

Stereospecific Pd(0)-Catalyzed Arylation of an Allylic Hydroxy Phosphonate Derivative: Formal Synthesis of (S)(+)-ar-Turmerone

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Supporting Information

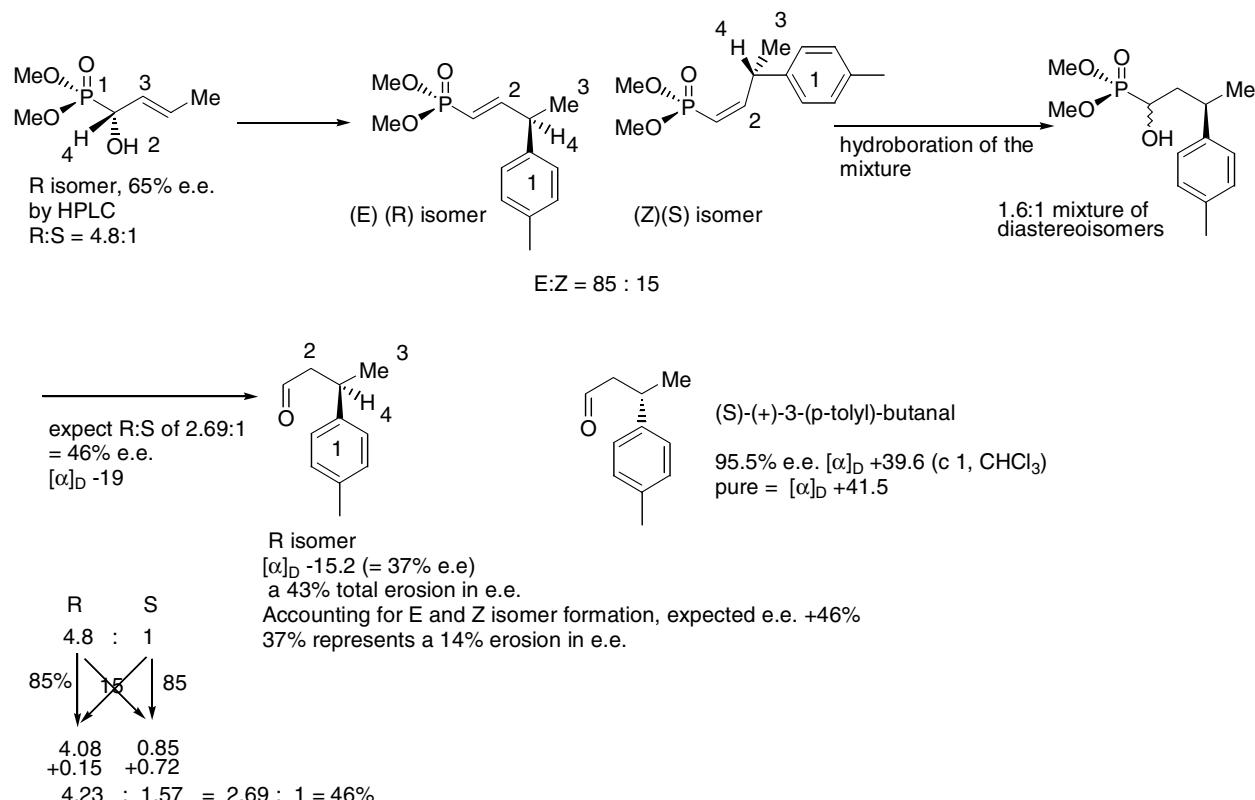
<i>Table of Contents</i>	S1-S2
General Experimental	S2
Stereochemical Analysis	S3
^1H NMR spectrum for 3	S4
^{13}C NMR spectrum for 3	S5
^1H NMR spectrum for 6E	S6
$^1\text{H} \{ ^{31}\text{P} \}$ NMR Spectrum for 6E	S7
^{13}C NMR spectrum for 6E	S8
^1H NMR spectrum for 6Z	S9
$^1\text{H} \{ ^{31}\text{P} \}$ NMR spectrum for 6Z	S10

¹³ C NMR spectrum for 6Z	S11
¹ H NMR spectrum for 7	S12
¹³ C NMR spectrum for 7	S13
³¹ P NMR spectrum for 8	S14
¹ H NMR spectrum for 8	S15
¹³ C NMR spectrum for 8	S16
A comparison of HPLC data for compound 7 prepared from (\pm) 6 , (3S,1E) 6 and (3R,1Z) 6	S17

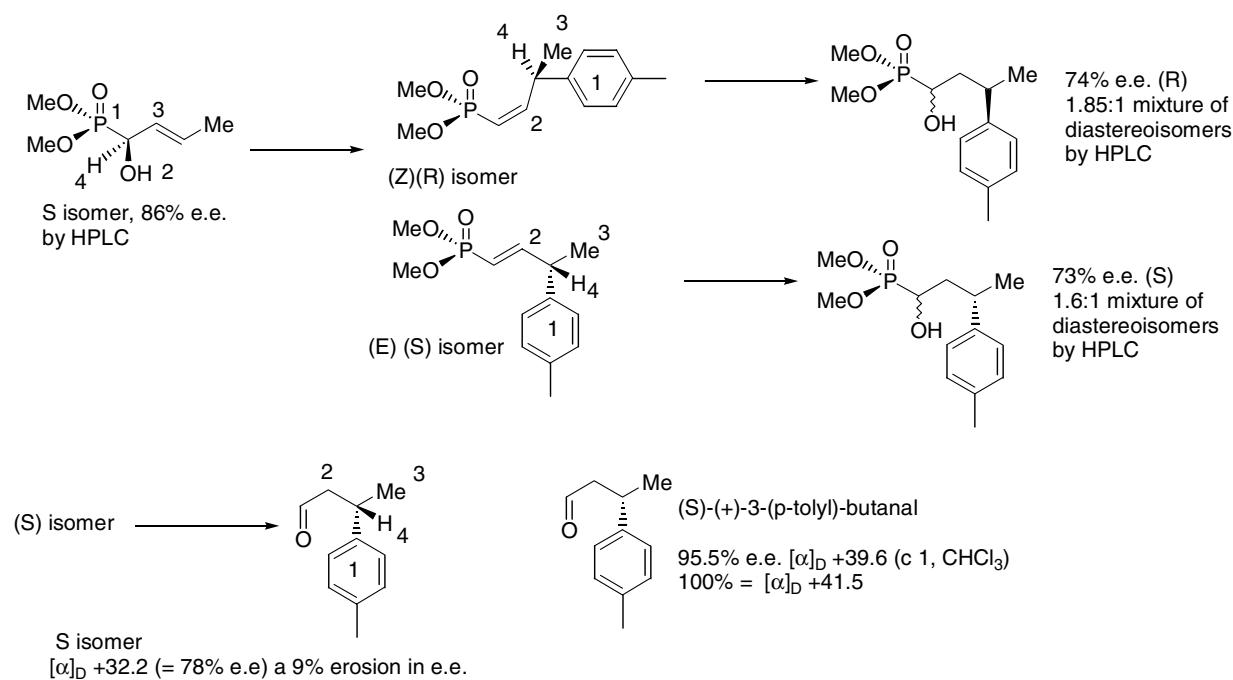
General Experimental: ¹H, ¹³C and ³¹P NMR spectra were recorded at 300, 75 and 121 MHz, respectively in CDCl₃. ¹H NMR spectra are referenced to internal tetramethylsilane (TMS, δ=0.00), ¹³C NMR spectra to the center-line of CDCl₃ (77.23 ppm) and ³¹P NMR spectra to external 85% H₃PO₄. Coupling constants, *J*, are reported in Hz. Enantiomer ratios were measured by chiral stationary-phase HPLC on a (*S,S*)-Whelk-O 1 column or a Chiralpak AS column. Optical rotations were determined using a polarimeter set at 589 nm. (1R) (2E) dimethyl (1-hydroxy-2-but enyl) phosphonate was prepared using published procedures.^{1,2} (1S,2E) Dimethyl (1-hydroxy-2-but enyl) phosphonate (1) was prepared according to published procedures using D-dimethyl tartrate as the ligand and the lipase from *rhizopus arrhizus* (Sigma) (2.6 g, 86% e.e.).²

