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for the Preparation
of Organic Compounds

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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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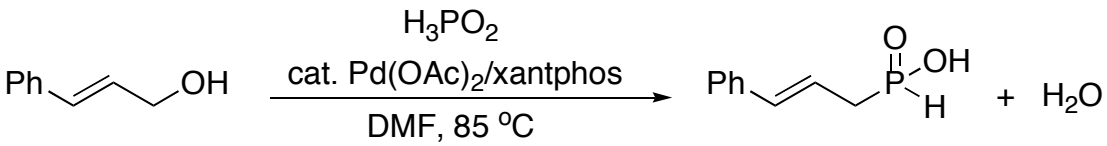
PALLADIUM-CATALYZED DEHYDRATIVE ALLYLATION OF HYPOPHOSPHOROUS ACID WITH ALLYLIC ALCOHOLS. PREPARATION OF CINNAMYL-*H*-PHOSPHINIC ACID



Submitted by Karla Bravo-Altamirano and Jean-Luc Montchamp.[1]
Checked by Alena Rudolph and Mark Lautens.

1. Procedure

A 1-L, round-bottomed flask (Note 1) equipped with a magnetic stirring bar is charged under air, with a solution of concentrated hypophosphorous acid (19.80 g, 300 mmol, 2.0 equiv) (Note 2) in *N*,*N*-dimethylformamide (300 mL, via a graduated cylinder) (Note 3). Mesitylene (20.9 mL, 18.03 g, 150 mmol, 1.0 equiv) (Note 4) and cinnamyl alcohol (19.7 mL, 20.57 g, 153 mmol, 1.0 equiv) (Note 5) are added via syringe (Note 6). The flask is then fitted with a rubber septum and placed on a magnetic stir plate. After stirring for 5 min, $Pd(OAc)_2$ (0.067 g, 0.300 mmol, 0.002 equiv) (Note 7), and 9,9-dimethyl-4,5-bis(diphenylphosphino)xanthene (0.190 g, 0.330 mmol, 0.0022 equiv) (Note 8) are added by temporarily removing the septum. Material adhering to the sides of the reaction flask is rinsed into the reaction mixture with 5 mL of *N*,*N*-dimethylformamide, resulting in a clear, brown solution. Stirring is maintained and the reaction flask is equipped with a Claisen adapter fitted with a reflux condenser with nitrogen inlet, and a thermocouple temperature probe adapter. Under a nitrogen atmosphere, the system is placed in a heating mantle filled with sand (Note 9) and the thermocouple is inserted through the adapter. The solution is heated at 85 °C (internal temperature) for 7 h (Notes 10, 11). Heating and stirring are then interrupted, and the resulting solution is allowed to cool to room temperature (Notes 12, 13). After removing the nitrogen inlet and the water condenser, the reaction mixture is concentrated for 1 h by rotary evaporation (50 °C, 0.5 mmHg). The residue is diluted with ethyl acetate (150 mL) and treated with activated charcoal (3.0 g) (Note 14). The resulting heterogeneous mixture is stirred

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for 30 min and filtered in vacuo through a Celite pad (Note 15) in a Büchner funnel. The Celite is carefully washed with three 100-mL portions of ethyl acetate (Note 16) and the combined washings are transferred to a 1-L separatory funnel. The organic layer is washed with aqueous HCl (2 M, 250 mL) (Note 17) and the aqueous phase is separated and extracted with two 125 mL portions of ethyl acetate. The combined organic layers are washed with 200 mL of brine, then are retreated with charcoal (1.0 g) and $MgSO_4$ (20 g) (Notes 18, 19), then are filtered through a second Celite pad in a Büchner funnel (Notes 15, 20), and concentrated under reduced pressure (45 °C, 150 mmHg). The resulting pale-yellow solid (around 26 g, 95% yield) (Note 21) is dissolved in 80 mL of hot dichloromethane (35–38 °C), and about 140 mL of hexane (38 °C) is added until a light-yellow, homogeneous solution is obtained. The solution is cooled at –15 °C for 2 h and the resulting white crystals (21.8 g) are collected by suction filtration on a Büchner funnel, and are washed with ice-cold hexane (100 mL). The filtrate is concentrated under reduced pressure (45 °C, 150 mmHg) and the residue is dissolved in 20 mL of dichloromethane (35 °C), and 30 mL of hexane (35 °C). The solution is cooled at –15 °C overnight and a second crop of crystals is collected by suction filtration, and are washed with ice-cold hexane (20 mL). The two crops of crystals are combined and dried overnight at 0.1 mmHg to provide 23.2 g of cinnamyl-*H*-phosphinic acid (83%) as white crystals (Note 22).

