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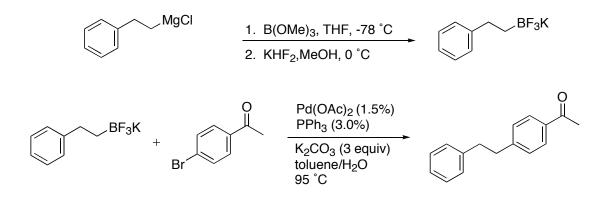
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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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## 1-(4-ACETYLPHENYL)-2-PHENYLETHANE FROM POTASSIUM 2-PHENETHYLTRIFLUOROBORATE AND 4-BROMOACETOPHENONE



Submitted by Gary A. Molander and Daniel E. Petrillo.<sup>1</sup> Checked by Scott E. Denmark and Christophe Eggertswyler.

### 1. Procedure

A. Preparation of Potassium 2-Phenethyltrifluoroborate. A singlenecked, 500-mL round-bottomed flask, equipped with a magnetic stirring bar, a 125-mL pressure-equalizing addition funnel, and connected by a three-way stopcock fitted with a rubber septum to a vacuum-argon manifold, is flame-dried under vacuum (0.3 mm Hg), then is flushed with dry argon. THF (90 mL) is added via syringe to the addition funnel, followed by trimethyl borate (10.0 mL, 89.2 mmol, 1.5 equiv) (Note 1). After the addition of the trimethyl borate to the round-bottomed flask, additional THF (30 mL) (Note 1) is added via syringe to rinse the addition funnel. The solution is then cooled in a dry ice/isopropyl alcohol bath. Phenethylmagnesium chloride (60.0 mL of a 1.0 M solution in THF, 60.0 mmol) (Note 2) is then added dropwise over 1 h via the addition funnel. The mixture is stirred in the dry ice/isopropyl alcohol bath for 1.5 h, then is allowed to warm to room temperature over 1 h. The resulting white slurry is then cooled in an ice/water bath and methanol (60 mL) (Note 3) is charged into the addition funnel by syringe. The methanol is added over 15 min to dilute the slurry (Note 4). The argon inlet is removed and a solution of potassium hydrogen fluoride (80.0 mL of 4.5 M aqueous solution, 360

mmol, 6.0 equiv) (Note 5) is then charged into the addition funnel and is added dropwise over 15 min. The resulting solution is stirred for 1 h in the ice/water bath, and then is concentrated on a rotary evaporator (60 °C water bath, 9 mm Hg). The resulting white solids are placed on a high-vacuum line (0.3 mm Hg) for 36 h until the solids appear dry (Note 6). Acetone (120 mL) (Note 7) is added and the solids are broken up into a fine powder with a spatula. The suspension is then placed on a rotary evaporator and heated (40) °C) with rotation under atmospheric pressure for 5–10 min and filtered over a 14 g pad of Celite in a glass-fritted filter funnel. Acetone (120 mL) is added to the solid remaining in the flask, the suspension is placed on a rotary evaporator and heated (40 °C) with rotation under atmospheric pressure for 5–10 min and filtered over the same Celite pad. This process is repeated one additional time (Note 8). The combined filtrates are concentrated on a rotary evaporator (60 °C water bath) to a white solid, which is dissolved in a minimal amount of dry acetone (~25 mL). Diethyl ether (30 mL) is then added to precipitate the product. The white slurry is allowed to cool first to 23 °C, and then is cooled in an ice/water bath for 15 min. The slurry is placed in a refrigerator (4 °C) for 1 h, and is then filtered to give 9.84 g (77%) of a white solid (Note 9).