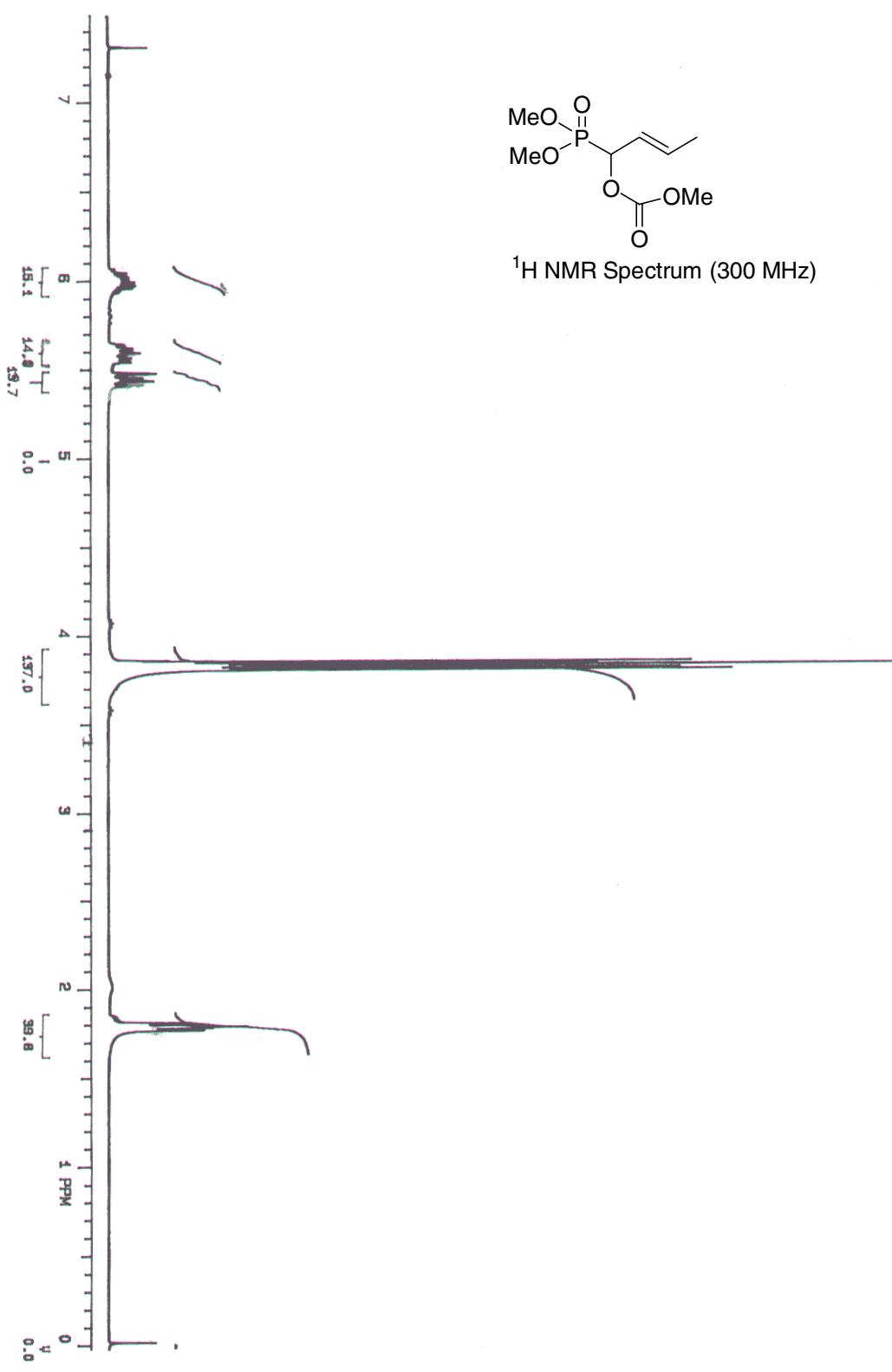
1. a) Texier-Boullet, F.; Foucaud, A. *Synthesis* 1982, 165; b) Baraldi, P. G.; Guarneri, M.; Moroder, F.; Pollini, G. P.; Simoni, D. *Synthesis* 1982, 653.
2. Rowe, B.J.; Spilling, C. D. *Tetrahedron Asymm.* **2001**, 12, 1701

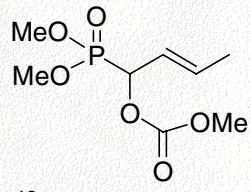
Stereochemical Analysis For the (R) Hydroxy Phosphonate (65% e.e.)



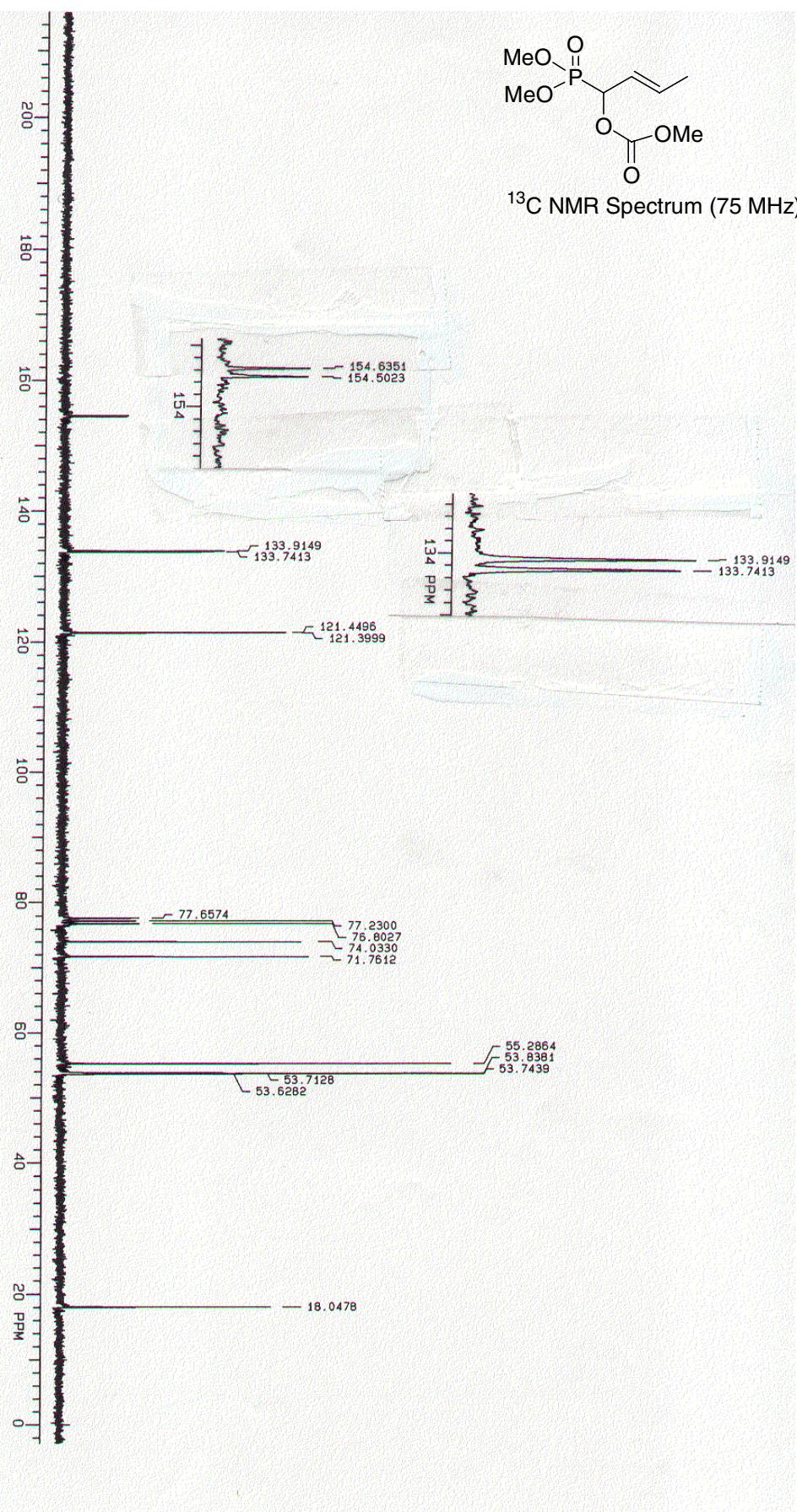
Stereochemical Analysis For the (S) Hydroxy Phosphonate (86% e.e.)



