

The Copper-Catalyzed *N*-Arylation of Imidazoles

Ryan A. Altman, Erica D. Koval and Stephen L. Buchwald*

*Department of Chemistry
Massachusetts Institute of Technology
Cambridge, MA 02139*

sbuchwal@mit.edu

Supporting Information

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General Considerations.

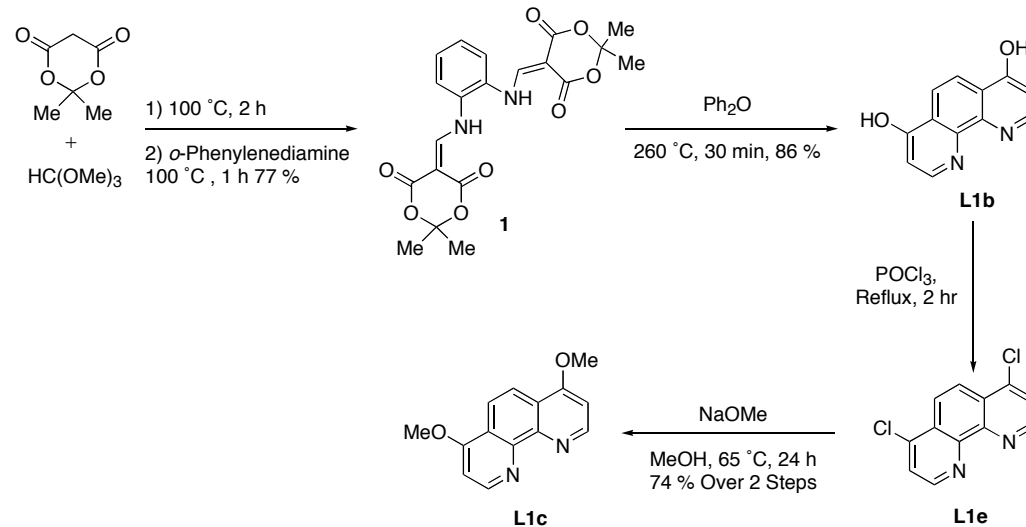
All reactions were carried out in resealable test tubes with teflon septa and run under a dry argon or nitrogen atmosphere. Copper (I) oxide (97%) was purchased from Aldrich company as a red powder. Anhydrous Cs_2CO_3 (99.9%) was purchased from Alfa Aesar; the bulk of the material was stored under nitrogen in a Vacuum Atmospheres glovebox. Small portions (~5 g) were removed from the glovebox in glass vials, stored in the air in a desiccator filled with anhydrous calcium sulfate, and weighed in the air. Poly(ethylene glycol) (M_n 3,400) was purchased from Aldrich. Generally, aryl halides and imidazoles were purchased from commercial sources and used without further purification. When necessary, aryl halides were filtered through neutral alumina, or distilled. Butyronitrile ($\geq 99\%$) was purchased from Aldrich and used without further purification. Anhydrous solvents (NMP, DMSO, and Acetonitrile) were purchased from Aldrich in Sure-Seal [®] bottles. Flash column chromatography was performed with EM Science silica gel 60 (230-400 mesh). In all cases, dichloromethane was used to load the crude reaction material onto a silica gel column. A gradient elution technique was used for column chromatography, beginning with hexane and continuing to the specified concentration of ethyl acetate in hexane.

Yields reported in the publication are isolated (except where noted) and represent an average of at least two independent runs. Yields reported in the supporting information refer to a single experiment. Compounds described in the literature were characterized by comparing their ¹H NMR, and melting point (m.p.) to the previously reported data; their purity was confirmed by gas chromatographic analyses (GC). For known compounds prepared using the new method

(conditions) described, a copy of the ^1H NMR spectrum, of each, is included. GC analyses were performed on a Hewlett Packard 6890 instrument with an FID detector and a Hewlett Packard 10 m x 0.2 mm i.d. HP-1 capillary column using dodecane as an internal standard. Previously unknown compounds were synthesized, purified and analyzed from a single run and were then repeated to determine an average yield. They were characterized by ^1H NMR, ^{13}C NMR, m.p., IR and elemental analysis. Elemental analyses were performed by Atlantic Microlabs, Inc., Norcross, GA. For those compounds that did not give a satisfactory elemental analysis, a copy of their ^1H NMR spectrum is included. ^1H NMR and ^{13}C NMR were recorded on Varian 300 MHz and 500 MHz instruments with chemical shifts reported relative to the deuterated solvent or TMS. IR spectra were recorded on a Perkin-Elmer System 2000 FT-IR instrument for all previously unknown compounds (KBr disc). Melting points (uncorrected) were obtained on a Mel-Temp II capillary melting point apparatus.

Synthesis of Ligands.

L1a, **L1f**, **L3**, **L4**, **L5**, **L7** and **L8** were purchased from commercial sources. **L3** was purified by recrystallization from hexane. **L1d**¹ and complex **L6**² were prepared according to literature precedent. The synthesis of **L1b**, **L1c** and **L1e** was adapted from literature precedent.³ A larger scale preparation of **L1b**, **L1c** and **L1e** can be performed as described in the following text. Alternatively, **L1b** and **L1c** can be purchased in small quantities from commercial sources.

Scheme 1. Synthesis of 4,7-Dimethoxy-1,10-Phenanthroline

1,2-Bis-[(2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)methyl]amino]benzene (**1**).

An oven-dried 2 L 2-neck flask equipped with a mechanical stirrer was charged with trimethyl orthoformate (850 mL, 7.8 mol) and Meldrum's acid (101 g, 0.700 mmol). The flask was fitted with a reflux condenser; the contents were flushed with N_2 and brought to a gentle reflux for 2 h. The resulting red solution was cooled ($\sim 80\text{ }^\circ\text{C}$) and phenylene diamine (32.4 g, 300 mmol) was added portionwise (*exothermic reaction*) resulting in the formation of a yellow solid. The mixture was heated to reflux, stirred vigorously for an additional hour and then cooled to room temperature. The resulting solid was filtered, washed with cold acetone (slightly soluble) and dried to afford 95 g (77%) of product as a flaky light-yellow solid. ^1H NMR (CDCl_3 , 400 MHz) δ 11.34 (br d, 2H), 8.50 (d, 2H), 7.41 (m, 4H), 1.74 (s, 12H). m.p. $208\text{--}210\text{ }^\circ\text{C}$, decomp. (Lit. 209, decomp.).^{3a}

4,7-Dihydroxy-1,10-phenanthroline (L1b). A 5 L 3-neck flask equipped with a mechanical stirrer and a large air-cooled reflux condenser was charged with 3 L of diphenyl ether and was heated to $240\text{ }^\circ\text{C}$ using a heating mantle. Precursor **1** was added in small portions resulting in

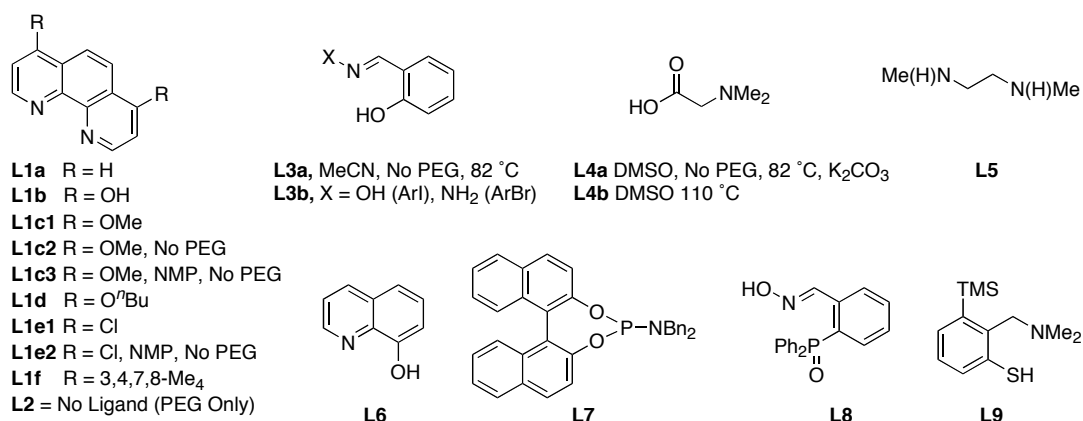
vigorous gas evolution. When the addition was complete, the mixture was brought to reflux (260 °C) for 30 min. The mixture was allowed to cool to 80 °C, and the precipitate was isolated by vacuum filtration and washed with acetone until the filtrate was colorless. The product was further washed with excess hexane and diethyl ether. Drying by vacuum filtration, then under hi-vac at 60 °C, afforded 41.5 g (86%) of a fine dark-brown powder. Although the title compound was essentially insoluble in common NMR solvents, a spectrum could be obtained using NaOH in D₂O. ¹H NMR (D₂O, NaOH, 400 MHz) δ 8.17 (d, 2H, *J* = 5.6 Hz), 7.75 (s, 2H), 6.43 (d, 2H *J* = 5.6 Hz). Anal Calc. for C₁₂H₈N₂: C 67.92, H 3.80. Found: C 67.60, H 3.59. m.p. stable up to 250 °C (Lit. 471-474, decomp.).⁴

4,7-Dichloro-1,10-phenanthroline (L1e). A 1 L 2-neck round bottom flask equipped with a stir bar, reflux condenser, and distillation apparatus was flame-dried and allowed to cool under an atmosphere of N₂. Phosphorous oxychloride (400 mL) and **L1b** (20.0 g, 94.3 mmol) were added to the flask under a N₂ purge. The apparatus was immersed in an oil bath and heated at reflux for 2 h (the condenser for the distillation apparatus was not filled with water at this time). After this period, the circulation of water for the distillation apparatus was turned on and roughly half of the excess phosphorous oxychloride was removed by gentle vacuum distillation. The solution was cooled to room temperature and crushed ice was slowly added to the reaction mixture (*very exothermic!*) while keeping the temperature below 30 °C with an ice bath. When HCl gas evolution ceased, the acidic solution was stirred for one hour at room temperature to dissolve the black solids that formed. The resulting dark cloudy solution was filtered through activated charcoal (Darco®) to give a translucent-beige solution, which was brought to pH 13 by the slow addition of 20% KOH solution while maintaining the temperature below 25 °C. The white

precipitate that formed was collected by suction filtration, washed with excess H₂O, and dried under vacuum overnight at 60 °C affording **L1e** as a white solid. The product was used in the subsequent step without further purification. ¹H NMR (DMSO, 400 MHz) δ 9.09 (d, 2H, *J* = 4.8 Hz), 8.41 (s, 2H), 8.08 (d, 2H, *J* = 4.8 Hz). m.p. 245-247 (Lit. 249-250).^{3c}

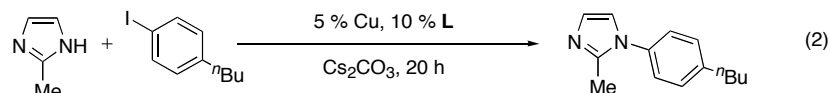
4,7-Dimethoxy-1,10-phenanthroline (L1c). An oven-dried 3-neck round bottom flask was cooled under a stream of nitrogen. Anhydrous methanol (1.2 L) was added, and purged with N₂ for 10 min. Sodium metal (9.20 g, 400 mmol) was slowly added in small pieces while the solution was stirred. A reflux condenser was attached, and **L1e** (all that was produced in the previous step) was added. The flask was heated to reflux for 24 hours under an atmosphere of N₂. Concentration of the resulting solution to ~30 mL and addition of cold water (250 mL) resulted in the precipitation of a tan solid. The flask was stored overnight in a refrigerator to allow complete precipitation of the solid. The product was collected by filtration, washed with excess water, and dried under vacuum overnight at 60 °C affording 16.7 g (74% over 2 steps) of a tan solid, which can be recrystallized from benzene. ¹H NMR (CDCl₃, 300 MHz) δ 8.93 (d, 2H, *J* = 5.3 Hz), 8.18 (s, 2H), 7.03 (d, 2H, *J* = 5.3 Hz), 4.09 (s, 6H). m.p. 210-212 (Lit. 209-210).^{3e}

Table of Ligands Commonly Used for Cu-Catalyzed *N*-Arylation of Heterocycles

Figure 1. Reported Ligands for Cu-Catalyzed *N*-Arylation of Imidazole and Benzimidazole

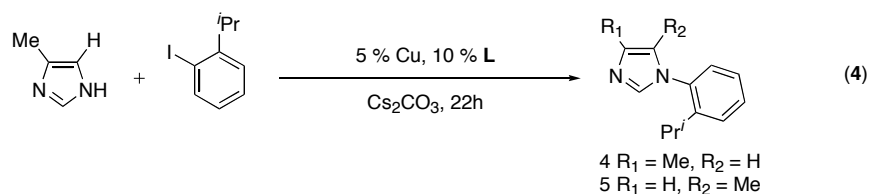
Procedure for screening of the coupling of aryl iodides with imidazoles.

To a screw-cap test tube, was added copper precatalyst (0.025 mmol), ligand (0.05 mmol, solid), imidazole (0.6 mmol), poly(ethylene glycol) (100 mg), Cs₂CO₃ (0.7 mmol), and a magnetic stir bar. The reaction vessel was fitted with a rubber septum. The vessel was evacuated and back-filled with argon or nitrogen. This was repeated two additional times. Aryl iodide (0.5 mmol), ligand (0.375 mmol, liquid) and solvent (0.25 mL) were added. The reaction tube was sealed and the contents were stirred in a pre-heated oil bath at 110 °C for the designated time period. The reaction mixture was cooled to room temperature, and 113 μL dodecane and dichloromethane (5 mL) were added. This mixture was stirred, then filtered through a small plug of celite into a vial for GC analysis. The average of two experiments is reported.



Ligand	Copper Source	Solvent	Additive	Temperature(°C)	GC Conv. (%)	GC Yield (%)
L1a	Cu ₂ O	PrCN	PEG	110	21	14
L1b	Cu ₂ O	PrCN	PEG	110	99	95
L1c1	Cu ₂ O	PrCN	PEG	110	100	96
L1c1	Cu ₂ O	PrCN	-	110	100	96
L1c2	Cu ₂ O	NMP	-	110	100	96
L1d	Cu ₂ O	PrCN	PEG	110	99	97
L1e1	Cu ₂ O	PrCN	PEG	110	89	86

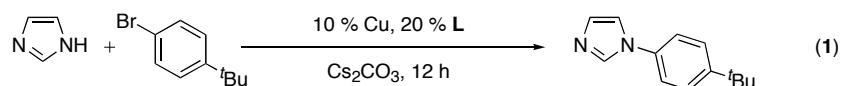
L1e2	Cu ₂ O	NMP	-	110	100	88
L1f	Cu ₂ O	PrCN	PEG	110	34	30
L2	Cu ₂ O	PrCN	PEG	110	11	8
L3a	Cu ₂ O	CH ₃ CN	-	82	6	3
L3b	CuI	PrCN	PEG	110	15	11
L4a	CuI	DMSO	-	82	23	15
L4b	CuI	DMSO	-	110	28	14
L5	CuI	PrCN	PEG	110	18	13
L6a	CuI	DMF/H ₂ O(10:1)	-	110	88	69
L6b	CuI	PrCN	PEG	110	100	96
L10	CuI	PrCN	PEG	110	75	72



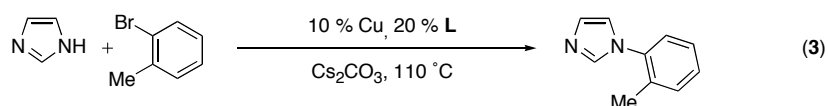
Ligand	Copper Source	Solvent	Additive	Temperature(°C)	GC Conv. (%)	GC Yield (%)	Ratio (4 Me / 5 Me)
L1a	Cu ₂ O	PrCN	PEG	110	32	31	11
L1b	Cu ₂ O	PrCN	PEG	110	56	51	42
L1c	Cu ₂ O	PrCN	PEG	110	68	61	41
L1c	Cu ₂ O	PrCN	-	110	64	53	30
L1c	Cu ₂ O	NMP	-	110	91	81	37
L1d	Cu ₂ O	PrCN	PEG	110	41	39	12
L1e	Cu ₂ O	PrCN	PEG	110	43	37	11
L1e	Cu ₂ O	NMP	-	110	77	62	32
L1f	Cu ₂ O	PrCN	PEG	110	39	35	19
L2	Cu ₂ O	PrCN	PEG	110	31	30	15
L3a	Cu ₂ O	CH ₃ CN	-	82	12	12	n. d.
L3b	CuI	PrCN	PEG	110	34	29	8
L4a	CuI	DMSO	-	82	4	8	n. d.
L4b	CuI	DMSO	-	110	79	66	9
L5	CuI	PrCN	PEG	110	40	36	15
L6a	CuI	DMF/H ₂ O(10:1)	-	110	99	81	64
L6b	CuI	PrCN	PEG	110	66	40	n.d.
L10	CuI	PrCN	PEG	110	28	26	11

Procedure for screening the coupling of aryl bromides with imidazoles.

To a screw-cap test tube, was added copper precatalyst (0.05 mmol), ligand (0.01 mmol, solid), imidazole (0.6 mmol), poly(ethylene glycol) (100 mg), Cs_2CO_3 (0.7 mmol), and a magnetic stir bar. The reaction vessel was fitted with a rubber septum. The vessel was evacuated and back-filled with argon or nitrogen. This procedure was repeated two times. Aryl bromide (0.5 mmol), ligand (0.01 mmol, liquid) and solvent (0.25 mL) were then added successively. The reaction tube was sealed and the contents were stirred in a pre-heated oil bath at 110 °C for the designated time period. The reaction mixture was cooled to room temperature and 113 μL dodecane and dichloromethane (5 mL) were added. This mixture was stirred, then filtered through a small plug of celite into a vial for GC analysis. The average of two experiments is reported.



Ligand	Copper Source	Solvent	Additive	Temperature (°C)	GC Conv. (%)	GC Yield (%)
L1a	Cu_2O	PrCN	PEG	110	30	28
L1b	Cu_2O	PrCN	PEG	110	74	75
L1c	Cu_2O	PrCN	PEG	110	97	97
L1c	Cu_2O	PrCN	-	110	68	67
L1c	Cu_2O	NMP	-	110	100	95
L1d	Cu_2O	PrCN	PEG	110	60	61
L1e	Cu_2O	PrCN	PEG	110	51	51
L1e	Cu_2O	NMP	-	110	87	82
L1f	Cu_2O	PrCN	PEG	110	47	41
L2	Cu_2O	PrCN	PEG	110	29	29
L3a	Cu_2O	CH_3CN	-	82	12	9
L3b	CuI	PrCN	PEG	110	21	19
L4a	CuI	DMSO	-	82	7	2
L4b	CuI	DMSO	-	110	46	38
L5	CuI	PrCN	PEG	110	32	33
L6a	CuI	DMF/H ₂ O(10:1)	-	110	60	49
L6b	CuI	PrCN	PEG	110	90	89
L10	CuI	PrCN	PEG	110	27	18



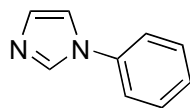
Ligand	Copper Source	Solvent	Additive	Temperature (°C)	GC Conv. (%)	GC Yield (%)
L1a	Cu ₂ O	PrCN	PEG	110	43	42
L1b	Cu ₂ O	PrCN	PEG	110	42	40
L1c1	Cu ₂ O	PrCN	PEG	110	76	78
L1c2	Cu ₂ O	PrCN	-	110	56	53
L1c3	Cu ₂ O	NMP	-	110	89	83
L1d	Cu ₂ O	PrCN	PEG	110	52	50
L1e1	Cu ₂ O	PrCN	PEG	110	49	47
L1e2	Cu ₂ O	NMP	-	110	71	66
L1f	Cu ₂ O	PrCN	PEG	110	42	41
L2	Cu ₂ O	PrCN	PEG	110	43	41
L3a	Cu ₂ O	CH ₃ CN	-	82	16	14
L3b	CuI	PrCN	PEG	110	51	52
L4a	CuI	DMSO	-	82	17	6
L4b	CuI	DMSO	-	110	56	42
L5	CuI	PrCN	PEG	110	37	27
L6a	CuI	DMF/H ₂ O(10:1)	-	110	93	61
L6b	CuI	PrCN	PEG	110	100	85
L10	CuI	PrCN	PEG	110	48	47

General procedure for the *N*-arylation of imidazoles with aryl iodides.

An oven-dried screw-cap test tube was charged with Cu₂O (0.025 mmol), **L1c** (0.075 mmol), imidazole (1.2 mmol), aryl iodide (1.00 mmol, if solid), poly(ethylene glycol) (200 mg), Cs₂CO₃ (1.4 mmol), and a magnetic stir bar. The reaction vessel was fitted with a rubber septum. The vessel was evacuated and back-filled with argon or nitrogen, and this sequence was repeated an additional time. Aryl iodide (1.00 mmol, if liquid) and solvent (0.5 mL) were then added successively. The reaction tube was sealed, and stirred in a pre-heated oil bath for the designated time period. The reaction mixture was cooled to room temperature, diluted with dichloromethane (10 mL), and filtered through a plug of celite, eluting with additional dichloromethane (50 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography to provide the desired product.

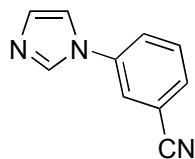
General procedure for the *N*-arylation of imidazoles with aryl bromides.

An oven-dried screw-cap test tube was charged with Cu₂O (0.05 mmol), **L1c** (0.15 mmol), imidazole (1.2 mmol), aryl bromide (1.00 mmol, if solid), poly(ethylene glycol) (200 mg), Cs₂CO₃ (1.4 mmol), and a magnetic stir bar. The reaction vessel was fitted with a rubber septum. The vessel was evacuated and back-filled with argon or nitrogen, and this sequence was repeated an additional time. Aryl bromide (1.00 mmol, if liquid), and solvent (0.5 mL) were then added successively. The reaction tube was sealed, immersed, and stirred in a preheated oil bath for the designated time period. The reaction mixture was cooled to room temperature, diluted with dichloromethane (10 mL), filtered through a plug of celite, and eluted with additional dichloromethane (50 mL). The filtrate was concentrated and the resulting residue was purified by flash chromatography to provide the desired product.

