New Mixed Phosphonate Esters by Transesterification of Pinacol-phosphonates and their use in Aldehyde and Ketone Coupling Reactions with Non-stabilized Phosphonates

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Supporting Information

The final products **12b**,¹ **12c**,² **12d**³ and **12e**⁴ are known compounds.

To circumvent the pinacol ester cleavage altogether, other hydrogen phosphites were investigated in the rhodium catalyzed alkene hydrophosphorylation reaction. For this study the hydrogen phosphonites and phosphinic acids in Figure 1 were selected because of their easy preparation (**i**),⁵ the structural homology they shared with **1** (**ii-iii** and **v**),⁶ and the commercial availability of precursors (**iv**). The hydrogen phosphites were prepared by hydrolysis of commercial chlorophosphines (**iv**), or by the condensation of PCl_3 with the corresponding diols (**ii-ii**), 2-mercaptoethanol (**iii**) or diamine (**v**). However, **ii-a** and **iii** were useless for hydrophosphorylation because they polymerized within hours of preparation,⁷ **ii-b** underwent explosive decomposition during purification by vacuum distillation (0.4 mm Hg, $\sim 130^\circ\text{C}$),⁸ and **ii-c** was insoluble in common organic solvents.⁶ The hydrophosphorylation of octene with the remaining hydrogen phosphonites **i**, **iv**, and **v** with Wilkinson's catalyst was unsuccessful. A similar lack of reactivity was observed in a palladium catalyzed olefin hydrophosphorylation study with $(\text{MeO})_2\text{P}(\text{O})\text{H}$, $(i\text{-PrO})_2\text{P}(\text{O})\text{H}$, $(\text{PhO})_2\text{P}(\text{O})\text{H}$ and **i-b**.⁹ Although alkyne hydrophosphorylation has been achieved with $\text{Ph}_2\text{P}(\text{O})\text{H}$,¹⁰ $\text{Ph}(\text{OMent})\text{P}(\text{O})\text{H}$,¹¹ $(\text{MeO})_2\text{P}(\text{O})\text{H}$ and $(\text{EtO})_2\text{P}(\text{O})\text{H}$ ¹² using a palladium catalyst,⁹ and intramolecular hydrophosphination of unactivated olefins has been reported,^{13,14,15} at its current state of development olefin hydrophosphorylation remains restricted to hydrogen phosphite **1**.

Figure 1. Hydrogen phosphonites screened with $(\text{PPh}_3)_3\text{RhCl}$.

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⁴ See e.g. Walker, L. F.; Bourghida, A.; Connolly, S.; Wills, M. J. *Chem. Soc., Perkin Trans. 1* **2002**, 7, 965-981.

⁵ Zwierzak, A. *Can. J. Chem.* **1967**, 45, 2501-2512.

⁶ Lucas, H. J.; Mitchell, F. W. Jr.; Scully, C. N. *J. Am. Chem. Soc.* **1950**, 72, 5491-5497.

⁷ Nifant'ev, E. E.; Nasonovskii, I. S.; Miklashevskii, A. V.; Zavalishina, A. I.; Smirnova, E. I. *Zh. Org. Khim.* **1975**, 11, 2206-2210.

⁸ A similar observation has been reported for **ii-a**, ref 6

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¹³ (a) Douglass, M. R.; Marks, T. J. *J. Am. Chem. Soc.* **2000**, 122, 1824-1825. (b) Douglass, M. R.; Stern, C. L.; Marks, T. J. *J. Am. Chem. Soc.* **2001**, 123, 10221-10238.

¹⁴ For an asymmetric version with activated alkenes, see: (a) Costa, E.; Pringle, P. G.; Smith, M. B.; Worboys, K. *J. Chem. Soc., Dalton Trans.* **1997**, 4277-4282. (b) Costa, E.; Pringle, P. G.; Worboys, K. *Chem. Commun.* **1998**, 49-50. (c) Kovacic, I.; Wicht, D. K.; Grewal, N. S.; Glueck, D. S.; Incarvito, C. D.; Guzei, I. A.; Rheingold, A. L. *Organometallics* **2000**, 19, 950-953.

¹⁵ For examples with alkynes, see: Kazankova, M. A.; Efimova, I. V.; Kochetkov, A. N.; Afanas'ev, V. V.; Beletskaya, I. P.; Dixneuf, P. H. *Synlett* **2001**, 4, 497-500.

Butyl-octyl-phosphinic acid 2-hydroxy-1,1,2-trimethyl-propyl ester (5). To a cooled ($-78\text{ }^{\circ}\text{C}$) solution of **4** (242 mg, 1.0 mmol) in THF (2.0 mL) was added 2.27 M BuLi in hexanes (0.50 mL, 1.1 mmol). After stirring at $-78\text{ }^{\circ}\text{C}$ for 1 h the reaction mixture was concentrated *in vacuo*. Flash chromatography (SiO_2 , gradient: EtOAc) afforded **5** (31 mg, 0.093 mmol) as a light yellow oil in 9% yield. R_f 0.40 (EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 0.86-0.96 (m, 6H), 1.24-1.56 (m, 30H), 1.71-1.74 (m, 2H), 4.06 (brs, 1H); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ 13.18 (CH_3), 14.3 (CH_3), 22.7 (CH_2), 22.9 (CH_2), 24.2 (CH_2), 24.8 (CH_2), 25.2 (CH_3), 25.5 (CH_3), 29.3 (CH_2), 31.3 (CH_2), 32.0 (CH_2), 74.6 (C), 91.3 (C), 91.4 (C); ^{31}P NMR (CDCl_3 , 121.5 MHz) δ 58.5; MS (EI) m/z 335 $[\text{M}+\text{H}]^+$, 235 $[\text{M}-t\text{BuC}(\text{O})\text{Me}+\text{H}]^+$; MS (EI) m/z 371 and 369 $[\text{M}+\text{Cl}]^-$; HRMS (CI) calcd for $\text{C}_{18}\text{H}_{39}\text{O}_3\text{P}$ $[\text{M}+\text{H}]^+$ 335.2715, found 335.2725.

Octyl-Phosphonic acid mono-(2-hydroxy-1,1,2-trimethyl-propyl) ester (6). To a cooled $-78\text{ }^{\circ}\text{C}$ solution of **4** (61 mg, 0.25 mmol) in THF (1.0 mL) was added KHMDS in THF (0.94 M, 0.29 mL, 0.25 mmol). After stirring at $-78\text{ }^{\circ}\text{C}$ for 1 h, AcOH (0.018 mL, 0.31 mmol) was added and the mixture was stirred for 16 h. The resulting suspension was concentrated *in vacuo*, redissolved in 50% MeOH in CHCl_3 and filtered through silica gel. Concentrated *in vacuo* afforded crude **6**. R_f 0.32 (50% MeOH in CHCl_3); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ 14.3 (CH_3), 22.9 (CH_2), 24.0 (CH_2), 29.0 (CH_2), 30.9 (CH_2), 31.3 (CH_2), 31.6 (CH_2), 32.1 (CH_2), 22.3 (CH_3), 24.5 (CH_3), 25.2 (CH_3), 75.8 (C), 85.8 (C), 85.9 (C); MS (EI) m/z 295 $[\text{M}+\text{H}]^+$, 277 $[\text{M}-\text{H}_2\text{O}+\text{H}]^+$, 195 $[\text{M}-t\text{BuC}(\text{O})\text{Me}+\text{H}]^+$; MS (EI) m/z 293 $[\text{M}-\text{H}]^-$; HRMS (CI) calcd for $\text{C}_{14}\text{H}_{31}\text{O}_4\text{P}$ $[\text{M}+\text{H}]^+$ 295.2038, found 295.2050.