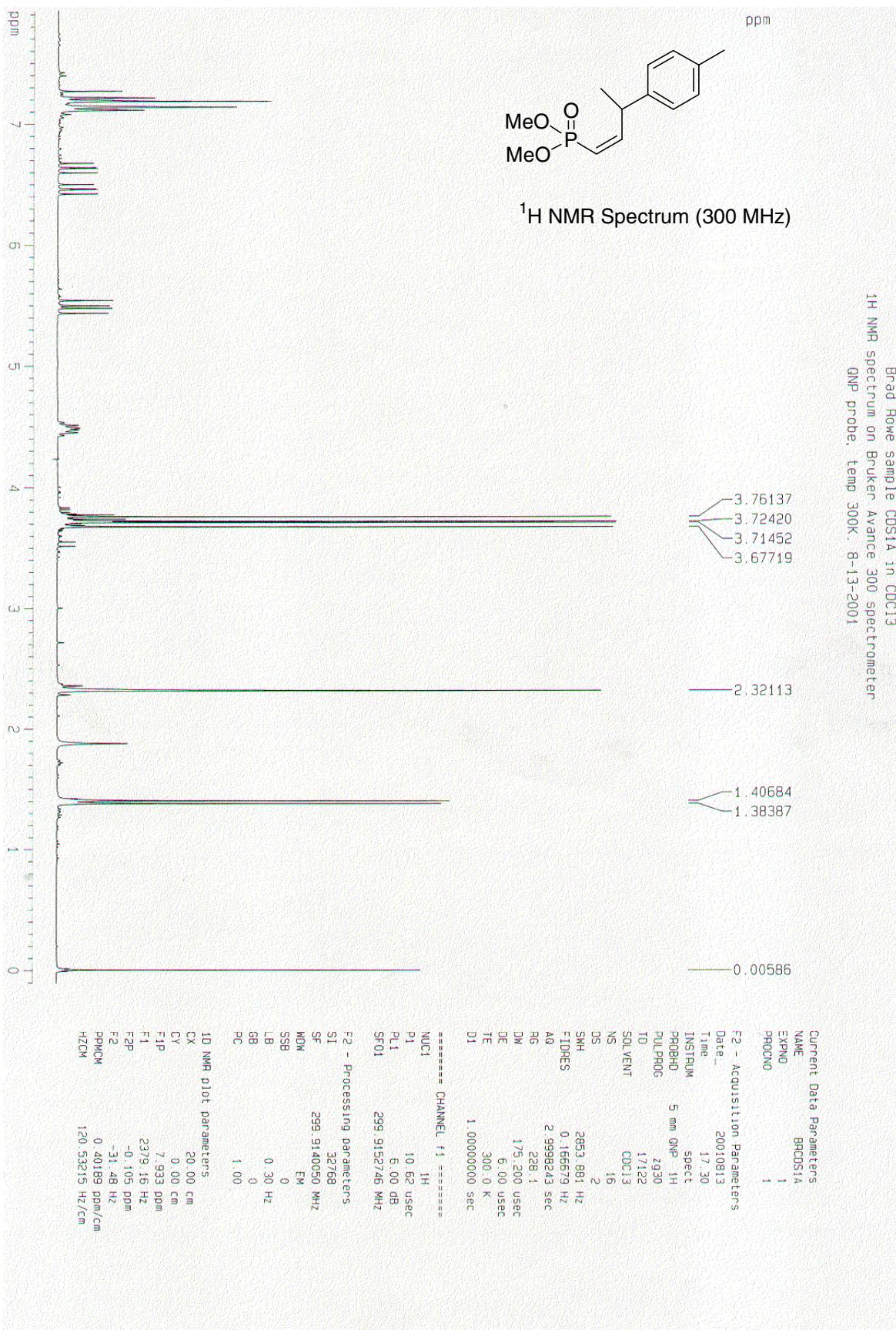


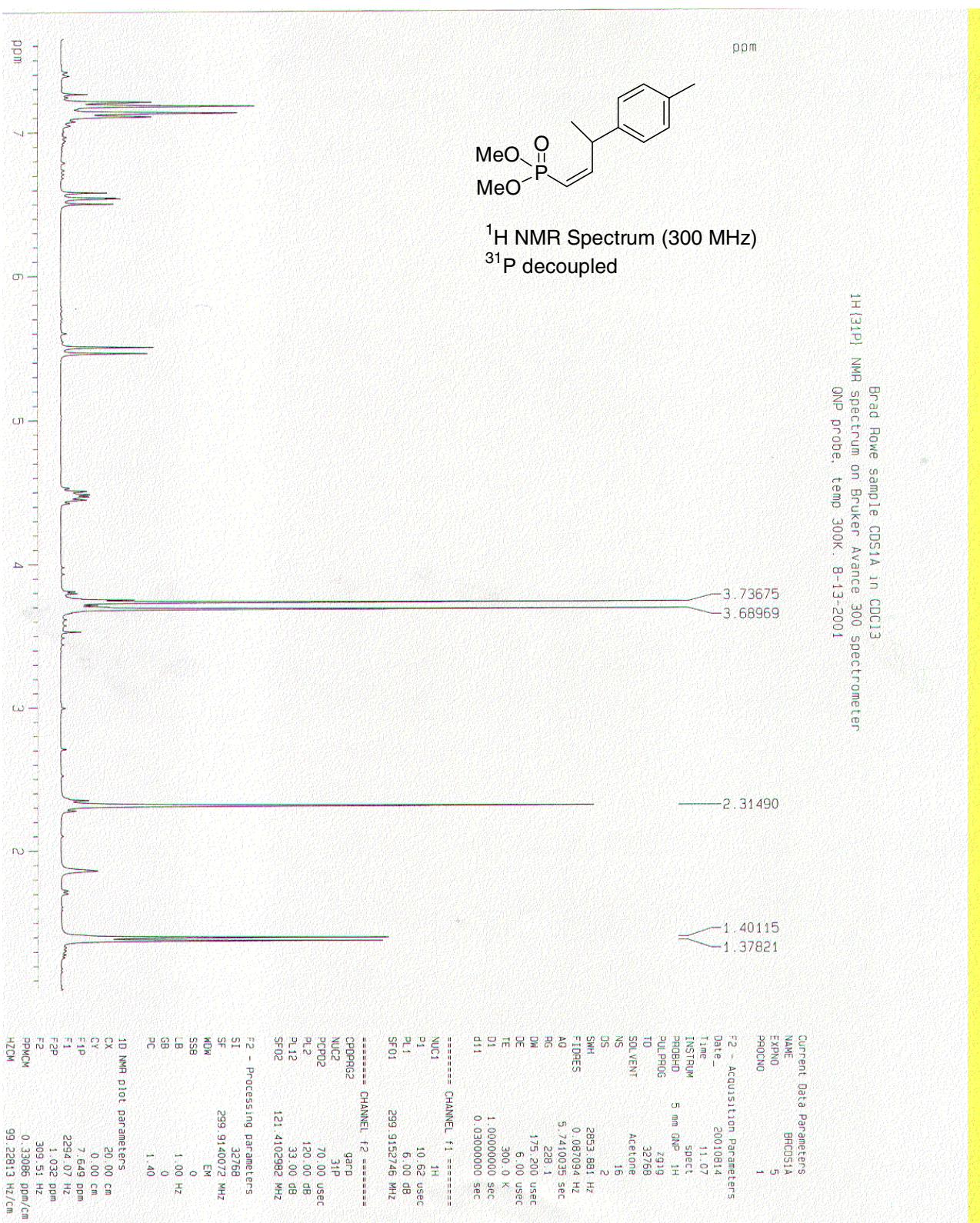