Experimental procedures for all compounds contained in Table 1**1-Phenyl-1*H*-imidazole (1a)**

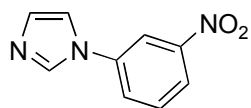
The general procedure was followed using Cu₂O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), iodobenzene (112 μ L, 1.00 mmol), and imidazole (82 mg, 1.2 mmol), with NMP (0.5 mL) as solvent for 3 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) afforded 1-phenyl-1*H*-imidazole (slightly yellow oil, 131 mg, 92%). The low catalyst loading experiment was performed using the general procedure with Cu₂O (0.4 mg, 0.0025 mmol), **L1c** (1.8 mg, 0.0075 mmol), PEG (2.0 g), Cs₂CO₃ (4.50 g, 14 mmol), iodobenzene (1.12 mL, 10 mmol), and imidazole (820 mg, 12 mmol), in butyronitrile (2.0 mL) as solvent for 48 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) provided 1-phenyl-1*H*-imidazole (slightly yellow oil, 1.34 g, 93%). ¹H NMR (300 MHz, CDCl₃) δ 7.82 (s, 1H), 7.47-7.41 (m, 2H),

7.36-7.29 (m, 3H), 7.25 (bs, 1H), 7.18 (bs, 1H).⁵



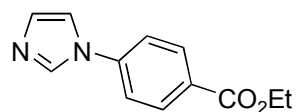
3-Imidazol-1-yl-benzonitrile (1b)

The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 3-iodobenzonitrile (229 mg, 1.00 mmol), and imidazole (82 mg, 1.2 mmol), with butyronitrile (0.5 mL) as solvent for 24 h at 90 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) 3-imidazol-1-yl-benzonitrile (white needles, 158 mg, 94%). ^1H NMR (500 MHz, CDCl_3) δ 7.89 (s, 1H), 7.71-7.60 (m, 4H), 7.30 (bs, 1H), 7.25 (bs, 1H). m.p. 151-154 °C (Lit. 156-157 °C).⁶



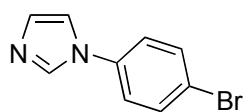
1-(3-Nitro-phenyl)-1H-imidazole (1c)

The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 3-nitroiodobenzene (249 mg, 1.00 mmol), and imidazole (83 mg, 1.2 mmol) with acetonitrile (0.5 mL) as solvent for 29 h at 80 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) 1-(3-nitro-phenyl)-1H-imidazole (white solid, 177 mg, 93%). ^1H NMR (500 MHz, CDCl_3) δ 8.25 (t, 1H, $J = 1.9$), 8.19 (ddd, 1H, $J = 1.1, 1.9, 7.9$ Hz), 7.94 (s, 1H), 7.75 (ddd, 1H, $J = 1.4, 2.2, 8.1$ Hz), 7.69 (t, 1H, $J = 8.0$ Hz), 7.36 (s, 1H), 7.23 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 149.1, 138.3, 125.5, 131.5, 131.2, 126.9, 122.1, 118.0, 116.2. m.p. 109-110 °C (Lit. 109-110 °C).⁷



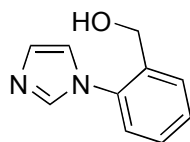
4-Imidazol-1-yl-benzoic acid ethyl ester (1d)

The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), ethyl-4-iodobenzoate (168 μL , 1.00 mmol), and imidazole (82 mg, 1.2 mmol), 3 Å molecular sieves (200 mg, powdered, flame activated) with acetonitrile (0.5 mL) as solvent for 23 h at 80 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) 4-imidazol-1-yl-benzoic acid ethyl ester (white crystals, 184 mg, 84%). ^1H NMR (500 MHz, CDCl_3) δ 8.18 (td, 2H, $J = 1.8, 8.5$ Hz), 7.96 (s, 1H), 7.48 (td, 2H, $J = 1.8, 8.8$), 7.37 (s, 1H), 7.26 (s, 1H), 4.43 (q, 2H, $J = 7.1$ Hz), 1.44 (t, 3H, $J = 7.0$ Hz). m.p. 101-103 °C (Lit. 100-102 °C).⁸



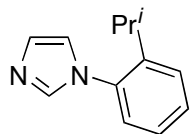
1-(4-Bromo-phenyl)-1H-imidazole⁹ (1e)

The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 1-bromo-4-iodobenzene (340 mg, 1.20 mmol), and imidazole (68 mg, 1.0 mmol) with butyronitrile (0.5 mL) as solvent for 24 h at 90 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) provided 1-(4-bromo-phenyl)-1H-imidazole (white crystals, 171 mg, 78%). ^1H NMR (300 MHz, CDCl_3) δ 7.83 (bs, 1H), 7.63 (m, 2H), 7.32-7.20 (m, 4H). m.p. 120-122 °C. GC/MS of the crude material showed an 6.1 : 1 mixture of 1-(4-bromo-phenyl)-1H-imidazole to 1-(4-iodo-phenyl)-1H-imidazole.



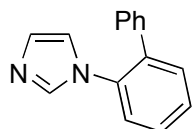
(2-Imidazol-1-yl-phenyl)-methanol (1f)

The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 2-iodobenzylalcohol (234 mg, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.3 mL) as solvent for 24 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) provided (2-imidazol-1-yl-phenyl)-methanol (clear crystals, 165 mg, 95%). ^1H NMR (300 MHz, CDCl_3) δ 7.62 (dd, 2H, $J = 1.7, 7.6$ Hz), 7.45 (td, 1H, $J = 1.4, 7.5$ Hz), 7.38 (td, 1H, $J = 1.4, 7.4$ Hz), 7.23 (dd, 1H, $J = 1.1, 7.7$ Hz), 7.13 (s, 1H), 7.08 (s, 1H), 4.90 (bs, 1H), 4.46 (s, 1H). m.p. 102-104 °C (Lit. 100.5-102.5 °C).¹⁰



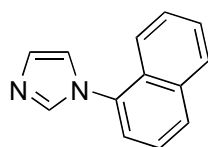
1-(2-Isopropyl-phenyl)-1H-imidazole (1g)

The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 2-isopropyl iodobenzene (246 mg, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.5 mL) as solvent for 48 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) provided 1-(2-isopropyl-phenyl)-1H-imidazole (white crystals, 171 mg, 92%). ^1H NMR (300 MHz, CDCl_3) δ 7.57 (bs, 1H), 7.46-7.42 (m, 2H), 7.31-7.11 (m, 3H), 7.05 (m, bs), 2.74 (heptet, 1H, $J = 6.9$ Hz), 1.16 (d, 6H, $J = 6.9$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 145.0, 135.1, 129.4, 129.2, 127.0, 126.7, 126.4, 121.1, 27.4, 24.0. m.p. 76-77 °C (Lit. 67-68 °C).¹¹ Anal. Calc. for $\text{C}_{12}\text{H}_{14}\text{N}_2$: C 77.38, H 7.58. Found: C 77.42, H 7.78.



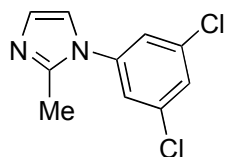
1-Biphenyl-2-yl-1*H*-imidazole (1h)

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 2-iodobiphenyl (176 μL, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.6 mL) as solvent for 72 h at 120 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) provided 1-biphenyl-2-yl-1*H*-imidazole (slightly yellow crystals, 179 mg, 81%). ¹H NMR (300 MHz, CDCl₃) δ 7.50-7.33 (m, 5H), 7.30-7.24 (m, 3H), 7.02 (bs, 1H), 6.82 (bs, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 137.8, 137.5, 135.2, 131.5, 128.7, 128.7, 128.6, 128.3, 127.8, 126.3. Anal. Calc. for C₁₅H₁₂N₂: C 81.79, H 5.49. Found: C 81.50, H 5.46. m.p. 93-95 °C.



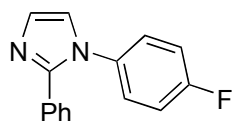
1-Naphthalen-1-yl-1*H*-imidazole (1i)

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 1-iodonaphthalene (146 μL, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with NMP (0.3 mL) as solvent for 24 h at 110 °C. The crude reaction mixture was dissolved in 20 mL of water, and extracted with dichloromethane (5 x 30 mL). The combined organic layers were dried with MgSO₄, and the solvent was removed *in vacuo*. Chromatographic purification (hexane / ethyl acetate 1 : 1) provided 1-naphthalen-1-yl-1*H*-imidazole (yellow-white solid, 179 mg, 92%). ¹H NMR (300 MHz, CDCl₃) δ 7.94-7.92 (m, 2H), 7.76, (bs, 1H), 7.61-7.48 (m, 4 H), 7.43 (d, 1H, J = 7.0 Hz), 7.30 (bs, 1H), 7.25 (bs, 1H). m.p. 63-64 °C (Lit. 62 °C).¹²



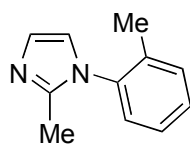
1-(3,5-Dichloro-phenyl)-2-methyl-1*H*-imidazole (1j)

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 1,3-dichloro-5-iodobenzene (273 mg, 1.00 mmol), 2-methylimidazole (100 mg, 1.2 mmol) with butyronitrile (0.5 mL) as solvent for 24 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) provided 1-(3,5-dichloro-phenyl)-2-methyl-1*H*-imidazole (white needles, 194 mg, 86%). ¹H NMR (300 MHz, CDCl₃) δ 7.44 (t, 1H, J = 1.8), 7.23 (d, 2H, J = 1.9), 7.05 (d, 1H, J = 1.2), 6.99 (d, 1H, J = 1.2), 2.40 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 139.8, 135.8, 128.5, 128.4, 124.1, 14.0. IR (KBr disc, cm⁻¹) 1534, 1501, 1463, 1451, 1405, 1305, 1176, 1143, 1115, 1099, 985, 850, 781. Anal. Calc. for C₁₀H₈N₂Cl₂: C 52.89, H 3.55. Found: C 52.95, H 3.44. m.p. 122-125 °C.



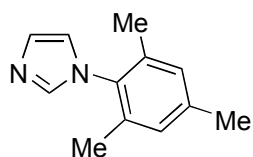
1-(4-Fluoro-phenyl)-2-phenyl-1*H*-imidazole (1k)

The general procedure was followed using Cu₂O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 4-fluoroiodobenzene (222 mg, 1.00 mmol), 2-phenylimidazole (173 mg, 1.2 mmol) with butyronitrile (0.3 mL) as solvent for 24 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 3 : 1) provided 1-(4-fluoro-phenyl)-2-phenyl-1*H*-imidazole (white solid, 211 mg, 89%). ¹H NMR (300 MHz, CDCl₃) δ 7.36-7.30 (m, 2H), 7.24-7.16 (m, 4H), 7.15-7.09 (m, 2H), 7.06 (s, 1H), 7.03-6.96 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 163.5, 160.2, 134.5, 129.0, 128.5, 128.4, 128.2, 127.6, 127.5, 116.5, 116.2. IR (KBr disc, cm⁻¹) 1509, 1501, 1466, 1414, 1303, 1285, 1233, 1212, 1151, 1128, 1091, 1068, 970, 915, 840, 775, 747, 715, 697. Anal. Calc. for C₁₅H₁₁N₂F: C 75.62, H 4.65. Found: C 75.39, H 4.62. m.p. 112-114 °C.



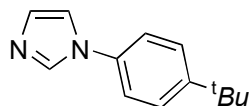
2-Methyl-1-*o*-tolyl-1*H*-imidazole (1l)

The general procedure was followed using CuI (19 mg, 0.10 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 2-iodotoluene (127 μL, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) except NMP (0.5 mL) was used as solvent and the reaction was carried out for 48 h at 140 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) afforded 2-methyl-1-*o*-tolyl-1*H*-imidazole (yellow oil, 149 mg, 86%). ¹H NMR (300 MHz, CDCl₃) δ 7.34-7.09 (m, 4H), 6.99 (bs, 1H), 6.81 (bs, 1H), 2.12 (s, 3H), 1.99 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 138.9, 137.6, 135.5, 133.5, 129.6, 129.1, 120.1, 21.1, 17.4. IR (KBr disc, cm⁻¹) 1524, 1501, 1461, 1416, 1303, 1178, 1141, 1093, 1045, 986, 770, 726, 697.

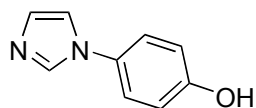


1-(2,4,6-Trimethyl-phenyl)-1*H*-imidazole (1m)

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 2-iodotoluene (127 μL, 1.00 mmol), 2-methylimidazole (100 mg, 1.2 mmol) except that DMSO (0.5 mL) was used as solvent and the reaction was carried out for 48 h at 150 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) afforded 1-(2,4,6-trimethyl-phenyl)-1*H*-imidazole (yellow oil, 96 mg, 51%). ¹H NMR (300 MHz, CDCl₃) δ 7.42 (s, 1H), 7.21 (s, 1H), 6.95 (s, 2H), 6.87 (s, 1H), 2.32 (s, 3H), 1.97, (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 145.1, 136.9, 135.3, 131.1, 129.1, 127.7, 127.4, 126.9, 120.4, 17.2, 13.1. m.p. 107-109 °C (Lit. 107-108 °C).¹³

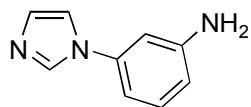
Experimental procedures for all compounds contained in Table 2**1-(4-*tert*-Butylphenyl)-1*H*-imidazole¹⁴ (2a)**

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.175 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 4-*t*-butylbromobenzene (173 μ L, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.25 mL) as solvent for 15 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) afforded 1-(4-*tert*-butylphenyl)-1*H*-imidazole (white crystals, 174 mg, 87%). ¹H NMR (300 MHz, CDCl₃) δ 7.78 (s, 1H), 7.3 (m, 2H), 7.25 (m, 2H), 7.20 (s, 1H), 7.15 (s, 1H), 1.30 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 150.6, 135.6, 134.8, 130.2, 126.7, 121.1, 118.3, 34.6, 31.3. IR (KBr disc, cm⁻¹) 1525, 1462, 1365, 1302, 1266, 1243, 1120, 1106, 1061, 903. Anal Calc. for C₁₃H₁₆N₂: C 77.96, H 8.05. Found: 77.56, H 8.00. m.p. 90-91 °C.

**4-Imidazol-1-ylphenol (2b)**

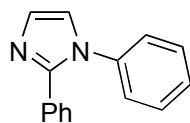
The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (400 mg), Cs₂CO₃ (1.0 g, 3.0 mmol), 4-bromophenol (172 mg, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.5 mL) as solvent for 15 h at 110 °C. After cooling to ambient temperature, the crude reaction mixture was dissolved in 20 mL 2M HCl_(aq), and washed once with diethyl ether. The aqueous layer was brought to pH 8 with Na₂CO₃, and extracted repeatedly with CH₂Cl₂. The combined organic layers were dried with anhydrous MgSO₄, and concentrated.

Chromatographic purification (1% ethanol in ethyl acetate, dry pack) afforded 4-imidazol-1-yl-phenol (white crystals, 198 mg, 90%). ^1H NMR (300 MHz, CD_3OD) δ 7.95 (bs, 1H), 7.41 (bs, 1H), 7.35-7.30 (m, 2H), 7.01 (bs, 1H), 6.92-6.87 (m, 2H). ^{13}C NMR (100 MHz, CD_3OD) δ 158.7, 130.8, 129.8, 124.3, 117.4, 24.0. m.p. 196-198 °C (Lit. 188-190°C [203-205 °C MeOH, H_2O]).¹⁵



3-Imidazol-1-yl-phenylamine (2c)

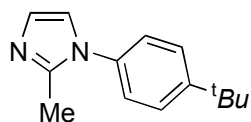
The general procedure was followed using Cu_2O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 3-bromoaniline (109 μL , 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.25 mL) as solvent for 20 h at 110 °C. Chromatographic purification (ethyl acetate) afforded 3-imidazol-1-yl-phenylamine (white powder, 141 mg, 88%). ^1H NMR (300 MHz, CDCl_3) δ 7.79 (bs, 1H), 7.21-7.15 (m, 3H), 6.72-6.68 (m, 1H), 6.64-6.60 (m, 2H). 4.01 (bs, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 148.2, 138.4, 135.6, 130.7, 130.1, 118.4, 114.0, 111.1, 107.7. m.p. 112-114 °C (Lit. 111-113 °C).¹⁶



1,2-Diphenyl-1*H*-imidazole (2d)

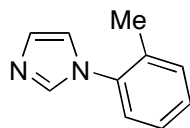
The general procedure was followed using Cu_2O (14.4 mg, 0.10 mmol), **L1c** (72 mg, 0.30 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 4-*t*-butylbromobenzene (173 μL , 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.6 mL) as solvent for 72 h at 120 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 1,2-diphenyl-1*H*-imidazole

(white crystals, 198 mg, 90%). ^1H NMR (300 MHz, CDCl_3) δ 7.42-7.32 (m, 5H), 7.28-7.16 (m, 6H), 7.14 (d, 1H, $J = 1.1$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 138.6, 130.4, 129.5, 129.1, 128.6, 128.4, 128.2, 128.2, 125.9, 123.0. m.p. 88-89 °C (Lit. 90 °C).¹⁷



1-(4-*tert*-Butyl-phenyl)-2-methyl-1*H*-imidazole (2e)

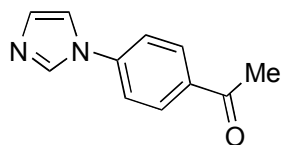
The general procedure was followed using Cu_2O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 4-*t*-butylbromobenzene (173 μL , 1.00 mmol), 2-methylimidazole (100 mg, 1.2 mmol) with butyronitrile (0.25 mL) as solvent for 48 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) afforded 1-(4-*tert*-butyl-phenyl)-2-methyl-1*H*-imidazole (yellow oil, 207 mg, 97%). ^1H NMR (300 MHz, CDCl_3) δ 7.41 (m, 2H), 7.12 (m, 1H), 6.94 (bs, 1H), 6.91 (bs, 1H), 2.28 (s, 3H), 1.29 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 151.1, 135.3, 127.5, 126.3, 124.9, 12.7, 34.6, 31.3, 13.8. IR (KBr Disc, cm^{-1}) 2962, 2870, 1608, 1579, 1513, 1463, 1419, 1365, 1302, 1269, 1178, 1139, 1114, 996, 986, 842, 730, 674, 571.



1-*ortho*-Tolyl-1*H*-imidazole (2f)

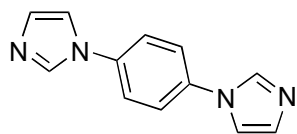
The general procedure was followed using Cu_2O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 2-bromotoluene (120 μL , 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.3 mL) as solvent for 28 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 1-*ortho*-tolyl-1*H*-imidazole (yellow oil, 140 mg,

89%). ^1H NMR (300 MHz, CDCl_3) δ 7.59 (bs, 1H), 7.37-7.28 (m, 3H), 7.23-7.20 (m, 2H), 7.06 (bs, 1H), 2.19 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 148.2, 138.4, 135.6, 130.7, 130.1, 118.4, 114.0, 111.1, 107.7.¹⁸



1-(4-Imidazol-1-yl-phenyl)-ethanone (2g)

The general procedure was followed using Cu_2O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 4-bromoacetophenone (199 mg, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.4 mL) as solvent for 48 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 3) afforded 1-(4-imidazol-1-yl-phenyl)-ethanone (white solid, 159 mg, 86%). ^1H NMR (300 MHz, CDCl_3) δ 8.04 (m, 2H), 7.92 (bs, 1H), 7.46 (m, 2H), 7.33 (bs, 1H), 7.19 (bs, 1H), 2.59 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 196.6, 140.7, 135.7, 135.4, 131.2, 130.4, 120.7, 117.8, 26.7. m.p. 112-114 °C (Lit. 110-112 °C).¹⁹

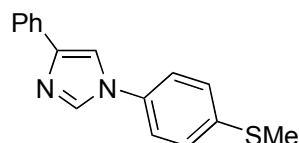


1,4-bis(Imidazol-1-yl)-benzene²⁰ (2h)

The general procedure was followed using Cu_2O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), Cs_2CO_3 (0.90 g, 2.8 mmol), 1,4-dibromobenzene (236 mg, 1.00 mmol), and imidazole (164 mg, 2.4 mmol) with NMP (0.5 mL) as solvent for 30 h at 110 °C. The crude reaction mixture was diluted in excess CH_2Cl_2 , and filtered through a celite plug. After removal of the solvent *in vacuo*, the product was crystallized from EtOAc and stored in a freezer at -23 °C overnight, to afford 1,4-bis(imidazol-1-yl)-benzene (white solid, 200 mg, 95%). ^1H NMR (500 MHz, CDCl_3) δ 7.90 (bs, 2H), 7.54 (s,

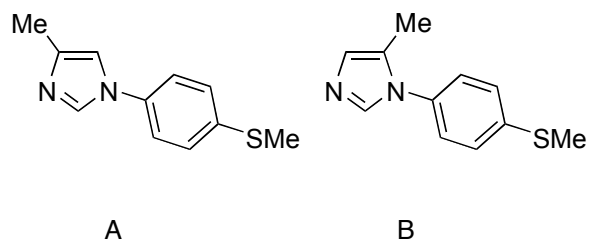
4H), 7.32 (bs, 1H), 7.26 (bs, 1H). m.p. 190 °C (dec.). IR (KBr disc, cm^{-1}) 1534, 1485, 1304, 1248, 1105, 1059. Anal. Calc. for $\text{C}_{12}\text{H}_{10}\text{N}_4$: C 68.56, H 4.79. Found C 68.49, H 4.90.

Experimental procedures for all compounds contained in Table 3



1-(4-Methylsulfonyl-phenyl)-4-phenyl-1H-imidazole (3a)

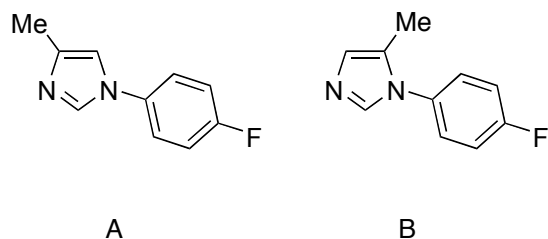
The general procedure was followed using Cu_2O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 4-bromothioanisole (203 mg, 1.00 mmol), 4-phenylimidazole (173 mg, 1.2 mmol) with butyronitrile (0.4 mL) as solvent for 17 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 1-(4-methylsulfonyl-phenyl)-4-phenyl-1H-imidazole (white crystals, 254 mg, 95%). GC analysis and ^1H NMR showed no trace of a second regioisomer. ^1H NMR (300 MHz, CDCl_3) δ 7.87-7.83 (m, 3H), 7.54 (d, 1H, 1.4 Hz), 7.44-7.34 (m, 6H), 7.32-7.26 (m, 2H), 2.53 (s, 3H). ^{13}C NMR (75.5 MHz, CDCl_3) δ 143.3, 138.4, 135.8, 134.5, 133.8, 128.8, 127.8, 127.3, 125.1, 122.0, 113.9, 16.1. IR (KBr disc, cm^{-1}) 1550, 1508, 1443, 1420, 1313, 1252, 1070, 957, 936, 920, 828, 817, 758, 703. Anal. Calc. for $\text{C}_{16}\text{H}_{14}\text{N}_2\text{S}$: C 72.15, H 5.30. Found: C 71.98, H 5.34. m.p. 122-124°C.



4-Methyl-1-(4-methylsulfonyl-phenyl)-1H-imidazole (3b)

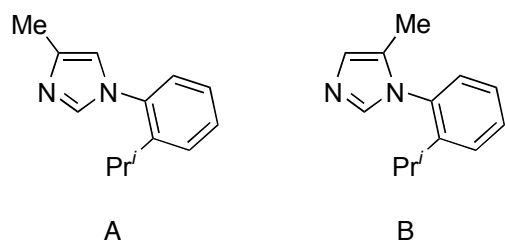
The general procedure was followed using Cu_2O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol),

PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 1-bromo-4-fluorobenzene (109 μL, 1.00 mmol), 4-methylimidazole (100 mg, 1.2 mmol) with butyronitrile (0.3 mL) as solvent for 48 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 4-methyl-1-(4-methylsulfanyl-phenyl)-1*H*-imidazole (yellow oil, 203 mg, 100%). ¹H NMR showed a 3.0 : 1 mixture of A : B. ¹H NMR (300 MHz, CDCl₃) δ 7.65 (bs, 1H), 7.28-7.11 (m, 4H), 6.90 (bs, 1H), 2.46 (s, minor regioisomer), 2.43 (s, major regioisomer). ¹³C NMR (100 MHz, CDCl₃) δ 138.5, 135.9, 134.6, 133.9, 128.9, 127.9, 125.1, 122.0, 113.9, 16.7, 15.6. Anal. Calc. for C₁₁H₁₂N₂S: C 64.67, H 5.92. Found: C 64.29, H 5.93.



