A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

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.

Organic Syntheses

A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

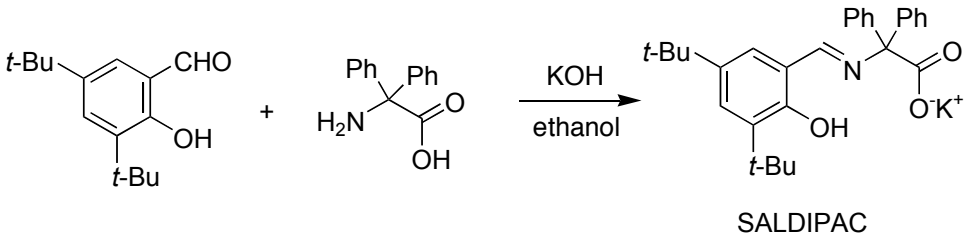
Working with Peroxy Compounds

Caution! Reactions and subsequent operations involving peracids and peroxy compounds should be run behind a safety shield. Peroxy compounds should be added to the organic material, never the reverse. For relatively fast reactions, the rate of addition of the peroxy compound should be slow enough so that it reacts rapidly and no significant unreacted excess is allowed to build up. The reaction mixture should be stirred efficiently while the peroxy compound is being added, and cooling should generally be provided since many reactions of peroxy compounds are exothermic. New or unfamiliar reactions, particularly those run at elevated temperatures, should be run first on a small scale. Reaction products should never be recovered from the final reaction mixture by distillation until all residual active oxygen compounds (including unreacted peroxy compounds) have been destroyed. Decomposition of active oxygen compounds may be accomplished by the procedure described in Korach, M.; Nielsen, D. R.; Rideout, W. H. Org. Synth. 1962, 42, 50 (Org. Synth. 1973, Coll. Vol. 5, 414). [Note added April 2018].

DOI:10.15227/orgsyn.087.0088

SYNTHESIS OF (3-CHLOROBUTYL)BENZENE BY THE COBALT-CATALYZED HYDROCHLORINATION OF 4-PHENYL-1-BUTENE

A.



SALDIPAC

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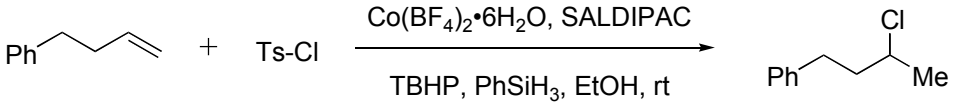

Submitted by Boris Gaspar, Jerome Waser, and Erick M. Carreira.[1]
Checked by David Hughes.

1. Procedure

In-situ preparation of 2-(3,5-di-tert-butyl-2-hydroxybenzylideneamino)-2,2-diphenylacetic acid potassium salt (SALDIPAC). A 500-mL, three-necked, round-bottomed flask is equipped with a rubber septum pierced with a thermocouple thermometer probe (Note 1), a gas adapter connected to a nitrogen line and oil bubbler, and a ground-glass stopper. A 3-cm Teflon-coated oval stir bar is added to the flask. The flask is charged with α,α-diphenylglycine (0.94 g, 4.1 mmol) and absolute ethanol (40 mL) (Note 2). The suspension is stirred at 22 °C and 0.5 M KOH in ethanol (10.0 mL, 5.0 mmol) is added (Note 3). After stirring for 10 min, most of the solids are dissolved. 3,5-Di-*t*-butyl-2-hydroxybenzaldehyde (1.00 g, 4.27 mmol) is added, forming a bright yellow solution. The solution is stirred at 22 °C for 16 h to provide 2-(3,5-di-*tert*-butyl-2-hydroxybenzylideneamino)-2,2-diphenylacetic acid potassium salt as a solution in ethanol (Note 4).

