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

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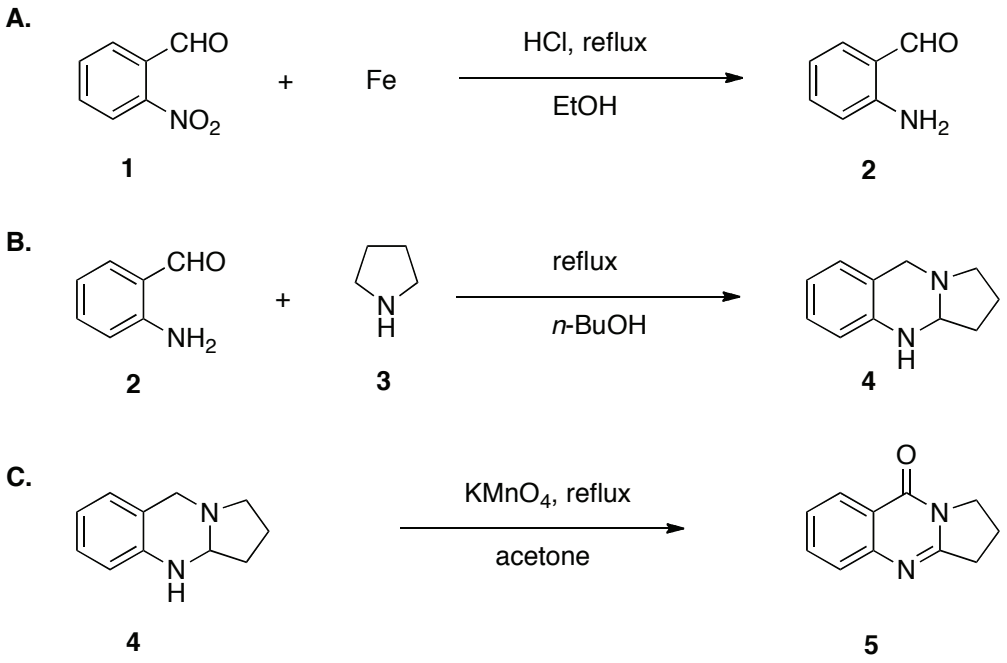
o-Aminobenzaldehyde, Redox-Neutral Aminal Formation and Synthesis of Deoxyvasicinone



Submitted by Chen Zhang, Chandra Kanta De, and Daniel Seidel.[1]
Checked by Liang Huang, Brenda Burke, and Margaret Faul.

1. Procedure

A. *o-Aminobenzaldehyde* (**2**). A 1-L, one-necked, round-bottomed flask, containing an egg-shaped magnetic stir bar (Note 1), is charged with *o*-nitrobenzaldehyde (**1**) (9.07 g, 60 mmol) (Note 2) and 170 mL of absolute ethanol (Note 3). The resulting mixture is stirred for one min, at which point a yellow solution is formed. Iron powder (10.05 g, 180 mmol, 3.0 equiv) (Note 4) is added to the stirring solution, followed by addition of diluted HCl (60 mL) (Note 5). The flask is then equipped with a reflux condenser containing a nitrogen inlet, and heated under reflux for 60 min. Under continued stirring, the reaction mixture is allowed to cool to room temperature over a period of 35 min. The reaction mixture is subsequently diluted with EtOAc (500 mL), followed by addition of anhydrous $MgSO_4$ (70 g) (Note 6). The mixture is allowed to stir at room temperature for 5 min and then filtered through a Büchner funnel packed with celite (Note 7). The filter cake is washed with EtOAc (4 x 100 mL) and the combined

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filtrates are concentrated to a volume of 15 mL (Notes 8 and 9). The resulting clear yellow solution is passed through a silica gel plug as quickly as possible, eluting with 20% EtOAc in hexanes (800 mL) under reduced pressure (Notes 10 and 11). The solvent is removed in vacuo (Note 8) and the residue dried under high vacuum (< 0.1 mmHg) for 2.5 h to obtain 6.95 g (74 wt% quantitative ^{1}H NMR, 57 mmol, 70%) of *o*-aminobenzaldehyde (**2**), which is used directly in the next step (Notes 12 and 13).