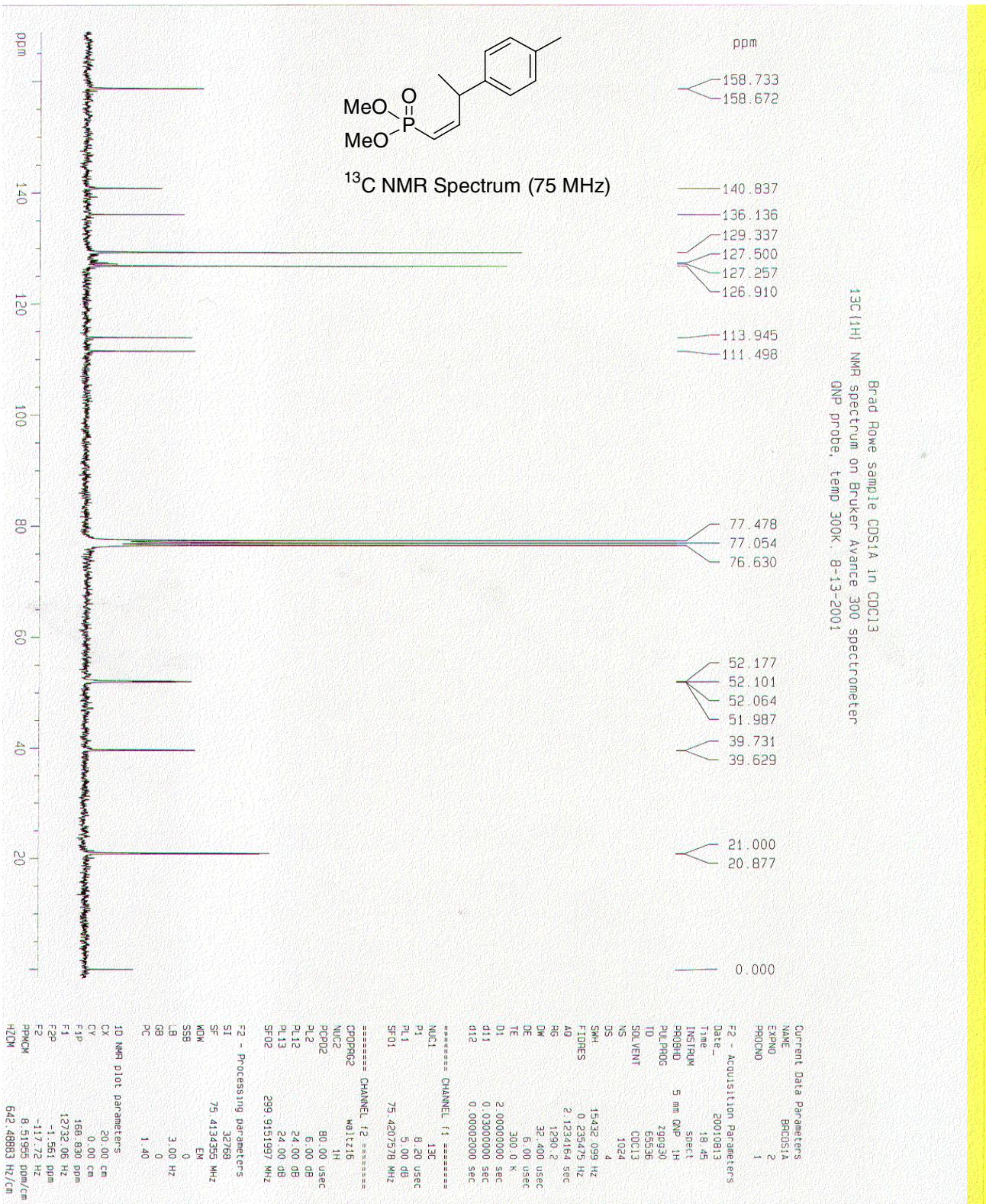
^{13}C NMR Spectrum (75 MHz)