B. Preparation of 1-(4-Acetylphenyl)-2-phenylethane. A threenecked, 250-mL, round-bottomed flask, equipped with a reflux condenser, a rubber septum, a glass stopper, a magnetic stirring bar, and connected to a vacuum-argon manifold, is charged sequentially with potassium 2phenethyltrifluoroborate (7.77)36.6 mmol. 1.05 equiv), 4g, bromoacetophenone (6.97 g, 35.0 mmol), potassium carbonate (14.5 g, 105 mmol, 3.0 equiv), triphenylphosphine (0.275 g, 1.05 mmol, 0.03 equiv) and palladium acetate (0.118 g, 0.53 mmol, 0.015 equiv) (Note 10). The flask is evacuated and filled with dry argon three times. Toluene (105 mL) (Note 11) and H<sub>2</sub>O (21 mL) are added, and the resulting two-phase mixture is stirred in a 95 °C oil bath until consumption of the 4-bromoacetophenone is indicated by GC analysis (Note 12). The mixture is cooled to room temperature and then is transferred to a 250-mL separatory funnel. The flask is rinsed with toluene (20 mL) and H<sub>2</sub>O (20 mL). The layers are shaken and separated. The organic layer is washed with 10% aq. citric acid solution (20 mL) (Note 13) and saturated aq. NaCl solution (20 mL). The organic layer is dried over anhydrous  $Na_2SO_4$  (0.780 g) and filtered through a medium porosity glass-fritted funnel, rinsing the drying agent with toluene (20 mL). The resulting solution is concentrated (50 °C water bath, 9 mmHg) to a light 318

yellow solid (Note 14), which is crystallized from methanol to give 5.79 g (74%, two crops) (Notes 15, 16) of a light tan solid. Recrystallization of 0.3 g of this solid from hot methanol (2 mL) provided white, crystalline material (0.25 g) of analytical purity (Note 17).

## 2. Notes

1. Trimethyl borate (98%) was obtained from Aldrich Chemical Company and was distilled from sodium. THF (Fischer, HPLC Grade) was dried by percolation through a column packed with neutral alumina and a column packed with Q5 (a supported copper catalyst for removing oxygen) under a positive pressure of argon.

2. Phenethylmagnesium chloride (1.0 M in THF) was obtained from Aldrich Chemical Company and was used as received.

3. Methanol (ACS Grade) was obtained from Aldrich Chemical Company and was distilled continuously from Mg(OMe)<sub>2</sub>.

4. At this stage the solution may be transferred to a Nalgene vessel to avoid etching of the glass flask by the KHF<sub>2</sub>.

5. Potassium hydrogen fluoride (p.a. >99%) was obtained from Fluka and was used as received.

6. Care was taken to ensure that the solids are free of water or else inorganic fluoride contaminated the final product.

7. Acetone (Spectroscopic Grade) was obtained from Aldrich Chemical Company and was used as received. Diethyl ether (ACS Grade) was obtained from Fischer Scientific and was used as received.

8. The desired product was soluble in acetone whereas the inorganic salts were not. This extractive process ensured that any product trapped within the inorganic solids would be dissolved.

9. Analytical data: mp: 332–335 °C (sealed tube). IR cm<sup>-1</sup>: 3026, 2921, 2847; <sup>1</sup>H NMR (600 MHz, acetone- $d^6$ )  $\delta$ : 0.51 (br, 2 H), 2.56 (dt, J = 8.8, 4.7 Hz, 2 H), 7.00–7.05 (m, 1 H), 7.14–7.17 (m, 4 H); <sup>13</sup>C NMR (150 MHz, acetone- $d^6$ )  $\delta$ : 32.1, 124.5, 127.9, 128.0, 148.5; <sup>11</sup>B NMR (192.4 MHz, acetone- $d^6$ )  $\delta$  5.48; <sup>19</sup>F NMR (470 MHz, acetone- $d^6$ )  $\delta$  –142.32; MS (ES): m/z 173.1 (100) Anal. Calcd for C<sub>9</sub>H<sub>9</sub>BF<sub>3</sub>K: C, 45.31; H, 4.28; F, 26.88. Found: C, 45.03; H, 4.29; F, 25.38.

10. 4-Bromoacetophenone (98%) was obtained from Aldrich Chemical Company and was used as received. Potassium carbonate (ACS Grade) was purchased from Fisher Scientific and was used as received. Triphenylphosphine (ReagentPlus Grade, 99%) was obtained from Aldrich Chemical Company and was used as received. Palladium acetate was obtained from Frontier Scientific and was used as received.