(3-Chlorobutyl)benzene. To the ligand solution prepared above is added absolute ethanol (140 mL) and $Co(BF_4)_2•6H_2O$ (1.40 g, 4.11 mmol, 8 mol%). The resulting dark red-brown solution is stirred at 22 °C for 10 min. To the vigorously stirred mixture (Note 5) is added 4-phenyl-1-butene (6.69 g, 50.6 mmol, 1.00 equiv) in one portion by syringe followed

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by *p*-toluenesulfonyl chloride (11.80 g, 61.89 mmol, 1.2 equiv). Then *t*-butyl hydroperoxide (2.8 mL, 14-17 mmol, 0.3 equiv) is added, followed by phenylsilane ($PhSiH_3$) (6.23 g, 57.6 mmol, 1.1 equiv); both are added by syringe in one portion. The temperature slowly rises to 38 °C over 10 min as the color changes to dark green and weak gas evolution occurs (Notes 6 and 7). The reaction mixture slowly cools to 22 °C over one h and is vigorously stirred at 22 °C for an additional 3 h (Note 8). The mixture is then transferred into a 1-L flask and the solvent is removed under reduced pressure (20 mmHg, 40 °C bath temperature) by rotary evaporation (Notes 9 and 10) to afford 30 g of a blue-green gum. Hexanes (200 mL) are added and the mixture is sonicated (Note 11) for 5 min to form a suspension, which is then filtered through a pad of Celite (50 g) in a 350-mL medium-porosity sintered glass funnel. The flask and the Celite pad are washed with hexanes (2 x 100 mL) and the combined filtrate is concentrated by rotary evaporation (20 mmHg, 40 °C bath temperature) to afford 17.5 g of crude product. The crude product is purified by chromatography on SiO_2 (Note 12) to afford (3-chlorobutyl)benzene (7.16 g, 84 %) as a colorless oil (Notes 13 and 14).

2. Notes

1. The internal temperature is monitored using a J-Kem Gemini digital thermometer with a Teflon-coated T-Type thermocouple probe (12-inch length, 1/8 inch outer diameter, temperature range -200 to +250 °C)

2. The following reagents and solvents used in this preparation were obtained from Sigma-Aldrich and used without further purification: 3,5-di-*t*-butyl-2-hydroxybenzaldehyde (99%), 4-phenyl-1-butene (99%), cobalt(II) tetrafluoroborate hexahydrate (99%), *p*-toluenesulfonyl chloride (99%), *t*-butyl hydroperoxide (5 – 6 M in decane), hexanes (ACS reagent, >98.5%), dichloromethane (ACS reagent, >99.5%), Celite 545, and silica gel (200-400 mesh, 60 Å). The following reagents were obtained from Acros and used without further purification: α,α-diphenylglylcine (98%), KOH (powdered, >85%) and phenylsilane (97%). Absolute ethanol was obtained from Pharmaco. Deionized tap water was used throughout the procedure.

3. A 0.5 M solution of KOH in ethanol is prepared by adding 3.2 g of 85% powdered KOH to 100 mL of absolute ethanol and sonicating for 5 minutes. (Note 11) The resulting hazy solution is allowed to settle overnight

to provide a clear solution with residual powdered solids in the bottom of the flask.

4. The reaction was monitored by ^{1}H NMR as follows. An approx. 0.05 mL aliquot of the reaction mixture was diluted with CD_3OD for NMR analysis. The resonances at 9.9, 7.7, and 7.6 ppm of the salicylaldehyde were readily observable and were integrated vs the imine resonances at 8.1 and 6.9 ppm. The reaction typically proceeded to 90% conversion of the aldehyde.

5. The mixture is stirred at 500 rpm on a magnetic stirring plate throughout the reaction.

6. For larger scale preparations, external cooling is recommended.

7. Slow bubbling started after *t*-butyl hydroperoxide addition and was most probably hydrogen gas. Hydrogen chloride can be excluded as a wet Riedel-de-Haen pH paper placed in the neck of the flask gave a negative test for acid.