Octyl-Phosphonic acid (7). To a solution of **4** (187 mg, 0.68 mmol) in CH_2Cl_2 (2.0 mL) was added TMSBr (0.35 mL, 2.65 mmol) and the mixture was stirred at rt for 16 h followed by concentration *in vacuo*. The residue was redissolved in CH_2Cl_2 (1.5 mL) and MeOH (0.5 mL) was then added. The clear solution was allowed to stir at rt for 2 h followed by concentration *in vacuo* to yield crude **7**. ^1H NMR (CDCl_3 , 300 MHz) δ 0.87 (t, $J = 6.6\text{ Hz}$, 3H), 1.21-2.01 (m, 12), 9.65 (brs, 2H); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ 14.3 (CH_3), 22.1 (CH_2), 22.8 (CH_2), 24.9 (CH_2), 25.0 (CH_2), 26.8 (CH_2), 29.2 (CH_2), 29.3 (CH_2), 29.9 (CH_2), 30.5 (CH_2), 30.8 (CH_2), 32.0 (CH_2); ^{31}P NMR (CDCl_3 , 121.5 MHz) δ 38.0; MS (EI) m/z 195 $[\text{M}+\text{H}]^+$.

Phenylethyl-phosphonic acid monomethyl ester (8b). The synthesis was performed according to the general procedure for **8a**. ^1H NMR (CDCl_3 , 300 MHz) δ 2.09-2.21 (m, 2H), 2.95-3.04 (m, 2H), 3.78 (d, $J = 11.1\text{ Hz}$, 3H), 7.23-7.36 (m, 5H); ^{31}P NMR (CDCl_3 , 121.5 MHz) δ 36.1.

Octyl phosphonic acid benzyl ester methyl ester (9c). The synthesis was performed according to the general procedure for **9b**. Purification by flash chromatography (SiO_2 , gradient: EtOAc:hexane 1:2 to 1:1 to EtOAc) afforded **9c** as a colorless oil (1.27 g, 4.3 mmol) in 82% yield. R_f 0.70 (EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 0.89 (t, $J = 6.8\text{ Hz}$, 3H), 1.26-1.36 (m, 10H), 1.54-1.80 (m, 4H), 3.68 (d, $J = 10.8\text{ Hz}$, 3H), 5.09 (d, $J = 8.4\text{ Hz}$, 2H), 7.28-7.42 (m, 5H); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ 14.3 (CH_3), 22.5 (CH_2), 22.6 (CH_2), 22.9 (CH_2), 24.7 (CH_2), 26.6 (CH_2), 29.2 (CH_2), 30.7 (CH_2), 30.9 (CH_2), 32.1 (CH_2), 52.3 (CH_3), 67.3 (CH_2), 67.4 (CH_2), 128.1 (CH), 128.6 (CH), 128.8 (CH), 136.8 (C), 136.9 (C); ^{31}P NMR (CDCl_3 , 121.5 MHz) δ 35.7; MS (CI) m/z 299 $[\text{M}+\text{H}]^+$; HRMS (CI) calcd for $\text{C}_{16}\text{H}_{27}\text{O}_3\text{P}$ $[\text{M}+\text{H}]^+$ 299.1776, found 299.1773.

(5-Hex-3-enyloxy-pentyl)-phosphonic acid *tert*-butyl ester methyl ester (9e). The synthesis was performed according to the general procedure for **9d**. Purification by flash chromatography (SiO_2 , gradient: EtOAc:hexane 1:2 to 1:1 to EtOAc) afforded **9e** (1.21 g, 3.8 mmol) as a colorless oil in 88% yield. R_f 0.52 (EtOAc); ^1H NMR (CDCl_3 , 300 MHz) δ 0.89 (t, $J = 7.4\text{ Hz}$, 3H), 1.33-1.67 (m, 8H), 1.43 (s, 9H), 1.92-2.04 (m, 2H), 2.15-2.28 (m, 2H), 3.31-3.36 (m, 4H), 3.62 (d, $J = 11.1\text{ Hz}$, 3H), 5.22-5.49 (m, 2H); ^{31}P NMR (CDCl_3 , 121.5 MHz) δ 30.6.

(1-Benzyl-2-ethyl-2-hydroxy-butyl)-phosphonic acid benzyl ester methyl ester (10b). The synthesis was performed according to the general procedure for **10a**. Purification by flash chromatography (SiO₂, gradient: EtOAc:hexane 1:4 to 1:1) afforded **10b** (380 mg, 1.01 mmol) as a colorless oil in 62% yield. *R_f* 0.70 (EtOAc); ¹H NMR (CDCl₃, 300 MHz) δ 0.92-1.03 (m, 6H), 1.52-1.90 (m, 4H), 2.45-2.65 (m, 1H), 2.80-2.95 (m, 2H), 3.28 and 3.64 (d, *J* = 10.8 Hz, 3H), 4.46-5.05 (m, 3H), 7.03-7.40 (m, 10H); ¹³C NMR (CDCl₃, 75.5 MHz) δ 6.94 (CH₃), 6.98 (CH₃), 7.25 (CH₃), 7.32 (CH₃), 27.97 (CH₂), 28.07 (CH₂), 28.13 (CH₂), 28.24 (CH₂), 30.22 (CH₂), 30.28 (CH₂), 30.31 (CH₂), 30.37 (CH₂), 31.79 (CH₂), 31.85 (CH₂), 31.93 (CH₂), 31.99 (CH₂), 45.1 (CH), 46.2 (CH), 46.9 (CH), 47.9 (CH), 51.45 (CH₃), 51.55 (CH₃), 51.48 (CH₃), 51.66 (CH₃), 66.72 (CH₂), 66.81 (CH₂), 66.96 (CH₂), 67.04 (CH₂), 74.95 (CH₂), 74.99 (CH₂), 125.93 (CH), 125.95 (CH), 126.53 (CH), 126.86 (CH), 127.80 (CH), 127.90 (CH), 127.97 (CH), 128.00 (CH), 128.10 (CH), 128.20 (CH), 128.32 (CH), 128.50 (CH), 128.63 (CH), 135.38 (C), 135.45 (C), 135.74 (C), 135.82 (C), 139.51 (C), 139.55 (C), 139.61 (C); ³¹P NMR (CDCl₃, 121.5 MHz) δ 36.6, 36.5; MS (CI) *m/z* 377 [M+H]⁺, 359 [M-H₂O+H]⁺; HRMS (CI) calcd for C₂₁H₃₀O₄P [M+H]⁺ 377.1882, found 377.1881.