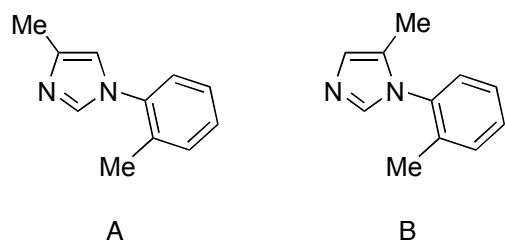
1-(4-Fluoro-phenyl)-4-methyl-1*H*-imidazole (3c)

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 1-bromo-4-fluorobenzene (109 μL, 1.00 mmol), 4-phenylimidazole (173 mg, 1.2 mmol) with butyronitrile (0.5 mL) as solvent for 15 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 1-(4-Fluoro-phenyl)-4-methyl-1*H*-imidazole (yellow solid/liquid, 161 mg, 92%). ¹H NMR showed a 5.0 : 1 mixture of regioisomers. By GC, the ratio of A : B was determined to be 5.7 : 1. ¹H NMR (300 MHz, CDCl₃) δ 7.65 (bs, 1H), 7.31-7.24 (m, 2H), 7.15-7.07 (m, 2H), 6.92 (bs, 1H), 2.26 (s, 1H, major regioisomer), 2.12 (s, 1H, minor regioisomer). ¹³C NMR (100 MHz, CDCl₃) δ 162.5, 160.5, 139.8, 134.9, 133.9, 123.1, 116.8, 13.7. IR (KBr disc, cm⁻¹) 1519, 1449, 1324, 1293, 1227, 1160, 1101, 1071, 1007, 974, 926, 816. Anal. Calc. for C₁₀H₉N₂F: C 68.17, H 5.15. Found: C 68.30, H 5.19. m.p. 22-24 °C



1-(2-Isopropyl-phenyl)-4-methyl-1*H*-imidazole (3d)

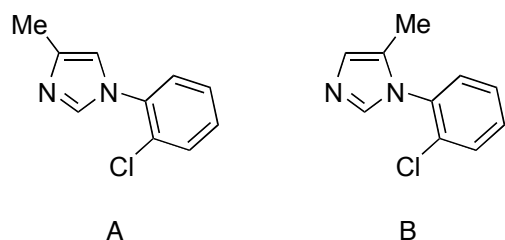
The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 2-isopropyl iodobenzene (160 μL , 1.00 mmol), 4-methylimidazole (100 mg, 1.2 mmol) with butyronitrile (0.3 mL) as solvent for 20 h at 110 $^\circ\text{C}$. GC and GC/MS analysis of the crude material showed an 82% yield and 41 : 1 ratio of A : B. (See GC screens on p. S8) ^1H NMR of the purified material showed a 16 : 1 ratio of A : B. ^1H NMR (300 MHz, CDCl_3) δ 7.48-7.40 (m, 3H), 7.28-7.15 (m, 2H), 6.75 (t, 1H, $J = 0.9$ Hz), 2.80 (heptet, 1H, $J = 6.9$ Hz), 2.31 (d, 3H, $J = 0.8$ Hz), 1.18 (d, 6H, $J = 6.9$ Hz)



4-Methyl-1-*o*-tolyl-1*H*-imidazole²¹ (3e)

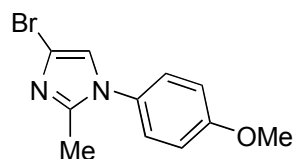
The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 2-iodotoluene (127 μL , 1.00 mmol), 4-methylimidazole (100 mg, 1.2 mmol), with butyronitrile (0.3 mL) as solvent for 24 h at 110 $^\circ\text{C}$. GC Analysis showed a 23 : 1 ratio of regioisomers. Chromatographic purification (hexane / ethyl acetate 1 : 3) provided 4-methyl-1-*o*-tolyl-1*H*-imidazole (yellow oil, 154 mg, 87%). ^1H NMR of the purified material showed a 16 : 1 ratio of A : B. ^1H NMR (300 MHz, CDCl_3) δ 7.44 (bs, 1H), 7.35-7.14 (m,

4H), 6.75 (bs, 1H), 2.28 (s, 3H, major isomer), 2.17 (s, 3H, major isomer), 2.11 (s, minor isomer), 1.97 (s, minor regioisomer). (KBr disc, cm^{-1}) 1507, 1448, 1386, 1364, 1294, 1269, 1231, 1191, 1122, 1073. 1002, 971.



1-(2-Chloro-phenyl)-4-methyl-1H-imidazole (3f)

The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 2-chloriodobenzene (122 μL , 1.00 mmol), 4-methylimidazole (100 mg, 1.2 mmol) with butyronitrile (0.3 mL) as solvent for 15 h at 110 $^\circ\text{C}$. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 1-(2-chloro-phenyl)-4-methyl-1H-imidazole (yellow oil, 191 mg, 99%). ^1H NMR showed a 12.1 : 1 mixture of A : B. By GC, the ratio was determined to be 19 : 1. ^1H NMR (300 MHz, CDCl_3) δ 7.49 (bs, 1H), 7.47-7.39 (m, 1H), 7.29-7.18 (m, 3H), 6.78 (bs, 1H), 2.21 (s, major regioisomer), 2.06 (d, minor regioisomer). ^{13}C NMR (100 MHz, CDCl_3) δ 135.1, 130.7, 129.4, 129.2, 127.7, 127.4, 13.6. IR (KBr disc, cm^{-1}) 1593, 1570, 1503, 1447, 1392, 1369, 1290, 1232, 1202, 1131, 1084, 1059, 1035, 1004, 973, 819, 761, 633. Anal. Calc. for $\text{C}_{10}\text{H}_9\text{N}_2\text{Cl}$: C 68.17, H 5.15. Found: C 68.30, H 5.19.

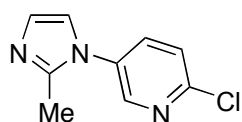


4-Bromo-1-(4-methoxy-phenyl)-2-methyl-1H-imidazole (3g)

The general procedure was followed using CuI (9.5 mg, 0.05 mmol), **L1c** (18 mg, 0.075 mmol), Cs_2CO_3 (0.45 g, 1.4 mmol), 4-iodoanisole (280 mg, 1.2 mmol), 4-bromo-2-methylimidazole (161

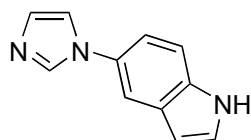
mg, 1.00 mmol) with acetonitrile (1.0 mL) as solvent for 24 h at 110 °C. Chromatographic purification (hexane / ethyl acetate 3 : 1) provided 4-bromo-1-(4-methoxy-phenyl)-2-methyl-1*H*-imidazole (white crystalline solid, 218 mg, 82%). ¹H NMR (300 MHz, CDCl₃) δ 7.09 (m, 2H), 6.89, (m, 2H), 6.82 (s, 1H), 3.79, (s, 3H), 2.12 (s, 3H) ¹³C NMR (100 MHz, CDCl₃) δ 159.6, 145.1, 129.6, 126.7, 119.7, 114.6, 113.8, 55.53, 13.4. IR (KBr disc, cm⁻¹) 1559, 1507, 1457, 1437, 1419, 1299, 1239, 1181, 1167, 1135, 1108, 1032, 1020, 949, 668. Anal. Calc. for C₁₁H₁₁BrN₂O: C 49.46, H 4.15. Found: C 49.51, H 4.03. m.p. 136-137 °C.

Experimental procedures for all compounds contained in Table 4



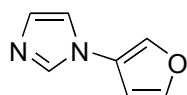
2-Chloro-5-(2-methyl-imidazol-1-yl)-pyridine (4a)

The general procedure was followed using Cu₂O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 5-iodo-2-chloropyridine (239 mg, 1.00 mmol), 2-methylimidazole (100 mg, 1.2 mmol) with butyronitrile (1.0 mL) as solvent for 24 h at 110 °C. Chromatographic purification (ethyl acetate) provided 2-chloro-5-(2-methyl-imidazol-1-yl)-pyridine (white solid, 143 mg, 74%). ¹H NMR (300 MHz, CDCl₃) δ 8.33, (d, 1H, J = 2.9 Hz), 7.59 (dd, 1H, J = 2.9, 8.7 Hz), 7.43 (d, 1H, 8.7, Hz), 6.99 (d, 1H, 1.2 Hz), 6.95 (d, 1H, J = 1.6 Hz), 2.31 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 139.9, 136.0, 128.7, 128.5, 124.4, 124.2, 120.5, 120.4, 14.1. IR (KBr disc, cm⁻¹) 1503, 1476, 1415, 1386, 1300, 1177, 1151, 1109, 993, 982. m.p. 147-149 °C.

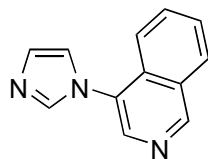


5-Imidazol-1-yl-1*H*-indole (4b)

The general procedure was followed using Cu₂O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 5-iodoindole (243 mg, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with butyronitrile (0.3 mL) as solvent for 13 h at 110 °C. Chromatographic purification (ethyl acetate) provided 5-imidazol-1-yl-1*H*-indole (white solid, 152 mg, 83%). ¹H NMR (300 MHz, CDCl₃/CD₃CN) δ 10.0 (bs, 1H), 7.9 (1H), 7.79-7.65 (1H), 7.60-7.48 (1H), 7.44-7.43 (2H), 7.28 (s, 1H), 7.21 (dd, 1H), 6.68-6.60 (m, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 202.2, 136.5, 135.4, 129.6, 128.4, 126.8, 119.9, 116.8, 114.3, 112.3, 102.6. IR (KBr disc, cm⁻¹) 1541, 1498, 1457, 1436, 1346, 1309, 1268, 1241, 1110, 1055, 916, 870, 725. Anal. Calc. for C₁₁H₉N₃: C 72.11, H 4.95. Found: C 72.43, H 4.95. m.p. 144-146 °C.

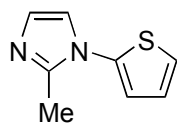
**1-Furan-3-yl-1*H*-imidazole (4c)**

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 2-bromofuran (108 μL, 1.2 mmol), and imidazole (68 mg, 1.00 mmol) with DMSO (0.5 mL) as solvent for 24 h at 110 °C. Chromatographic purification (ethyl acetate / hexane 3 : 1) provided 1-furan-3-yl-1*H*-imidazole (white solid, 89 mg, 67%). ¹H NMR (300 MHz, CDCl₃) δ 7.71 (bs, 1H), 7.62 (dd, 1H, J = 0.8, 1.7 Hz), 7.42 (dd, 1H, J = 1.7, 1.9 Hz), 7.22-7.06 (bs, 2H), 6.54 (dd, 1H, J = 0.8, 1.9 Hz). ¹³C NMR (100 MHz, CDCl₃) δ 143.9, 133.0, 126.0, 106.2. IR (neat, cm⁻¹) 2092, 1523, 1489, 1406, 1317, 1252, 1173, 1025. m.p. 39-41 °C.



4-Imidazol-1-yl-isoquinoline (4d)

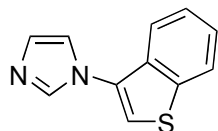
The general procedure was followed using Cu_2O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 4-bromoisoquinoline (250 mg, 1.2 mmol), and imidazole (68 mg, 1.00 mmol) with DMSO (0.5 mL) as solvent for 24 h at 110 °C. Chromatographic purification (ethyl acetate / hexane 3 : 1) provided the title compound (clear crystals, 165 mg, 85%). ^1H NMR (300 MHz, CDCl_3) δ 9.27 (d, 1H, 0.9 Hz), 8.48 (s, 1H), 8.08 (m, 1H), 7.78-7.62, (m, 4H), 7.30 (bs, 1H), 7.25 (bs, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 153.3, 139.6, 138.2, 132.1, 131.9, 130.2, 129.2, 128.8, 128.4, 127.9, 121.5, 121.2. IR (KBr disc, cm^{-1}) 1589, 1508, 1491, 1406, 1382, 1307, 1260, 1250, 1194, 1107, 1078, 1038, 942, 913, 782, 756, 660, 589. Anal. Calc. for $\text{C}_{12}\text{H}_9\text{N}_3$: C 73.43 H 4.65. Found: C 73.43, H 4.63. m.p. 67-71 °C.



2-Methyl-1-thiophen-2-yl-1H-imidazole (4e)

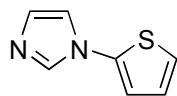
The general procedure was followed using Cu_2O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 2-iodothiophene (132 mg, 1.2 mmol), 2-methylimidazole (83 mg, 1.00 mmol) with DMSO (0.3 mL) as solvent for 24 h at 110 °C. Workup was performed under an atmosphere of N_2 . Chromatographic purification (ethyl acetate / hexane 3 : 1) provided 2-methyl-1-thiophen-2-yl-1H-imidazole (yellow oil, 90 mg, 55%). ^1H NMR (500 MHz, CDCl_3) δ 7.25 (dd, 1H, $J = 1.4, 5.5$ Hz), 7.03-6.99 (m, 3H), 6.96 (dd, 1H, 1.5, 3.7 Hz), 2.39 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 128.0, 126.2, 124.1, 123.5, 122.3, 100.0, 13.7. IR (neat, cm^{-1}) IR (KBr disc, cm^{-1}) 1555, 1496, 1451, 1406, 1305, 1287, 1172, 1137, 987, 941. This compound

turns dark brown upon exposure to air or after standing for 2-3 of days as room temperature under an argon atmosphere.



1-Benzo[*b*]thiophen-3-yl-1*H*-imidazole (4f)

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.05 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 3-bromothiophene (131 μL, 1.00 mmol), and imidazole (82 mg, 1.2 mmol) with DMSO (0.5 mL) as solvent for 48 h at 110 °C. The crude reaction mixture was dissolved in 20 mL H₂O, and the organic material was extracted repeatedly with CH₂Cl₂. The combined organic extracts were washed once with brine, then dried over MgSO₄ and concentrated to an oil. Chromatographic purification (ethyl acetate / hexane 3 : 1) 1-benzo[*b*]thiophen-3-yl-1*H*-imidazole (yellow oil, 188 mg, 94%). ¹H NMR (300 MHz, CDCl₃) δ 7.89-7.85 (m, 1H), 7.80 (bs, 1H), 7.68-7.63 (m, 1H), 7.46-7.38 (m, 3H), 7.26 (bs, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 139.0, 133.8, 130.5, 130.0, 125.8, 125.3, 123.44, 121.0, 119.7. IR (KBr Disc, cm⁻¹) 3114, 1570, 1539, 1509, 1486, 1431, 1385, 1332, 1267, 1254, 1228, 1106, 1081, 1061, 1035, 912, 821, 731, 757, 659. After 2-3 days at room temperature under an argon atmosphere, the compound turned dark brown in color.



1-Thiophen-2-yl-1*H*-imidazole (4g)

The general procedure was followed using Cu₂O (3.6 mg, 0.025 mmol), **L1c** (18 mg, 0.075 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 2-iodothiophene (132 mg, 1.2 mmol), imidazole (68 mg, 1.00 mmol) with DMSO (0.3 mL) as solvent for 24 h at 110 °C. Chromatographic purification

(ethyl acetate / hexane 3 : 1) provided 1-thiophen-2-yl-1*H*-imidazole (yellow oil, 125 mg, 83%). ¹H NMR (300 MHz, CDCl₃) δ 7.71 (bs, 1H), 7.15 (bs, 1H), 7.11 (bs, 1H), 7.09 (dd, 1H, J = 2.0, 5.2 Hz), 6.96-6.92 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 139.0, 137.0, 130.2, 126.4, 121.8, 120.3, 119.0. This oil turned dark brown after 2-3 days at room temperature under an argon atmosphere.

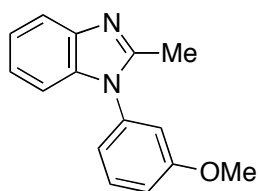
General procedure for the *N*-arylation of benzimidazoles with aryl bromides using Cs₂CO₃ as base.

An oven-dried screw-cap test tube was charged with Cu₂O (0.05 mmol), **L1c** (0.15 mmol), benzimidazole (1.2 mmol), aryl bromide (1.00 mmol, if solid), poly(ethylene glycol) (200 mg), Cs₂CO₃ (1.4 mmol), and a magnetic stir bar. The reaction vessel was fitted with a rubber septum. The vessel was evacuated and back-filled with argon, and this sequence was repeated an additional time. Aryl bromide (1.00 mmol, if liquid) and DMSO (0.5 mL) were then added successively. The reaction tube was sealed, immersed, and stirred in a 110 °C preheated oil bath for the designated time period. The reaction mixture was cooled to room temperature, diluted with dichloromethane (10 mL), filtered through a plug of celite, and eluted with additional dichloromethane (50 mL). The crude reaction mixture was dissolved in 20 mL of ammonium hydroxide and extracted with dichloromethane (3 x 30 mL). The combined organic layers were then extracted with 20 mL of brine. The resulting aqueous phase was extracted with dichloromethane (3 x 30 mL). The combined organic layers were dried with anhydrous MgSO₄, and the solvent was removed *in vacuo*. The resulting residue was purified by flash chromatography to provide the desired product.

General procedure for the *N*-arylation of substituted benzimidazoles with aryl bromides using MTBD as base.

An oven-dried screw-cap test tube was charged with Cu₂O (0.10 mmol), **L1c** (0.20 mmol), the benzimidazole derivative (1.2 mmol), aryl bromide (1.00 mmol, if solid), 1,3,4,6,7,8-Hexahydro-1-

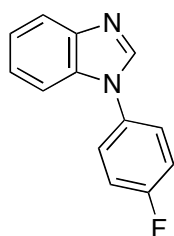
methyl-2H-pyrimido[1,2-a]-pyrimiding (MTBD) (200 μ L), and a magnetic stir bar. The reaction vessel was fitted with a rubber septum. The vessel was evacuated and back-filled with argon, and this sequence was repeated an additional time. Aryl bromide (1.00 mmol, if liquid) and DMSO (0.5 mL) were then added successively. The reaction tube was sealed, immersed, and stirred in a 130 $^{\circ}$ C preheated oil bath for the designated time period. The reaction mixture was cooled to room temperature, diluted with dichloromethane (10 mL), filtered through a plug of celite, and eluted with additional dichloromethane (50 mL). The crude reaction mixture was dissolved in 20 mL of ammonium hydroxide and extracted with dichloromethane (3 x 30 mL). The combined organic layers were then extracted with 20 mL of brine. The resulting aqueous phase was extracted with dichloromethane (3 x 30 mL). The combined organic layers were dried with anhydrous MgSO_4 , and the solvent was removed *in vacuo*. The resulting residue was purified flash chromatography to provide the desired product.



1-(3-methoxyphenyl)-2-methyl-1H-benzo[d]imidazole (5a)

The general procedure was followed using Cu_2O (14.3 mg, 0.10 mmol), **L1c** (48 mg, 0.20 mmol), MTBD (200 μ L, 1.4 mmol), 3-bromoanisole (126 μ L, 1.00 mmol), and 2-methylbenzimidazole (159 mg, 1.2 mmol) with DMSO (0.5 mL) as solvent for 24 h at 130 $^{\circ}$ C. The crude reaction mixture was dissolved in 20 mL of ammonium hydroxide and extracted with dichloromethane (3 x 30 mL). The combined organic layers were then extracted with 20 mL of brine. The resulting aqueous phase was extracted with dichloromethane (3 x 30 mL). The combined organic layers were dried with anhydrous MgSO_4 , and the solvent was removed *in vacuo*. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 1-(3-methoxyphenyl)-1H-benzo[d]imidazole (white solid, 192 mg,

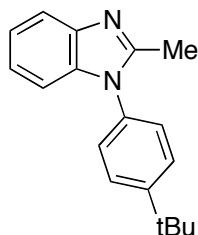
81%). ^1H NMR (300 MHz, CDCl_3) δ 7.64 (d, 1H, $J = 8.0$), 7.37 (t, 1H, $J = 8.1$ Hz), 7.19-7.05 (m, 3H), 6.95 (ddd, 1H, $J = 0.9, 2.5, 8.5$ Hz), 6.80-6.87 (m, 2H), 3.75 (s, 1H), 2.40 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3) δ 160.7, 151.6, 142.6, 137.2, 136.4, 130.7, 122.6, 122.4, 119.3, 119.0, 114.4, 112.9, 110.1, 55.6, 14.5. IR (KBr disc, cm^{-1}) 1601, 1516, 1492, 1464, 1392, 1322, 1288, 1255, 1222, 1165, 1054, 1024, 928, 879, 830, 786, 751, 697, 435. Anal. Calc. for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}$: C 75.61, H 5.92. Found C 75.22, H 5.79. m.p. 132.5-133.5 $^\circ\text{C}$.



1-(4-fluorophenyl)-1H-benzop[d]imidazole (5b)

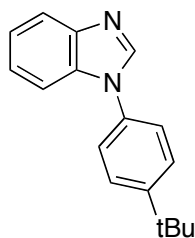
The general procedure was followed using Cu_2O (14.3 mg, 0.10 mmol), **L1c** (48 mg, 0.20 mmol), PEG (200 mg), Cs_2CO_3 (0.45 g, 1.4 mmol), 1-bromo-4-fluorobenzene (109 μL , 1.00 mmol), and benzimidazole (142 mg, 1.2 mmol) with DMSO (0.5 mL) as solvent for 9 h at 110 $^\circ\text{C}$. The crude reaction mixture was dissolved in 20 mL of ammonium hydroxide and extracted with dichloromethane (3 x 30 mL). The combined organic layers were then extracted with 20 mL of brine. The resulting aqueous phase was extracted with dichloromethane (3 x 30 mL). The combined organic layers were dried with anhydrous MgSO_4 , and the solvent was removed *in vacuo*. Chromatographic purification (hexane / ethyl acetate 1 : 2) afforded 1-(4-fluorophenyl)-1H-benzop[d]imidazole (white solid, 180 mg, 85%). ^1H NMR (300 MHz, CDCl_3) δ 7.92 (s, 1H), 7.76-7.72 (m, 1H), 7.34-7.30 (m, 3H), 7.30-7.09 (m, 4H). ^{13}C NMR (125 MHz, CDCl_3) δ 162.9, 160.9, 143.9, 142.3, 133.8, 132.3, 126.0, 125.9, 123.8, 122.9, 120.6, 117.1, 116.9, 110.2. IR (KBr disc, cm^{-1}) 3416, 3061, 1911, 1781, 1734, 1666, 1614, 1511, 1486, 1458, 1317, 1289, 1235, 1217, 1148, 1094, 1010, 980, 936, 887, 868, 845, 823, 815, 783, 766, 750, 716, 668, 647, 619, 589,

567, 531, 483, 436, 411. Anal. Calc. for $C_{13}H_9FN_2$: C 73.57, H 4.27. Found C 73.37, H 4.24. m.p. 118.5-119.5 °C (Lit. 114-115 °C).²²



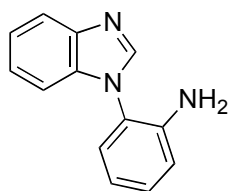
1-(4-*tert*-butylphenyl)2-methyl-1*H*-benzo[*d*]imidazole (5c)

The general procedure was followed using Cu_2O (14.3 mg, 0.10 mmol), **L1c** (48 mg, 0.20 mmol), MTBD (200 μ L, 1.4mmol), 1-bromo-4-*tert*-butylbenzene (173 μ L, 1.00 mmol), and 2-methylbenzimidazole (159 mg, 1.2 mmol) with DMSO (0.5 mL) as solvent for 24 h at 130 °C. The crude reaction mixture was dissolved in 20 mL of ammonium hydroxide and extracted with dichloromethane (3 x 30 mL). The combined organic layers were then extracted with 20 mL of brine. The resulting aqueous phase was extracted with dichloromethane (3 x 30 mL). The combined organic layers were dried with anhydrous $MgSO_4$, and the solvent was removed *in vacuo*. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 1-(4-*tert*-butylphenyl)2-methyl-1*H*-benzo[*d*]imidazole (white solid, 191 mg, 72%). 1H NMR (300 MHz, $CDCl_3$) δ 7.63 (d, 1H, $J=7.9$ Hz), 7.44 (d, 2H, $J=8.4$), 7.15-7.13 (m, 3H), 7.05-7.03 (m, 2H), 2.39 (s, 3H), 1.29 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.9, 151.7, 142.6, 136.6, 133.3, 126.8, 126.5, 122.4, 122.2, 118.9, 110.1, 34.9, 31.4, 14.5. IR (KBr disc, cm^{-1}) 3399, 3050, 3038, 2964, 2868, 2717, 2320, 1924, 1883, 1847, 1806, 1766, 1664, 1612, 1587, 1518, 1477, 1456, 1397, 1367, 1324, 1314, 1285, 1269, 1248, 1204, 1186, 1145, 1122, 1108, 1033, 1011, 998, 973, 942, 875, 860, 842, 764, 741, 704, 678, 644, 633, 593, 566, 534, 497, 429, 406. Anal. Calc. for $C_{18}H_{20}N_2$: C 81.78, H 7.63. Found C 81.69, H 7.62. m.p. 132-133 °C.