8. The reaction was monitored by TLC on Merck silica gel 60 F_{254} TLC glass plates and visualized with UV light and permanganate stain. The R_f values in hexane:CH_2Cl_2 (7:1) are 0.65 for 4-phenylbutene and 0.56 for (3-chlorobutyl)benzene. The reaction was also followed using ^{1}H NMR as follows: One drop of the reaction mixture was added to 1 mL of $CDCl_3$, then filtered through a plug of Celite. The multiplet at 6 ppm of the starting material was monitored to assess reaction completeness vs. the doublet at 1.6 ppm of the product. *p*-TsCl (doublet at 7.9 ppm) and phenylsilane (singlet at 4.2 ppm) could also be monitored. The reaction was >95% complete within the first hour. More concentrated samples caused line broadening.

9. Solids in the flask require slow lowering of the pressure during the concentration to prevent bumping.

10. TLC and ^{1}H NMR analysis of the ethanol distillate from the concentration indicated the presence of a small amount of product. In one run, the ethanol distillate was added to 200 mL of water then extracted with 200 mL of hexanes. The hexanes extract was washed with water (2 x 200 mL), then concentrated to 1.2 g which was purified by chromatography on 15 g of SiO_2 using 7:1 hexanes: dichloromethane to afford 0.30 g of product (3.5% yield).

11. Sonication is carried out using a Fisher Scientific Ultrasonic Cleaner, Model FS20, having a capacity of 2.8L and power of 143 watts.

12. Chromatography conditions: 270 g of SiO_2 packed and eluted with hexanes:CH_2Cl_2 (7:1), column diameter 5 cm, fraction volume 40 mL. After loading the crude product on the column, 150 mL of eluent is collected before fractions are collected. The product appears in fractions 13-23.

13. The physical and spectroscopic properties of (3-chlorobutyl)benzene are as follows: IR (film) 3064 (w), 3028 (w), 2971 (w), 2927 (w), 2863 (w), 2361 (w), 1604 (w), 1496 (w), 1454 (m), 1379 (w), 1275 (w), 1117 (w), 1030 (w), 820 (w), 748 (m), 699 (s), 612 (w), 574 (w), 506 (w), 454 (w) cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ: 1.55 (d, 3 H, *J*=6.5 Hz), 2.01–2.08 (m, 2 H), 2.73–2.91 (m, 2 H), 3.97–4.06 (m, 1 H), 7.20–7.33 (m, 5 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 25.6, 33.1, 42.1, 58.1, 126.3, 128.68, 128.72, 141.3; HRMS (EI) *m/z* calcd. for $C_{10}H_{13}Cl$ $[M]^+$ 168.0700; found 168.0700; Anal. calcd. for $C_{10}H_{13}Cl$: C, 71.21, H, 7.77; found: C, 70.91, H, 7.68.

14. An analytically pure sample was prepared by dissolving 200 mg of the chromatographed product in 5 mL of pentane, filtering the solution through a 0.45 micron Teflon filter, and concentrating to dryness by rotary evaporation, then further removing residual solvent under vacuum at room temperature (20 mm Hg) for 3 h.

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

Olefins are inexpensive and readily available starting materials for organic synthesis. For this reason, the direct heterofunctionalization of the C=C has been of interest for many years, especially in a regioselective manner.[2] Among these, the hydrochlorination reaction belongs to one of the first fundamental reactions discussed in introductory organic chemistry. However, the process is very limited in scope, as the addition at useful rates occurs only to highly substituted or strained olefins[3] and to styrene-like substrates.[4] Lewis acid or surface mediated reactions of HCl were reported for simple olefins such as cyclohexene and cycloheptene.[5] In attempts to

avoid strongly acidic conditions different precursors were recognized to form HCl in small amounts *in situ*, however these methods are still limited to polysubstituted or activated alkenes and acid sensitive functional groups are not tolerated.[6]

The cobalt-catalyzed hydrochlorination described above is applicable to a range of unactivated alkenes and tolerates a variety of functional groups.[7] Importantly, the reaction displays complete Markovnikov selectivity and operates under very mild conditions (EtOH as solvent, room temperature). Furthermore, all the reaction components are commercially available. In a broader sense, the role of *p*-TsCl as a Cl-transfer reagent is intriguing and may have additional applications in other processes.[8,9]