[1-(Hydroxy-phenyl-methyl)-octyl]-phosphonic acid benzyl ester methyl ester (10c). The synthesis was performed according to the general procedure for **10a**. Purification by flash chromatography (SiO₂, gradient: EtOAc:hexane 1:2 to 1:1) afforded **10c** (322 mg, 0.80 mmol) as a colorless oil in 80% yield. *R_f* 0.35 (EtOAc:hexane 1:1); ¹H NMR (CDCl₃, 300 MHz) δ 0.84 (t, *J* = 6.8 Hz, 3H), 1.06-1.70 (m, 12H), 2.09-2.19 (m, 1H), 3.56-3.64 (m, 3H), 4.07-5.38 (m, 3H), 7.19-7.40 (m, 5H); ³¹P NMR (CDCl₃, 121.5 MHz) δ 35.9, 36.1, 36.4; MS (CI) *m/z* 405 [M+H]⁺, 387 [M-H₂O+H]⁺; HRMS (CI) calcd for C₂₃H₃₃O₄P [M+H]⁺ 405.2195, found 405.2197.

[1-(1-Hydroxy-3-phenyl-propyl)-octyl]-phosphonic acid benzyl ester methyl ester (10d). The synthesis was performed according to the general procedure for **10a**. Purification by flash chromatography (SiO₂, gradient: EtOAc:hexane 1:2 to 1:1 to EtOAc) afforded **10d** (247 mg, 0.57 mmol) as a colorless oil in 57% yield. *R_f* 0.74 (EtOAc); ¹H NMR (CDCl₃, 300 MHz) δ 0.91 (t, *J* = 6.8 Hz, 3H), 1.25-2.06 (m, 15H), 2.60-2.97 (m, 2H), 3.63-3.73 (m, 3H), 3.83-3.99 (m, 1H), 5.07-5.13 (m, 2H), 7.17-7.40 (m, 5H); ³¹P NMR (CDCl₃, 121.5 MHz) δ 36.0, 36.2, 36.4, 36.4; MS (CI) *m/z* 433 [M+H]⁺, 415 [M-H₂O+H]⁺; HRMS (CI) calcd for C₂₅H₃₇O₄P [M+H]⁺ 433.2508, found 433.2500.

(1-Benzyl-2-hydroxy-4-phenyl-butyl)- phosphonic acid tert-butyl ester methyl ester (10e). The synthesis was performed according to the general procedure for **10a**. Purification by flash chromatography (SiO₂, gradient: EtOAc:hexane 1:4 to 1:2) afforded **10e** (206 mg, 0.53 mmol) as a colorless oil in 53% yield. *R_f* 0.65 (EtOAc); ³¹P NMR (CDCl₃, 121.5 MHz) δ 30.1, 30.3, 30.4; MS (CI) *m/z* 391 [M+H]⁺; HRMS (CI) calcd for C₂₂H₃₁O₄P [M+H]⁺ 391.2038, found 391.2026.

[5-Hex-3-enyloxy-1-(5-Hex-3-enyloxy-pentyl)]-phosphonic acid tert-butyl ester methyl ester (10f). The synthesis was performed according to the general procedure for **10a**. Purification by flash chromatography (SiO₂, gradient: EtOAc:hexane 1:2 to 1:1) afforded **10f** (338 mg, 0.75 mmol) as a colorless oil in 82% yield. *R_f* 0.56 (EtOAc); ¹H NMR (CDCl₃, 300 MHz) δ 0.95 (t, *J* = 7.5 Hz, 3H), 1.14-2.08 (m, 12H), 1.47 (s, 9H), 2.19-2.32 (m, 2H), 2.58-2.72 (m, 1H), 2.83-2.91 (m, 1H), 3.35-3.39 (m, 4H), 3.64-4.10 (m, 5H), 5.27-5.53 (m, 2H), 7.12-7.28 (m, 5H); ³¹P NMR (CDCl₃, 121.5 MHz) δ 31.4, 31.6; MS (CI) *m/z* 455 [M+H]⁺; HRMS (CI) calcd for C₂₅H₄₃O₅P [M+H]⁺ 455.2926, found 455.2920.

1-Phenyl-1-nonene (12c). The synthesis was performed according to the general procedure for **12b**. Purification by flash chromatography (SiO₂, hexane) afforded **12c** (99 mg, 0.38 mmol) as a colorless oil in 59% yield. *R_f* 0.65 (hexanes); ¹H NMR (CDCl₃, 300 MHz) δ 0.94-0.98 (m, 3H), 1.35-1.55 (m, 10H), 2.25-2.45 (m, 2H), 5.70-5.79 (m, 0.8H), 6.25-

6.35 (m, 0.2H), 6.43-6.51 (m, 1H), 7.23-7.43 (m, 5H); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ 14.4 (CH_3), 23.0 (CH_2), 29.0 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.7 (CH_2), 30.3 (CH_2), 32.1 (CH_2), 33.4 (CH_2), 126.2 (CH), 126.7 (CH), 127.0 (CH), 128.4 (CH), 128.7 (CH), 129.0 (CH), 130.0 (CH), 131.5 (CH), 133.6 (CH), 138.1 (C); MS (CI) m/z 203 $[\text{M}+\text{H}]^+$.

1-Phenyl-3-undecene (12d). The synthesis was performed according to the general procedure for **12b**. Purification by flash chromatography (SiO_2 , hexane) afforded **12d** (99 mg, 0.382 mmol) as a colorless oil in 55% yield. R_f 0.65 (hexanes); ^1H NMR (CDCl_3 , 300 MHz) δ 0.89-0.93 (m, 3H), 1.29 (brs 10H), 1.99 (brs, 2H), 2.35-2.42 (m, 2H), 2.66-2.72 (m, 2H), 5.40-5.46 (m, 2H), 7.18-7.33 (m, 5H); ^{13}C NMR (CDCl_3 , 75.5 MHz) δ 14.4 (CH_3), 22.9 (CH_2), 27.5 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.8 (CH_2), 29.9 (CH_2), 30.0 (CH_2), 32.1 (CH_2), 32.8 (CH_2), 34.7 (CH_2), 36.3 (CH_2), 36.4 (CH_2), 125.9 (CH), 126.0 (CH), 128.5 (CH), 128.7 (CH), 128.9 (CH), 129.5 (CH), 131.0 (CH), 131.5 (CH), 142.4 (C); MS (CI) m/z 231 $[\text{M}+\text{H}]^+$.