2. Notes

1. The success of the reaction does not depend on having previously dried the glassware, or on adding the reagents under a nitrogen atmosphere.

2. Aqueous hypophosphorous acid (50 wt.%) was purchased from Aldrich Chemical Company, Inc. and concentrated before reaction, according to the following procedure. The 1-L round-bottomed reaction flask was charged with 39.6 g of 50% aqueous hypophosphorous acid. The acid was concentrated for 30 min by rotary evaporation (40 °C, 0.3 mmHg).

3. Reagent grade *N*,*N*-dimethylformamide (≥99.8%) was purchased from Aldrich Chemical Company, Inc. and used as received.

4. Mesitylene (standard for GC, ≥99.8%) was purchased from Fluka and used as received. This reagent does not interfere with the reaction; it works only as internal standard for GC-monitoring of the reaction progress and can be omitted. The checkers omitted the use of mesitylene.

5. Cinnamyl alcohol (98%) was purchased from Aldrich Chemical Company, Inc. and used without further purification.

6. Due to the low melting point of cinnamyl alcohol (30–33 °C), the reagent was immersed in a water bath at 45 °C for 30 min before use to facilitate its addition via syringe. A preheated (45–50 °C) 20–mL glass syringe fitted with a short needle (50 mm) was used in order to avoid solidification of the reagent during the addition.

7. Palladium (II) acetate, min. 98% (99.9+%-Pd) was purchased from Strem Chemicals, Inc. and used as received.

8. 9,9-Dimethyl-4,5-bis(diphenylphosphino)xanthene (Xantphos) (97%) was purchased from Aldrich Chemical Company, Inc. and used as received. The checkers purchased Xantphos (min 98%) from Strem Chemicals, Inc. and used it as received.

9. The surface of the solution was below the sand level and good stirring was maintained along the process.

10. A J-KEM Scientific, Inc. temperature controller model 150 with a Teflon-coated thermocouple was used with the heating mantle. The thermocouple was placed inside the solution (1-2 inches) and the temperature was set to 85 °C. The reaction time was measured once the internal temperature of the solution was stabilized at 85±3 °C, which took about 20-30 min. The checkers used a mercury thermometer to monitor the internal temperature of the reaction.

11. The reaction was terminated when TLC analysis indicated that all the cinammyl alcohol was consumed. TLC was conducted using Merck silica gel 60 F-254 plates (elution with hexanes/ethyl acetate, 7:1; visualization by UV, and by immersion in anisaldehyde stain (by volume: 93% ethanol, 3.5% sulfuric acid, 1% acetic acid, and 2.5% anisaldehyde) followed by heating; R_f cinnamyl alcohol = 0.13, blue spot on anisaldehyde. The progress of the reaction was also monitored by gas chromatography. GC analysis was performed on a HP5890 Gas Chromatograph equipped with a HP5 capillary column (30-m x 0.32-mm x 0.25-μm) and FID detector, under the following conditions: flow(He) = 0.9 mL/min at 60 ºC, under constant pressure at 5 psi; inlet temp 200 °C; oven temp 60 °C, 1 min; ramp1 5 °C/min; final temp1: 160 °C, final time1: 0 min; then ramp2: 25 °C/min; final temp2: 280 °C, final time2: 20 min; detector 280 °C; split mode with constant make-up. For GC analysis, 3 drops of sample was diluted in 1 mL of diethyl ether. The solution was washed with 1 mL of saturated aqueous $NaHCO_3$ solution and 1-μL of the organic solution was injected into the GC;

t_R (cinnamyl alcohol), 18.72 min; t_R (mesitylene). 8.69 min. The checkers monitored the reaction by TLC only.