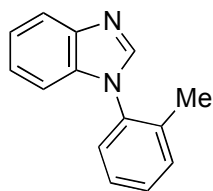
1-(4-*tert*-butylphenyl)-1*H*-benzo[*d*]imidazole²² (5d)

The general procedure was followed using Cu₂O (14.3 mg, 0.10 mmol), **L1c** (48 mg, 0.20 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 1-bromo-4-*tert*-butylbenzene (173 μ L, 1.00 mmol), and benzimidazole (142 mg, 1.2 mmol) with DMSO (0.5 mL) as solvent for 6 h at 110 °C. The crude reaction mixture was dissolved in 20 mL of ammonium hydroxide and extracted with dichloromethane (3 x 30 mL). The combined organic layers were then extracted with 20 mL of brine. The resulting aqueous phase was extracted with dichloromethane (3 x 30 mL). The combined organic layers were dried with anhydrous MgSO₄, and the solvent was removed *in vacuo*. Chromatographic purification (hexane / ethyl acetate 1 : 1) afforded 1-(4-*tert*-butylphenyl)-1*H*-benzo[*d*]imidazole (white crystals, 240 mg, 96%). ¹H NMR (300 MHz, CDCl₃) δ 7.94 (s, 1H), 7.77-7.74 (dd, 1H, J = 2.9, 6.2 Hz), 7.43-7.37 (m, 3H), 7.27-7.15 (m, 4H), 1.25 (s, 9H). ¹³C NMR (125 MHz, CDCl₃) δ 151.2, 144.0, 142.3, 133.8, 133.7, 126.9, 123.6, 123.6, 123.5, 122.6, 120.5, 110.5, 34.7, 31.3. IR (KBr disc, cm⁻¹) 3429, 3112, 3055, 2959, 2902, 2867, 1736, 1609, 1520, 1488, 1461, 1416, 1369, 1361, 1323, 1303, 1267, 1232, 1212, 1162, 1142, 1123, 1107, 1026, 1009, 977, 932, 890, 871, 846, 827, 783, 765, 741, 645, 621, 589, 565, 548, 500, 431. Anal. Calc. for C₁₇H₁₈N₂: C 81.56, H 7.25. Found C 81.12, H 7.23. m.p. 150-151.5 °C.



2-(1*H*-benzo[*d*]imidazol-1-yl)aniline (5e)

The general procedure was followed using Cu₂O (14.3 mg, 0.10 mmol), **L1c** (48 mg, 0.20 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 2-bromoaniline (109 μL, 1.00 mmol), and benzimidazole (142 mg, 1.2 mmol) with DMSO (0.5 mL) as solvent for 15 h at 110 °C. The crude reaction mixture was dissolved in 20 mL of ammonium hydroxide and extracted with dichloromethane (3 x 30 mL). The combined organic layers were then extracted with 20 mL of brine. The resulting aqueous phase was extracted with dichloromethane (3 x 30 mL). The combined organic layers were dried with anhydrous MgSO₄, and the solvent was removed *in vacuo*. Chromatographic purification (with Biotage system with gradient of pure hexane to pure ethyl acetate to hexane / ethyl acetate 1 : 1) afforded 2-(1*H*-benzo[*d*]imidazol-1-yl)aniline (orange crystals, 157 mg, 75%). ¹H NMR (300 MHz, CDCl₃) δ 7.83 (s, 1H), 7.73 (dd, 1H, J = 2.2, 6.3 Hz), 7.20-7.11 (m, 4H), 7.03 (dd, 1H, J = 1.4, 7.7 Hz), 6.80-6.71 (m, 2H), 3.67 (s, 2H) ¹³C NMR (125 MHz, CDCl₃) δ 143.5, 143.3, 142.9, 133.9, 130.3, 128.2, 123.6, 122.8, 121.1, 120.4, 118.6, 116.6, 110.9. IR (KBr disc, cm⁻¹) 3227, 3202, 1625, 1507, 1486, 1454, 1308, 1288, 1227, 1157, 977, 890, 785, 744, 503, 442, 428. Anal. Calc. for C₁₃H₁₁N₃: C 74.62, H 5.30. Found C 74.45, H 5.25. m.p. 115-116 °C (Lit. 112.5-113 °C).²³

**1-*o*-tolyl-1*H*-benzo[*d*]imidazole²⁴ (5f)**

The general procedure was followed using Cu₂O (7.2 mg, 0.05 mmol), **L1c** (36 mg, 0.15 mmol), PEG (200 mg), Cs₂CO₃ (0.45 g, 1.4 mmol), 2-bromotoluene (120 μL, 1.00 mmol), and benzimidazole (142 mg, 1.2 mmol) with DMSO (0.5 mL) as solvent for 24 h at 130 °C. The crude reaction mixture was dissolved in 20 mL of ammonium hydroxide and extracted with

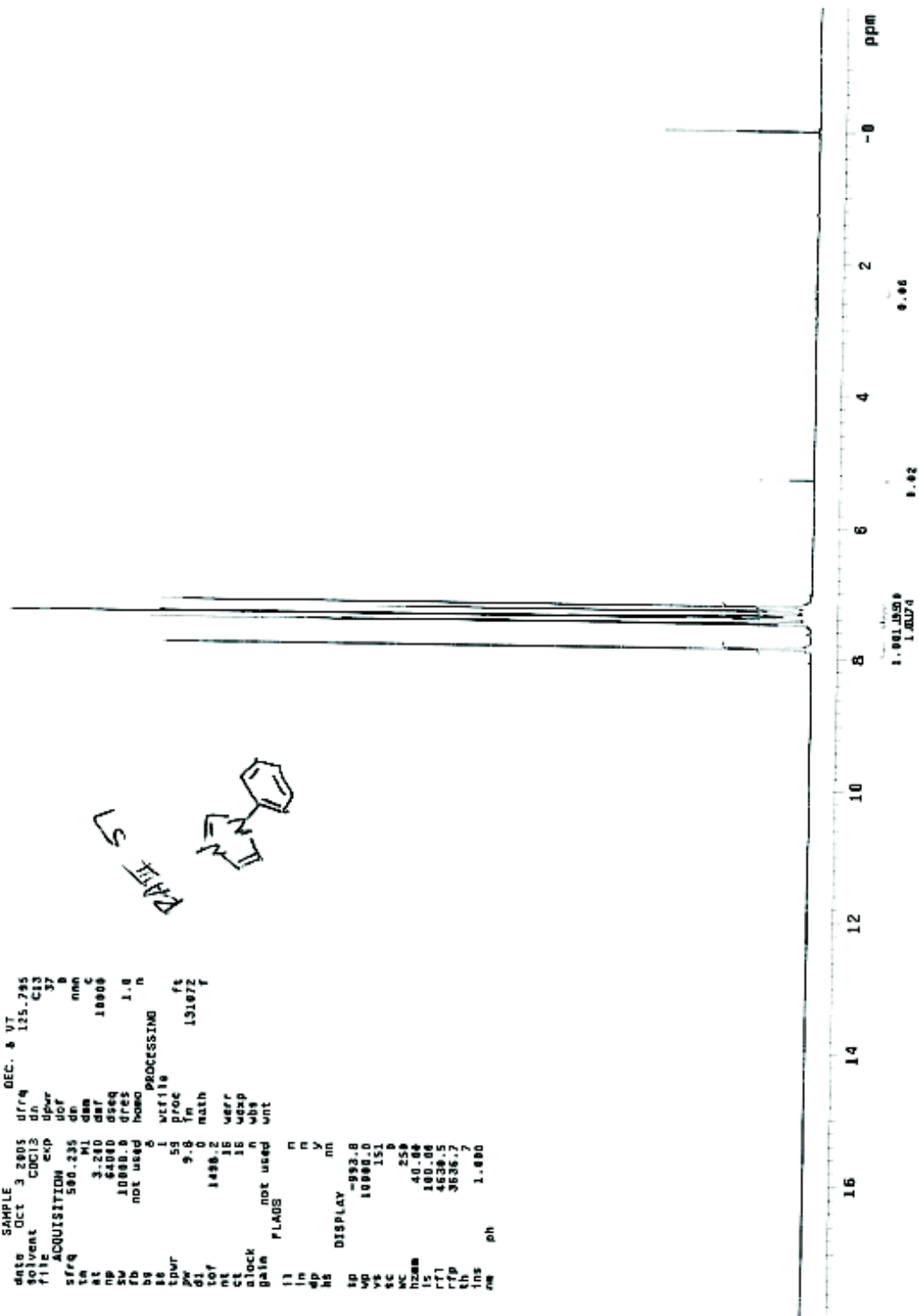
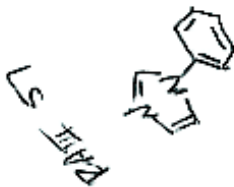
dichloromethane (3 x 30 mL). The combined organic layers were then extracted with 20 mL of brine. The resulting aqueous phase was extracted with dichloromethane (3 x 30 mL). The combined organic layers were dried with anhydrous MgSO_4 , and the solvent was removed *in vacuo*. Chromatographic purification (with Biotage system with gradient of pure hexane to hexane / ethyl acetate 1 : 1) afforded 1-*o*-tolyl-1*H*-benzo[*d*]imidazole (yellow oil, 186 mg, 78%). ^1H NMR (300 MHz, CDCl_3) δ 7.88 (s, 1H), 7.81 (d, 1H, $J = 7.9$ Hz), 7.95-7.18 (m, 6H), 7.05 (dd, $J = 7.9, 0.6$ Hz), 2.01 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ 143.5, 143.1, 135.5, 134.9, 134.8, 131.7, 129.5, 127.8, 127.3, 123.6, 122.6, 120.6, 110.6, 17.8

STANDARD PROTON PARAMETERS

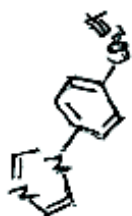
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clock 16 warr
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hs m
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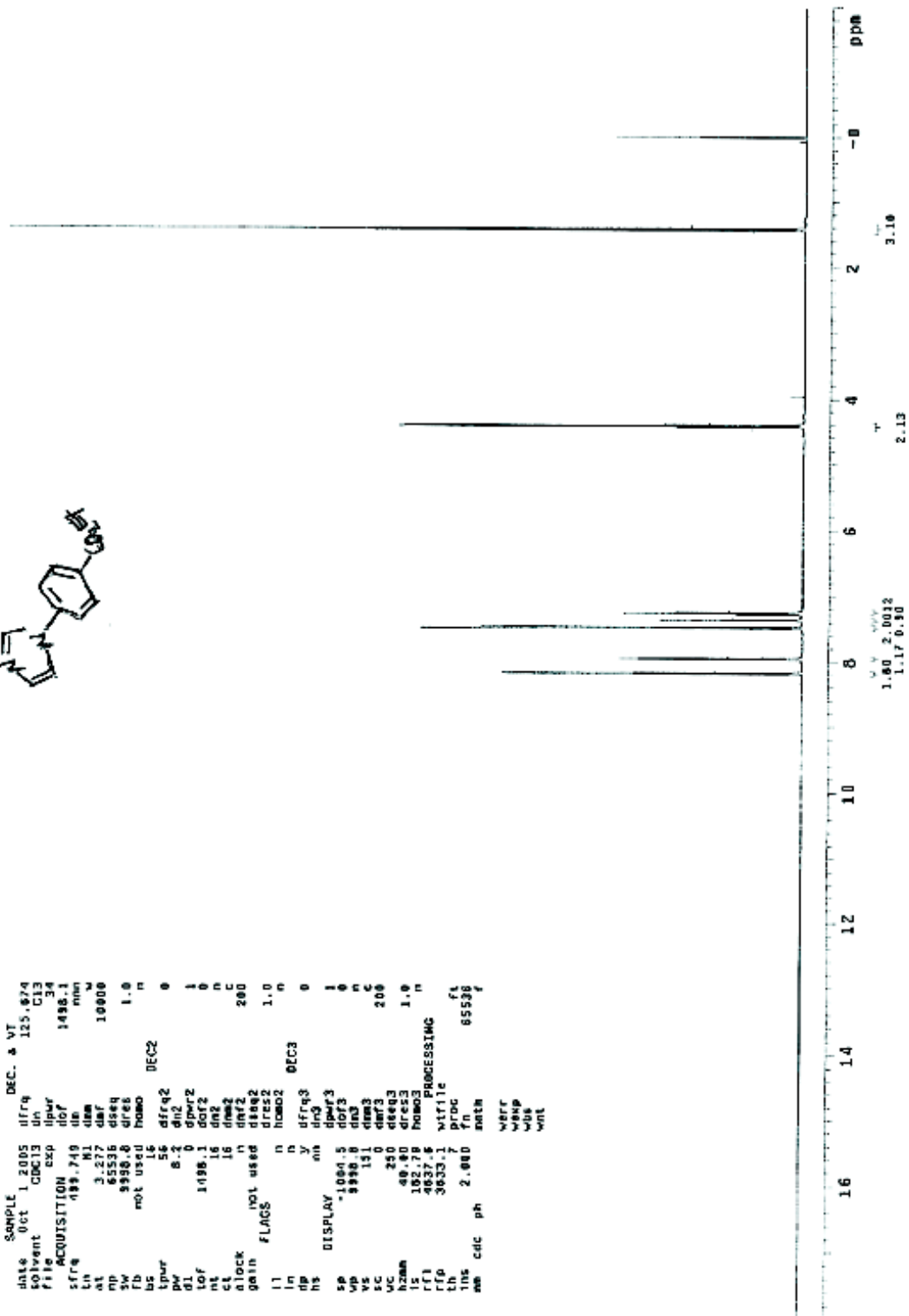
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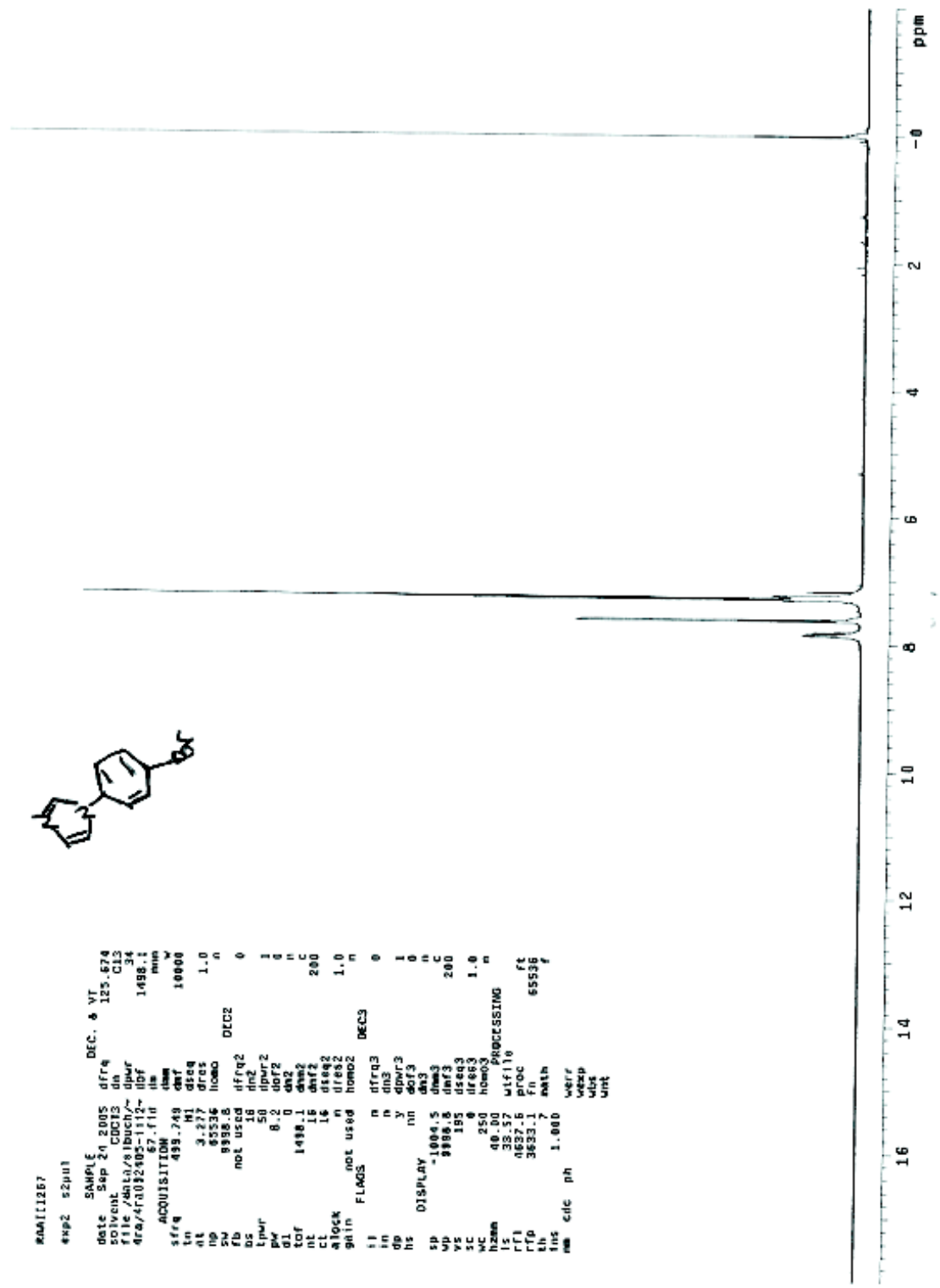
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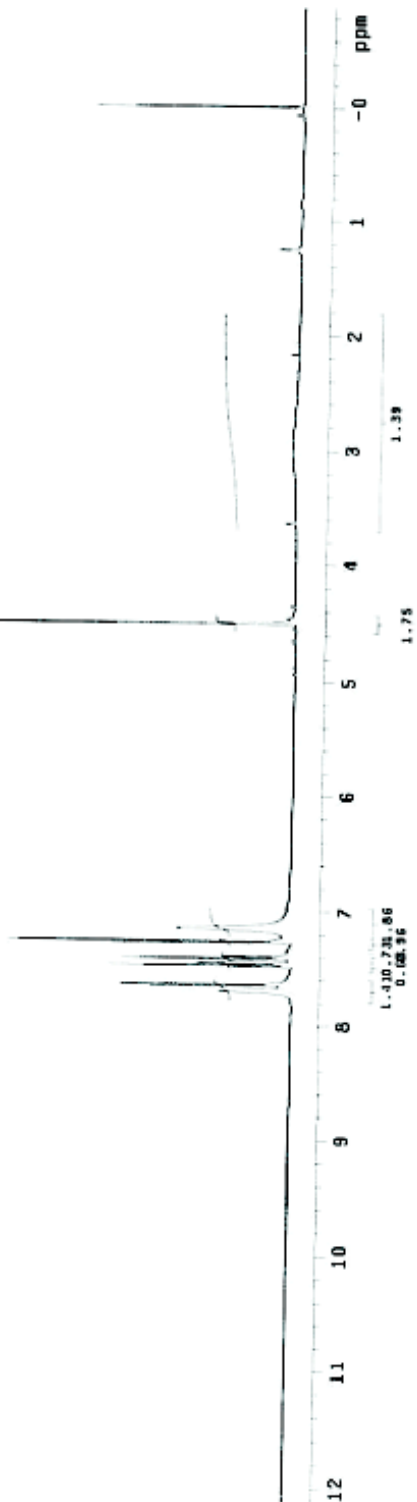
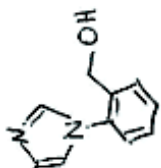
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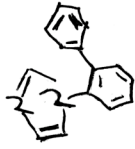
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RAMIT 212





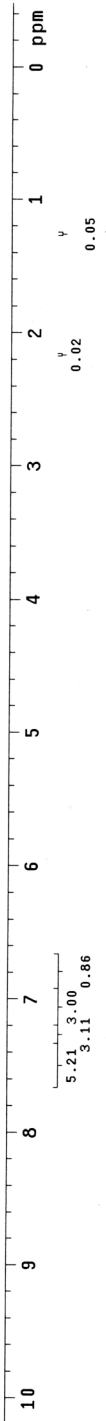
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Byron Atteem 1-im-naphthalene

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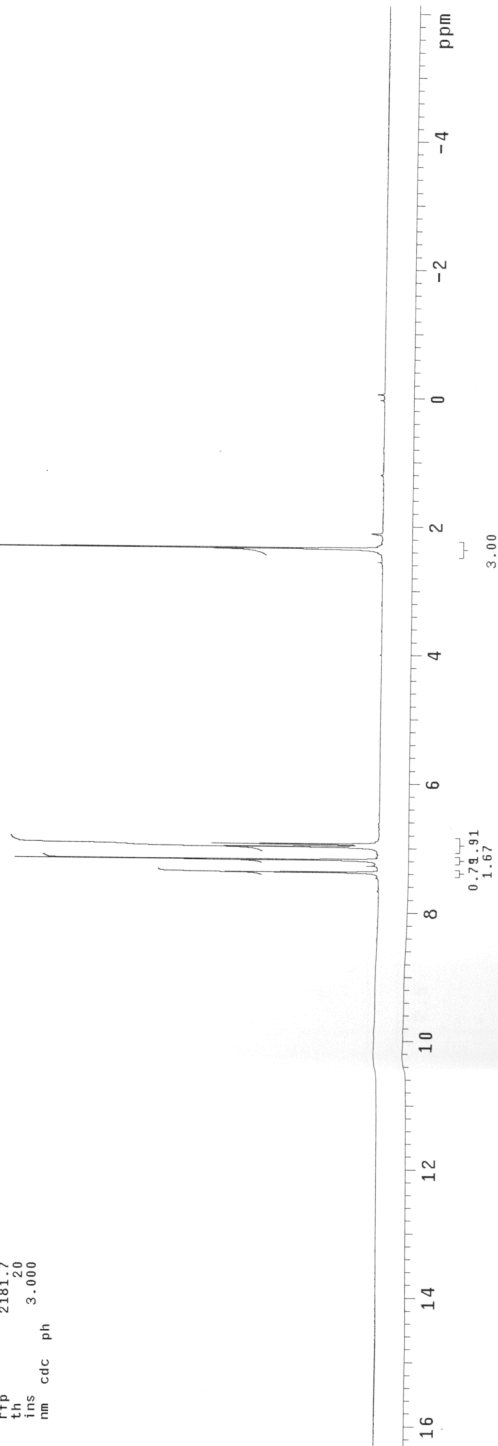
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 l1306 n

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exp5 std1h

```

SAMPLE          DEC. & VT
date    Feb 28 2005  dfrq    300.100
solvent  CDC13      dn      H1
file    /data/export/~ dpwr    30
home    /s/libuch/ara/mk~ dof    0
nat     /ra022806-rip2~ dm    nnn
          dm          dmf
ACQUISITION .fid
sfrq    300.100  wf file    C
tn      1.997   proc      not used
np      26982  fn
fw      6756.0 werr
bs      not usd wexp
tpwr    54     wbsxp
pw      7.0   wnt
d1      1.000
tof     0
ct      16
ct      16
alock   n
gain    not used
          FLAGS
ll      n
ln      n
dp      y
          DISPLAY
SP      -1841.7
WP      6756.3
VS      151
SC      0
WC      250
Hzmm    27.03
SFL     4023.8
rfl     2181.7
th      20
ins     3.000
nm      cdc  ph
    
```