(8-Hex-3-enyloxy-oct-3-enyl)-benzene (12f). The synthesis was performed according to the general procedure for **12e**. Purification by flash chromatography (SiO_2 , gradient: hexane to EtOAc:hexane 1:19) afforded **12f** (151 mg, 0.53 mmol) as a colorless oil in 61% yield. R_f 0.33 (EtOAc:hexane 1:19); ^1H NMR (CDCl_3 , 300 MHz) δ 1.00-1.06 (m, 3H), 1.34-1.49 (m, 2H), 1.56-1.72 (m, 2H), 2.04-2.16 (m, 4H), 2.33-2.46 (m, 4H), 2.70-2.76 (m, 2H), 3.43-3.50 (m, 4H), 5.40-5.58 (m, 4H), 7.21-7.36 (m, 5H); MS (CI) m/z 287 $[\text{M}+\text{H}]^+$; HRMS (CI) calcd for $\text{C}_{20}\text{H}_{30}\text{O}$ $[\text{M}+\text{H}]^+$ 287.2375, found 287.2369.

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tn         C13
at         1.777
np         64000
sw         18009.9
fb         10000
bs         16
tpwr       50
pw         6.0
p1         30.0
d1         2.000
d2         0.007
d3         0.001
tof        586.5
nt         512
ct         162
a1ock      n
gain       60
          FLAGS
il         n
in         n
dp         n
hs         nn
          DISPLAY
sp         -853.9
wp         18009.9
vs         116
sc         0
wc         250
hzmm       72.04
          500.00
          100.000
          200
          ph
          180 160 140 120 100 80 60 40 20 0 ppm

```

SAMPLE		DEC. & VT	
date	Jul 25 01	dfrq	300.141
solvent	CDCl3	dn	H1
file	exp	dpr	36
ACQUISITION		dof	0
sfrq	121.489	dm	yyy
tn	P31	dmm	w
at	0.640	dmf	7000
np	64000	dseq	
sw	50000.0	dres	1.0
fb	27600	homo	n
bs	16	PROCESSING	
tpwr	57	lb	2.00
pw	2.0	wtfile	
d1	2.000	proc	ft
tof	-75.6	fn	not used
nt	16	math	f
ct	16	werr	
alock	n	wexp	
gain	40	wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-24751.7		
wp	50000.0		
vs	7196		
sc	0		
wc	250		
hzmm	200.00		
is	354.40		
rfl	24751.7		
rfp	0		
th	20		
ins	100.000		
ai	no	ph

DEC. & VT 300.142

solvent	cd3od	exp	30	HI
file	ACQUISITION	file	30	0
sfrq	300.142	dm	nmn	c
tn	3.813	dmf	200	
at	32000	dseq	1.0	n
np	4196.4	dres	0.10	
sw	2400	homo		
fb	16	PROCESSING		
bs	60	lb		
tpwr	3.0	wfile		
pw	1.000	proc	ft	
d1	152.3	fn	not used	
tof	160	math	f	
nt	35	werr		
ct	not used	wexp		
alock	FLAGS	wbs		
gain		wnt		
il	n			
in	n			
dp	y			
hs	nn			
sp	-453.4			
wp	4196.4			
vs	198			
sc	0			
wc	250			
hzmm	16.79			
ls	752.78			
rfl	1443.9			
rfl	990.5			
rfl	20			
th	3.000			
ins				
nm	cdc			
ph

SAMPLE Sep 11 01
 date Sep 11 01
 solvent CDC13
 file exp
 ACQUISITION
 sfrq 121.499
 tn P31
 at 0.640
 np 64000
 sw 50000.0
 fb 27600
 bs 16
 tpwr 57
 pw 2.0
 d1 2.000
 tof -75.6
 nt 16
 ct 16
 alock n
 gain 40
 FLAGS n n n n
 il n
 in n
 dp n
 hs nn
 DISPLAY
 sp -24751.7
 wp 50000.0
 vs 3552
 sc 0
 wc 250
 hzmm 200.00
 ls 354.40
 rfl 24751.7
 rfp 0
 th 20
 ins 100.000
 ai no ph

DEC. & VT
 dfrq 300.141
 dn H1
 dpwr 36
 dof 0
 dm yvy
 dmm w
 dmf 7000
 dseq 1.0
 dres n
 homo
 PROCESSING
 lb 2.00
 wtfile
 proc ft
 fn not used
 math f
 werr
 wexp
 wbs
 wnt

SAMPLE Jan 25 02 DEC. & VT
 date CDC13 300.141
 solvent dn H1
 file exp 30
 ACQUISITION dof 0
 sfrq 300.141 dm mnn
 tn H1 c
 at 3.813 dmf 200
 np 32000 dseq
 sw 4196.4 dres 1.0
 fb 2400 homo n
 bs 16 PROCESSING
 tpwr 60 lb not used
 pw 3.0 wfile
 di 1.000 proc ft
 tof 152.3 fn not used
 nt 8 math f
 ct 8
 alock n werr
 gain not used wexp
 FLAGS n wbs
 il n wnt
 in n
 dp y
 nn
 :SPLAY -150.1
 3001.2
 287
 0
 250
 12.01
 950.20
 445.2
 0
 20
 3.000
 ph

```

--u1
SAMPLE  Jan 25 02  DEC. & VT 300.141
date      Jan 25 02  dfrq      300.141
solvent   CDC13     dn        H1
file      exp       dpwr      36
ACQUISITION
sfrq      121.499   dm        YVY
tn        P31      dmm        W
at        0.640     dmf        7000
np        64000     dseq
sw        50000.0   dres      1.0
fb        27600     homo      n
bs        16       PROCESSING
tpwr      57       lb        2.00
pw        2.0      wtfile
d1        2.000     proc      ft
tof       -75.6     fn        not used
nt        16       math      f
ct        16       werr
alock     n        wexp
gain      40       wbs
flags     n        wnt
il        n
in        n
dp        n
hs        nn
DISPLAY
sp        -24751.7
wp        50000.0
vs        16705
sc        0
wc        250
hzmh      200.00
ls        354.40
rfl       24751.7
rfp       0
th        20
ins       100.000
ai no. ph

```

SAMPLE date Jun 21 01 DEC. & VT 300.141
 solvent CDC13 dn H1
 file exp 30
 ACQUISITION
 sfrq 300.141 dm nm
 tn H1 dmm C
 at 3.813 dmf 200
 np 32000 dseq
 sw 4196.4 dres 1.0
 fb 2400 homo n
 bs 16 PROCESSING
 tpwr 60 lb 0.10
 pw 3.0 wfile
 d1 1.000 proc ft
 tof 152.3 fn not used f
 nt 8 math
 ct 8
 alock n werr
 gain not used wexp
 flags n wbs
 il n wnt
 in n
 dp y
 hs nn
 DISPLAY
 sp -150.1
 wp 3001.2
 vs 456
 sc 0
 wc 250
 hzmm 12.01
 ls 777.46
 rfl 445.2
 rfp 0
 th 8
 ins 6.000
 nm cdc ph

SAMPLE		DEC. & VT	
date	Jun 21 01	dfrq	300.141
solvent	CDCl3	dn	H1
file	exp	dpwr	36
ACQUISITION		dof	0
sfrq	121.499	dm	yyv
tn	0.640	dmm	7000
at	64000	dseq	
np	50000.0	dres	1.0
sw	27600	homo	n
fb	16	PROCESSING	
bs	57	lb	2.00
tpwr	2.00	wfile	
pw	2.000	proc	ft
d1	-75.6	fn	not used
tof	16	math	f
nt	16	werr	
ct	40	wexp	
alock		wbs	
gain		wnt	
FLAGS			
il	n		
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-24751.7		
wp	50000.0		
vs	256		
sc	0		
wc	250		
hzmm	200.00		
ls	354.40		
rfl	24751.7		
rfp	0		
th	20		
ins	100.000		
ai	no	ph	

36.249

200 150 100 50 0 -50 -100 -150 ppm