B. 1,2,3,3a,4,9-Hexahydropyrrolo[2,1-b]quinazoline (**4**). An oven-dried, 500-mL one-necked, round-bottomed flask, containing a magnetic stir bar (Note 1), is charged with *n*-butanol (200 mL) (Note 14) and pyrrolidine (8.81 g, 10.17 mL, 123.8 mmol, 3.0 equiv) (Note 15). The flask is then equipped with a reflux condenser capped with a septum and a nitrogen inlet, and the reaction mixture is heated under reflux. Using a cannula that is inserted through the septum located on top of the reflux condenser, a solution of *o*-aminobenzaldehyde (**2**) (5.0 g, 41.3 mmol, 1.0 equiv) in *n*-butanol (24 mL) is added slowly via syringe pump over 24 h. Subsequent to the addition, the reaction mixture is heated under reflux for an additional 14 h. The reaction mixture is then allowed to cool to room temperature and is concentrated under reduced pressure (Note 16). The crude product is purified by flash column chromatography, eluting with 500 mL of 50% hexanes in EtOAc, followed by 4 L of 5% MeOH in EtOAc (Note 17). The solvent is removed in vacuo (Note 8) and the residue dried under high vacuum (< 0.1 mmHg) for 24 h to obtain 5.20–5.66 g (100 wt% by quantitative ^{1}H NMR, 32.1 mmol, 72–77%) of **4** as a light yellow microcrystalline solid (Notes 18 and 19).

C. *Deoxyvasicinone* (**5**). A 1-L one-necked, round-bottomed flask, containing a magnetic stir bar (Note 1), is charged with **4** (5.0 g, 28.7 mmol), and acetone (285 mL) (Note 20). The resulting mixture is stirred for one min at which point a light yellow solution is formed. $KMnO_4$ (22.67 g, 143 mmol) (Note 21) is added into the stirring solution. The flask is then equipped with a reflux condenser containing a nitrogen inlet, and the reaction mixture is heated under reflux for 3 h (Note 22). Under continued stirring, the reaction mixture is allowed to cool to room temperature over a period of 30 min and then filtered through a Büchner funnel packed with celite (Note 7). The filter cake is washed with acetone (2 x 50 mL) and the combined filtrates are concentrated under reduced pressure (Note 23). The combined filtrate is concentrated in vacuo (Note 8) and the residue dried

under high vaccum (<0.1 mmHg) for 24 h to obtain 4.9–5.0 g (96 wt%, quantitative ^{1}H NMR, 87–89%) of (**5**) as a light yellow solid (Notes 24 and 25).

2. Notes

1. The egg-shaped magnetic stir bar was of 3.8 cm length. A stirring speed of 1000 rpm was maintained throughout the reaction.

2. *o*-Nitrobenzaldehyde (98+%, Cat. No. 552-89-6) was obtained from Alfa Aesar and used as received.

3. Ethanol (200 Proof - Absolute, Anhydrous ACS/USP grade, Cat No. 111ACS200) was obtained from Decon Laboratories, Inc and used as received.

4. Baker analyzed reagent grade iron powder (99.3%, Cat. No. 7439-89-6) was obtained from Mallinckrodt Baker and used as received.

5. Diluted HCl solution was prepared by dissolving 1 mL of concentrated HCl in 66 mL of distilled water.

6. Magnesium sulfate certified anhydrous (Cat. No. 7487-88-9) was obtained from Fisher Chemicals and used as received.

7. Celite (50 g) was used in a Büchner funnel with a 16.5 cm diameter.

8. A standard rotary evaporator was used (25 °C bath temperature, 50 mmHg).

9. Allowing the solution to go to dryness or remain on the rotary evaporator for prolonged periods of time will result in a significantly lower yield.

10. Silica gel (standard grade, 230 x 400 mesh) was obtained from Sorbent Technologies and used as received.

11. A coarse fritted glass funnel (diameter: 7 cm, height of silica gel: 4 cm) with a wet-packed silica gel plug was used in combination with a vacuum filter flask.

12. The initially formed yellow oil turns into a yellow solid if kept in a –20 °C freezer. However, **2** should not be stored for prolonged periods of time as it degrades gradually even at this temperature. The reaction was also performed with 36.3 g of starting material, which resulted in a yield of 23.5 g (68%) of product with similar purity.