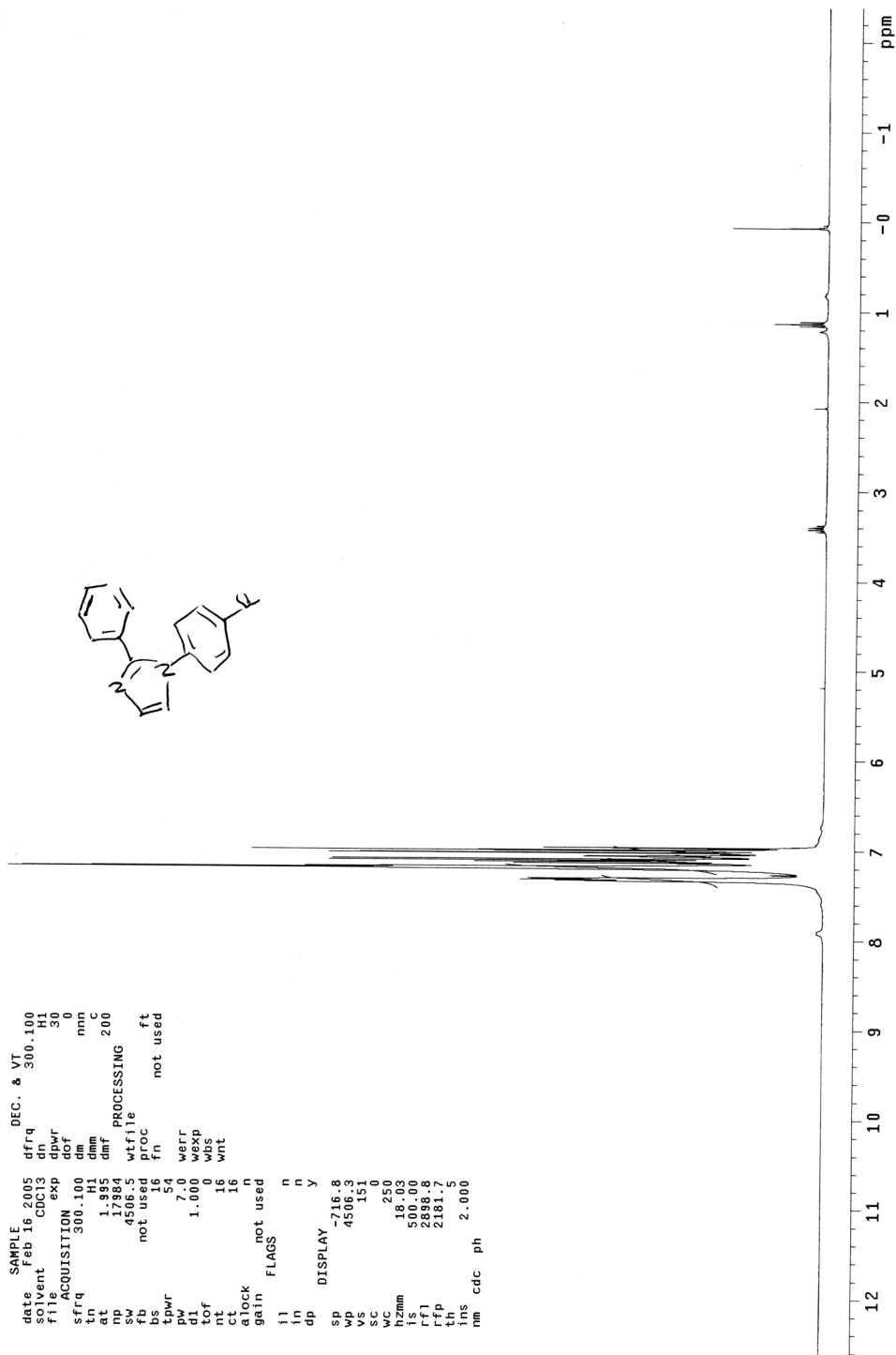
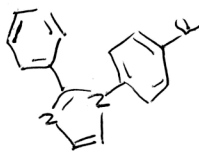


Ryan Altman II p 236

```

exp5 stdih
SAMPLE
date Feb 16 2005 dfrq DEC. & VT 300.100
solvent CDC13 dn H1
file ACQUISITION exp dpwr 30
sfrq 300.100 dm nnn
acq 1.91 dmm
nt 17984 dmr PROCESSING 200
sw 4508.5 wtfile
fb not used proc ft
bs 16 fn not used
tpwr 54
pv 7.0 werr
pw 1.000 wexp
tof 16 wnt
nt 16
ct 16
alock n
gain not used
il n
in n
dp n
DISPLAY
sp -716.8
wp 4506.3
xs 151
vc 0
hzmm 250
is 18.03
rfl 500.00
rfp 2898.8
lrc 2181.7
lrs 5
lms cdc ph 2.000
mm

```



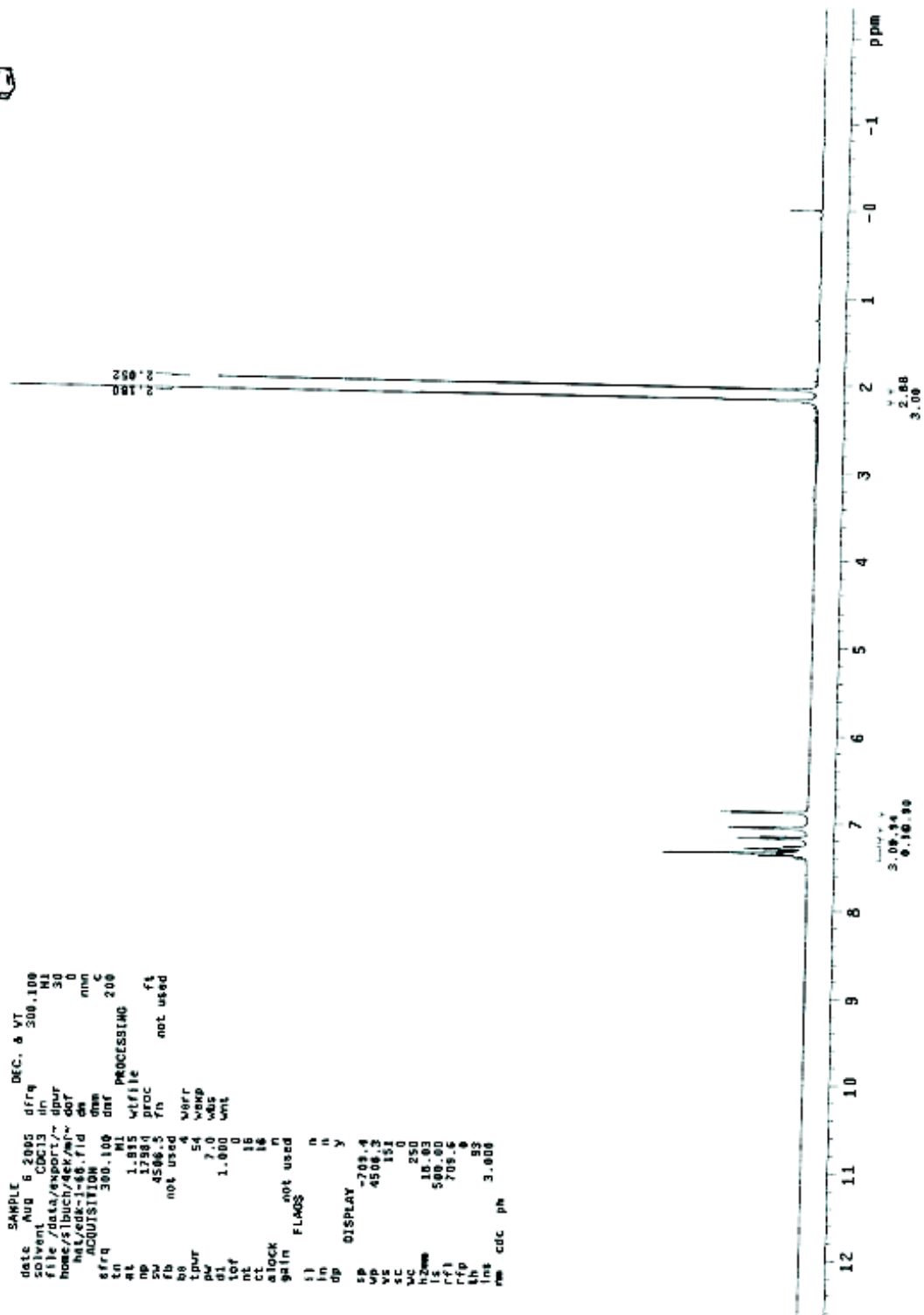
2.0010276
3.01260



STANDARD III OBSERVE

```

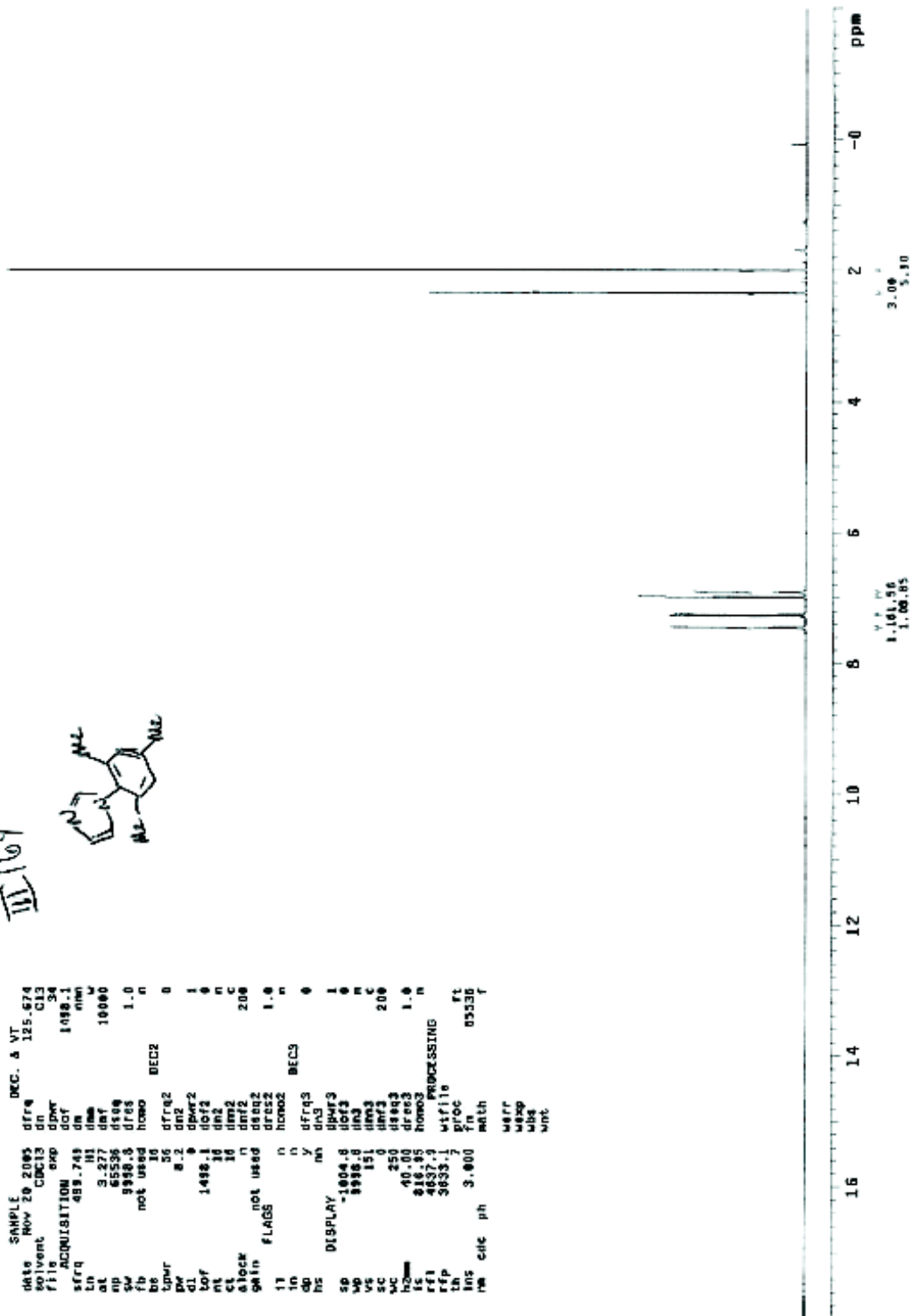
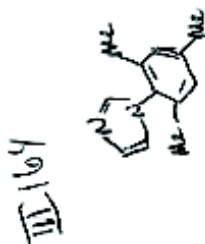
expl stdsh
SAMPLE DEC. & VT
date Aug 6 2005 dfrq 300.100
solvent CDC13 in MI
file /data/report/~ dpr 30
name /data/ack/~ dpr 0
hit /data/ack/~ dpr 0
hit ACQUISITION.FID on
ACQUISITION.FID on
sfrq 300.100 dnm 200
in MI PROCESsing C
rl 1.815 wfile
np 17884 proc fl
sv 4568.5 fh not used
bb not used 4 wpr
tpur 54 wkp
pr 7.0 wbs
vl 1.000 wnt
ntf 0
ct 16
at 16
gain not used
FLMOS
ii n
in n
dp y
DISPLAY -709.6
wv 4508.3
ve 15
sc 15
wc 250
hzmm 10.03
ls 500.00
rfi 709.6
rfp 0
lh 53
ms cdc ph 3.000
  
```



STANDARD PROTON PARAMETERS

```

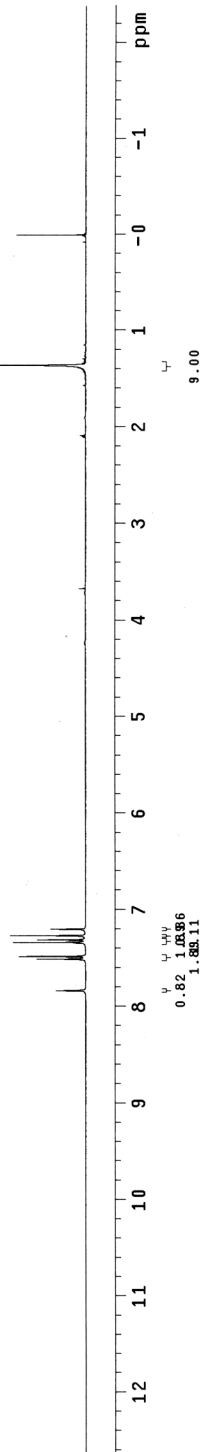
exp1 42jul1
SAMPLE DEC. & VT
date Row 20 2085 dfrq 125.674
solvent CBC13 dn C13
file exp dpr 34
ACQUISITION exp dpr 1498.1
sfrq 489.749 dn
ln 31 dm
at 225 dfr
sp 988.8 dfrs
fb not used homo 1.0
br 36 DECD
tpr 56 dfr2 0
pw 8.2 dn2 0
dl 9 dpr2 1
tor 1498.1 dfr2 0
nt 30 dn2 n
ct 30 dn2 n
clock not used dpr2 200
sh th not used dpr2 1.0
FLAGS n homo2 BECS
ln 0 dfr3
dn 0 dn3
hs nh dn3 0
DISPLAY dpr3 1
sp -1004.8 dfr3 0
wp 9916.8 dn3 n
rs 151 dn3 200
mc 250 dn33
h2mm 40.00 dfr3 1.0
rs 468.95 homo3 n
rfr 4637.9 PROCESSING n
rff 3833.7 wfile
lns 3.000 fnc 0530 f
rm cdc ph math
warr
wasp
wbs
wnt
  
```





```

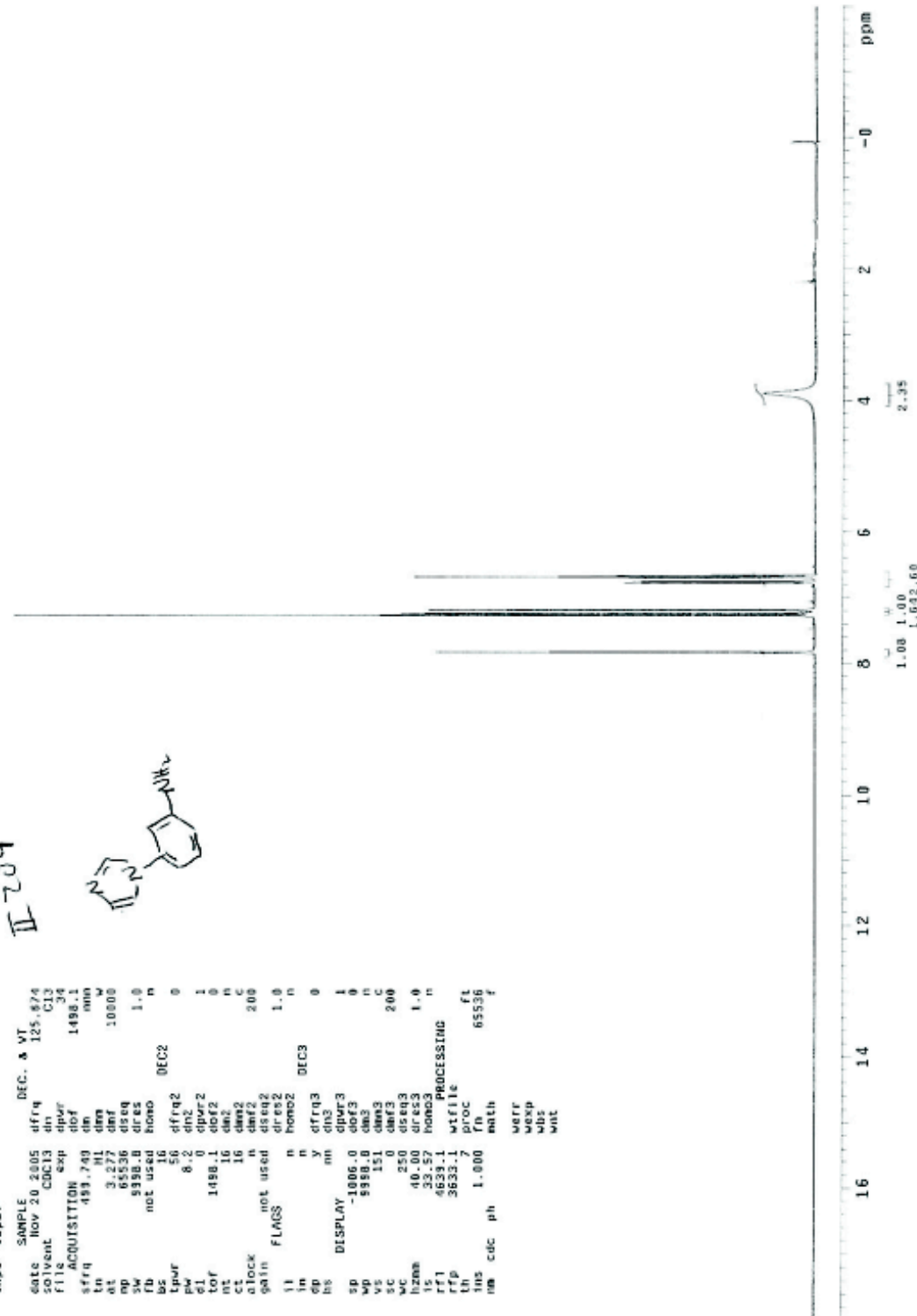
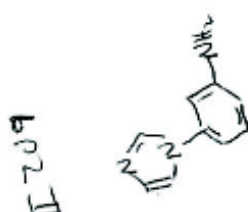
Ryan Altman
exp2 stdih
SAMPLE 8 2004 DEC. & VT
date Dec CDC13 dfrq 300.100
solvent CHCl3 dn 30
file /data/export/~ dpwr 0
home/sibuch/4ra/mr~ dof 0
hat/4ra-1-lm-4-tbu~ dm nnn
-benzene.fid dnm C
ACQUISITION dnr PROCESSING 200
sfrq 300.100 wtfile
tn q 1.995 proc ft
at 17984 fn not used
np 4506.5
sw not used werr
fb exp
ps 54
dpwr 7.0 wnt
d1 1.000
tof 0
nt 16
ct 16
a lock
gain not used
ll in n
in in y
dp DISPLAY 716.7
sp 4506.2
wp 167
SC 0
WC 250
hzmm 18.03
ls 2092.29
rfi 2886.7
tff 2181.20
ttr
ins 9.000
nm cdc ph
  
```



STANDARD PROTON PARAMETERS

```

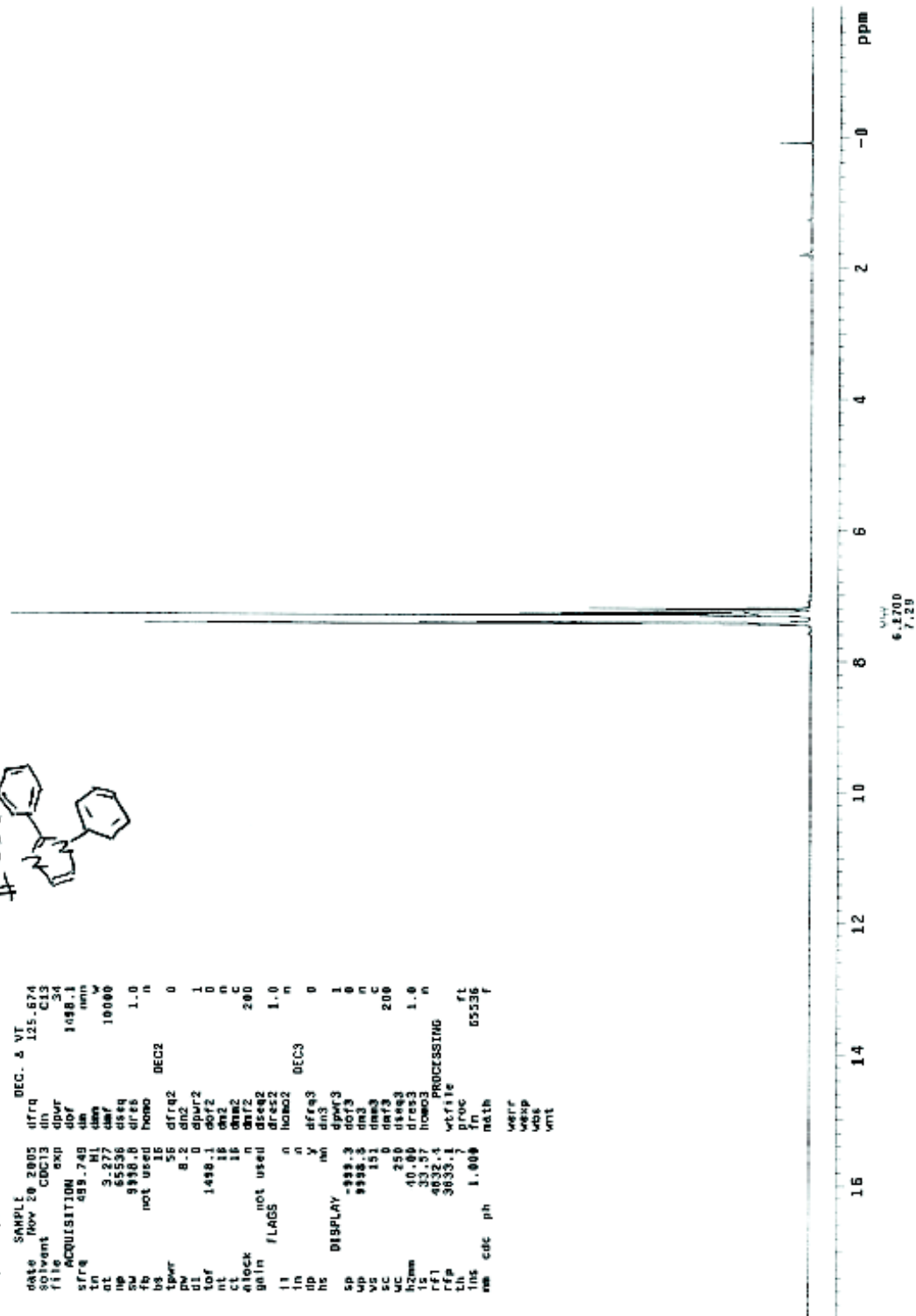
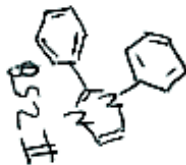
exp1 12pul
SAMPLE
date Nov 20 2005 DEC. & VT
solvent CDC13 dn 125.874
file ACQUISITION exp dprf 34
sfrq 499.749 dn 1498.1
ln ml dmw 10000
at 3.277 dmf
sp 6536 dseq
fb not used dres 1.0
bc 16 homo DEC2
Lpvr 56 dfrq2 0
pw 8.2 dn2
d1 0 dprf2 1
tof 1498.1 ddf2 0
nt 16 dm2
ct 16 dmw2 C
atlock not used ddf2 200
gain not used dres2 1.0
FLAG n homo2 DEC3
ln n dfrq3 0
gp y dn3
hs DISPLAY nm dprf3 1
sp -1006.0 ddf3 0
wp 5988.0 dm3
vs 151 dmw3 C
vc 250 ddf2
hzam 40.00 ddf3 1.0
rs 33.57 homo3
rf1 4639.1 wifile
rfp 3633.1 proc
th 1.000 Fh 65536
ms cdc ph anth
      usff
      wexp
      wbs
      wnt
  