```

IPLE Jun 18 02 dfrq 300.141 DEC. & VT
  solvent CDC13 dn H1
  file exp 30
  ACQUISITION dOf 0
  sfrq 300.141 dm nnn
  tn H1 c
  at 3.813 dmf 200
  rp 32000 dseq
  sw 4186.4 dres 1.0
  fb 2400 homo n
  bs 16
  tpwr 60 lb PROCESSING 0.10
  pw 3.0 wtfile
  d1 1.000 proc ft
  tof 152.3 fn not used
  nt 8 math f
  ct 8
  alock n
  gain not used
  FLAGS n n
  il in y
  dp ns mn
  ns
  DISPLAY
  sp -150.1
  wp 3001.2
  vs 173
  SC 0
  WC 250
  hzmm 12.01
  is 725.79
  rfl 445.2
  rfp 0
  th 11
  ins 3.000
  nm cdc ph

```

date Jun 18 02
 solvent cdcl3
 file exp
 ACQUISITION
 sfrq 75.478
 tn C13
 at 1.777
 np 64000
 sw 18009.9
 fb 10000
 bs 16
 tpwr 50
 pw 6.0
 p1 30.0
 d1 2.000
 d2 0.007
 d3 0.001
 tof 586.5
 nt 256
 ct 38
 alock n
 gain 60
 FLAGS
 il n
 in n
 dp n
 hs mn
 DISPLAY
 sp -887.2
 wp 18009.9
 vs 394
 sc 0
 vc 250
 pz 179.000
 f1 100.000
 f2 100.000
 f3 100.000

DEC. & VT 300.141
 dn H1
 dpwr 40
 dof 400.0
 dm yny
 dmm w
 dseq 8000
 dres 1.0
 homo n
 PROCESSING
 lb 1.00
 wf file
 proc ft
 fn not used
 math f
 werr
 wexp
 wbs
 wnt
 proclot
 testsn

220 200 180 160 140 120 100 80 60 40 20 0 ppm

```

E          DEC. & VT
Jun 19 02  dfrq 300.141
solvent CDC13 dn H1
file exp dpwr 36
ACQUISITION dof 0
sfrq 121.499 dm yvy
tn P31 dnm W
at 0.640 dmf 7000
np 64000 dseq
sw 50000.0 dres 1.0
fb 27600 homo n
bs 16 PROCESSING
tpwr 57 lb 2.00
pw 2.00 wfile
d1 2.000 proc ft
tof -75.6 fn not used
nt 16 math f
ct 16
alock n werr
gain 40 wexp
il 1 n wbs
in n wnt
dp n
hs nn
FLAGS
il 1 n
in n
dp n
hs nn
DISPLAY
sp -24751.7
wp 50000.0
vs 405
sc 0
wc 250
hzm 200.00
is 354.40
rfl 24751.7
rfp 0
th 7
ins 100.000
ai no ph

```

33.787

200 150 100 50 0 -50 -100 -150 ppm

```

DEC. & VT
01 dfrq 300.141
C13 dn H1
exp 30
dpwr 0
dof 0
dm nnn
tn H1
at 3.813
np 32000
sw 4196.4
fb 2400
bs 16
tpwr 60
pw 3.0
d1 1.000
tof 152.3
      ft
      not used
      f

oek n
in not used
      n
      n
      y
      nn
      mm
      227
      0
      250
      12.01
      878.88
      445.2
      0
      6
      3.000
      s cdc ph
  
```

```

DISPLAY
-150.1
3001.2
227
0
250
12.01
878.88
445.2
0
6
3.000
s cdc ph
  
```

DEC. & VT

date	Oct 19 01	dfrq	300.141
solvent	cdcl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yny
tn	C13	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	16	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
p1	30.0	proc	ft
d1	2.000	fn	not used
d2	0.007	math	f
d3	0.001		
tof	586.5	werr	
nt	256	wexp	procplot
ct	163	wbs	testsn
gain	60	wnt	
FLAGS			
il	n		
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-853.9		
wp	18009.9		
vs	208		
sc	0		
mc	250		
h2	172.04		
h3	150.00		
h4	100.00

DEC. & VT 300.141

file	exp	ifrq	dn	H1
ACQUISITION	300.141	300.141	30	0
sfrq	H1	dm	dof	nmn
at	3.813	dmf	dm	C
np	32000	dseq	200	
sw	4196.4	dres	1.0	
fb	2400	homo	n	
bs	16	PROCESSING		
tpwr	60	lb	0.10	
pw	3.0	wtfile		
dl	1.000	proc	ft	
tof	152.3	fn	not used	
nt	8	math	f	
ct	8			
alock	n	werr		
gain	not used	wexp		
FLAGS		wps		
il	n	wnt		
in	n			
dp	y			
hs	nn			

WISPLAY

-150.1
3001.2
1043
0
250
12.01
1059.54
445.2
0
9
3.000

dc ph

DEC. & VT

file	12	dfrq	300.141
exp	13	dn	H1
dpwr	40	dpwr	40
dof	400.0	dof	400.0
sfrq	75.478	dm	yny
tn	Cl3	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	16	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
p1	30.0	proc	ft
d1	2.000	fn	not used
d2	0.007	math	f
d3	0.001	werr	
tof	586.5	wexp	procplot
nt	512	wbs	testsn
ct	244	wnt	
alock	n		
gain	60		
FLAGS			
il	n		
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-881.7		
wp	18009.9		
vs	331		
sc	0		
wc	250		
hzmm	72.04		
is	500.00		
th	18		
1ns	100.000