11. Toluene (ACS Grade, 99.5+%) was obtained from Aldrich Chemical Company and was used as received.

12. The reaction was assayed as follows: an aliquot (20  $\mu$ L) was removed and filtered through a small pad of silica gel in a Pasteur pipette, rinsing with EtOAc (1.5 mL). The sample was then injected on an HP 5890 GC with an HP 1 column (100 °C for 1 min., ramp to 250 °C at 25 °C/min.). Typical retention times were: 4-bromoacetophenone: 4.1 min, 1-(4-acetylphenyl)-2-phenylethane: 7.4 min. The reaction generally required 16–20 h.

13. Citric acid monohydrate (Baker Analyzed Reagent) was obtained from J. T. Baker Chemical and was used as received.

14. The submitters found that the crude product was a dark yellow solid that needed to be decolorized with activated carbon in MeOH prior to recrystallization.

15. The crude product was slurried in MeOH (25 mL) in a 100-mL flask and heated to 50 °C in an oil bath. After dissolution, the solution was allowed to cool to room temperature. It was then placed in an ice/water bath for 1 h, after which crystals were removed by filtration. The filtrate was concentrated (50 °C water bath) to ~ 10 mL and cooled to 0 °C, after which a second crop of the crystals was collected via filtration. This material had a melting point of 66–68 °C.

16. The submitters noted that the product may also be purified by silica gel column chromatography (10% EtOAc/hexanes as eluent,  $R_f = 0.34$ ) to give an 89% yield of product of unspecified purity.

17. Analytical data: mp: 68–72 °C (lit. mp: 68–70 °C).<sup>2</sup> IR cm<sup>-1</sup>: 1676, 1601; <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$ : 2.58 (s, 3 H), 2.92–3.01, (m, 4 H), 7.15-7.30 (m, 7 H), 7.88 (d, J = 8.3 Hz, 2 H); <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>)  $\delta$ : 26.5, 37.3, 37.8, 126.1, 128.3, 128.4, 128.5, 128.7, 135.1, 141.0, 147.4, 197.7. MS (EI, 70 eV): m/z 224 (M<sup>+</sup>, 44), 209 (12), 181 (8), 133 (12), 105 (8), 91 (100), 65 (7); Anal. Calcd for C<sub>16</sub>H<sub>16</sub>O: C, 85.68; H, 7.19. Found: C, 85.59; H, 7.31.

## Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

### 3. Discussion

The metal-catalyzed cross-coupling reaction between organometallic reagents with electrophiles has emerged as one of the most powerful methods for carbon-carbon bond formation.<sup>3</sup> The Suzuki-Miyaura<sup>4</sup> reaction using organoboron reagents is among the most important, due its tolerance of a broad range of functional groups as well as its formation of non-toxic byproducts. The use of alkylboron reagents has received special attention because of their utility in natural product synthesis.<sup>5</sup>

The coupling of alkylboron reagents has generally been performed with trialkylboranes ( $R_3B$ ), alkylborinate esters ( $R_2BOR'$ ), alkylboronic acids [ $RB(OH)_2$ ] or boronate esters [ $RB(OR')_2$ ]. Trialkylboranes such as 9-BBN<sup>6</sup> derivatives are among the most common, although they present problems such as functional group intolerance, air sensitivity, and a lack of atom economy. The use of alkylboronate esters often results in low yields in coupling reactions unless highly toxic thallium bases are used.<sup>7</sup> Additionally, these species are often hydrolytically unstable. The coupling of alkylboronic acids<sup>8</sup> has shown more promise, although their tendency to form cyclic anhydrides (boroxines) often makes determining their precise stoichiometry difficult.