```



STANDARD PROTON PARAMETERS

```

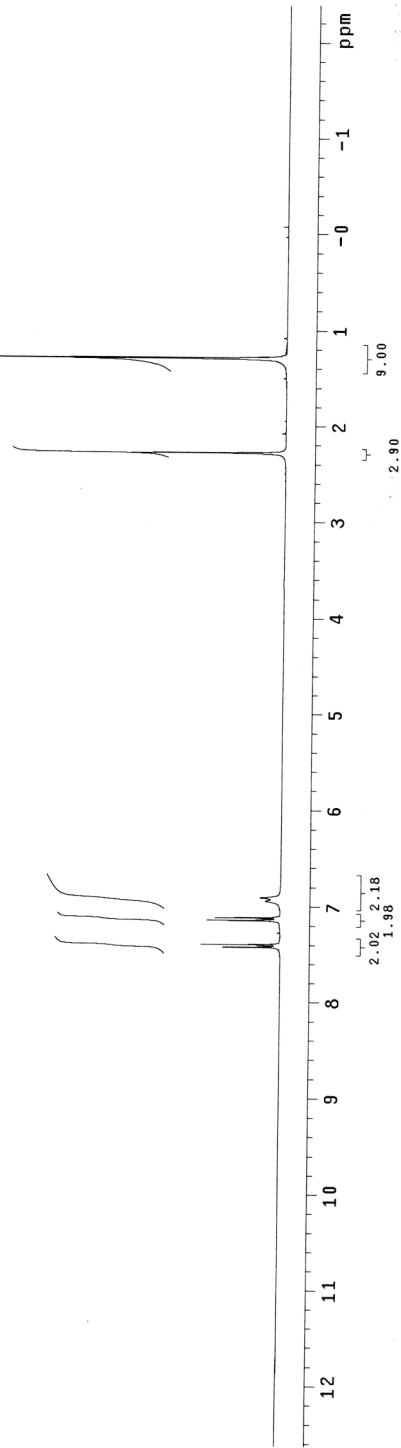
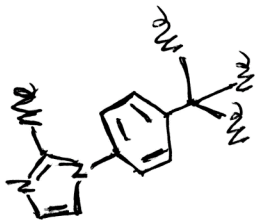
exp1 s2ju1
SAMPLE DEC. & VI
date Nov 20 2005 155-674
solvent CDC13 dn C13
file exp dprf 34
ACQUISITION exp dprf 1098.1
sfrq 499.749 uh 10000
in n1
dt 2.227 dar
sp 9888 dreq
fb not used nms 1.0
bs not used nms DEC2 n
tpwr 56 dfrq2 0
pw 8.2 dn2 1
d1 0 dprf2 1
lor 1498.1 dorf2 0
nt 18 dn2 n
ct 18 dn2 n
dlock not used nms 200
gn in not used dreq2 1.0
FLAGS n hnm22 DEC3 n
in n dfrq3 0
dp y dn3
hs nm dn3
DISPLAY dprf3 1
sp -999.3 rorf3 0
wp 9398.3 dn3 n
xs 151 dnms3 C
vc 250 dnrs3 200
h2nm 40.00 dnrs3 1.0
ls 33.37 lcom3 n
rf1 4832.4 wtfile
rfp 3833.7 proc
ins em cdc ph 1.009 in 65536 f
          usrf
          wexp
          vds
          vmt
  
```



Ryan Altman II p245

```

exp5 stdih
SAMPLE DEC. & VT
date Feb 23 2005 dfrq 300.100
solvent CDCl3 d1
file CDCl3 exp d1
ACQUISITION exp 30
sfrq 300.100 dm nnn
tn H1 dmm
at 1.995 dmf C
sw 45065 wtf file
fb not used PROCESsing
bs 16 ft
tpwr 54 fn not used
pw 7.0 werr
d1 1.000 wexp
ntf 10 wbs
ct 10 wnt
gain not used
alock n
gain not used
FLAGS n
l1 n
l2 n
dp n
sp -717.1 y
wp 4506.3
vs 151
sc 0
Hz 250
Hz/mm 18
is 500.00
rf1 2899.1
rfp 2181.7
th 20
ins 9.000
nm cdc ph
    
```

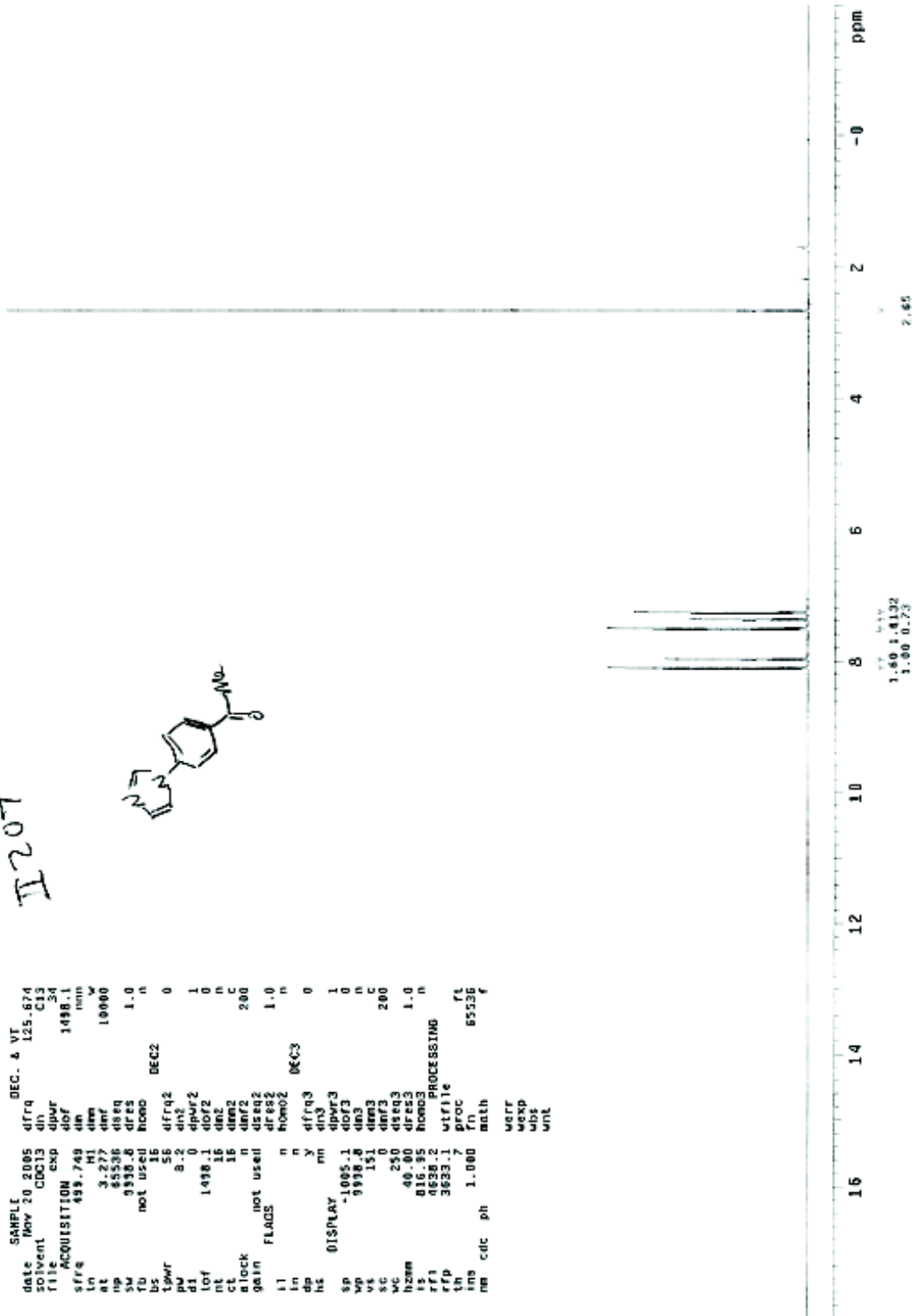
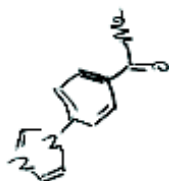


STANDARD PROTON PARAMETERS

```

exp1 02pm1
SAMPLE
date Nov 20 2005 DEC. & VT
solvent CDC13 d1h 125.674
file 34 d1h 013
ACQUISITION exp 1498.1 d1h 34
sffrq 499.749 d1h 1498.1
ln h1 dnm 10000
at 3.277 dnm
sp 9998.8 dnm
T0 not used NMR 1.0
bs 16 dnm DEC2
LWRT 56 dfrq2 0
pw 8.2 d1h2 0
d1 0 d1h2 1
lof 1498.1 d1h2 0
nt 16 d1h2 n
ct 16 dnm2 C
d1ock not used d1h2 200
gain not used d1h2 1.0
FLAGE n hnm02 DEC3
l1 n dfrq3 0
da y d1h3 0
hs nm d1h3 1
DISPLAY nm d1h3 0
sp -1095.1 d1h3 0
vp 9998.8 d1h3 n
vt 151 dnm3 C
vc 250 dnm3 200
hzam 46.80 d1h3 1.0
is 016.95 hnm03
rfi 0628.2
rff 3623.1 wifile
th 7 proc
ms cdc ph 1.000 fn 85236
math
warr
wexp
wbs
wnt
    
```

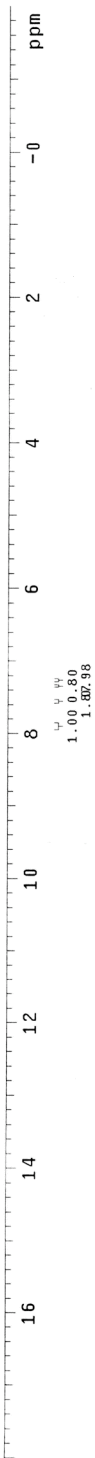
I207

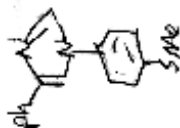




```

RAA III 259
exp1 s2pu1
SAMPLE
date Sep 24 2005 dfrq DEC. & VT 125.674
solvent CDC13 dn C13
file exp dpwr 34
ACQUISITION dof 1498.1
sfrq 499.749 dm nnn
at 3.271 dnm 10000
np 65536 dseq 1.0
sw 9998.8 dres 1.0
fb not used homo n
bs 16
tpwr 56 dfrq2 0
pw 8.2 dnc 1
dfr 1498.1 dfr2 0
nt 16 dm2 0
ct 16 dnm2 C
alock gain n dmf2 200
gain not used dseq2 1.0
FLAGS n homo2 n
il n homo3 0
in y dfrq3 0
dp in nn dpwr3 1
hs DISPLAY -899.3 dof3 0
sp wp 9998.8 dms 0
sc 15.0 dmr3 C
wc 250 dseq3 200
hzmm 40.00 dres3 1.0
is 33.57 homo3 n
rf1 4632.4 wtfile
tfp 3633.1 proc
ins 1.000 math 65536
nm cdc ph werr
wexp
wbs
wnt
    
```

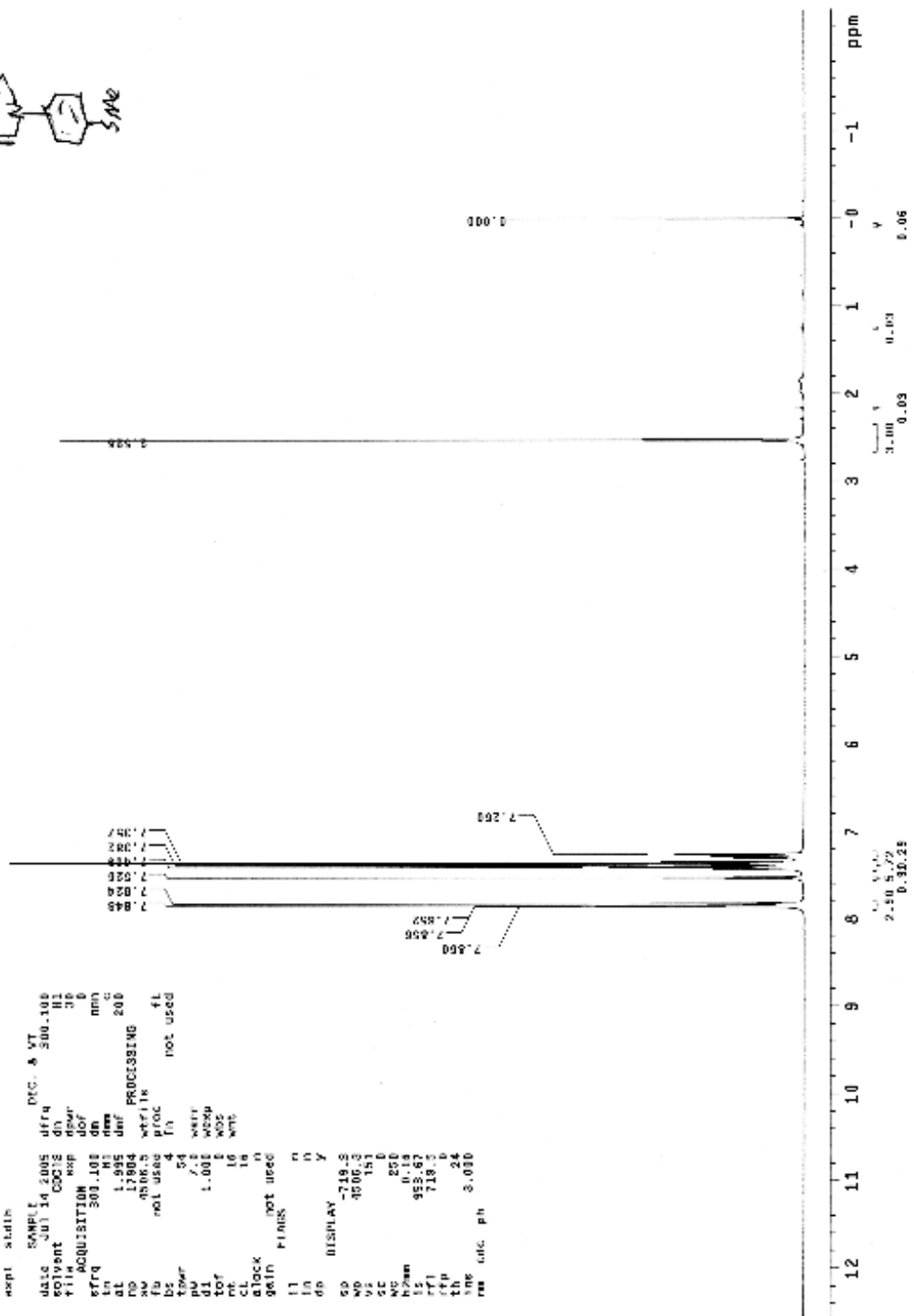


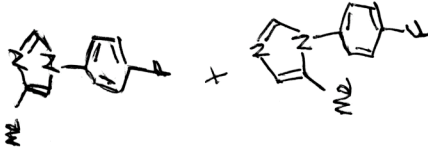


STARBOARD IN UNSERVY

```

expt statin
NAMEI
date JUL 14 2005 DEC. 8 VT
solvent CDCl3 dn dfrq 300.100
filn exp dnmr 30
ACQUISITION exp dn dnmr 300.100
an 1.000 raw
pb 1.000 dmf PROCESSING 200
sv dm00.0 wxtlik
fu mol used proc fl
bs 4 fn not used
tpr 54
pu 7.000 wxt
pv 1.000 exp
tpf 1.000 proc
mt 16 wxt
cl 16
alack n
gain not used
ll f1ms n
l0 n
l1 n
dp DISPLAY Y
gp -718.8
wp 4500.3
vz 15.1
nc 250
h2am 0.14
s5 953.67
rf1 718.7
cfp 0
th 0.24
re calc ph 3.010
    
```

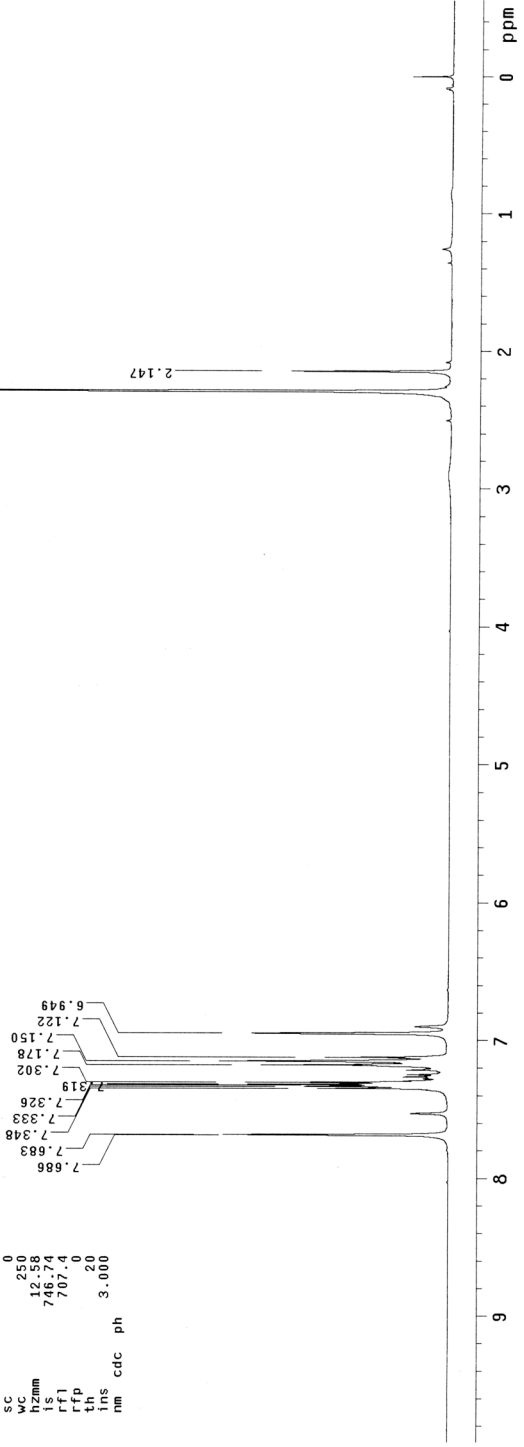


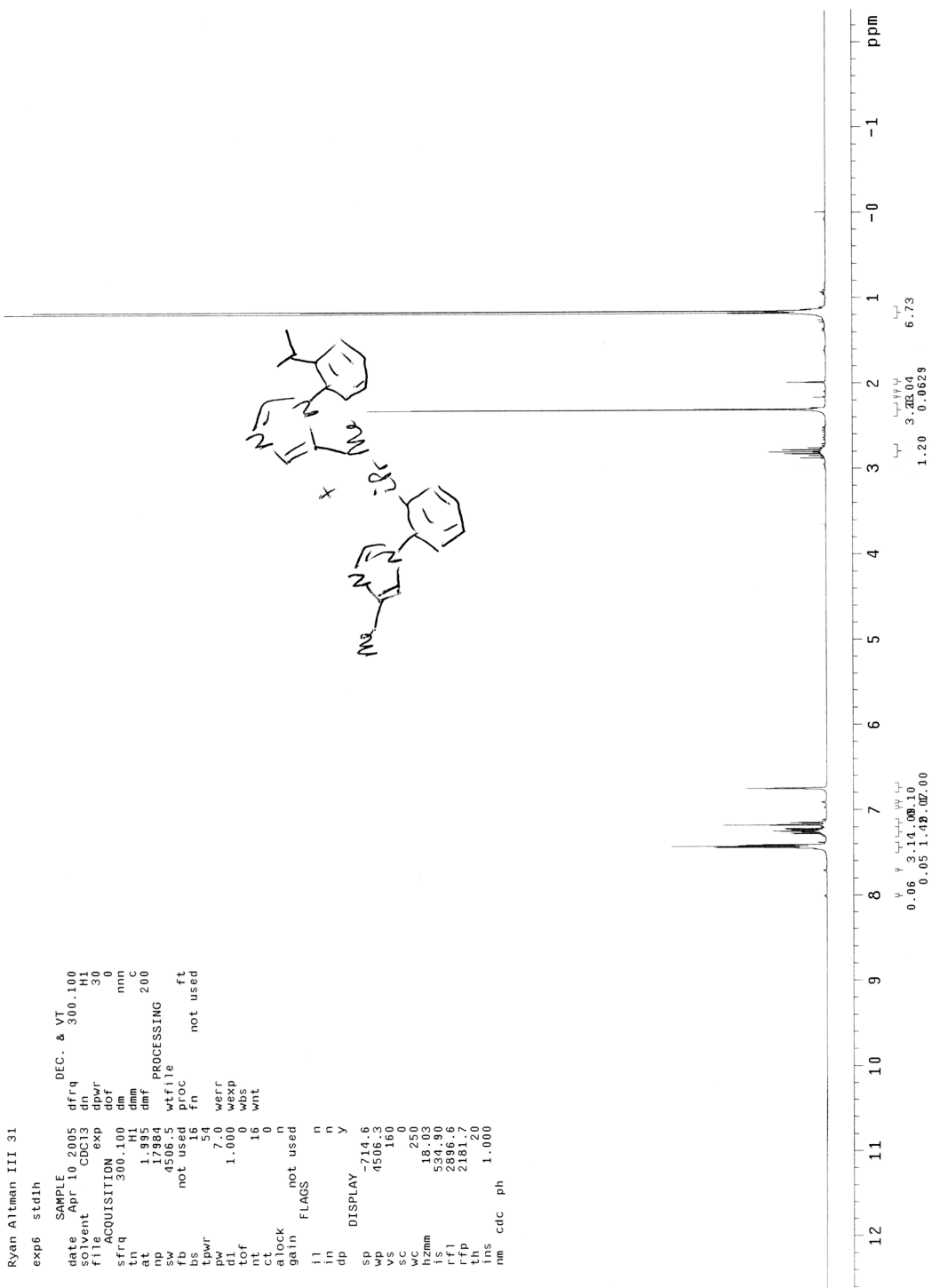


STANDARD 1H OBSERVE

```

exp1 stdih
SAMPLE
date Jul 21 2005 DEC. & VT
solvent C6Cl3 dfrq 300.100
file /data/export/~dowr do 30
home/slbuch/4ek/mr~ dof 0
hat/edk-1-56.fid dm nnn
ACQUISITION dmm
$frq 300.100 dmf PROCESSING 200
at 1.995 wifile
np 17984 proc ft
sw 4506.5 fn not used
bs not used
tpwr 16 werr
dw 54 wexp
ds 70 wss
dt 1.000 wnt
nt 0
ct 16
alock 0
gain not used
flags n
il n
in n
dp DISPLAY
sp -168.3
wp 3144.2
sc 1.1
wc 250
hzmm 12.58
rfl 746.74
tff 707.4
tfs 0
ins 0
nm cdc ph 3.000
  