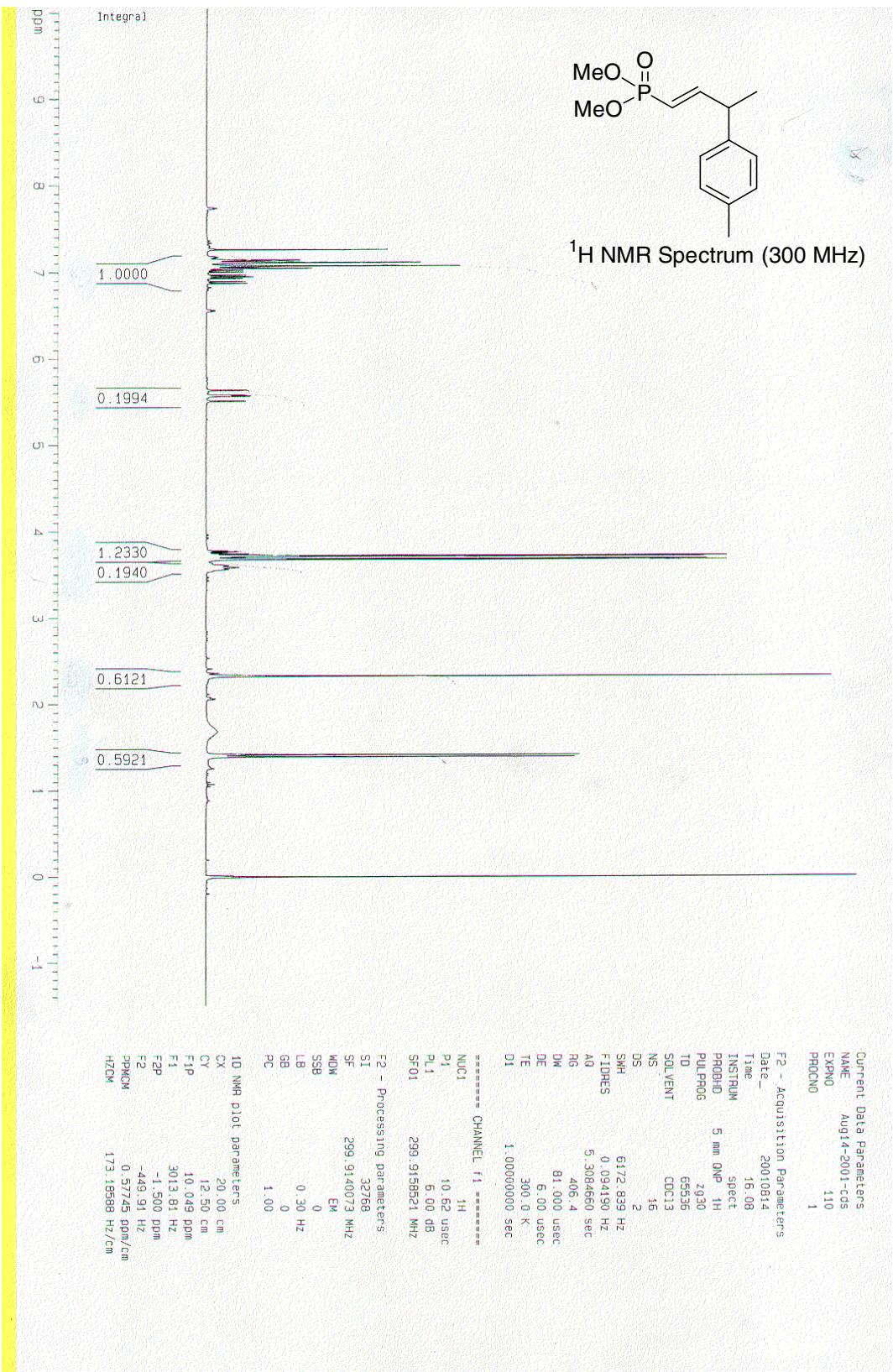


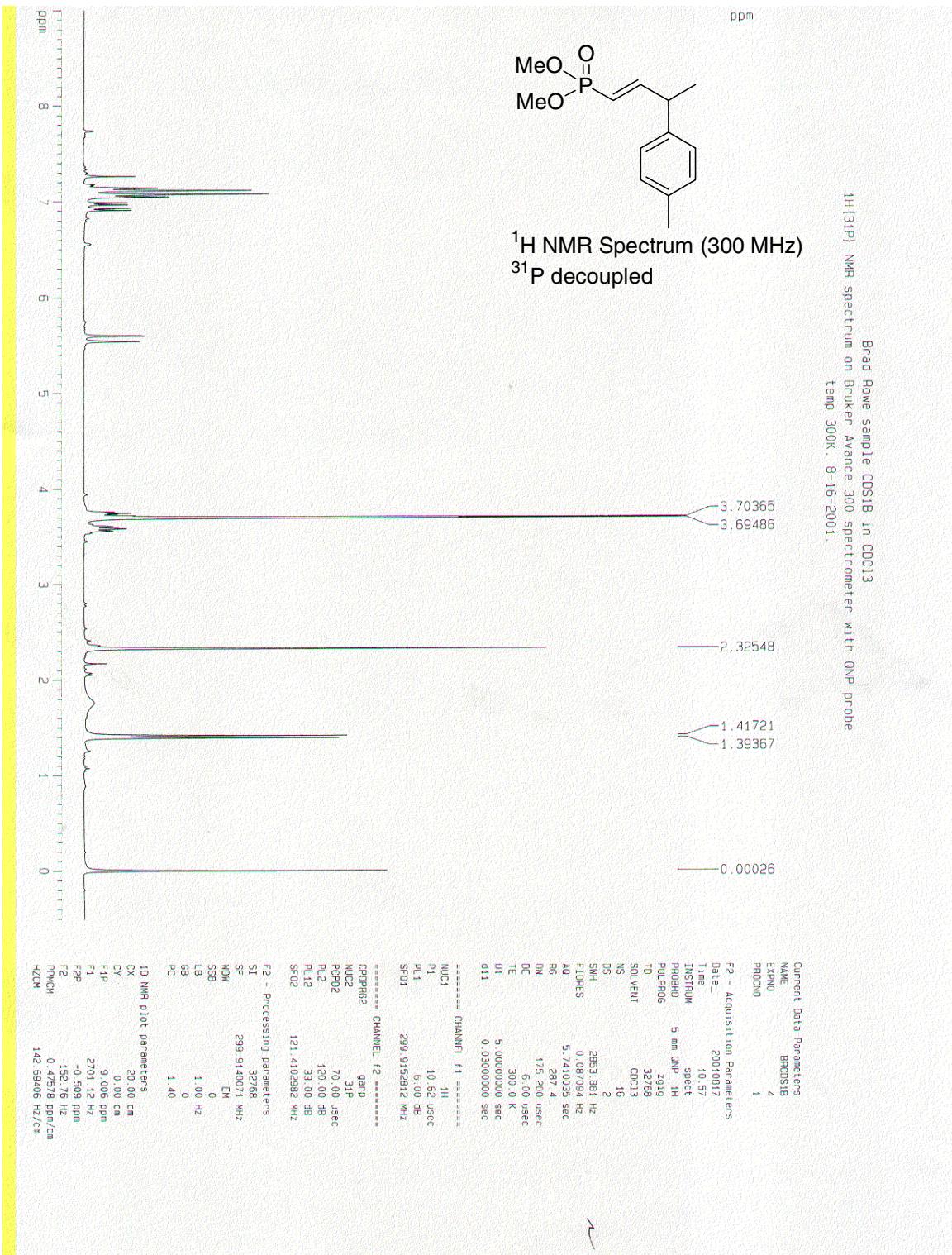
Brad Rowe sample CDS1A in CDCl₃
 1H NMR spectrum on Bruker Avance 300 spectrometer
 QNP probe, temp 300K. 8-13-2001