Recently, potassium organotrifluoroborates (RBF<sub>3</sub>K) have been shown to be highly effective coupling partners in the Suzuki-Miyaura reaction.<sup>9</sup> They are easily prepared on large-scale (>200 g) by the addition of KHF<sub>2</sub> (an inexpensive fluoride source) to various organoboron reagents such as boronic acids, boronate esters, and organodihaloboranes. Their advantages over traditional organoboron reagents include their greater nucleophilicity, air stability, and atom economy. Initial reports described their use in the palladium-catalyzed coupling with arenediazonium<sup>10</sup> or diaryliodonium<sup>11</sup> salts. Previous reports from our laboratory have shown the successful cross-coupling of potassium alkyl-,<sup>12</sup> alkenyl-,<sup>13</sup> aryl-,<sup>14</sup> and alkynyltrifluoroborates<sup>15</sup> with aryl- and alkenyl halides as well as triflates using catalytic PdCl<sub>2</sub>(dppf)•CH<sub>2</sub>Cl<sub>2</sub> and Cs<sub>2</sub>CO<sub>3</sub> or Et<sub>3</sub>N as a base. In the case of aryltrifluoroborates, it was also shown that ligandless conditions using Pd(OAc)<sub>2</sub> and K<sub>2</sub>CO<sub>3</sub> can be used.<sup>16</sup> Batey and Quach have reported similar cross-coupling reactions using tetraalkylammonium trifluoroborates.<sup>17</sup> Recently, functionalized organotrifluoroborates such as epoxidized<sup>18</sup> and dihydroxylated<sup>19</sup> potassium alkyltrifluoroborates have been coupled to electrophiles under palladium catalysis. The procedure described above represents a typical coupling reaction in which a commercially available, easily-handled potassium alkyltrifluoroborate is coupled to a common aryl electrophile.

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## Appendix Chemical Abstracts Nomenclature; (Registry Number)

Trimethyl borate; (121-43-7)

Phenethylmagnesium chloride: Magnesium, chloro(2-phenylethyl)-; (90878-19-6)

Potassium hydrogen fluoride: (7789-29-9)

Potassium 2-phenethyltrifluoroborate: Borate(1-), trifluoro(2-phenylethyl)-, potassium, (T-4)-: (329976-74-1)

4-Bromoacetophenone: Ethanone, 1-(4-bromophenyl)-: (99-90-1)

Triphenylphosphine; (603-35-0)

Palladium acetate; (3375-31-3)

# 1-(4-Acetylphenyl)-2-phenylethane: 1-[4-(2-Phenylethyl)phenyl]-ethanone; (785-78-4)



Professor Gary Molander received his B.S. degree at Iowa State University in 1975, working with Professor Richard C. Larock. He obtained his Ph.D. degree in 1979 under the direction of Professor Herbert C. Brown. He joined Professor Barry Trost's group at the University of Wisconsin, Madison as a National Institutes of Health postdoctoral fellow in 1980, and in 1981 he accepted an appointment at the University of Colorado, Boulder. In 1999 he joined the faculty at the University of Pennsylvania. His research interests focus on the development of new synthetic methods for organic synthesis and natural product synthesis.