1. Laboratory of Organic Chemistry, ETH Zürich, Wolfgang-Pauli-Strasse 10, 8093, Zürich.

2. Beller, M.; Seayad, J.; Tillack, A.; Jiao, H. *Angew. Chem. Int. Ed.* **2004**, *43*, 3368-3398.

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8. For a recent example of Pd-catalyzed chlorination with TsCl see: Zhao, X.; Dimitrijevic, E.; Dong V. M. *J. Am. Chem. Soc.* **2009**, *131*, 3466–3467.

9. For the use of related cobalt catalysts in a wide range of other olefin functionalization reactions, see: (a) Waser, J.; Carreira, E. M. *J. Am. Chem. Soc*. **2004**, *126*, 5676–5677; (b) Waser, J.; Carreira, E. M. *Angew. Chem. Int. Ed*. **2004**, *43*, 4099–4102; (c) Waser, J.; Nambu, H.; Carreira, E. M. *J. Am. Chem. Soc*. **2005**, *127*, 8294–8295; (d) Waser, J.; González-Gómez, J. C.; Nambu, H.; Huber, P.; Carreira, E. M. *Org. Lett*. **2005**, *7*, 4249–4252; (e) Waser, J.; Gaspar, B.; Nambu, H.; Carreira, E. M. *J. Am. Chem. Soc*. **2006**, *128*, 11693–11712; (f) Gaspar, B.; Waser, J.; Carreira, E. M. *Synthesis* **2007**, *24*, 3839–3845.

Appendix
Chemical Abstracts Nomenclature; (Registry Number)

(3-Chlorobutyl)benzene; (4830-94-8)

2-(3,5-Di-*tert*-butyl-2-hydroxybenzylideneamino)-2,2-diphenylacetic acid potassium salt, (SALDIPAC); (858344-69-1)

α,α-Diphenylglycine: Benzeneacetic acid, α-amino-α-phenyl-; (3060-50-2)

3,5-Di-*t*-butyl-2-hydroxybenzaldehyde: Benzaldehyde, 3,5-bis(1,1-dimethylethyl)-2-hydroxy-; (37942-07-7)

Cobalt(II) tetrafluoroborate hexahydrate; (15684-35-2)

p-Toluenesulfonyl chloride: Benzenesulfonyl chloride, 4-methyl-; (98-59-9)

t-Butyl hydroperoxide: Hydroperoxide, 1,1-dimethylethyl; (75-91-2)

Phenylsilane: Benzene, silyl-; (694-53-1)

4-Phenyl-1-butene: Benzene, 3-buten-1-yl-; (768-56-69)

Prof. Erick M. Carreira obtained a B.S. degree in 1984 from the University of Illinois at Urbana-Champaign and a Ph.D. degree in 1990 from Harvard University. After carrying out postdoctoral work with Peter Dervan at the California Institute of Technology through late 1992, he joined the faculty at the same institution as an assistant professor of chemistry and subsequently was promoted to the rank of full professor. Since September 1998, he has been professor of chemistry at the ETH Zürich. Most recently, he is the recipient of the Tetrahedron Chair Award, Thieme Prize, the Springer Award, American Chemical Society Award in Pure Chemistry, Nobel Laureate Signature Award, Young Investigator Awards from Merck, Novartis, Pfizer, Eli Lilly, as well as Astra Zeneca, and a recipient of the David and Lucile Packard foundation Fellowship in Science and Engineering.

Boris Gaspar was born in 1982 in Nove Zamky, Slovakia. He completed his undergraduate studies in chemistry at the Comenius University in Bratislava while working in the group of Associate Professor M. Salisova. During his undergraduate studies, he also worked with Professor A. Solladiè-Cavallo, (Université Louis Pasteur, Strasbourg, France) as a Socrates-Erasmus exchange fellow and carried out an internship at Syngenta (Basel, Switzerland). He recently completed his Ph.D. studies at the ETH Zürich in the group of Professor E. M. Carreira where he was involved in the development of metal-catalyzed functionalizations of olefins.