```

file ACQUISITION exp
sfrq 121.489
tn p31 dmm 7000
at 0.640 dmf 7000
np 64000 dseq 1.0
sw 50000.0 dres 1.0
fb 27600 homo n
bs 16 PROCESSING
tpwr 57 lb 2.00
pw 2.0 wtfile
di 2.000 proc ft
tof -75.6 fn not used
nt 16 math f
ct 16
gain n werr
alock n wexp
il 40 wbs
in n wht
dp n
hs mn
flags
DISPLAY
sp -24751.7
wp 50000.0
vs 797
sc 0
wc 250
hzmm 200.00
is 354.40
rfl 24751.7
rfp 0
th 8
ins 100.000
ai no ph

```

DEC. & VT

dfrq 300.141

dn H1

dopr 36

dof 0

dm YYY

dmf 7000

dseq 1.0

dres 1.0

homo n

PROCESSING

lb 2.00

wtfile

proc ft

fn not used

math f

werr

wexp

wbs

wht

mn

sp -24751.7

wp 50000.0

vs 797

sc 0

wc 250

hzmm 200.00

is 354.40

rfl 24751.7

rfp 0

th 8

ins 100.000

ai no ph

29.128

200 150 100 50 0 -50 -100 -150 ppm

```

date      Mar 19 02
solvent   CDCl3
file      ACQUISITION exp
sfrq      300.141
tn        3.813
np        32000
sw        4196.4
fb        2400
bs        15
tpwr      60
pw        3.0
d1        1.000
tof       152.3
nt        8
ct        8
alock     not used
gain      not used
flags     n n n n n
l         n n n n n
lp        y
is        nn
DISPLAY
ip        -150.1
rp        3001.2
rs        1020
ic        0
rc        250
izmm      12.01
s         919.49
-fl       445.2
-fp       0
ch        3
ins       4.000
nm        cdc ph
  
```

date	Mar 19 02	dfreq	300.141
solvent	cdcl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yn
tn	75.478	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	r
bs	16	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
p1	30.0	proc	ft
d1	2.000	fn	not used
d2	0.007	math	f
d3	0.001		
tof	586.5	werr	procplot
nt	512	wexp	testsn
ct	242	wbs	
alock	n	wnt	
gain	60		
FLAGS			
il	n		
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-881.7		
wp	18009.9		
vs	297		
sc	0		
wc	250		
h2mm	72.04		
h1mm	500.00		
h0mm	1000.00		
ph	13		
ns	100.000		
no	200		
ph	180

SAMPLE 19 02 DEC. & VT
 date Mar 19 02 dfrq 300.141
 solvent CDC13 dn H1
 file exp 36
 ACQUISITION 121.499
 sfrq 121.499 dm 0
 tn P31 dmm YVY
 at 0.640 dmf W
 np 64000 dseq 7000
 sw 50000.0 dres 1.0
 fb 27600 homo n
 bs 16 PROCESSING
 tpwr 57 lb 2.00
 pw 2.00 wfile
 dl 2.000 proc ft
 tof -75.6 fn not used
 nt 16 math f
 ct 16
 alock n werr
 gain 40 wexp
 flags wbs
 il n wnt
 in n
 dp n
 hs nn
 DISPLAY
 sp -24751.7
 wp 50000.0
 vs 282
 sc 0
 wc 250
 hzmm 200.00
 is 354.40
 rfl 24751.7
 rfp 0
 th 5
 ins 100.000
 al no ph

30.635

```

DEC. & VT
dfrq 300.141
dn H1
dpr 30
dof 0
dm nnn
dmm c
dmf 200
dseq 1.0
dres n
homo n
PROCESSING
lb 0.10
wf file
proc ft
fn not used
math f
werr n
wexp wbs
wnt n

```

```

n
at 3.813
np 32000
sw 4196.4
fb 2400
bs 16
tpwr 60
pw 3.0
d1 1.000
tof 152.3
nt 8
ct 8
alock n
gain not used
FLAGS n n y nn

```

```

DISPLAY
-150.1
3001.2
324
0
250
12.01
795.48
445.2
0
4
cdc ph 6.000

```

```

27 01 dfrq DEC. & VT 300.141
20C13 dn H1 36
exp dpwr 0
i dof yyv 7000
.499 dm 1.0
P31 dmm 2.00
0.640 dmf 7000
64000 dseq 1.0
50000.0 dres n
27600 homo PROCESSING
16 57 lb 2.00
tpwr 2.0 wfile ft
pw 2.000 proc not used
dl -75.6 fn f
nt 16 math
ct 16 n werr
gain alock 40 wexp
FLAGS whs
il n wnt
in n
dp n
hs nn
DISPLAY
sp -24751.7
wp 50000.0
vs 195
sc 0
wc 250
hzmm 200.00
ls 354.40
rfl 24751.7
rfp 0
th 20
ins 100.000
al no ph

```

39.125

200 150 100 50 0 -50 -100 -150 ppm

```

.E 19.02 dfrq 300.141 DEC. & VT
in CDC13 dn H1
exp 30 0
dopr 0
dof 0
dm c
dmn 200
dmf 1.0
dseq 1.0
dres 1.0
dres 1.0
homo n
PROCESSING
lb 0.10
pw 3.0
wfile ft
dl 1.000 proc
tof 152.3 fn not used
nt 8 math f
ct 8
alock n
gain not used
werr n
wexp wbs
wnt wnt
FLAGS n n
nn
DISPLAY
sp -150.1
wp 3001.2
vs 202
wc 250
hzm 12.01
hzm 845.63
hzm 445.2
hzm 0
hzm 5
hzm 6.000

```

E 19 02 dfrq 300.141
 n CDC13 H1
 exp 36
 ACQUISITION
 sfrq 121.499
 tn P31 dmm w
 at 0.640 dmf 7000
 np 64000 dseq
 sw 50000.0 dres 1.0
 fb 27600 homo n
 bs 16
 tpwr 57
 pw 2.0 wfile 2.00
 d1 2.000 proc ft
 tof -75.6 fn not used
 nt 16 math f
 ct 16
 alock n
 gain 40 werf
 wexp
 wbs
 wnt
 FLAGS
 il n
 in n
 dp n
 hs nn
 DISPLAY
 sp -24751.7
 wp 50000.0
 vs 336
 sc 0
 wc 250
 hzmm 200.00
 is 354.40
 rfl 24751.7
 rfp 0
 th 7
 ins 100.000
 ai no ph

36.487
36.437

200 150 100 50 0 -50 -100 -150 ppm

```

DEC. & VT 300.141
:2.01 dfrq 300.141 H1
:DC13 dn 30
exp 0
... ACQUISITION
sfrq 300.141 nmh 200
tn 3.813 C
at 32000 dmf 1.0
np 4196.4 dseq n
sv 2400 homo
fb 16
hs 60 lb 0.10
pwr 3.0 lb wtfile
w 1.000 proc ft
l 152.3 fn not used
of 8 math f
t 8
t lock n werr
aln not used wbs
FLAGS n wnt
1 n
n n
p y
s nn
DISPLAY
p -150.1
p 3001.2
p 437
s 0
c 250
c 12.01
zmm
s 950.22
f1 445.2
fp 0
h 5
ns 10.000
im cdc ph

```

DEC. & VT 300.141
 solvent cdc13
 file exp
 ACQUISITION
 sfrq 75.478
 tn 1.777
 at 64000
 np 18009.9
 sw 10000
 fb 16
 bs 50
 tpwr 6.0
 pw 30.0
 p1 2.000
 d1 0.007
 d2 0.001
 d3 586.5
 tof 512
 nt 251
 ct 60
 alock n
 gain n
 FLAGS
 il n
 in n
 dp n
 hs nn
 DISPLAY
 sp -890.0
 wp 18009.9
 vs 225
 sc 0
 wc 250
 hzmm 72.04
 is 500.00
 th 100.000
 200 no ph 200