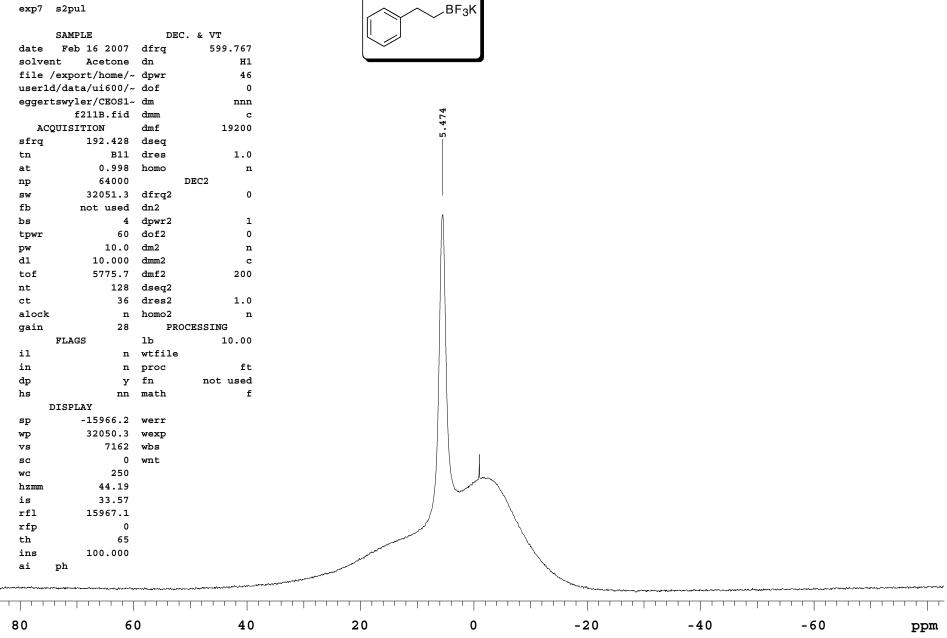
Daniel E. Petrillo received an A.B. in chemistry with honors from Princeton University in 1999. His senior thesis research was conducted in the laboratory of Professor Martin F. Semmelhack. From 1999 to 2002 he was employed as a chemist in the department of Process Research at Merck. He then entered graduate school at the University of Pennsylvania where his research has centered on synthetic methods using organotrifluoroborates under the guidance of Professor Gary A. Molander.



Christophe Eggertswyler was born and raised in Switzerland. He received his Diploma In Chemistry from the University of Fribourg, where he performed undergraduate research on the synthesis and ab initio computation of vibrational optical activity of sulfoxides. After graduation, he decided to stay in Fribourg where he joined the research group of Professor Titus A. Jenny. He investigated the synthetic potential of new iron tricarbonyl complexes in an organometallic approach to the taxane skeleton synthesis. After completion of his Ph.D. thesis in 2004, he joined the research group of Professor Scott E. Denmark at the University of Illinois at Urbana Champaign in 2005 as a postdoctoral research associate. His research focuses on the mechanistic study of the transmetallation event in crosscoupling reactions.