12. The submitters removed the sand bath and replaced it with a water bath. The checker removed the reaction mixture from the sand bath, continued stirring for 30 min and subsequently removed the solvent under reduced pressure as described in the procedure. The reaction mixture was not fully cooled to room temperature in a water bath prior to evaporation of the solvent.

13. One mL of the reaction mixture at room temperature was placed in an NMR tube for analysis. ^{31}P NMR (121.47 MHz, DMF) δ: 28.35, ~ 118% (dt, J_{HP} = 529 Hz, J = 19 Hz, Product), 4.70, ~ 48% (t, J_{HP} = 526 Hz, H_3PO_2), 3.02, ~ 34% (d, J_{HP} = 641 Hz, H_3PO_3). The ^{31}P NMR yields were determined by integration of all the resonances in the ^{31}P NMR spectra. The checkers did not perform this analysis.

14. Activated charcoal (Purum p.a.) was purchased from Fluka and used as received.

15. Celite 545 was purchased from Fischer Scientific Co. A slurry mixture of 40 g of Celite in ethyl acetate (about 50 mL) was placed in a Büchner funnel (7 cm diameter, 10-15 μ). The checkers used technical grade Celite from ACP Chemicals Inc.

16. A milky suspension was obtained with some dark, gel-like precipitate.

17. The submitters observed an emulsion with some black precipitate is formed at the interphase, which can be broken by using a stirring rod. Some black precipitate goes into the organic phase. The checkers did not observe the formation of an emulsion with black precipitate.

18. Magnesium sulfate (anhydrous, ≥97%) was purchased from Aldrich Chemical Company, Inc.

19. The mixture was stirred with activated charcoal (Note 15) for 15 min, then $MgSO_4$ was added and stirring was continued for another 15 min.

20. The Celite pad was washed with two 50-mL portions of ethyl acetate.

21. Cinnamyl-*H*-phosphinic acid was pure according to the melting point (84–85 °C), and NMR analysis (^{31}P, ^{1}H, ^{13}C). The material was recrystallized to remove any traces of Pd.

22. Full characterization of the product was as follows: mp 83–85 °C; ^{1}H NMR (400 MHz,$CDCl_3$) δ: 2.75 (dd, J_{HP} = 19.4, 7.6 Hz, 2 H), 6.04–6.13 (m, 1 H), 6.51 (dd, J = 15.8, 5.8 Hz, 1 H), 7.01 (d, J_{HP} = 558.7 Hz, 1

H), 7.20–7.35 (m, 5 H), 11.90 (bs, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 34.7 (d, J_{PC} = 90.9 Hz, CH_2), 116.8 (d, J_{PCC} = 10.1 Hz, CH), 126.5 (d, J_{PCCCCC} = 2.2 Hz, CH), 128.0 (CH), 128.8 (CH), 136.2 (d, J_{PCCC} = 14.6 Hz, CH), 136.7 (d, J_{PCCCC} = 4.1 Hz, C); ^{31}P NMR (161.82 MHz, $CDCl_3$) δ: 35.51 (d, J_{PH} = 557.9 Hz); IR (thin film, NaCl), cm^{-1}: 2619 and 1682 (P-O-H); 2422, 2326 and 2177 (P-H); 1217 (P=O); 970 and 728; UV (EtOH, c≈8 μM) λ_{max} = 255 nm; MS *m*/*z* (relative intensity): 182 (28), 118 (19), 117 (100), 116 (13), 115 (41), 91 (19). HRMS (EI) *m/z* Calcd for $C_9H_{11}O_2P$: 182.0497. Found: 182.0497. Anal. Calcd. for $C_9H_{11}O_2P$: C, 59.34; H, 6.09. Found: C, 58.89; H, 6.10. Analysis by Reverse Phase Ion-Pairing HPLC:[2] t_R 1.43 min. Agilent Zorbax® Eclipse XDB-C8 column (4.6 x 150 mm, 5μm) with a guard column (Agilent Zorbax® ODS, 4.6 x 12.5 mm, 5μm), 1 mL/min flow (isocratic), using as mobile phase a buffer (5 mM hexadecyl-trimethylammonium bromide, 50 mM ammonium acetate, and 2% MeOH. pH 4.85, adjusted with acetic acid). Injection volume: 5 μL, C≈0.24 mg/mL (EtOH). The checkers did not verify the HPLC data.