```

DEC. & VT
: 01 dfrq 300.141
PC13 dn H1
exp 36
sfrq 121.499 dm yyy
tn p31 dnm 7000
at 0.640 dmf
np 64000 dseq 1.0
sw 50000.0 dres n
fb 27600 homo
bs 16 PROCESSING
tpwr 57 lb 2.00
pw 2.0 wfile
d1 2.000 proc ft
tof -75.6 fn not used
nt 16 math f
ct 16 werr
alock n wexp
gain 40 wbs
il n wnt
in n
dp n
hs nn
DISPLAY 4056.3
sp 528.0
wp 299
vs 0
sc 250
wc 2.11
hzmm 354.40
is 24751.7
rfl 0
rfp 8
th 100.000
ins ai no ph

```

DEC. & VT 300.141

1 dfrq 300.141 H1 30 0

solvent exp 3.0 30 0

file exp 3.0 30 0

ACQUISITION

sfrq 300.141 dm nm 200

tn 3.813 dmf 200

np 32000 dseq

sw 4198.4 dres

fb 2400 homo

bs 16

tpwr 60

pw 3.0 wfile

dl 1.000 proc

tof 152.3 fn not used

nt 8 math

ct 8

gain not used

alock n

flags n

il n

in n

dp y

hs mn

DISPLAY

sp -150.1

wp 3001.2

vs 166

sc 0

wc 250

hzmm 12.01

is 1017.44

rfl 445.2

rfl 0

rfl 4

th 4

ins 2.000

nm cdc ph

DEC. & VI

solvent	cacl ₂	rq	300.141
file	exp	HI	40
ACQUISITION			
sfreq	75.478	dof	400.0
tn	C13	dm	yny
at	1.777	dmm	w
np	64000	dseq	8000
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	16	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
p1	30.0	proc	ft
d1	2.000	fn	not used
d2	0.007	math	f
d3	0.001		
tof	586.5	werr	
nt	512	wexp	procplot
ct	261	wbs	testsn
alock	n	wnt	
gain	60		
FLAGS			
il	n		
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-892.7		
wp	18009.9		
vs	304		
sc	0		
wc	250		
hzmm	72.04		
is	500.00		
ph	7		
th	100.000

DEC. & VT

solvent	CDCl3	dn	300.141
file	exp	dpwr	H1
	dof		36
			0
sfrq	121.499	dm	yyv
tn	0.640	dmm	w
at	0.640	dmf	7000
np	64000	dseq	
sw	50000.0	dres	1.0
fb	27600	homo	n
bs	16	PROCESSING	
tpwr	57	lb	2.00
pw	2.0	wfile	
d1	2.000	proc	ft
tof	-75.6	fn	not used
nt	16	math	
ct	16		
alock	n	werr	
gain	40	wexp	
il	n	whs	
in	n	wnt	
dp	n		
hs	nn		
sp	3502.4		
wp	1477.1		
vs	761		
sc	0		
wc	250		
hzm	5.91		
is	354.40		
rfl	24751.7		
rfl	0		
th	20		
ins	100.000		
ai	no	ph

DEC. & VT 300.141

file ACQUISITION
 sfrq 75.478
 tn C13
 at 1.777
 np 64000
 sw 18009.9
 fb 10000
 bs 16
 tpwr 50
 pw 6.0
 pl 30.0
 d1 2.000
 d2 0.007
 d3 0.001
 tof 586.5
 nt 512
 ct 268
 alock n
 gain 60
 flags
 il n
 in n
 dp n
 hs nn
 sp -888.3
 wp 18009.9
 vs 271
 sc 0
 wc 250
 hzmm 72.04
 is 500.00
 th 100.000

dm 400.0
 yny w
 dmf 8000
 dseq 1.0
 dres n
 homo 1.00
 lb 1.00
 wfile ft
 proc not used
 fn f
 math f
 werr
 wexp
 wbs
 wnt
 proplot
 testn

```

DEC. & VT 300.141
file 02 dfrq 300.141 H1
exp 213 dn 36
acq 121.499 dm 0
sfrq 121.499 dm 0
tn 0.640 dmm 7000
at 64000 dmf 7000
np 50000.0 dseq 1.0
sw 27600 dres 1.0
fb 16 homo n
bs 16
tpwr 57 lb 2.00
pw 2.00 wfile
dl 2.000 proc ft
tof -75.6 fn not used
nt 16 math f
ct 16
gain 40 werr n
il 16 wbs n
in 16 wnt n
dp 16
hs nn
flags
il 16 n
in 16 n
dp 16 n
hs nn
sp -24751.7
wp 50000.0
vs 4814
sc 0
wc 250
hzmm 200.00
is 354.40
rfl 24751.7
rfp 0
th 12
ins 100.000
ai no ph

```

30.283
30.147
29.140

0.141
 HI
 30
 0
 nnn
 c
 200
 1.0
 n
 2400
 homo
 PROCESSING
 16
 60
 lb
 0.10
 pw
 3.0
 wfile
 1.000
 proc
 152.3
 fn
 not used
 nt
 8
 math
 ct
 8
 n
 werr
 gain
 not used
 wexp
 wbs
 wnt
 il
 n
 y
 in
 n
 dp
 y
 hs
 nn
 DISPLAY
 -150.1
 3001.2
 796
 0
 250
 12.01
 715.89
 445.2
 0
 5
 4.000
 .h

```

NAME          C13
IN            1.777
NP            64000
SW            18009.9
FB            10000
BS            16
TPWR          50
PW            6.0
P1            30.0
D1            2.000
D2            0.007
D3            0.001
TOF           586.5
NT            256
CT            162
ALOCK         60
GAIN          60
          FLAGS
          IL      N
          IN      N
          DP      N
          HS      NM
          DISPLAY
SP      -887.2
WP      18009.9
VS      308
SC      0
WC      250
H2MM    72.04
H1MM    500.00
          100.000
          200
          220 no ph
          100.000
          180
          160
          140
          120
          100
          80
          60
          40
          20
          0 ppm
  
```