```



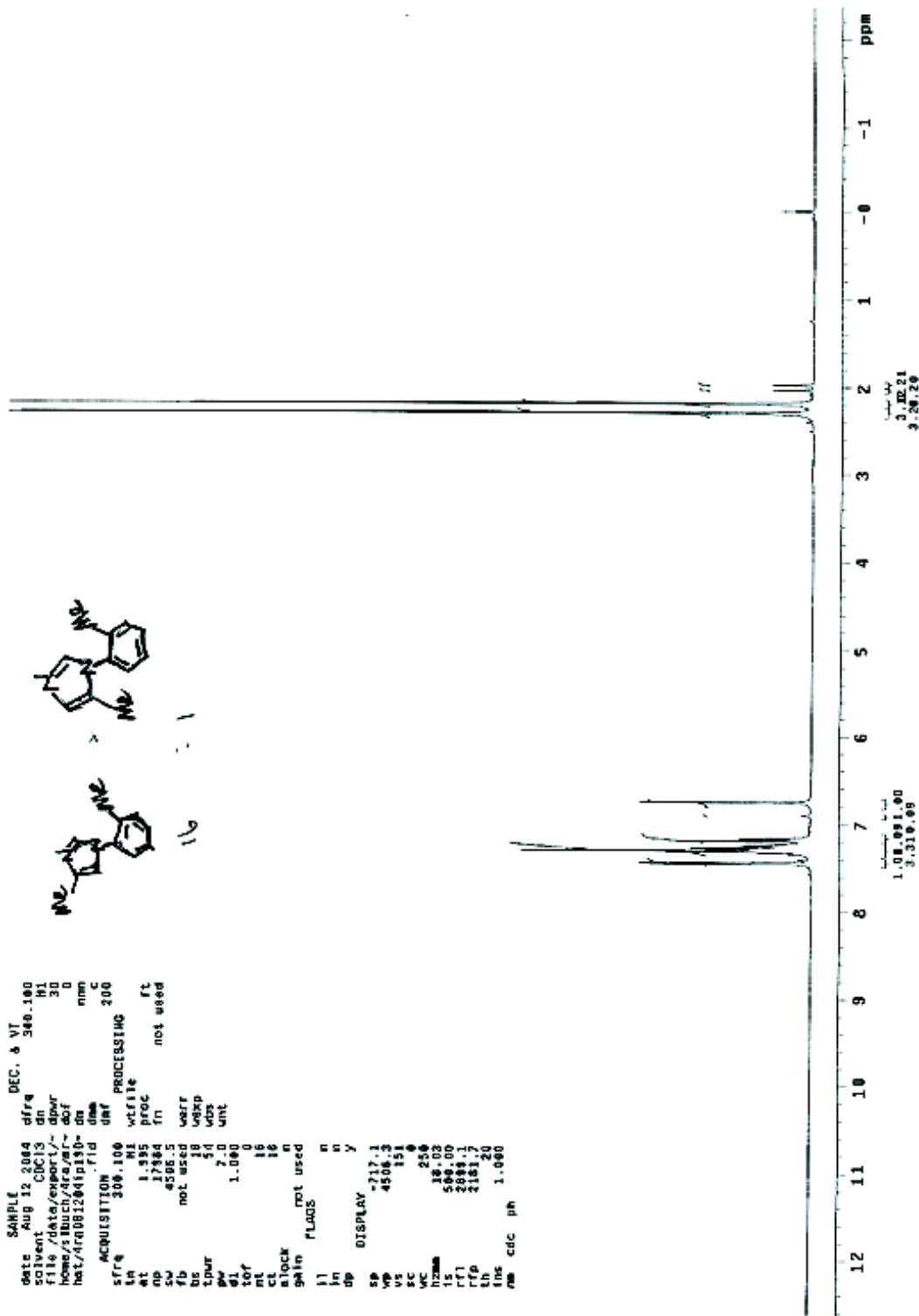


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exp2 stadih

```

SAMPLE          DEC. & VT
date Aug 12 2004 dfrq 300.100
solvent CDCl3 dn
file /data/exp01/- dpar 30
name /118117/ dfr 0
nat/4r0012/1p10- dm mm
ACQUISITION .fid dmw
sfrq 300.100 wtrfile
in 1.995 proc ft
ap 17884 fn not used
sw 4500.5
fb not used warr
bs 18 wexp
tpur 54 vbs
pw 7.0 wnt
ql 1.000
at 0
ct 10
cl 10
alock n
gain not used
flags
ll n
ln n
op n
SP -217.1
VS 4500.5
SC 15
VC 250
hzmax 10.03
ls 500.00
rf1 2000.1
rfp 4181.7
th 1.20
ms cdc ph 1.000
    
```

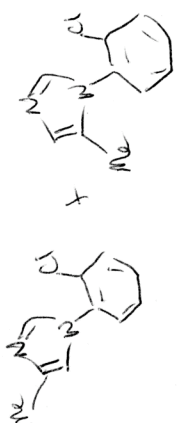


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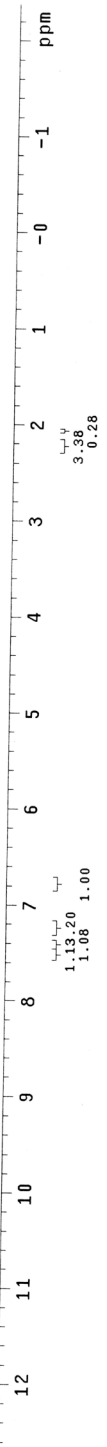
exp6 std1h

```

SAMPLE      8 2005      DEC. & VT
date        Mar      dfrq    300.100
solvent     CDC13     dn      30
file        exp      dpwr    0
ACQUISITION 300.100  dof     0
           300.100  dm      nnn
           1.995   dm      C
at          17984   dmf     PROCESSING 200
sw          4506.5  wf      file
fb          not used proc
tpwr       16      fn      not used
pw         7.0     werr   0
d1         1.000   wexp   16
nt         16     wbs    16
ct         16     wnt    16
alock      not used
gain       not used
il         n
in         n
dp         y
DISPLAY    -715.5
wp         450000
ve         151
sc         1.0
wc         250
hzmm      18.03
ls         500.00
rfi        2837.5
th         2101.20
ins        1.000
nm         cdc
ph
    
```



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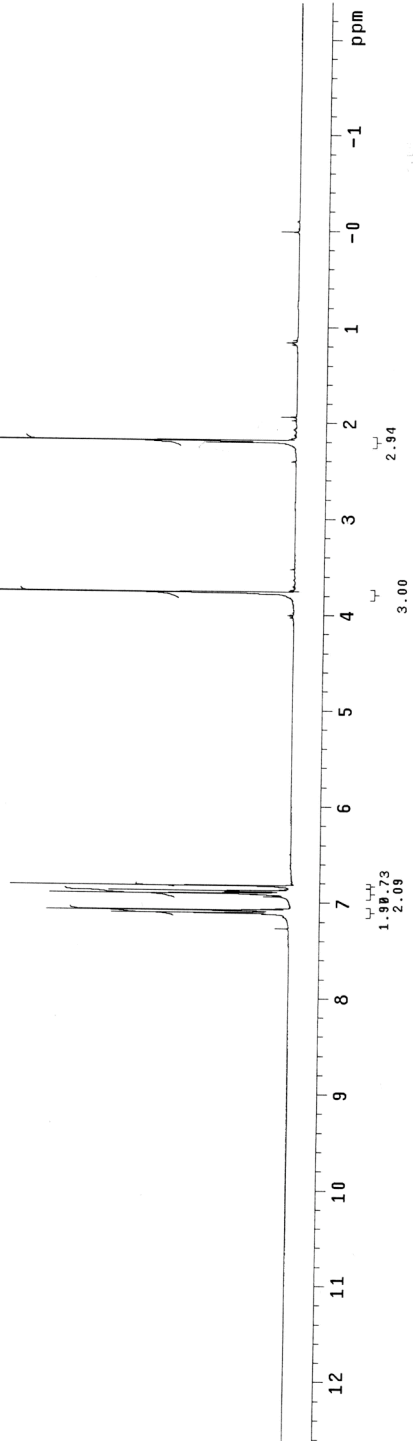
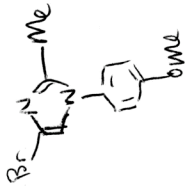


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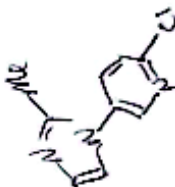
exp3 std1h

```

SAMPLE          DEC. & VT
date Jan 12 2005  gfrq 300.100
event CDC13      dn    H1
file (data/s1)  dn    30
4ra/4ra011205-II1~  dof  3
                    dmm  mn
                    dmm  c
ACQUISITION    dmf 200
sfrq 300.100    dmf 200
in      1       H1
ct      17884   wfile
nd      4506.5  proc
sw      not used  fn
fb      not used  werr
bs      16      wexp
tpwr    54      wbs
pw      7.0     wnt
rf      1.000   wnt
nt      16
ct      16
alock   not used  n
gain    not used  n
flags   n
in      n
dp      y
DISPLAY  -717.7
sp      4506.3
wp      151
vs      250
wc      18.03
hzmm    500.00
is      2899.7
rf1     2181.7
rfp     20
tms     3.000
mm      cdc  ph
    
```



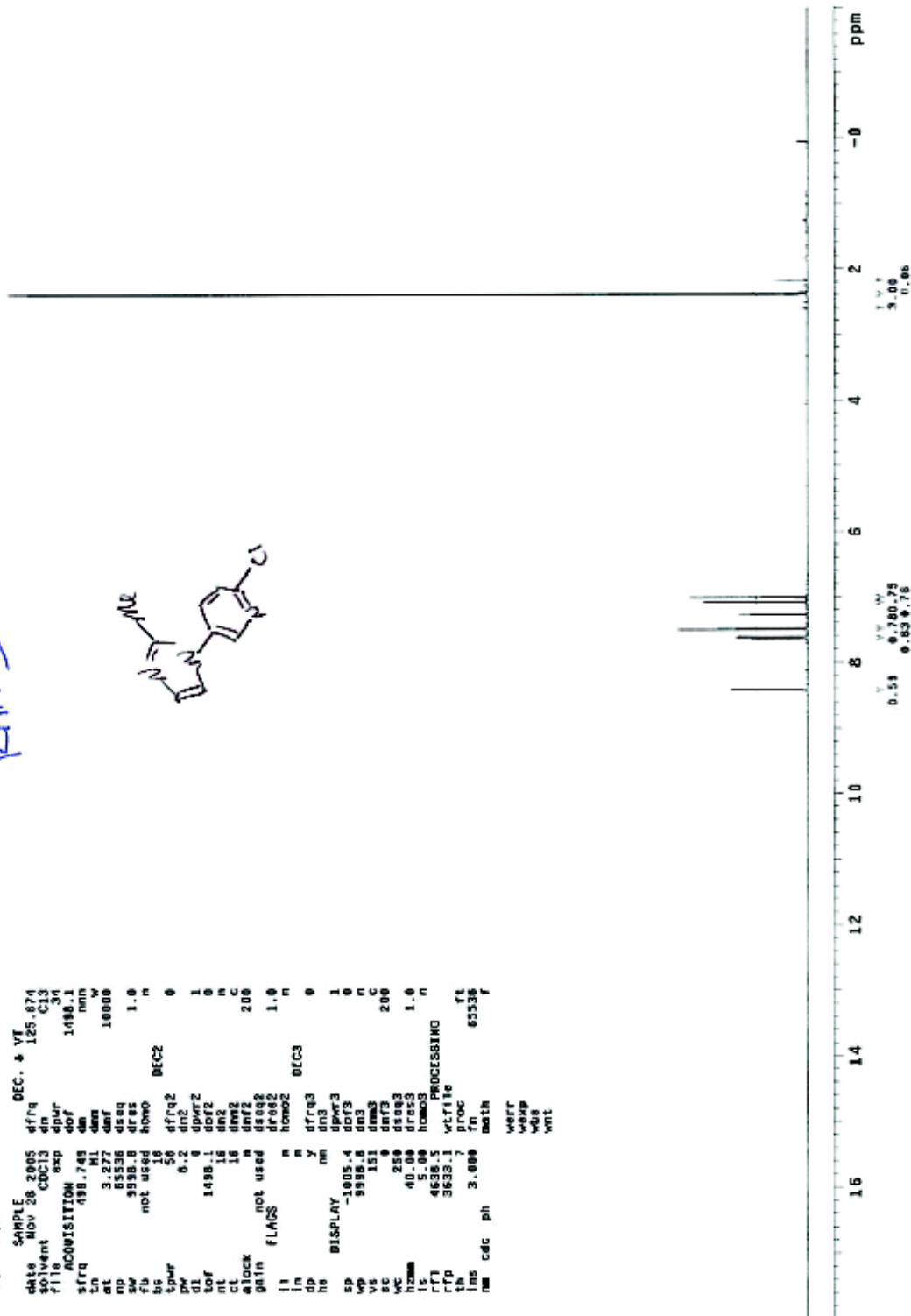
ZAA-III 95

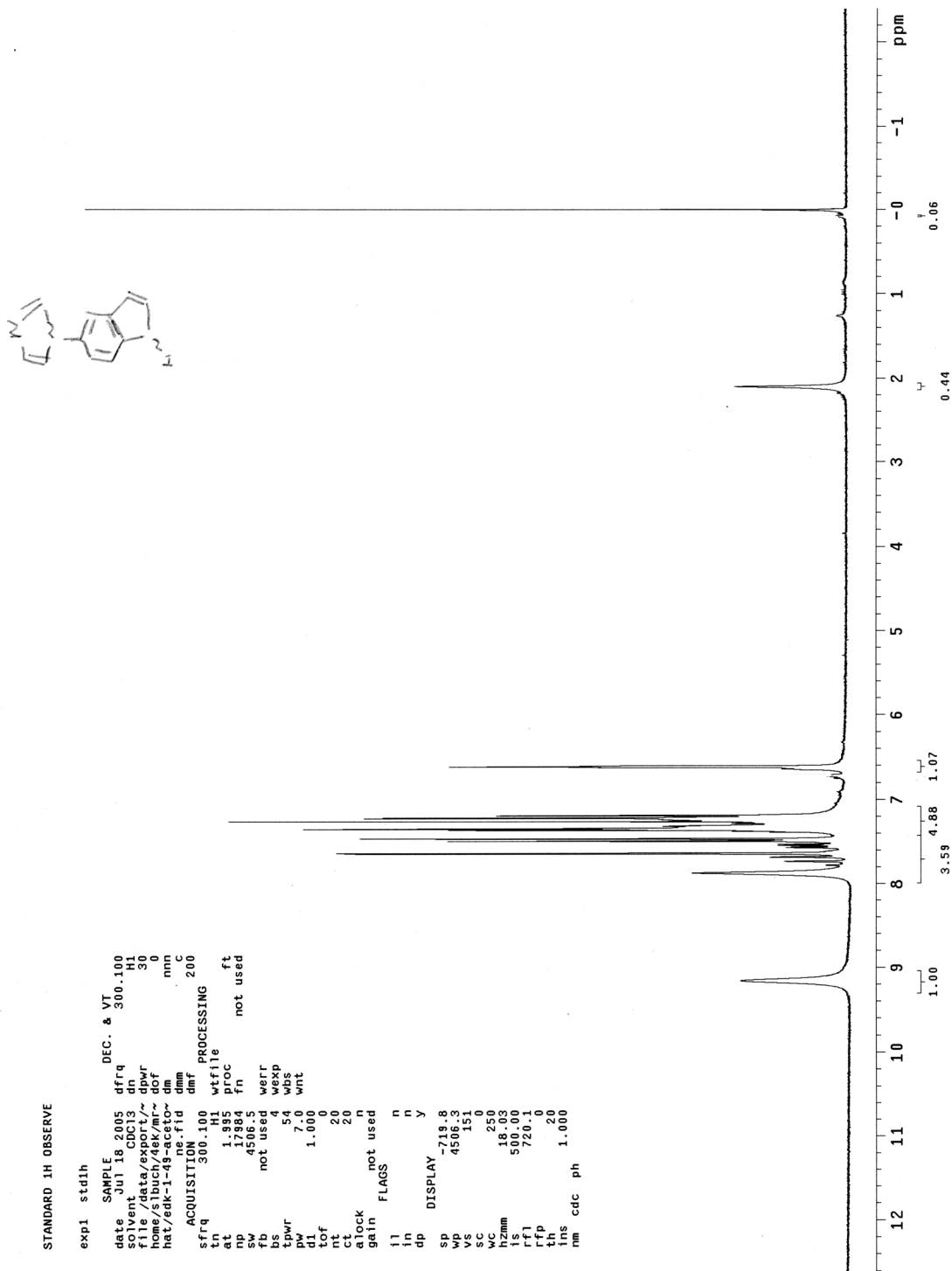


STANDARD PROTON PARAMETERS

```

exp1 52pu1
SAMPLE
date Nov 28 2005 DEC. 5 VT
solvent CDC13 d1 125.874
file exp 34
ACQUISITION
sfrq 498.743 dm 1488.1 nmh
ln M1 dmw 10000
at 3.277 dmf
np 8536 dsdq
fu 3338.8 dres 1.0
ts not used hore DECE
tpur 50 dfrq2 0
pw 6.2 dn2
d1 0 dpar2 1
tof 1488.1 dof2 0
ct 16 dn2
clock 16 dn2 200
dftn not used dsq2
flags dfo62 1.0
      homo2
      homo3 DECE
      y dfrq3 0
      nm dn3
      nm dpar3 1
DISPLAY
sp -1005.4 dof3 0
wp 9998.8 dn3
vs 151 dnms C
sc 258 dnf3 200
hzam 40.00 dres3 1.0
ls 5.00 homo3
rf1 4638.5 wtfile
rf2 3633.1 proc
lns cdc ph 3.000 math
nm cdc ph 3.000 math
      wdrf
      wdrp
      wdr
      wdt
  
```



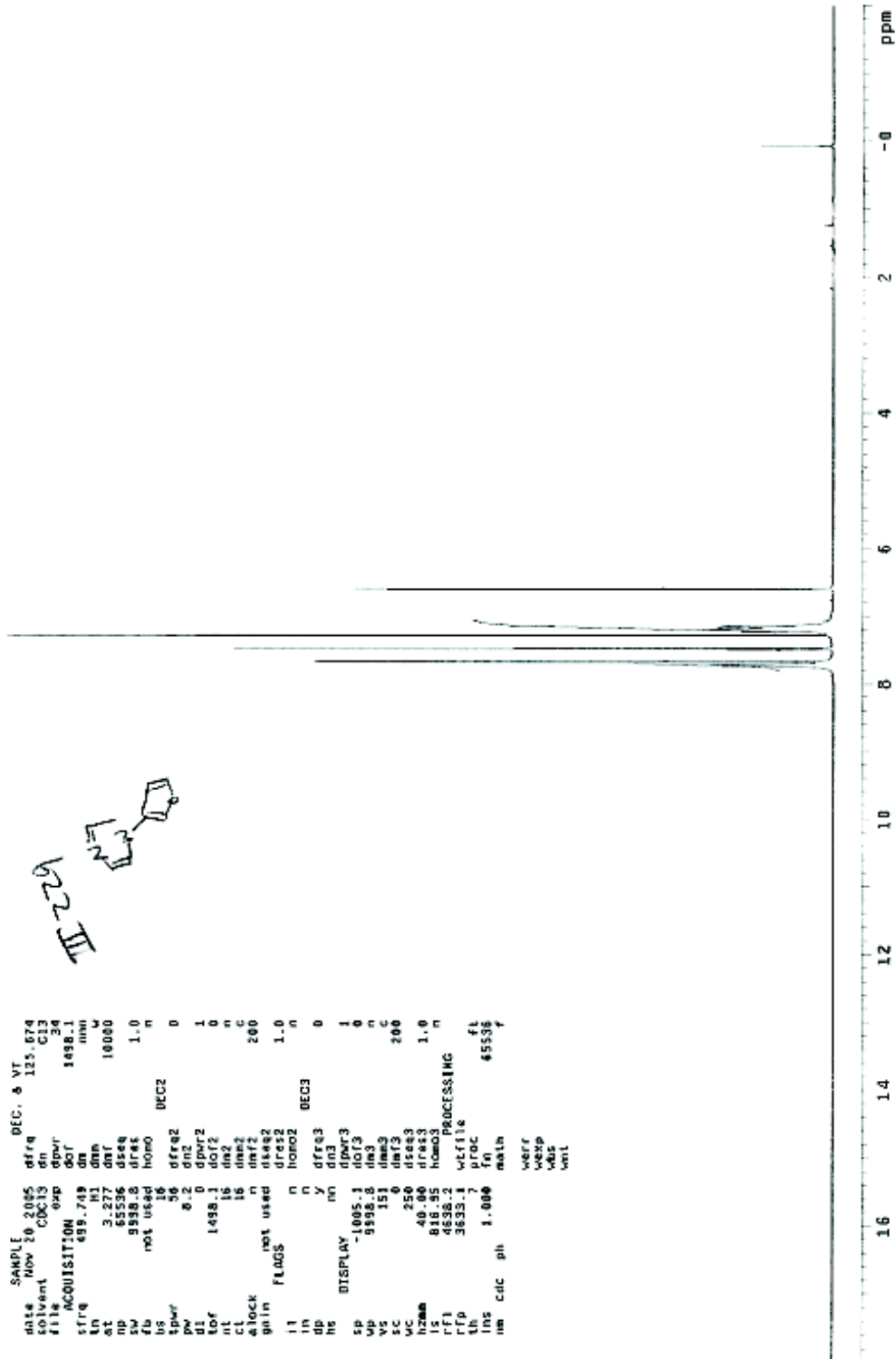


STANDARD PROTON PARAMETERS

```

exp1 s2pu1
SAMPLE
date Nov 20 2085 DEC. 8 VT
file CBC13 dn C13
ACQUISITION exp 34
sfreq 499.749 dn 1498.1
ln n1 dnw 10000
at 3.277 dnf
np 8558 useq
pw 8388.3 freq 1.0
hs not used homo 0K2
ls 56 dfrs2 0
pw 0.2 dn2 dpr2 1
dl 1498.1 dof2 0
nt 16 dne2 n
cl 16 dne2 c
dlock not used dne2 200
gulin FLAGS
l1 n homo2 1.0
l2 n dfrs3 0
dp Y dn3
hs DISPLAY no dpr3 1
sp -1005.1 dof3 0
wp 5998.8 dne3 n
vs 151 dne3 c
vc 250 dne3 200
hzam 40 dn2 dfrs3 1.0
ls 818.95 homo3 n
rf1 4658.2 PROCESSING
rfp 3633.1 wtfl1
th 7 proc fl
ims calc ph 1.000 fa 45536 y
          wdrf
          wdrp
          wdr
          wdr
  
```

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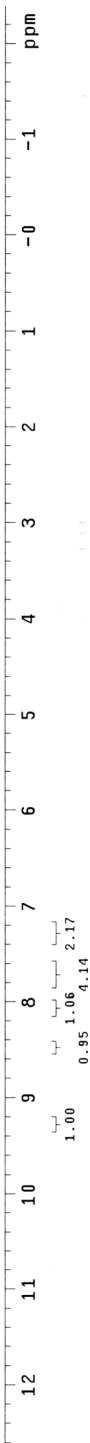


1.9939.05
1.18 66 1.13

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```

exp5 stdh
SAMPLE
date Feb 16 2005 dfrq DEC. & VT 300.100
solvent CDC13 dn H1
file exp dpwr 30
ACQUISITION exp dof 0
sfrq 300.100 dm nnn
tn H1 dmm
nt 1.984 dmm 200
sw 17884 dmf PROCESSING
fb not used wtfile ft
bs 16 fn not used
tpwr 54
pw 7.0 werr
t0f 1.000 wexp
nt 16 wnt
ct 16
alock not used n
gain FLAGS
il n
in n
dp y
DISPLAY
sp -716.8
wp 4506.3
vs 151
vc 250
hzm 18.03
is 500.00
rfl 2898.8
rfp 2181.7
th 20
ins 1.000
nm cdc ph
  
```

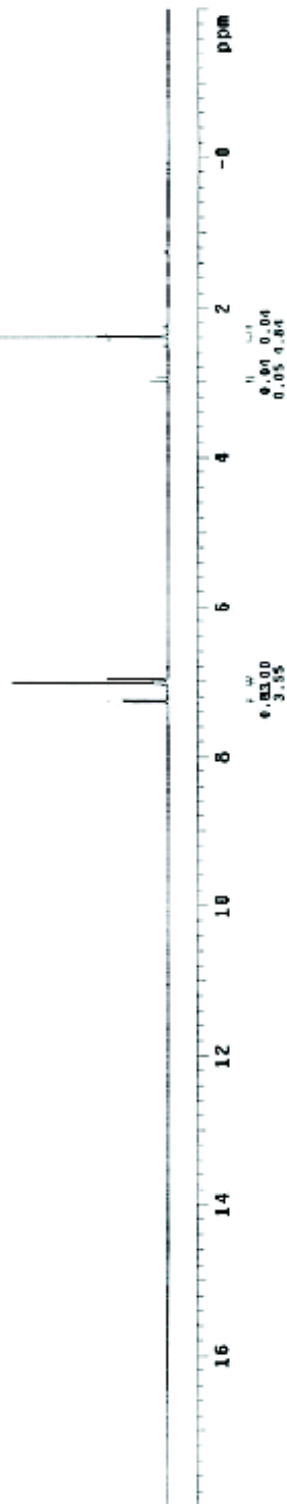
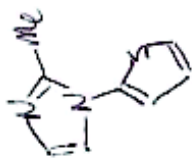


STANDARD PROTON PARAMETERS