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

Palladium-catalyzed dehydrative substitutions of allylic alcohols with carbon, oxygen, nitrogen, and sulfur nucleophiles have started to emerge as efficient and atom-economical processes in organic synthesis.[3,4] These reactions directly use allylic alcohols instead of activated esters or halides, which are prepared from the corresponding allylic alcohols. Though attractive, this type of allylic substitution is generally slow, and often requires the addition of catalytic or stoichiometric amounts of activating agents.[3] Metal-catalyzed C-P bond formation through allylation processes are by far less developed and just a couple of examples involving the use of allylic acetates or carbonates as electrophiles, in presence of stoichiometric amounts of base or silylating agent, are known.[5] This approach is not practical due to the formation (P(III) species) and/or manipulation of air sensitive compounds (nickel(0) catalysts). Hypophosphorous compounds

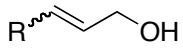
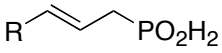
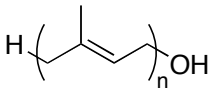
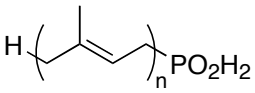
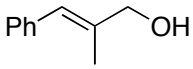
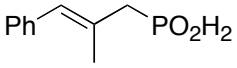
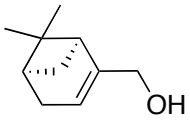
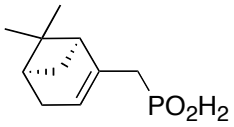
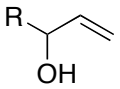
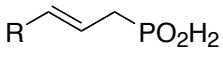
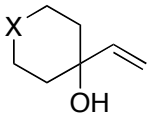
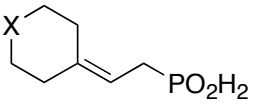
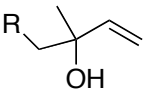
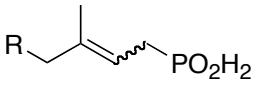
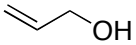
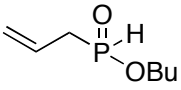
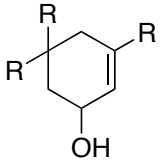
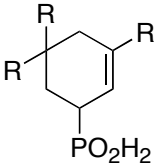
($ROP(O)H_2$) exist in a tautomeric equilibrium between the P(V) and P(III) forms due to the presence of labile hydrogen atoms. Under the influence of transition metals they are known to undergo transfer hydrogenation reactions, but their reactivity can be harnessed through adequate selection of catalysts.[6] The challenge to overcome the reductive pathway in palladium-catalyzed allylation reactions was initially addressed by the development of a cross-coupling reaction of allylic halides, acetates, benzoates and carbonates with amine salts of esters of hypophosphorous acid.[7,8] Subsequently, a palladium-catalyzed dehydrative allylation of H_3PO_2 with allylic alcohols *in the absence of any additives* was successfully achieved.[9] The present procedure describes a sound and environmentally friendly approach for the preparation of allylic-*H*-phosphinic acids, having water as byproduct (Table 1). In most cases, primary *H*-phosphinic acids can be isolated in moderate to good yields after a simple extractive workup (>95% purity). Since substrates that possess a terminal double bond and secondary or tertiary alcohols in the allylic position undergo rearrangement to form a primary C-P bond, the reaction is considered to proceed via π-allylpalladium intermediates. When using low molecular weight allylic alcohols (3-4 carbons), an *in situ* esterification with alkoxysilanes to the corresponding *H*-phosphinate esters improves the yield significantly. The reaction is highly selective towards formation of the *E*-isomers. Secondary allylic alcohols react successfully in this reaction, although they require more concentrated conditions and slightly higher catalyst loading. The reaction is water and air tolerant, and requires as little as 0.2 mol% of Pd (Pd_2dba_3, $Pd(OAc)_2$ or $PdCl_2$), with Xantphos as a ligand. *N*,*N*-Dimethylformamide (85 °C) is required as a solvent. Good yields of products are obtained even with equimolar amounts of H_3PO_2 and allylic alcohols, but 2 equivalents of H_3PO_2 appear to improve the yield.