```

solvent      CDCl3      dn      0.141
file         exp      dpwr      H1
              ACQUISITION  dof      36
              tn      dm      0
              sfrq    121.499  dmm    yyy
              at      0.640  dmf      w
              np      64000  dseq     7000
              sw      50000.0  dres    1.0
              fb      27600  homo     n
              bs      16      PROCESSING
              tpwr    57      lb      2.00
              pw      2.0      wfile
              d1      2.000  proc     ft
              tof     -75.6  fn      not used
              nt      16      math     f
              ct      16
              alock   gain     n      werr
              gain    40      wexp    wbs
              il      n      wnt
              in      n
              dp      n
              hs      nn
              DISPLAY  sp      3580.2
              wp      468.5
              vs      483
              SC      0
              WC      250
              hzmm    1.87
              ls      354.40
              rfl     24751.7
              rfp      0
              th      34
              ins     100.000
              ai      no      ph

```

```

DEC. & VT
... 22 02 dfrq 300.141
file CDC13 dn H1
ACQUISITION exp dpwr 30
sfrq 300.141 dm dof 0
tn H1 dnm nnn
at 3.813 dmf C
np 32000 dseq 200
sw 4196.4 dres 1.0
fb 2400 homo n
ls 16 PROCESSING
tpwr 60 lb 0.10
pw 3.0 wfile ft
d1 1.000 proc not used
tof 152.3 fn f
nt 8 math
Ct 8
alock n werr
gain not used wexp
FLAGS wbs
il n wnt
in n
dp y
hs nm
DISPLAY
sp -150.1
wp 3001.2
vs 231
sc 0
wc 250
hzmm 12.01
fs 1473.09
rfl 445.2
rfp 0
th 9
ins 2.000
nm cdc ph

```

solvent file cdc13
 ACQUISITION
 sfrq 75.478
 tn 1.777
 np 64000
 sw 18009.9
 fb 10000
 bs 16
 tpwr 50
 pw 6.0
 pl 30.0
 d1 2.000
 d2 0.007
 d3 0.001
 tof 586.5
 nt 512
 ct 34
 alock n
 gain 60
 flags
 il n
 in n
 dp n
 hs nn
 DISPLAY
 sp -879.5
 wp 18009.9
 vs 484
 sq 0
 wc 250
 Hz 120.04
 100000
 200 no ph

DEC. & VT 300.141
 dn H1
 dpwr 40
 dof 400.0
 dm yny
 dmm w
 dmf 8000
 dseq 1.0
 dres n
 homo n
 PROCESSING
 lb 1.00
 wfile ft
 proc not used
 fn math f
 werr
 wexp
 wbs
 wnt
 procplot
 testsn

date Sep 27 01 DEC. & VT
 solvent cdc13 dn H1
 file exp dpwr 40
 ACQUISITION
 sfrq 75.478
 tn C13 dm w
 at 1.777 dmf 8000
 np 64000 dseq 1.0
 sw 18009.9 dres n
 fb 10000 homo n
 ps 16
 tpwr 50 lb
 pw 6.0 wfile 1.00
 p1 30.0 proc ft
 d1 2.000 fn not used
 d2 0.007 math f
 d3 0.001
 tof 586.5 werr
 nt 256 wexp
 ct 83 wbs
 alock n
 gain 60 wnt
 FLAGS
 il n
 in n
 dp n
 hs nn
 DISPLAY
 sp -853.9
 wp 18009.9
 vs 218
 sc 0
 wc 250
 hzmm 72.04
 500.00
 100.000
 200
 180
 160
 140
 120
 100
 80
 60
 40
 20
 0 ppm

date Sep 14 01
 solvent CDC13
 file exp
 ACQUISITION
 sfrq 300.141
 tn H1
 at 3.813
 np 32000
 sw 4196.4
 fb 2400
 bs 16
 tpwr 60
 pw 3.0
 dl 1.000
 tof 152.3
 nt 8
 ct 8
 alock not used
 gain not used
 il n
 in n
 dp y
 hs nn
 DEC. & VT 300.141
 dn H1
 dpwr 30
 dof 0
 dm nnn
 dmm c
 dmf 200
 dseq 1.0
 dres n
 homo n
 PROCESSING
 lb 0.10
 wfile
 proc ft
 fn not used
 math f
 werr
 wexp
 wbs
 wnt
 DISPLAY
 -150.1
 3001.2
 535
 0
 250
 12.01
 818.72
 445.2
 0
 11
 2.000
 cdc ph

date Sep 14 01
 solvent cdc13
 file exp
 ACQUISITION
 sfrq 75.478
 tn C13
 at 1.777
 np 64000
 sw 18009.9
 fb 10000
 bs 16
 tpwr 50
 pw 6.0
 pl 30.0
 d1 2.000
 d2 0.007
 d3 0.001
 tof 586.5
 nt 512
 ct 483
 alock n
 gain 60
 FLAGS
 il n
 in n
 dp n
 hs nn
 DISPLAY
 sp -853.9
 wp 18009.9
 vs 354
 sd 250
 vc 120
 id 120

DEC. & VT 300.141
 H1
 40
 400.0
 yny
 w
 8000
 1.0
 n
 PROCESSING
 lb 1.00
 wfile
 30.0
 proc
 fn not used
 math
 f
 werr
 wexp
 wbs
 wnt
 procplot
 testsn

date 12-01-14
 solvent CDCl₃
 file exp
 ACQUISITION
 sfrq 75.478
 tn C13
 at 1.777
 np 64000
 sw 18009.9
 fb 10000
 bs 16
 tpwr 50
 pw 6.0
 p1 30.0
 d1 2.000
 d2 0.007
 d3 0.001
 tof 588.5
 nt 512
 ct 277
 alock n
 gain 60
 flags n
 il n
 in n
 dp n
 hs nn
 DISPLAY
 sp -853.9
 wp 18009.9
 vs 323
 sc 0
 wc 250
 hzmm 72.04
 is 500.00
 ch 16
 ins 100.000

DEC. & VT 300.141
 dn H1
 dpwr 40
 dof 400.0
 dm yny
 dmm w
 dmf 8000
 dseq 1.0
 dres n
 homo n
 PROCESSING
 lb 1.00
 wfile ft
 proc not used
 fn f
 math f
 werr
 wexp
 wbs
 wnt
 procplot
 testn

EC. & VT 300.141

solvent	CDC13	dn	300.141
file	exp	dpwr	H1
ACQUISITION	300.141	dof	30
sfrq	300.141	dm	0
tn	3.813	dmf	nmn
at	32000	dseq	C
np	4196.4	dres	200
sw	2400	homo	1.0
fb	16	lb	n
bs	60	wfile	0.10
tpwr	3.0	proc	ft
pw	1.000	fn	not used
dl	152.3	math	f
tof	8	werr	
nt	8	wexp	
ct	not used	wbs	
alock	FLAGS	wnt	
gain	n	nn	
il	n		
in	y		
dp	nn		
hs			
sp	-150.1		
wp	3001.2		
vs	173		
sc	0		
wc	250		
h2mm	12.01		
is	1800.50		
rfl	445.2		
rfp	0		
tn	14		
ins	4.000		
nm	cdc		
ph