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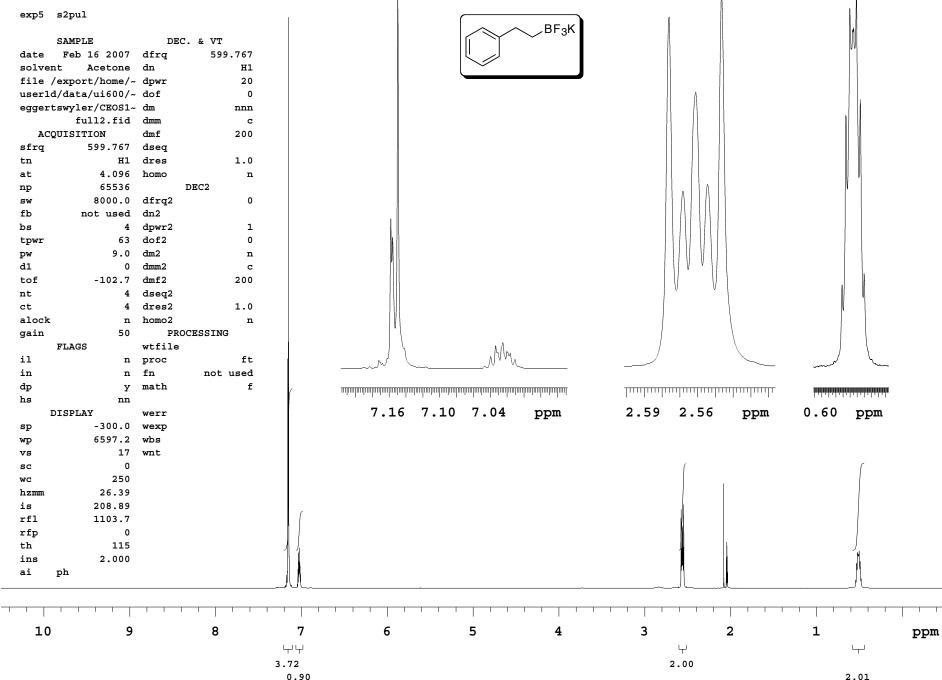
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fullc.fild dum w         AcQUISITION       dn f       19200         sfrq       150.825       dseq         in       Cli 3 dres       1.0         st       1.769       homo       n         app       131072       temp       25.0         ws       37037.0       DEC2         bb       not used       dfrq2       0         app       131072       temp       25.0         bb       not used       dfrq2       0         api       131072       temp       25.0         bb       not used       dfrq2       0         api       1.000       dma2       n         th       256       dmf2       10000         tt       256       dmf2       10000         tt       256       dmf2       10000         tt       256       dmf2       1.0         pin       n       tfile       n       n         pp       -754.6       n       ff       128.8       128.6       128.4       ppm         gr       37.8       ppm       37.8       ppm       37.8       ppm         rt1								
AcQUISITION       dmf       19200         ffrq       150.826       deeq         nn       013       dress       1.0         at       1.769       homo       n         ap       131072       temp       25.0         aw       37037.0       DEC2       DEC         bh       not used       dfrq2       0         bas       4       dn2       1         pw       7.0       dof2       0         bas       4       dn2       1         pw       7.0       dof2       0         bas       4       dn2       1         pw       7.0       dof2       0         alcok       n       dress2       1.0         gain       60       homo 2       n         fll       n       th       ft         pg       -754.6       pgm       3177.7         warr       33177.7       warr       3177.7         se       0       warp       se       128.8       128.6       128.4       ppm         37.8       ppm         rf1       14029.6       1       1       1			_					
frq       150.826       desq         in       0.13       dirs       1.0         at       1.769       homo       n         np       131072       temp       25.0         with       07037.0       DEC2         Eb       not used       dirq2       0         ps       4       da2         tpwr       58       dywr2       1         pv       7.0       dof2       0         at       1.000       mal       n         tof       2664.9       dmm2       c         th       256       dmf2       10000         tt       256       dmf2       10000         tt       256       dmf2       1.0         pain       60       homo2       n         pickis       procCessing       1       128.8       128.4       ppm       37.8         tin       n       ft       1402.6       1       128.8       128.4       ppm       37.8       ppm         sc       0       wsp       wsp </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>								
m       C13 dres       1.0         tt       1.769 homo       n         jp       131072 temp       25.0         w       37037.0       DEC2         bh       notused       dfrq2       0         se       4       da2       1         pyr       58       dpwr2       1         w       7.0       dof2       0         ll       1.000       dm2       n         of       2648.9       dm2       c         tt       256       dseq2       1.0         llock       n       dtec2       1.0         rain       60       nomo2       n         pISPLAY       math       f       o         pJSPLAY       math       f       o         pJ3177.7       werr       s       128.8       128.6       128.4       pp         3112       wexp       s       s       50.0       s       37.8       ppm         rf1       1602.6                   rfp       16162.4								
ti 1.769 komo n tp 131072 temp 25.0 tw 37037.0 DEC2 tb not used dfrq2 0 os 4 dn2 tpwr 58 dpwr2 1 tw 7.0 dof2 0 11 1.000 dm2 n cof 2648.9 dmm2 c tt 256 dmf2 10000 tt 256 dseq2 tlock n dres2 1.0 gain FLAGS PROCESSING tl n 1b 0.50 tm n fn not used DISPLAY math f tp 33177.7 verr rs 112 vexp tc 250 wt txumm 132.71 ts 500.00 rf1 14029.6 rfp 11612.4 th 6 100.000	-	-	1.0					
mm       37037.0       DEC2         hh       not used       dfr2       0         hy       not used       dfr2       0         ppwr       58       dpwr2       1         iii       1.000       dm2       n         icit       256       dmf2       10000         tt       256       dmf2       10000         tt       256       dmf2       1.0         pain       60       homo2       n         PLAGS       PROCESSING       128.8       128.6       128.4       ppm         051       120       wexp       128.8       128.6       128.4       ppm       37.8       ppm         ins       100.000	t 1.769		n					
bh       not used       dfrq2       0         ss       4       dn2         spwr       58       dpwr2       1         ww       7.0       dof2       0         ul       1.000       dma2       c         ucf       2648.9       dmm2       c         ut       256       deq2	p 131072	temp	25.0					
se       4       dn2         pyvr       58       dywr2       1         wr       7.0       dof2       0         ll       1.000       dm2       n         of       2648.9       dmm2       c         tt       255       dmf2       10000         tt       255       dseg2       10000         tt       255       dseg2       100         lock       n       db       0.50         n       n       b       0.50         n       n       b       0.50         n       n       not used       n         pp       -754.6       not used       128.8       128.6       128.4       ppm         rs       112       wexp       wexp       37.8       ppm         rc       250       wnt       wexp	w 37037.0		DEC2					