Jérôme Waser was born in Sierre, Valais, Switzerland in 1977. He studied chemistry at ETH Zurich and obtained his Diploma in 2001. In 2002, he started his Ph.D. studies at ETH Zurich with Prof. Erick M. Carreira**,** working on the development of metal-catalyzed amination reactions of olefins. In 2006, he joined Prof. Barry M. Trost at Stanford University and accomplished the total synthesis of Pseudolaric Acid B, a diterpene natural product. Since October 2007, he is working as tenure-track assistant professor at EPF Lausanne, focusing on the development and application of catalytic methods for the synthesis of bioactive compounds.

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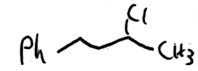

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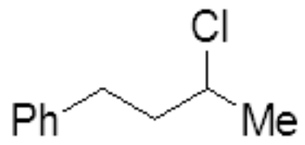

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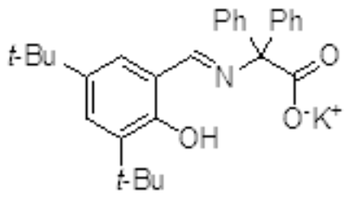

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5	7.2709	2907.4148	1.99
6	7.2366	2893.6993	7.63
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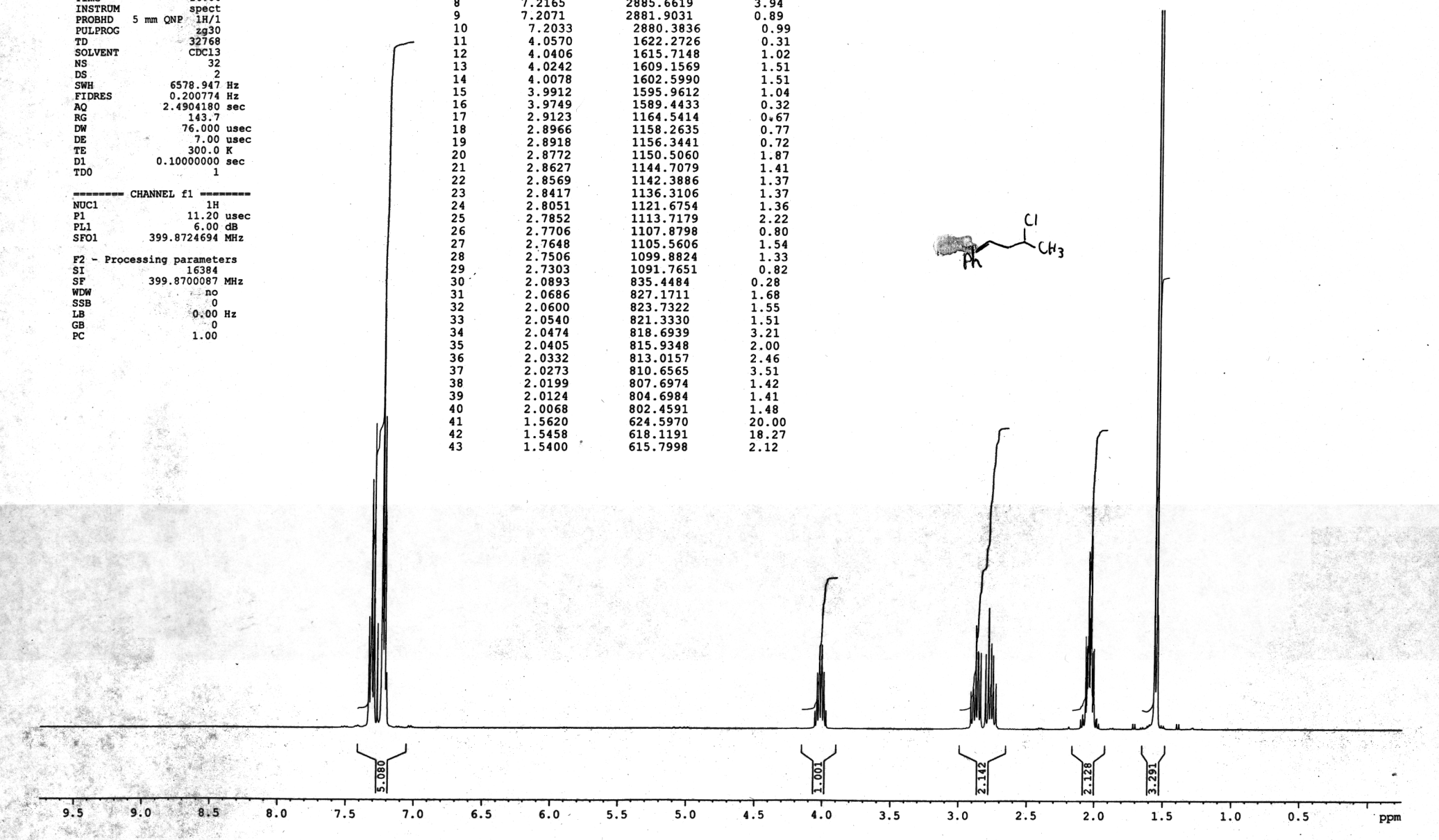
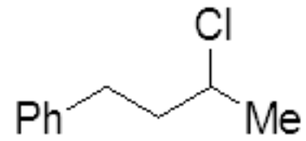
36	2.0332	813.0157	2.46
37	2.0273	810.6565	3.51
38	2.0199	807.6974	1.42
39	2.0124	804.6984	1.41
40	2.0068	802.4591	1.48
41	1.5620	624.5970	20.00
42	1.5458	618.1191	18.27
43	1.5400	615.7998	2.12