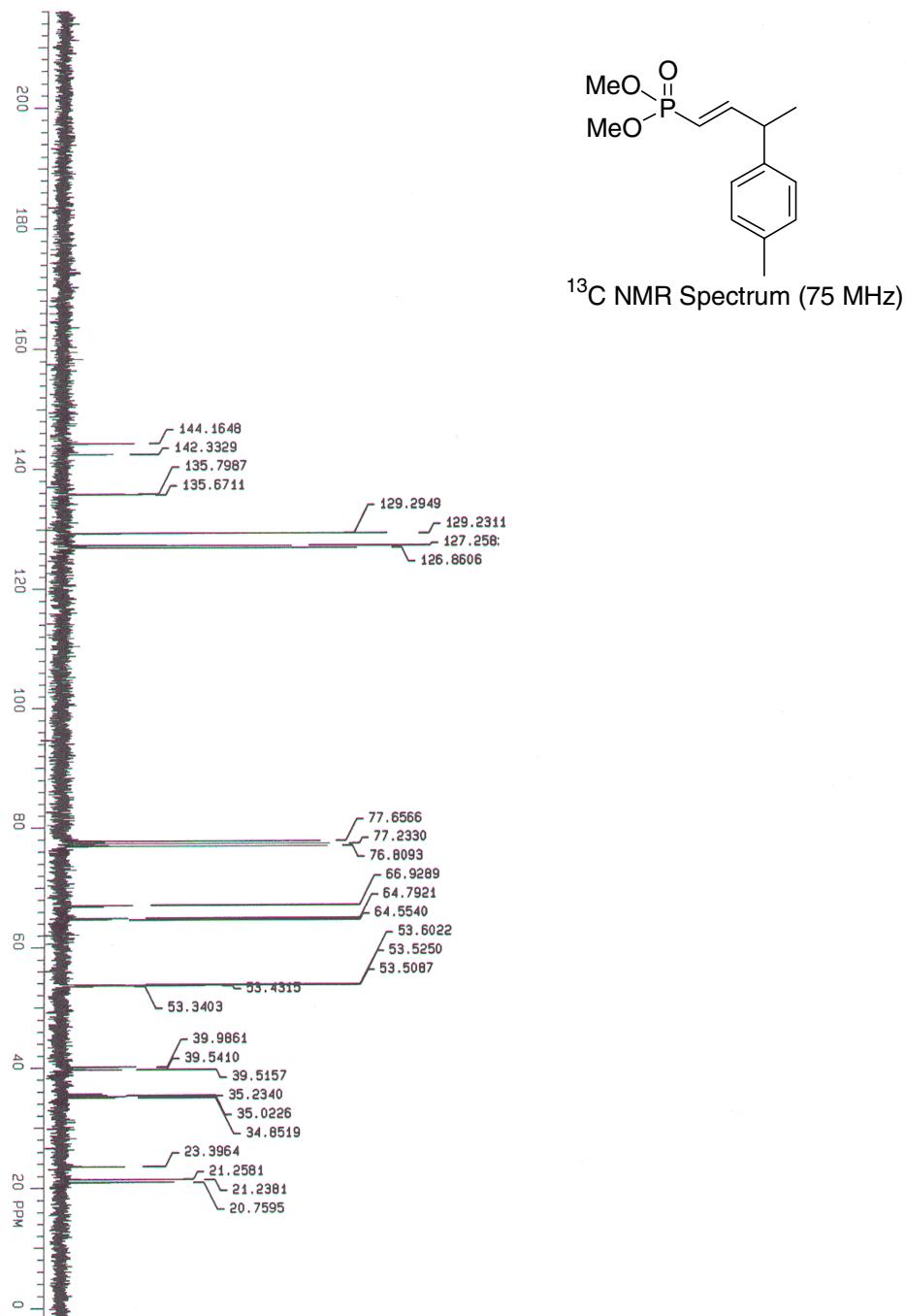


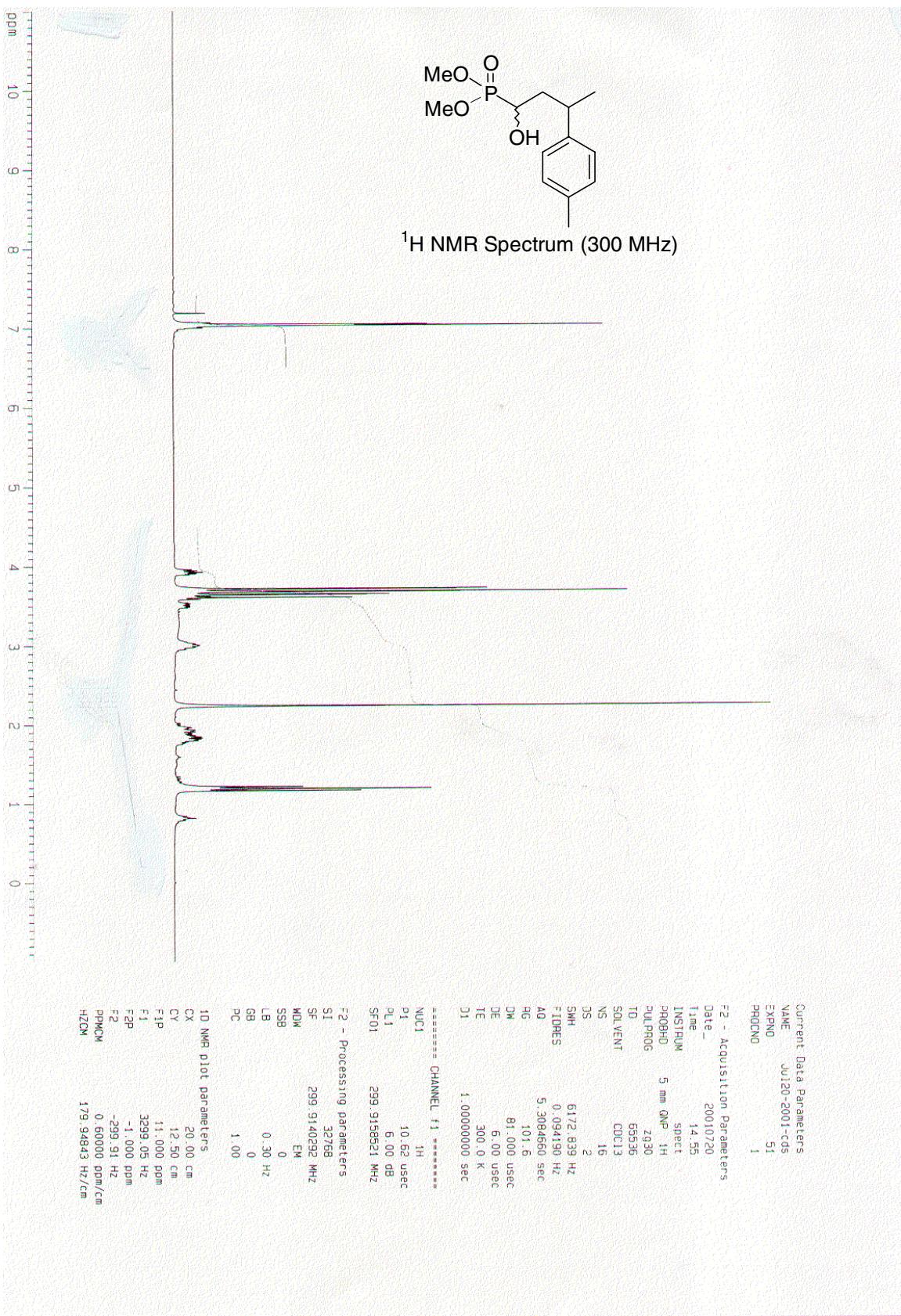


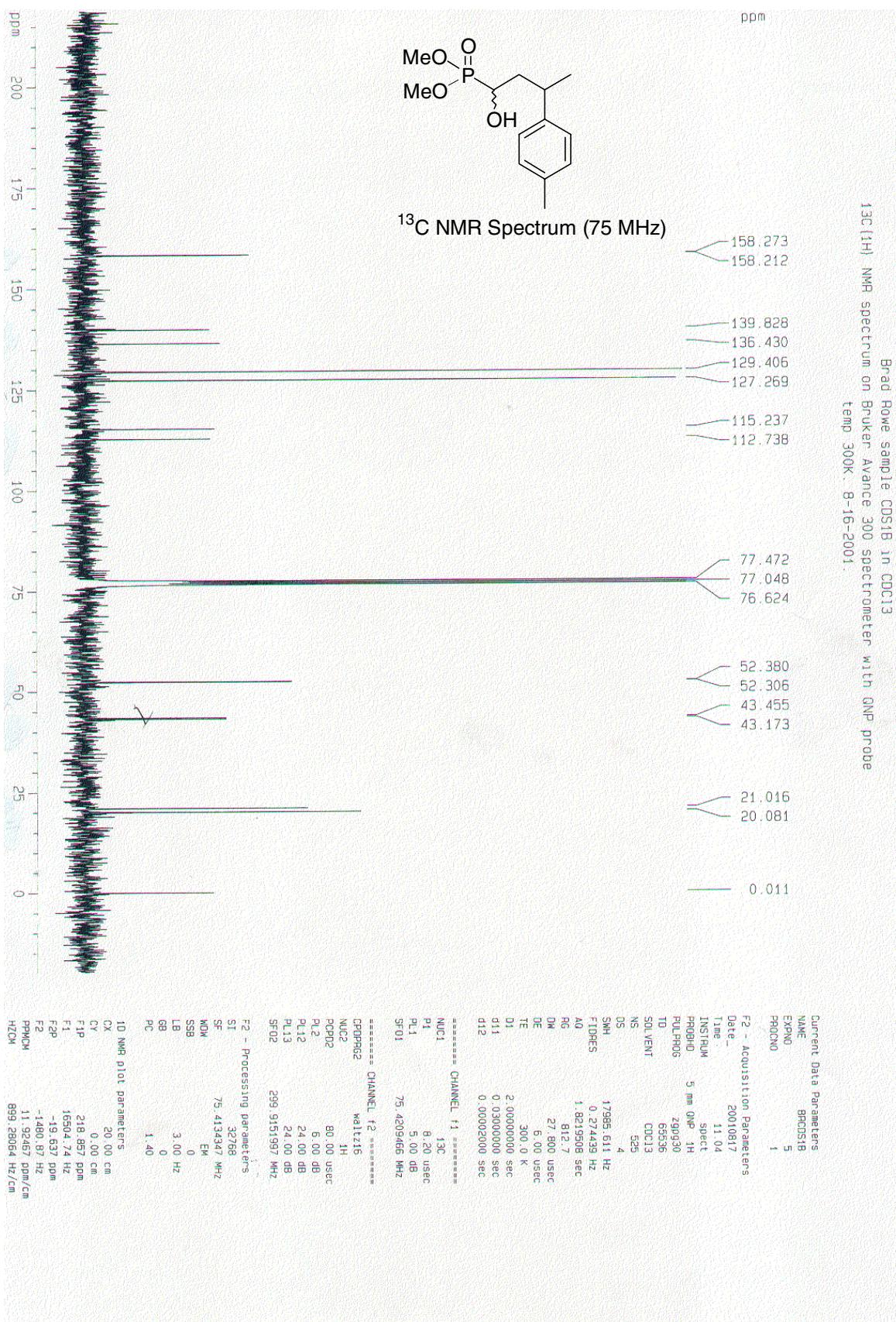