pwr       58       dywr2       1         w       7.0       dof2       0         1       1.000       dm2       n         of       2648.9       dmm2       c         t       256       dmf2       10000         t       256       dmf2       10000         t       256       dmf2       10000         t       256       dmf2       10000         ot       256       dmf2       10000         lock       n       dtes2       1.0         ain       60       bom2       n         r       PROCESSING       r       r         1       n       th       0.50         n       fn       not used       not used         DISPLAY       math       f         p       -754.6       r         p       -317.7       werr         s       112       wexp         c       0       whs         c       250       wnt         xmm       132.71       r         s       500.00       r         f1       14029.6         fn <t< td=""><td>b not used</td><td>dfrq2</td><td>0</td><td></td><td></td><td></td><td></td><td></td></t<>	b not used	dfrq2	0					
<pre>w 7.0 dof2 0 1 1.000 dm2 n of 2648.9 dm2 c t 256 dseq2 lock n dres2 1.0 ain 60 homo2 n FLAGS PROCESSING 1 n lb 0.50 n n wtfile p y proc ft s nn fn not used DISPLAY math f p -754.6 p 33177.7 werr s 112 wexp c 0 wbs c 250 wnt zmm 132.71 s 500.00 f1 14029.6 fp 11612.4 h 6 ns 100.00</pre>	s 4	dn2						
1       1.000       dm2       n         of       2648.9       dmm2       c         t       256       dmf2       10000         t       256       dmf2       10000         ti       256       dmf2       1.0         ain       60       homo2       n         plant       1       n       10       0.50         n       n       1       n       10       0.50         n       n       ff       not used       128.8       128.6       128.4       ppm         p       -754.6       -754.6       -754.6       -754.7       werp       37.8       ppm         c       0       wbs       -       -       37.8       ppm         c       0       wbs       -       -       -       -       -         c       0       wbs       -	pwr 58	dpwr2	1					
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rain       60       homo2       n         FLAGS       PROCESSING         1       n       1b       0.50         n       wtfile         p       y       proc       ft         nm       fn       not used       nummun         DISPLAY       math       f       128.8       128.6       128.4       ppm         rs       112       wexp       wexp       37.8       ppm         rc       250       wnt       min       128.8       128.4       ppm         rd       14029.6       intervention       intervention       intervention         ff       14029.6       intervention       intervention       intervention         ff       100.000       intervention       intervention       intervention	t 256	dseq2						
FLAGS       PROCESSING         .1       n       lb       0.50         .n       n       wtfile         up       y       proc       ft         DISPLAY       math       f         up       -754.6       128.8       128.4       ppm         y       33177.7       werr       37.8       ppm         rs       112       wexp       37.8       ppm         rc       250       wnt       weth       128.8       128.4       ppm         rs       112       wexp       weth       weth       14       14029.6       14       14029.6       14       14029.6       14       14029.6       14			1.0					
n1       n       lb       0.50         nn       n       wtfile         lp       y       proc       ft         ls       nn       fn       not used         DISPLAY       math       f       128.8       128.4       ppm       37.8       ppm         rs       112       wexp       wexp       37.8       ppm       37.8       ppm         rs       112       wexp       wexp <td></td> <td></td> <td></td> <td></td> <td></td> <td>/\</td> <td></td> <td></td>						/\		
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s       nn fn not used       128.8 128.6 128.4 ppm       37.8 ppm         p       -754.6       -754.6       -754.6       -754.6         p       33177.7 werr       -       -       -         s       112 wexp       -       -       -         c       0 wbs       -       -       -         c       250 wnt       -       -       -       -         s       500.00       -       -       -       -       -         fl       14029.6       -       <								
s nn fn not used DISPLAY math f p -754.6 p 33177.7 werr s 112 wexp c 0 wbs c 250 wnt zmm 132.71 s 500.00 f1 14029.6 fp 11612.4 h 6 ns 100.000		-				<del>,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,</del>	*****	արարարարարարություն
p       -754.6         p       33177.7 werr         s       112 wexp         c       0 wbs         c       250 wnt         zmm       132.71         s       500.00         fl       14029.6         fp       11612.4         h       6         ns       100.000								
p     33177.7 werr       s     112 wexp       c     0 wbs       c     250 wnt       zmm     132.71       s     500.00       f1     14029.6       fp     11612.4       h     6       ns     100.000		math	Í		]	128.8 12	128.4 ppm	37.8 ppm
s     112     wexp       c     0     wbs       c     250     wnt       zmm     132.71       s     500.00       f1     14029.6       fp     11612.4       h     6       ns     100.000								
c     0     wbs       c     250     wnt       zmm     132.71       s     500.00       f1     14029.6       fp     11612.4       h     6       ns     100.000	=							
c     250 wnt       zmm     132.71       s     500.00       f1     14029.6       fp     11612.4       h     6       ns     100.000		-						
zmm     132.71       s     500.00       fl     14029.6       fp     11612.4       h     6       ns     100.000								
s 500.00 fl 14029.6 fp 11612.4 h 6 ns 100.000								
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	1	180	160	140	120	100 80	·	40 20