2009-084
chromatographed
nmr400b h-1




```
Current Data Parameters
NAME            2009-084
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090906
Time               16.00
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             6578.947 Hz
FIDRES          0.200774 Hz
AQ              2.4904180 sec
RG                 143.7
DW                76.000 usec
DE                 7.00 usec
TE                300.0 K
D1             0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.20 usec
PL1                 6.00 dB
SFO1         399.8724694 MHz

F2 - Processing parameters
SI                 16384
SF           399.8700087 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity
1	7.3337	2932.5267	2.00
2	7.3142	2924.7292	4.51
3	7.3049	2921.0104	1.00
4	7.2965	2917.6515	5.48
5	7.2709	2907.4148	1.99
6	7.2366	2893.6993	7.63
7	7.2209	2887.4213	5.70
8	7.2165	2885.6619	3.94
9	7.2071	2881.9031	0.89
10	7.2033	2880.3836	0.99
11	4.0570	1622.2726	0.31
12	4.0406	1615.7148	1.02
13	4.0242	1609.1569	1.51
14	4.0078	1602.5990	1.51
15	3.9912	1595.9612	1.04
16	3.9749	1589.4433	0.32
17	2.9123	1164.5414	0.67
18	2.8966	1158.2635	0.77
19	2.8918	1156.3441	0.72
20	2.8772	1150.5060	1.87
21	2.8627	1144.7079	1.41
22	2.8569	1142.3886	1.37
23	2.8417	1136.3106	1.37
24	2.8051	1121.6754	1.36
25	2.7852	1113.7179	2.22
26	2.7706	1107.8798	0.80
27	2.7648	1105.5606	1.54
28	2.7506	1099.8824	1.33
29	2.7303	1091.7651	0.82
30	2.0893	835.4484	0.28
31	2.0686	827.1711	1.68
32	2.0600	823.7322	1.55
33	2.0540	821.3330	1.51
34	2.0474	818.6939	3.21
35	2.0405	815.9348	2.00
36	2.0332	813.0157	2.46
37	2.0273	810.6565	3.51
38	2.0199	807.6974	1.42
39	2.0124	804.6984	1.41
40	2.0068	802.4591	1.48
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