13. Analytical data of **2**: R_f = 0.48 in 20% EtOAc in hexanes; mp = 32–34 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.11 (br s, 2 H), 6.64–6.66

(m, 1 H), 6.76–6.77 (ddd, 1 H), 7.29–7.31 (ddd, 1 H), 7.47–7.49 (app dd, 1 H), 9.87 (s, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 116.0, 116.4, 118.9, 135.2, 135.7, 149.9, 194.0; IR (ATR) 3463, 3329, 3051, 2839, 2759, 1666, 1612, 1582, 1550, 1480, 1396, 1348, 1322, 1288, 1191, 1145, 1041, 881, 750 cm^{-1}; HRMS *m/z* calcd for C_7H_8N0 $[M+H]^+$: 122.05276, found 122.05957.

14. *n*-Butanol (99.9%, Cat. No. 71-36-3) was obtained from Sigma-Aldrich and used as received.

15. Pyrrolidine (99%, Cat. No. 123-75-1) was obtained from Alfa Aesar and was freshly distilled before use.

16. A standard rotary evaporator was used (60 °C bath temperature, 33 mmHg).

17. A wet-packed silica gel column was used (diameter: 8.0 cm, Height: 17 cm). The crude material was loaded as oil on top of the column.

18. The use of a syringe pump is not essential but allows for reproducibly higher yields. For instance, the submitters report that direct mixing of *o*-aminobenzaldehyde (6.06 g, 55 mmol) and three equivalents of pyrrolidine in *n*-butanol (220 mL) for 21 h under reflux gives rise to aminal **4** in 69% isolated yield.

19. Analytical data of **4**: R_f = 0.39 in 10% MeOH in EtOAc; mp = 63–64 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 1.61–1.68 (tdd, 1 H), 1.87–1.96 (m, 1 H), 1.97–2.07 (m, 1 H), 2.09–2.18 (m, 1 H), 2.66–2.70 (dt, 1H), 3.01–3.05 (dt, 1 H,), 3.67 (br s, 1 H), 3.90 (d, 1 H,), 4.04 (d, 1H), 4.13–4.17 (t, 1 H), 6.54 (d, 1 H), 6.70 (t, 1 H), 6.95 (d, 1 H), 7.01–7.02 (t, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.1, 31.8, 50.3, 50.5, 71.2, 114.9, 118.1, 119.4, 127.0, 127.2, 142.9; IR (ATR) 3040, 2967, 2826, 1608, 1585, 1476, 1380, 1252, 744 cm^{-1}; HRMS *m/z* calcd for $C_{11}H_{14}N_2$ $[M+H]^+$: 175.11570, found 175.12248.

20. Acetone CHROMASOLV for HPLC (> 99.9%, Cat. No. 67-64-1) was obtained from Sigma-Aldrich and used as received.

21. Potassium Permanganate, Baker Analyzed A.C.S. Reagent, J.T. Baker (99.0% min, Cat. No. 7722-64-7) was obtained from Mallinckrodt Baker and used as received.

22. The reaction is analyzed for complete consumption of starting material by ^{1}H NMR. The reaction will cleanly convert the starting material to product although the time required for the reaction may vary depending on the quality of $KMnO_4$. It is recommended that the reaction is driven to completion or final purification of the product is extremely difficult.

23. A standard rotary evaporator was used (25 °C bath temperature, 100 mmHg).

24. While it is recommended that efforts be made to ensure this reaction goes to completion, separation of the starting material and purification the product, although difficult, can be performed by flash column chromatography using a wet-packed silica gel column (diameter: 5.5 cm, height: 15 cm). The crude material was loaded as an oil on top of the column and the product eluted with 4 L of 3% MeOH in EtOAc.

25. Analytical data of **5**: R_f = 0.45 in 10% MeOH in EtOAc; mp = 99–102 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.22–2.34 (m, 2 H), 3.16 (t, 2 H), 4.18 (t, 2 H), 7.42 (t, 1 H), 7.62 (d, 1 H), 7.70 (t, 1 H), 8.25 (d, 1 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 19.7, 32.7, 46.6, 120.6, 126.4, 126.5, 126.9, 134.3, 149.3, 159.6, 161.1; IR (ATR) 1669, 1609, 1558, 1464, 1424, 1383, 1334, 1322, 770, 693 cm^{-1}; HRMS *m/z* calcd for $C_{11}H_{10}N_2$ $[M+H]^+$: 187.07931, found 187.08591.

Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academies Press; Washington, DC, 2011.

3. Discussion

o-Aminobenzaldehydes are important building blocks that are widely used in the synthesis of quinolines via the classic Friedländer condensation.[2] The present procedure describes a convenient and improved approach for the preparation of *o*-aminobenzaldehyde from *o*-nitrobenzaldehyde.[3] In our evaluation of published methods for the reduction of *o*-nitrobenzaldehyde,[4] we found that the use of iron dust in combination with hydrochloric acid provides the best yields of *o*-aminobenzaldehyde.

