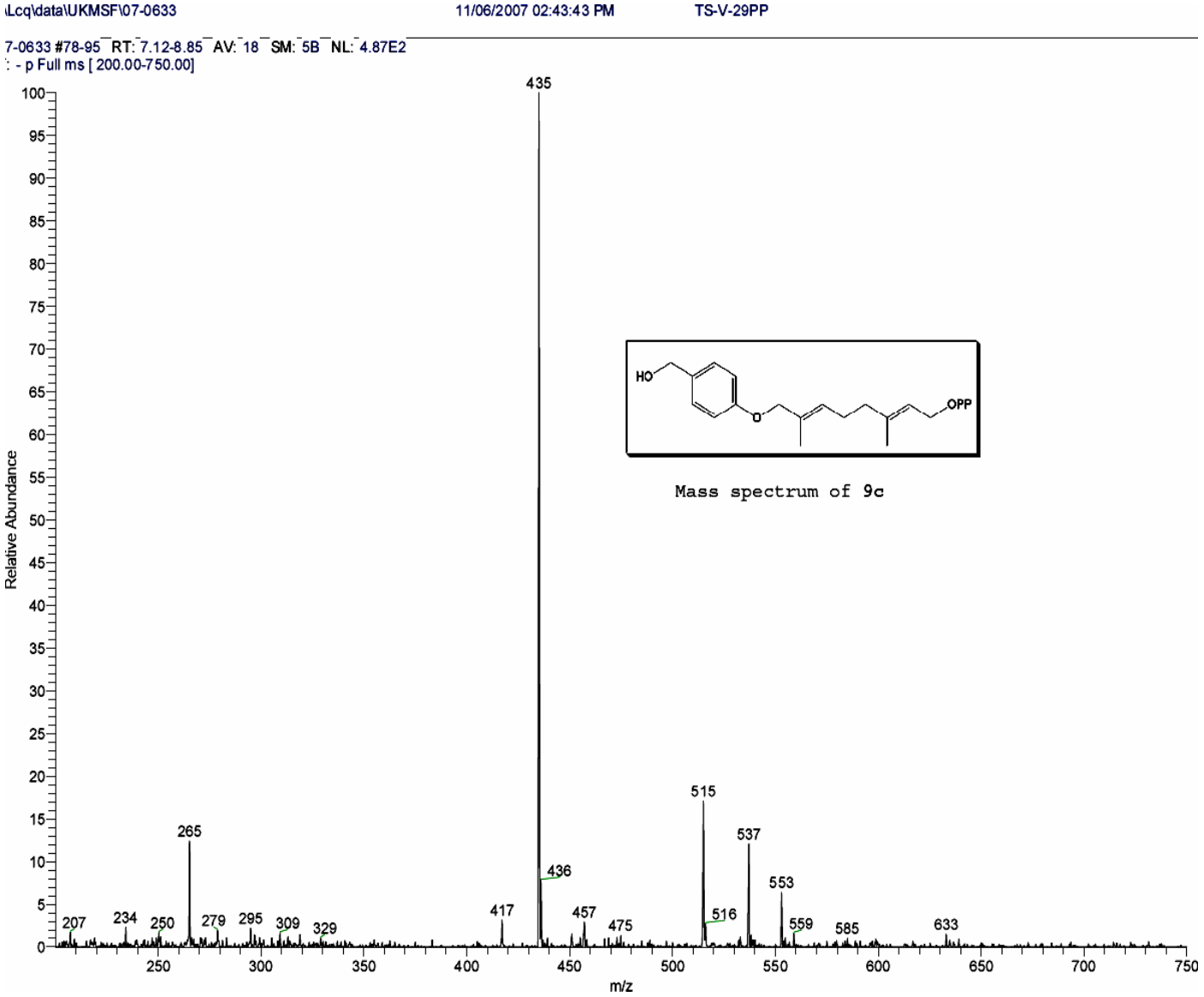


Phosphorus NMR of 9c





Mass spectrum of 9c