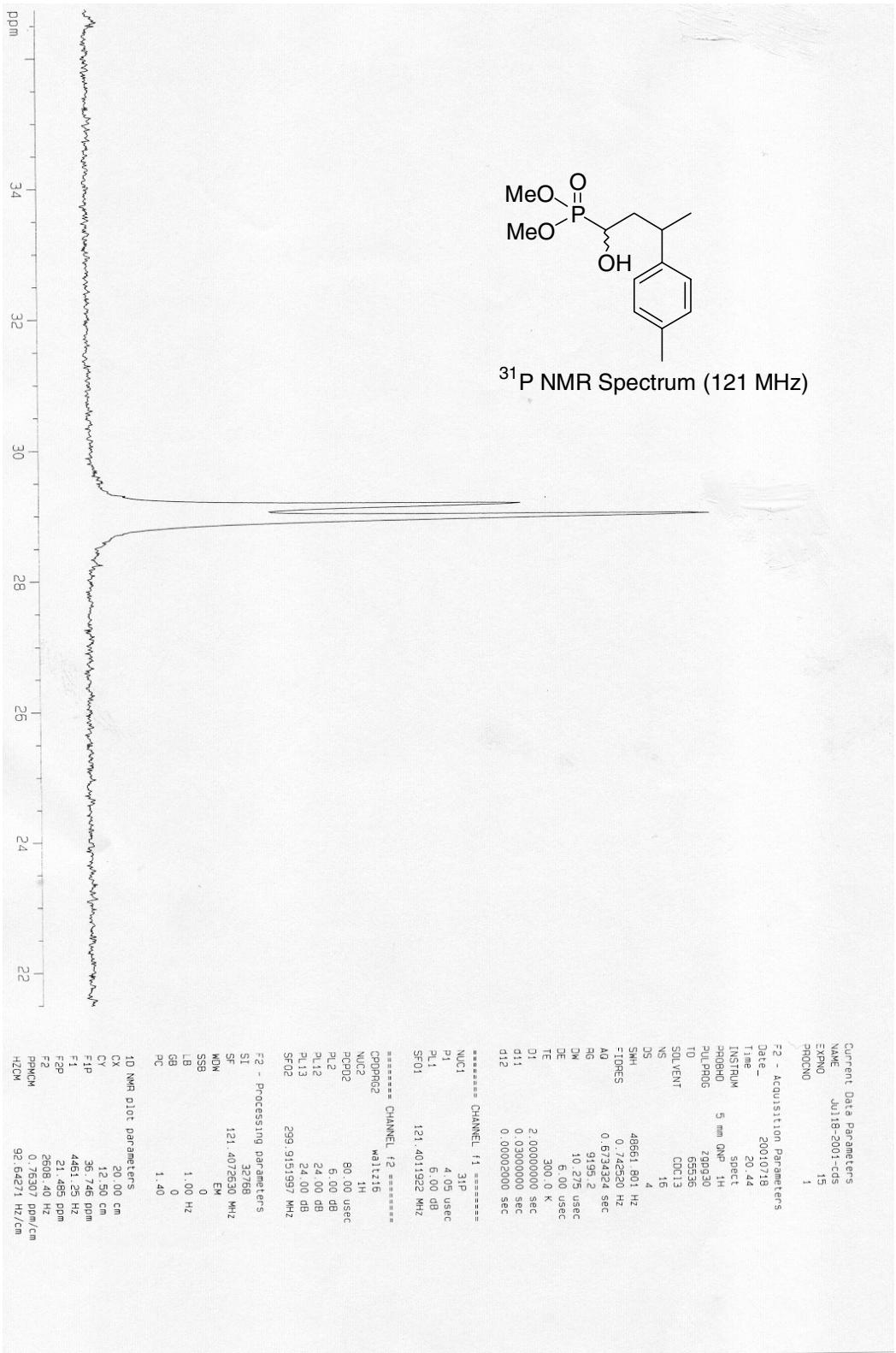


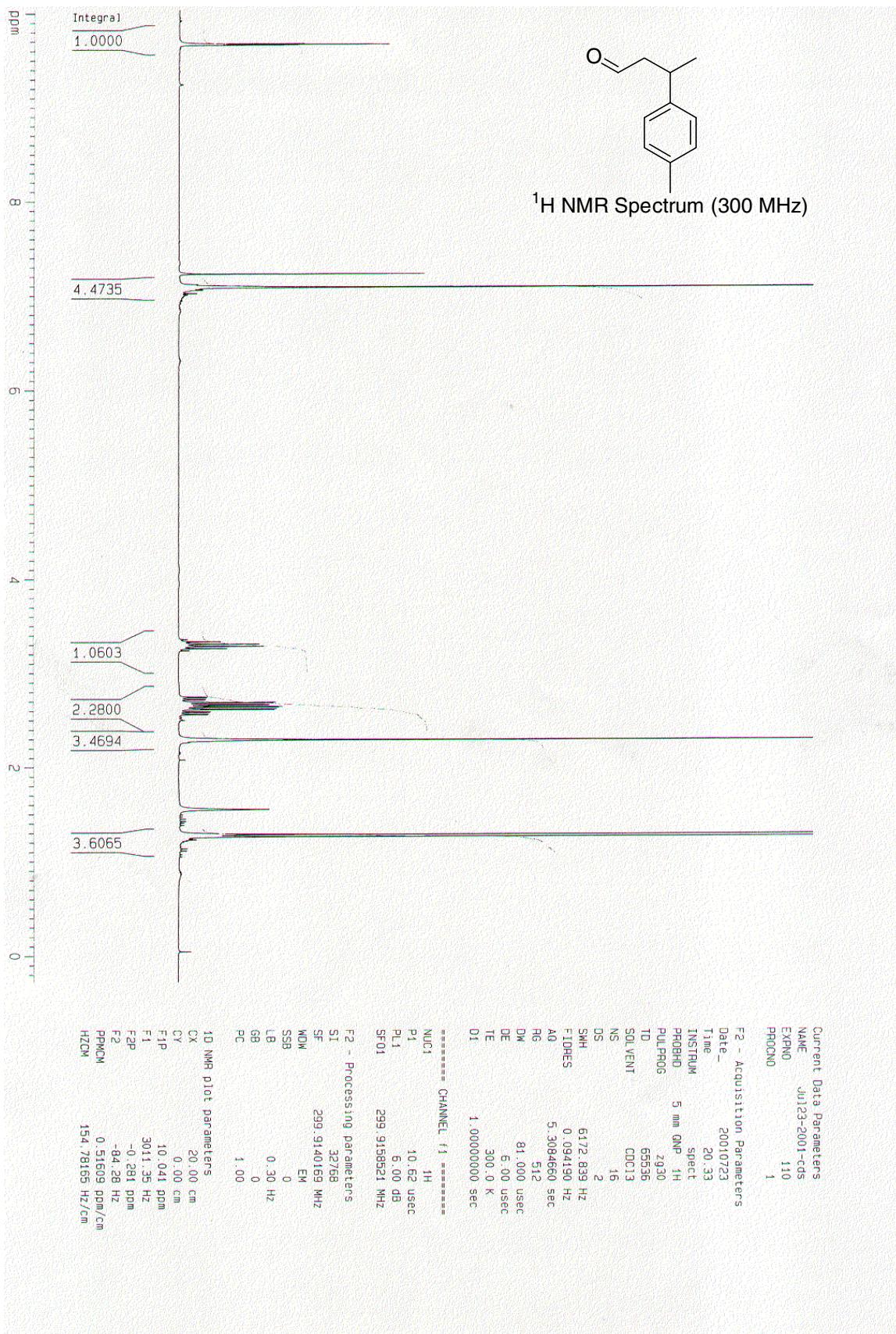


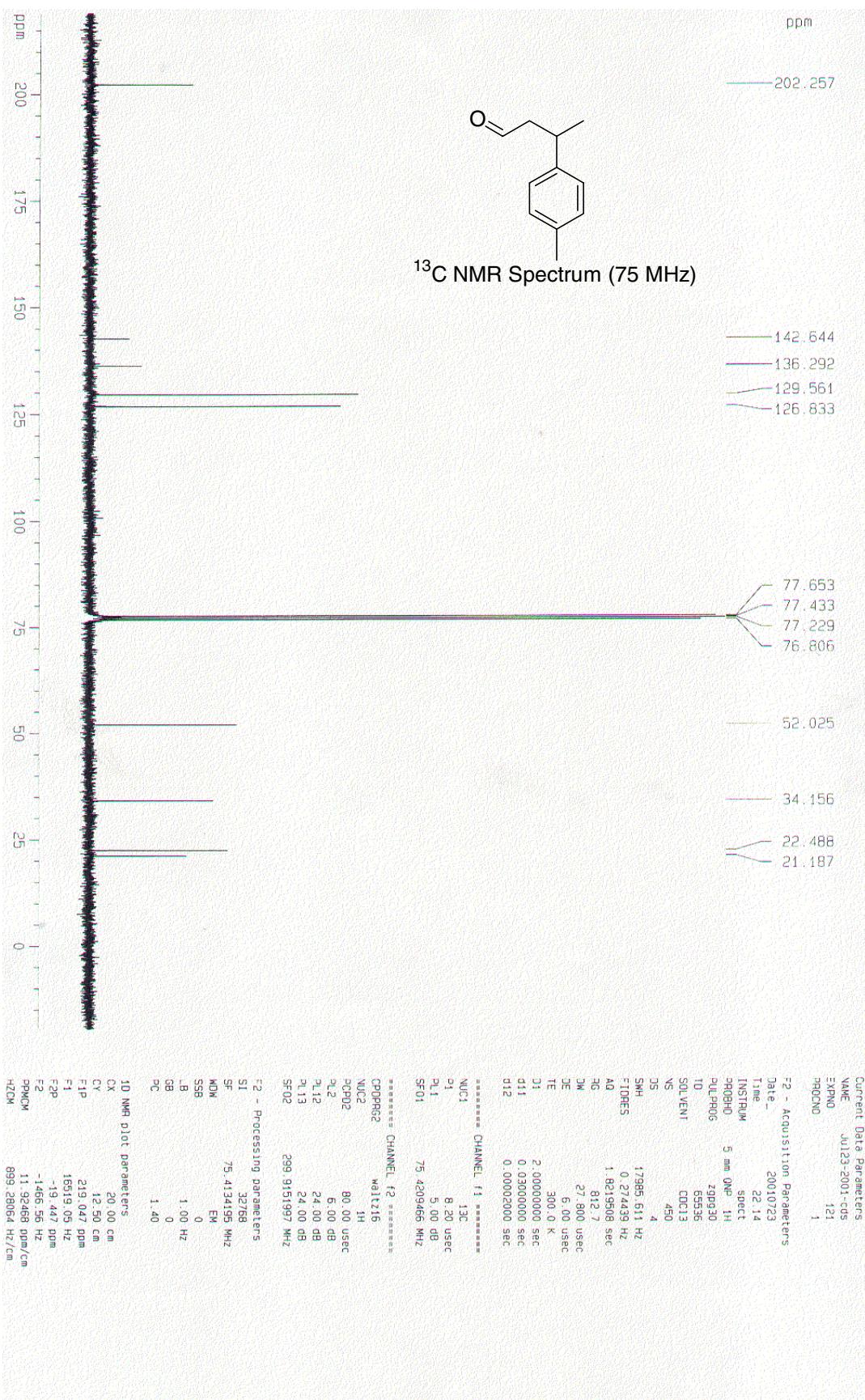












HPLC Data:

Dual column system: WhelkO S,S column followed by a ChiralPak AS column, detection at 205 nm, 95:5 EtOH hexane, 1 mL per minute