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exp6 s2pul

	SAMPLE		C. & VT						
date	Feb 12 2007	dfrq	499.435		BF <sub>3</sub> K				
solvent	t Acetone	dn	H1						
file /e	export/home/~	dpwr	20						
user1d/	/data/vxr500~	dof	0						
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1 f	Eull219F.fid	dmm	с						
ACQI	JISITION	dmf	200						
sfrq	469.937	dseq							
tn	F19	dres	1.0						
at	0.328	homo	n						
np	131072		DEC2						
- sw	200000.0	dfrg2	0						
fb		dn2							
bs		dpwr2	1						
tpwr		dof2	0						
pw	7.5	dm2	n						
d1	1.000	dmm2	c						
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nt		dseq2	200					142.138	
ct		dres2	1.0						
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gain	not used		DCESSING						
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sp	-99690.6								
wp	199996.9								
vs	818								
SC		wnt							
WC	250								
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ى بىغانلى مەنبىلىشادىغى. بېيەندە بېرىمە يايەردۇغەن	الله المانية وما يكون المارية المراجعة المراجعة ومن من المراجعة المعالمة المعاقبة المعاقبة المعالمة المعالمة ا المحال المحالية المارية المواجعة المراجعة ومن المراجع المحال المراجع المحال المحالية المحالية المحالية المحالية	ا منظ <sup>ر</sup> استخلاف المارسة. مربق محجود والمحجود والمحجو	nen en sektelen men stad få pårt av stil den som som attense, som stad ståde atten attense en stad stad. Here som til stad attense for granne ståd som etter stad attense og som for stød på som etter stød stad attense	nen diten til efter her som ander det er til et er som blever at her bis er er som at her bis er er som at her Er er efter som er er forser er e	a le su d'a esta a ser a se la seconda se	en der bestehen der Allen eine Berneter eine Berneter eine Berneter als der Berneter Berneter Berneter Bernete Angeneren eine Berneter Berneter an der Berneter an der Berneter Berneter Berneter Berneter Berneter Berneter Be	المركز المركز المركز المركز	the next of the second s	الم المركز الم المركز المر المركز المركز
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