Allylic *H*-phosphinic acids have been prepared previously from the reaction of an allylic halide with $(TMSO)_2PH$.[10] However, this method requires wasteful silylation, a halide-containing electrophile, and it is difficult to prevent the formation of symmetrically disubstituted products.[11]

Another synthetic approach to allylic-*H*-phosphinate esters is a base-promoted direct alkylation of alkyl phosphinates,[12] or a Michaelis-Becker reaction of masked hypophosphorous synthons.[13]

This novel catalytic phosphorous-carbon bond formation reaction is a powerful and atom-economical entry into allylic organophosphorus compounds from readily available allylic alcohols.

Table 1. Scope of the Pd-Catalyzed Allylation[a]

Entry	Alcohol	*H*-phosphinic acid	isolated yield %
1a	R = *E*-Pr		62
1b	R = *E*-Pr		92
1c	 R = Me		50 (88)[d]
1d	R = CH_2OBn		67[d]
1e	R = CO_2Et		77[d]
2a	R = *E*-Pr		52
2b	 R = *E*-Pr		100
2c	R = Me		86
3			68
4			74
5a	R = Et		92
5b	 R = *p*-FC_6H_4		96
6a	R = CH_2		78
6b	 R =NCO_2Et		73
7a	R = H		82
7b	 R = prenyl		93[c]
7c	R = geranyl		98[c]
8			43[c,d]
9a	 R = H		45[d,e]
9b	R = Me		52[e]

a See Reference 9 for details. Reactions were conducted in DMF over 4Å sieves (0.2 M) at 85 °C, with 0.5 mol% Pd/xantphos, and n equiv H_3PO_2. n = 1, entries 1a-c, 2-3, 7; n = 2, entries 4-6; n = 2.5, entries 1d-e; n = 3, entries 8-9. Reaction times: 2-8 h. b Isolated yield after extractive work-up. c 1:1 mixture of isomers. d After esterification and chromatographic purification. e Conditions: 2 mol% Pd/xantphos, 3 equiv H_3PO_2, 85 °C, DMF (2 M).

1. Department of Chemistry, Texas Christian University, Fort Worth, TX 76129. Financial support by the National Science Foundation (CHE-0242898) is gratefully acknowledged. K.B.A. was supported in part by a Texas Christian University Graduate Fellowship.

2. Richardson, D.; Sadi, B. B. M.; Caruso, J. A. in *Ultra-Trace Analysis of Organophosphorus Chemical Warfare Agent Degradation Products by HPLC-ICP-MS.* Agilent Technologies, Online Literature at www.agilent.com/chem.

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Appendix
Chemical Abstracts Nomenclature; (Registry Number)

Hypophosphorous acid: Phosphinic acid; (6303-21-5)

Cinnamyl alcohol: 3-Phenyl-2-propen-1-ol; (104-54-1)

Palladium(II) acetate: $Pd(OAc)_2$; (3375-31-3)

9,9-Dimethyl-4,5-bis(diphenylphosphino)xanthene: Xantphos; (161265-03-8)

Cinnamyl-H-phosphinic acid: [(2*E*)-3-phenyl-2-propenyl]-Phosphinic acid; (911128-46-6)

Jean-Luc Montchamp was born in Lyon, France. He completed his undergraduate studies at the Ecole Superieure de Chimie Industrielle de Lyon (ESCIL), now known as CPE. He obtained his Ph.D. from Purdue University in 1992, under the direction of Professor John W. Frost. After postdoctoral experiences at Michigan State University and at the Scripps Research Institute, he returned to Purdue University for a postdoctoral stay with Professor Ei-ichi Negishi. He became Assistant Professor at Texas Christian University in 1998, and was promoted to Associate Professor in 2004. His research interests include the development of methodology for phosphorus-carbon bond formation, especially using hypophosphorous derivatives and the medicinal chemistry of phosphorus-containing analogs of natural products.