```

exp1 $2pul
SAMPLE
date Nov 28 2005 DEC. & VT 123.674
solvent cdcl3 dn 123.674
file 34
ACQUISITION exp dpar 34
498.743 dn 1498.1
sfrq 498.743 dn 1498.1
ln 34 dn 10000
at 5.256 dnf
sp 1498.1 dnf
fb not used hno 1.0
br 16 hno DEC2
tpr 56 dfrq 0
pw 8.2 dn2
d1 0 dpar2 1
lor 1498.1 dnf2 0
ct 16 dn2
clock 16 dn2
gh in not used hno2 200
ii FLAGS in hno2 1.0
in in hno2 DEC3
dp y dfrq 0
hb dn3
DISPLAY dn3 dpar3 1
sp -1005.1 dnf3 0
ys 9998.0 dn3
xs 140 dn3
xc 250 dn3
hzam 40.00 dn33 1.0
ls 5.00 hno33
rf1 4638.2 vrf1le
rfp 3633.7 proc
ins 1.000 fn $5536
nm cdc ph math
veff
veamp
vds
vnt
  
```

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ZAA IV 64

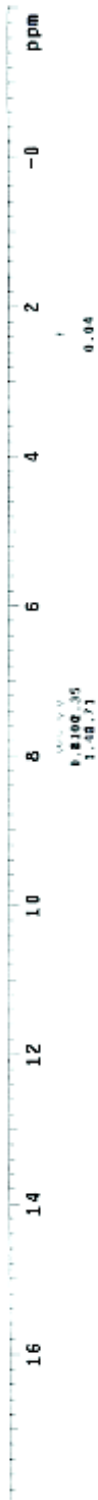


STANDARD PROTON PARAMETERS

```

exp1 52pul
SAMPLE
date NOV 28 2005 DEC. & VT 125.674
solvent CDCl3 dn C13
file 34
ACQUISITION exp 1488.1
sfrq 499.749 dn
ln H1
dt 3.277 msf
rg 933.8
rb not used PRES 1.0
ls 16 DECD
ls 16 DECD
tavr 56 dfrq2 0
pu 8.2 dn2
d1 0 dpar2 1
to1 1498.1 dorf2 0
rt 16 dms2 n
ct 16 dms2 c
dlock not used dorf2 200
gain not used dorf2 1.0
FLAG5 n ROR22 DECD
ln n ROR22 DECD
dp n y dfrq3 0
hs n dn3 dpar3 1
sp -1005.1 dorf3 0
vs 9338.8 dn3
vc 151 dn3
vc 250 dn3
hzam 40.00 dfrs3 1.0
ls 5.00 homo3
rf1 4038.2
rfp 3833.1 wfile
ln6 1.009 fn 65536
nm cdc ph math
worf
wexp
wds
wnt

```

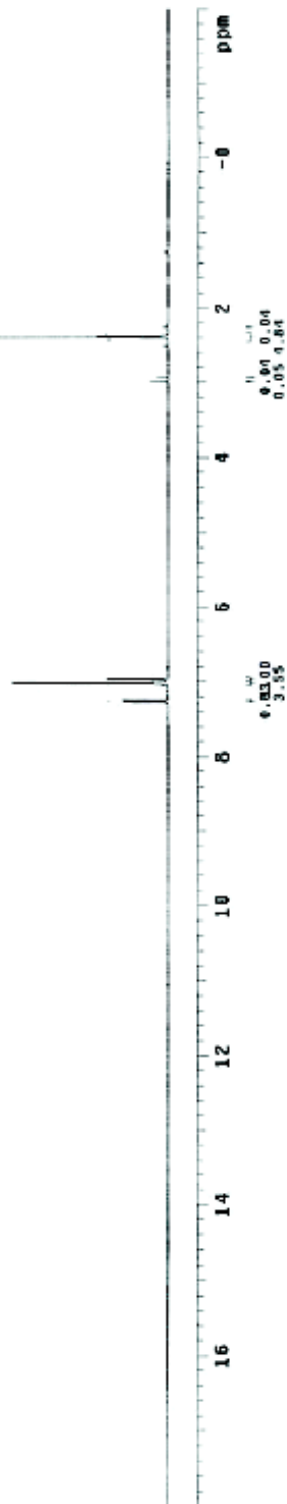
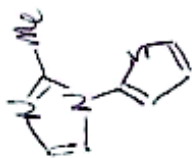


STANDARD PROTON PARAMETERS

```

exp1 $2pul
SAMPLE
date Nov 28 2005 DEC. 6 VT 123.674
solvent CDC13 dn 123.674
file exp dpmf 34
ACQUISITION exp dpmf 1498.1
sfrq 498.749 dn
ln 31 dms 10000
at 5.256 dmf
cp 3498.8 drcq
fb not used hrcs 1.0
br 16 hrcs
tprf 56 dfrq2 0
pw 8.2 dn2 0
d1 0 dpmf2 1
lor 1498.1 ddf2 0
cl 16 dn2 n
ct 16 dms2 200
atock not used hrc2 1.0
gh in hrc2
ii in hrcs2 1.0
in in hrcs3 0
dp y dfrq3 0
hb DISPLAY dn dn3 1
sp -1005.1 ddfs 0
ws 9998.8 dn3 n
xs 140 dms 200
xc 250 dms2
h2nm 40.00 dfrs3 1.0
ls 5.00 hrcs3 n
rf1 4638.2 vrf1file
rfp 3633.7 proc
ins 1.000 fn $5536
nm cdc ph
      lefff
      veemp
      vds
      unit
  
```

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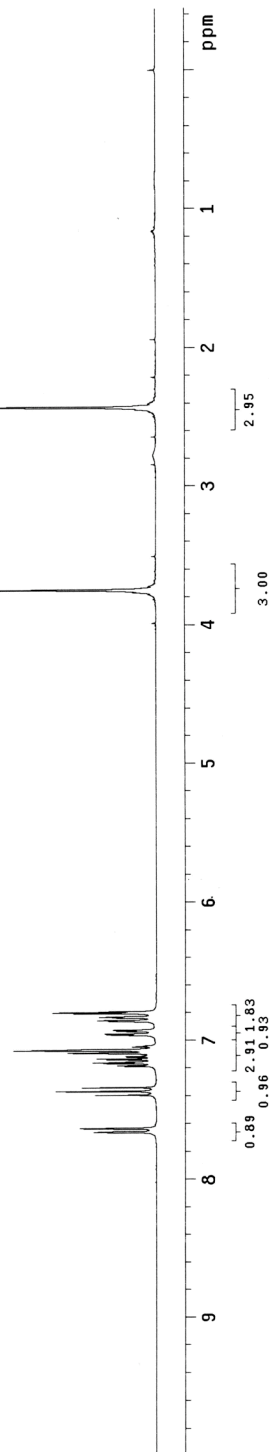
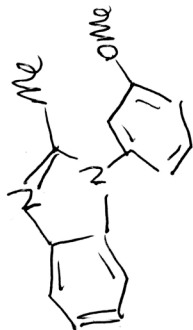


STANDARD 1H OBSERVE

```

exp1 stdih
SAMPLE
date      Jun 21, 2006      DEC. & VT
solvent   CDC13           dfrq    300.100
file      /data/slbucl3/~ dpwr    30
4ek/ek-1-146.fid  dof    0
ACQUISITION  dm      nnn
sfrq     300.100      dmm    200
ac       1.985        dmf    PROCESSING
np       17884        wtfil  e
sw       4506.5      proc   ft
fb       not used    fn     not used
bs       54          werr   4
tpwr     1.000       wexp   1.000
nt       16          wpr    1.000
ct       16          wnt    0
alock    not used   n
gain     not used   n
flags    n
in       n
dp       y
DISPLAY
sp       -133.7
wp       3132.3
vs       151
sc       250
h2mm     12.53
ls       500.00
rf1      743.5
rfp      0
tn       0
lms      0
nm       3.000
cdc      ph

```

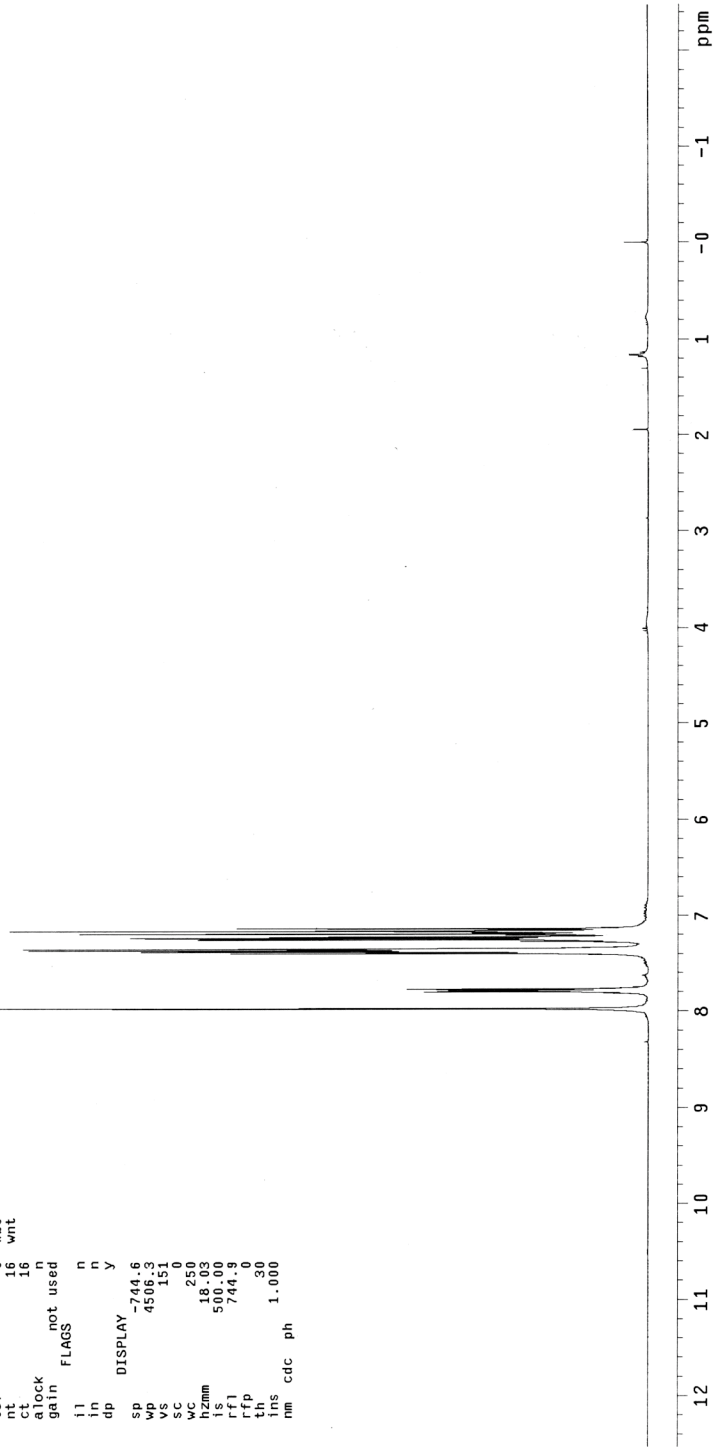
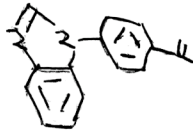




STANDARD 1H OBSERVE

```

exp1 stdih
SAMPLE
date Apr 11 2006 DEC. & VT
ent CD010 dfrq 300.100
file exp dpwr 30
ACQUISITION exp nnn
sfrq 300.100 dm
tn H1 dmm
at 1.995 dmf
np 457864 wf file
fb not used proc ft
bs not used 4 fn
tpwr 54 not used
pw 7.0 werr
d1 1.000 wexp
tof 0 wbs
ct 16 wnt
clock 16
gain in
not used
FLAGS
il n
in n
dp n y
SP -744.6
WP 4506.3
VS 151
SC 0
WC 250
hzmm 18.03
f1 500.00
rfp 744.70
th 30
ins 1.000
nm cdc ph
  
```

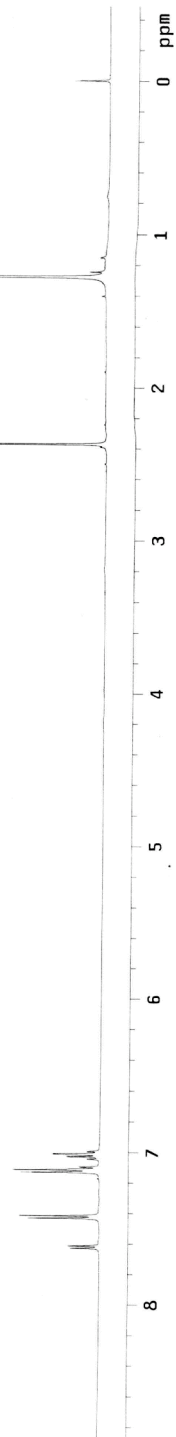
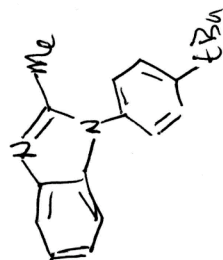


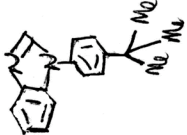
STANDARD PROTON PARAMETERS

```

exp1 s2pu1
SAMPLE
date Jul 25 2006 dfrq DEC. & VT 125.795
solvent CDCl3 dn C13 37
file exp dpwr 0
ACQUISITION 235 dof 0
sfrq 300.235 dnm nnn
tn 3.200 dmf 10000
at 64000 dseq
np 10000.0 dres 1.0
sw not used homo n
cs 4 PROCESSING
ss 4 wfile
tpwr 54 proc ft
pw 9.8 fn 131072
d1 0 math
tof 1498.2 werr
nt 16 wexp
ct 16 wexp
alock n was
gain not used wnt
FLAGS
il n
in n
dp y
hs nn
SP DISPLAY-244.9
WP 4691.1
VS 151
SC 0
WC 250
hzmm 18.76
f2 100.00
rfp 1046.5
th 7
ins 3.000
nm ph

```

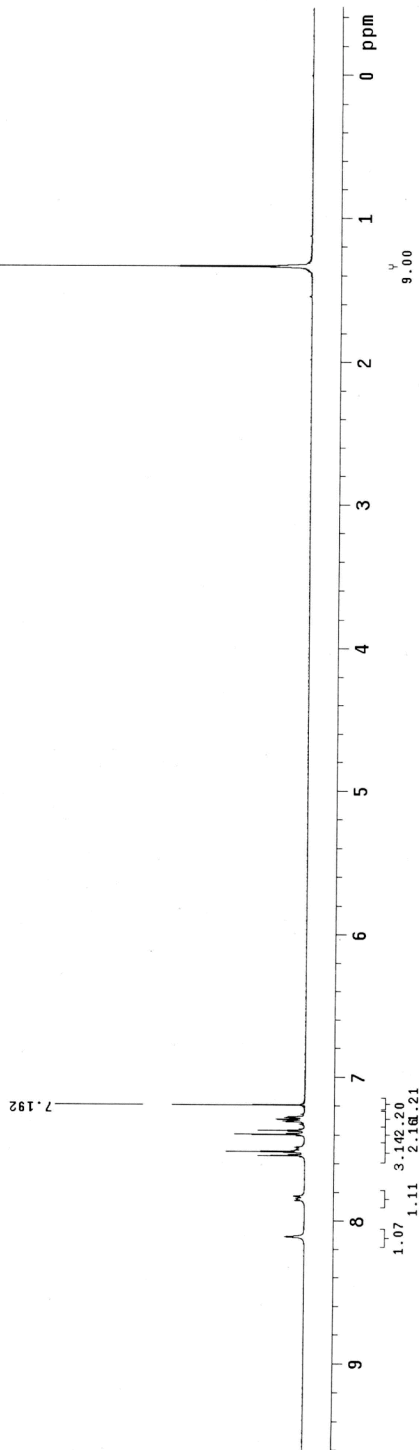


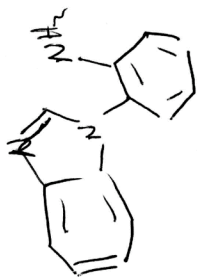


STANDARD 1H OBSERVE

```

exp1 std1h
SAMPLE
date Mar 13 2006 DEC. & VT
solvent CDC13 dfrq 300.100
file exp dpwr 30
ACQUISITION exp dcf 0
$frq 300.100 dm mnc
t 1.985 dmr
nt 17984 dmr PROCESSING 200
sw 4506.5 wtfile
fb not used proc ft
bs 4 fn not used
tpwr 54
w 750 werr
dt 1.060 exp
tof 0 whs
nt 16 wnt
ct 16
alock n
gain not used
il n
in n
dp n
DISPLAY
sp -141.9
wp 3025.6
vs 156
vc 250
h2mm 12.10
ls 570.00
rf1 740.2
rfp 0
th 20
ims cdc ph 9.000
  
```

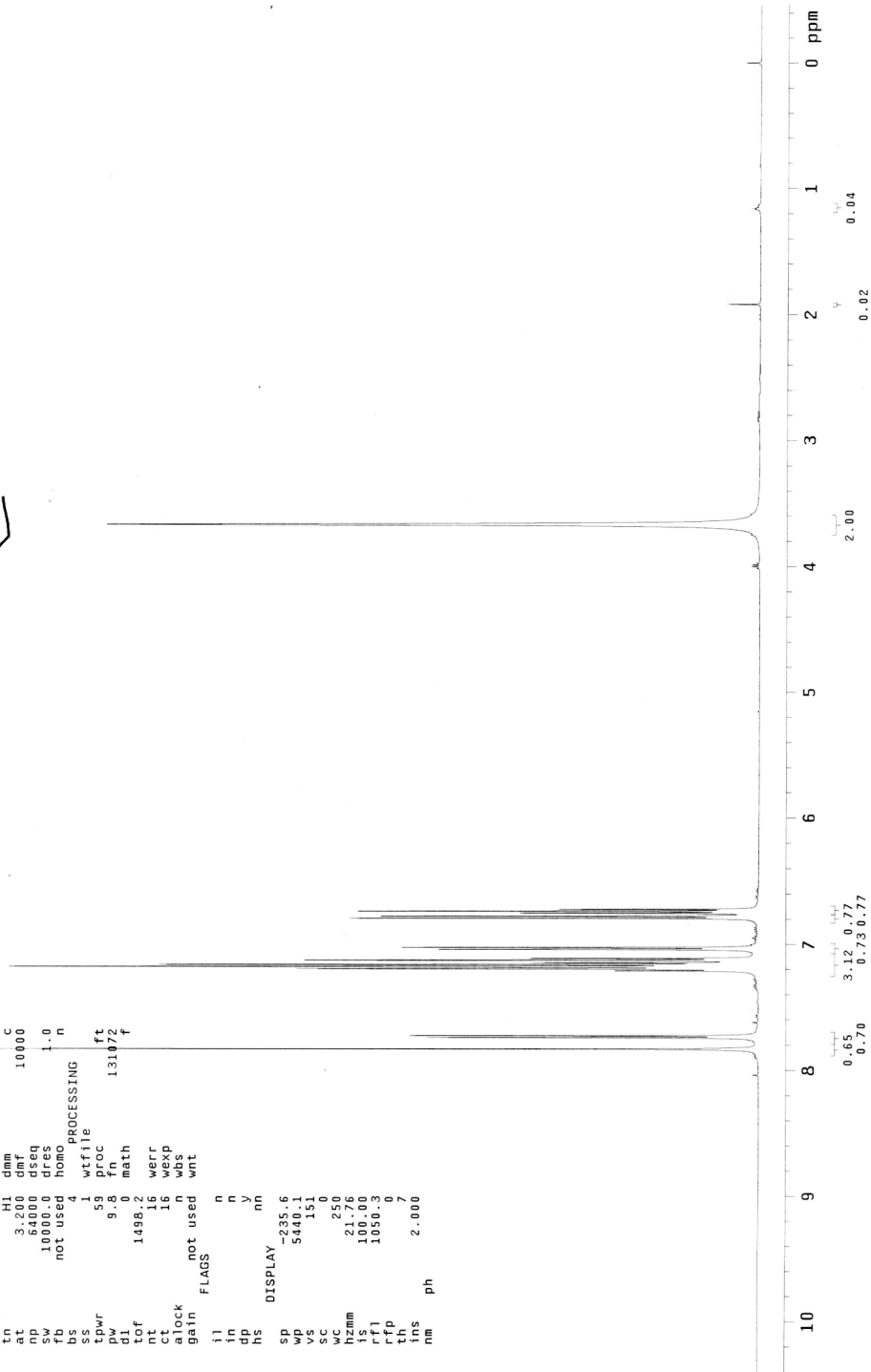




STANDARD PROTON PARAMETERS

```

exp1 s2pu]
SAMPLE
date Jul 25 2006 dfrq DEC. & VT 125.795
solvent CDC13 dn C13
file exp dpwr 37
ACQUISITION
sfrq 500.235 dm nnn
at 3.200 dmf 10000
np 64000 dseq
sw 10000.0 dres 1.0
fb not used homo n
bs 4
ss 1 wfile
dpwr 99 proc ft
d1 9.0 math 131072
tof 1498.2
nt 16 werr
ct 16 wexp
alock n
gain not used wnt
flags
il n
in n
dp y
hs nn
SP 235.6
WC 544.51
SC 15
WC 250
h2mm 21.76
ls 100.00
rfi 1050.3
tbp 7
ins 2.000
nm ph
  
```



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```

expl s2pu1
date SAMPLE 2 2006
solvent CDCl3
file ACQUISITION exp
sfrq 500.235
tn H1
at 3.000
sw 64000
fb 10000.0
bs not used
ss 8
tpwr 59
pw 9.0
tof 1498.2
ct 16
alock 16
gain not used
flags not used
wnt
il n
in n
dp y
hs nn
DISPLAY
sp -1043.6
wp 10000.0
sc 151
wc 250
hzmm 40.00
ls 100.00
rf1 1043.6
rfp 7
ins 3.000
nm ph

```

```

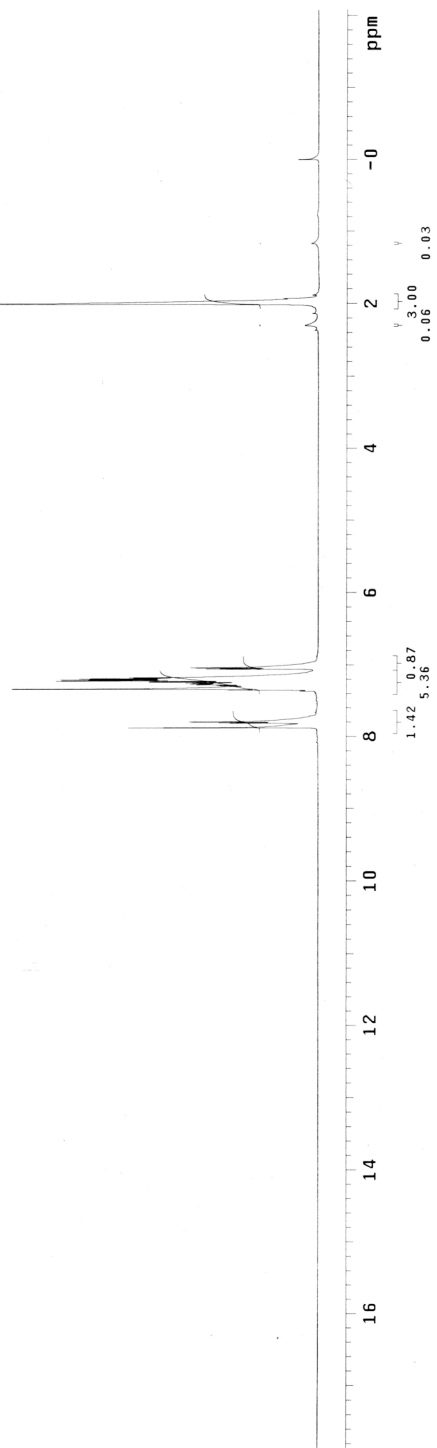
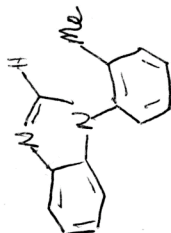
DEC. & VT 125.795
dn C13
dpwr 37
dof 0
dm nnn
dmm nnn
dref 10000
dres 1.0
dss n
homo 1.0
n n

```

```

PROCESSING
1 wfile
59 proc
9.0 fmath
131072 f

```



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