Karla Bravo-Altamirano was born in 1979, in Oaxaca, Mexico. She received a B.S. degree in Chemistry in 2002 from Universidad de las Américas Puebla, Mexico, where she conducted research for the group of Prof. Cecilia Anaya Berrios. She is currently pursuing graduate studies at Texas Christian University, under the guidance of Prof. Jean-Luc Montchamp. Her research focuses on the development of new methodologies for the synthesis of *H*-phosphinic acid derivatives and their *P*-chiral counterparts.

Alena Rudolph was born in Ottawa, Canada in 1978. She received her Bachelor of Science degree (Honors, Cooperative) in Chemistry from the University of Waterloo in 2002, where she conducted research in the group of Prof. Eric Fillion. After working in the department of Medicinal Chemistry at Abbott Bioresearch Center in Worcester, MA, she returned to Canada and is currently pursuing her Ph.D. under the supervision of Prof. Mark Lautens at the University of Toronto. Her research is currently focused on the development of tandem processes to generate functionalized heterocycles via a C–H activation pathway.

ar-09-67-xtal-13C

Data Collected on:
pompompurin-mercury300
Archive directory:

Sample directory:

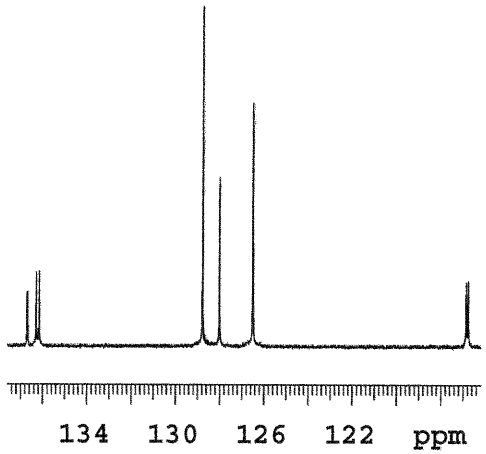
File: 20070831-ar0967xtal1_Carbon-002

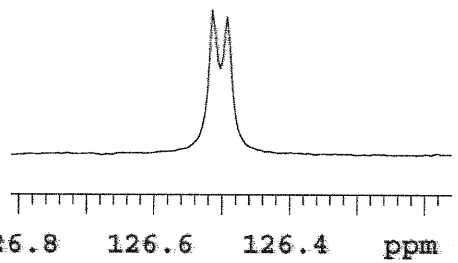
Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Operator: arudolph

Relax. delay 0.200 sec
Pulse 45.0 degrees
Acq. time 1.337 sec
Width 24509.8 Hz
704 repetitions
OBSERVE C13, 100.5167045 MHz
DECOUPLE H1, 399.7500357 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 35 min

INDEX	FREQUENCY	PPM	HEIGHT
1	13741.882	136.712	12.5
2	13737.768	136.671	12.6
3	13699.621	136.292	17.0
4	13685.036	136.147	17.3
5	12943.414	128.769	78.6
6	12867.494	128.013	39.1
7	12716.776	126.514	56.3
8	12714.532	126.492	53.8
9	11746.272	116.859	14.9
10	11736.174	116.758	15.1
11	7795.068	77.550	34.0
12	7762.905	77.230	35.3
13	7731.116	76.914	34.8
14	3534.575	35.164	13.5
15	3443.695	34.260	13.8





ar-09-67-xtal

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pompompurin-mercury300
Archive directory:

Sample directory:

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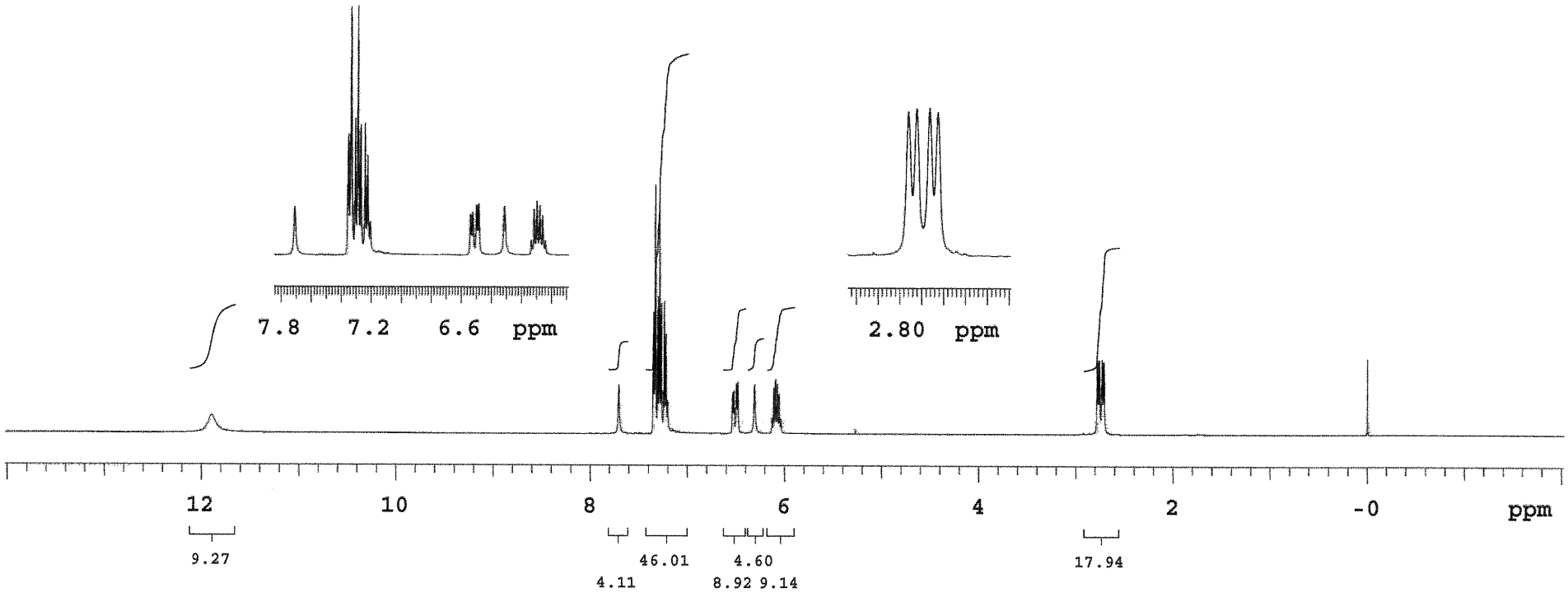
Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Operator: arudolph

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7480375 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min

INDEX	FREQUENCY	PPM	HEIGHT
1	4755.852	11.897	2.8
2	3081.493	7.709	7.6
3	2938.295	7.350	19.1
4	2931.057	7.332	39.1
5	2920.102	7.305	21.5
6	2913.059	7.287	39.3
7	2905.430	7.268	20.5
8	2894.279	7.240	20.8
9	2887.041	7.222	15.6
10	2879.803	7.204	5.2
11	2612.970	6.537	6.4
12	2607.297	6.522	6.7
13	2597.124	6.497	7.9
14	2591.451	6.483	8.1
15	2522.786	6.311	7.7
16	2451.383	6.132	2.4
17	2443.949	6.114	7.2
18	2436.320	6.095	8.6
19	2428.299	6.075	7.8
20	2420.670	6.055	6.3
21	2413.236	6.037	2.3
22	1111.348	2.780	11.4
23	1103.719	2.761	11.7
24	1091.982	2.732	11.7
25	1084.352	2.713	11.4
26	-0.000	-0.000	11.9



ar-09-67-xtal-31P

INDEX	FREQUENCY	PPM	HEIGHT
1	5728.371	35.399	85.7

Data Collected on:
pompompurin-mercury300
Archive directory:

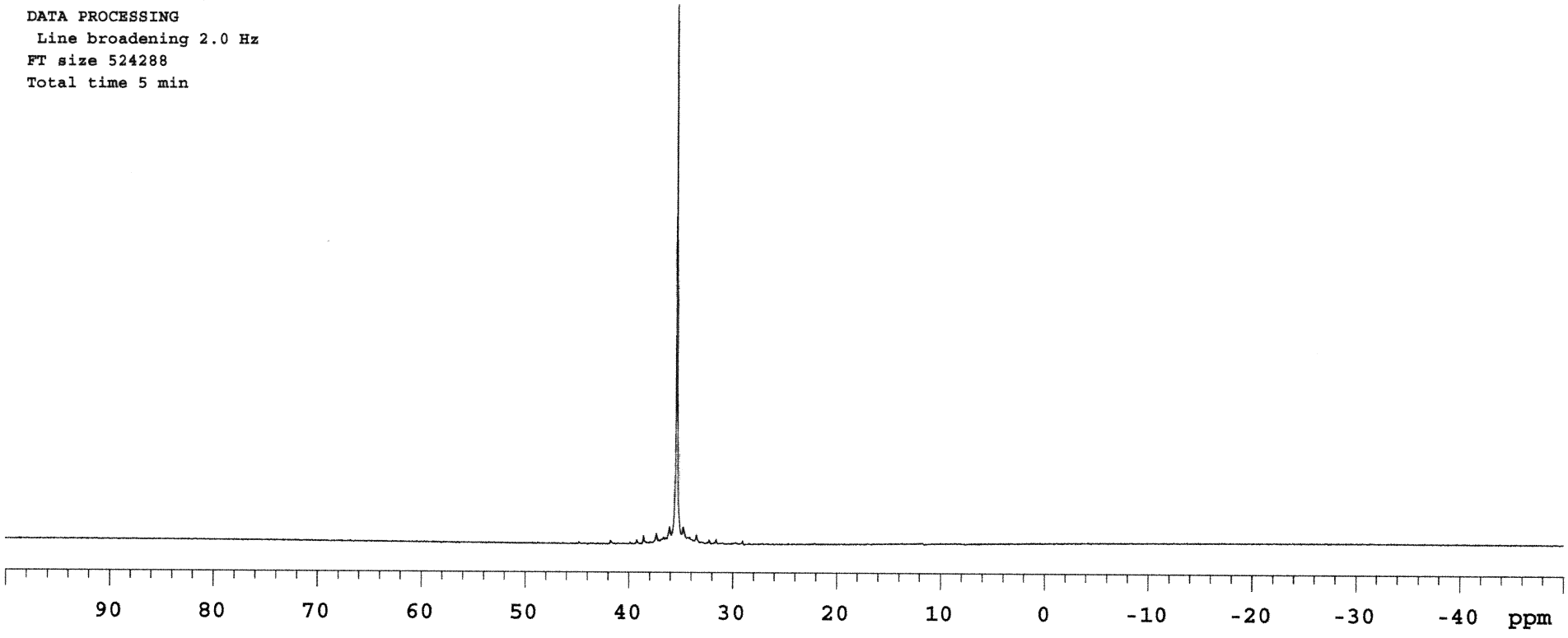
Sample directory:

File: 20070831-ar0967xtal1_Phosphorus-001

Pulse Sequence: s2pul
Solvent: cdcl3

Temp. 25.0 C / 298.1 K
Operator: arudolph

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.258 sec
Width 104.2 kHz
64 repetitions
OBSERVE P31, 161.8207907 MHz
DECOUPLE H1, 399.7500357 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 524288
Total time 5 min



ar-09-67-coupled 31P

INDEX	FREQUENCY	PPM	HEIGHT
1	6024.408	37.229	105.5
2	5466.508	33.781	108.3

Automation directory: /export/home/arudolph/vnmrsys/data/auto_2007.09.19
File : /export/home/arudolph/vnmrsys/data/20070919-ar0967_phos_coupled_Phosphorus-001.fid
Sample id : s_20070919_01
Sample : ar0967_phos_coupled

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: arudolph
File: 20070919-ar0967_phos_coupled_Phosphorus-001
VNMRS-400 "vnmr400"

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 1.258 sec
Width 104.2 kHz
256 repetitions
OBSERVE P31, 161.8207907 MHz
DECOUPLE H1, 399.7500357 MHz
Power 40 dB
off during acquisition
on during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 524288
Total time 5 min, 49 sec