As part of a program aimed at developing redox neutral functionalizations of amines,[5] we have recently reported novel reactions of *o*-aminobenzaldehydes and secondary amines that give rise to ring-fused aminals.[5a] In the course of our attempts to adapt this procedure to a larger scale production of these versatile building blocks, we found that reactions conducted in *n*-butanol (as opposed to the originally used ethanol) provide faster reaction rates and higher yields on larger scale. In addition, slow

addition of *o*-aminobenzaldehyde via syringe pump further improved the yield of this process. A low concentration of *o*-aminobenzaldehyde minimizes the well-known propensity of this substrate to undergo self-condensation.[6]

The aminal structural motif[7] is found in a number of natural products.[8] In addition, aminals such as **4** represent reduced versions of quinazolinone alkaloids, compounds that have attracted significant attention due to their diverse array of biological activities.[9] The selective oxidation of ring fused aminal **4**, reported herein, provides rapid access to deoxyvasicinone (**5**).[10,11] The latter has been reported to possess antimicrobial, anti-inflammatory, and antidepressant activities.[10]

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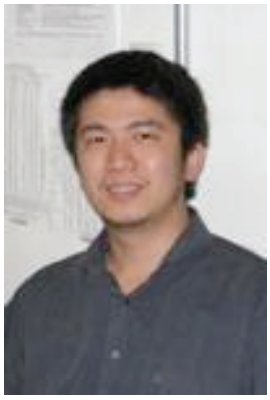
Appendix
Chemical Abstracts Nomenclature; (Registry Number)

o-Aminobenzaldehyde: Benzaldehyde, 2-amino-; (529-23-7)
o-Nitrobenzaldehyde: Benzaldehyde, 2-nitro-; (552-89-6)
Iron powder; (7439-896)

Pyrrolidine; (123-75-1)
1,2,3,3a,4,9-Hexahydropyrrolo[2,1-b]quinazoline: (495-58-9)
Deoxyvasicinone: Pyrrolo[2,1-b]quinazolin-9(1*H*)-one, 2,3-dihydro-; (530-53-0)
Potassium permanganate; (7722-64-7)

Daniel Seidel was born in Mühlhausen, Thüringen (Germany) in 1972. He obtained his Diplom at the Friedrich-Schiller-Universität Jena in 1998. He then joined the group of Prof. Jonathan L. Sessler at the University of Texas at Austin, where he received his Ph.D. in 2002. From 2002–2005, Daniel was an Ernst Schering Postdoctoral Fellow in the group of Prof. David A. Evans at Harvard University. He started his independent career at Rutgers University in August of 2005. Research in his group is focused on new concepts for asymmetric catalysis and the development of synthetic methods.



Chen Zhang was born in Fuzhou (P. R. of China) in 1982. He received his B.Sc. degree from the University of Science and Technology of China in 2005. In September of 2005, he joined the Seidel group as a Ph.D. student. His research is focused on the development of redox neutral reaction cascades.

Chandra Kanta De was born in West Bengal (India) in 1984. He obtained his B.Sc. (Honors in Chemistry) degree from RKMV Belur Math College (University of Calcutta, India) in 2004 and his M.Sc. degree in Chemistry from Indian Institute of Technology Bombay (India) in 2006. In September of 2006, he joined the Seidel group as a Ph.D student. His research is focused on asymmetric catalysis and the development of redox neutral reaction cascades.

Liang Huang obtained his M.S. degree in Chemistry from Murray State University in 1997. He then began working at AlliedSignal as a synthetic organic chemist. In 1998 he moved to CB Research and Development, Inc. taking a position as Scientist. He has been working in Amgen's Chemical Process Research and Development group as a Process Chemist since 1999.

Brenda J. Burke was born in Naples, NY in 1977. She graduated from the University of Rochester in 1999 *cum laude* with a B.S. degree in Chemistry. She completed her doctoral studies at the University of California, Irvine in 2004 under the direction of Professor Larry Overman after which time she moved to the University of Cambridge to begin her post-doctoral studies in the labs of Professor Steven Ley. In 2006, Brenda became a member of Amgen's Chemical Process Research and Development group, and in 2011 she started a position in the Process Research group Gilead Sciences.

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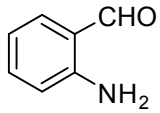

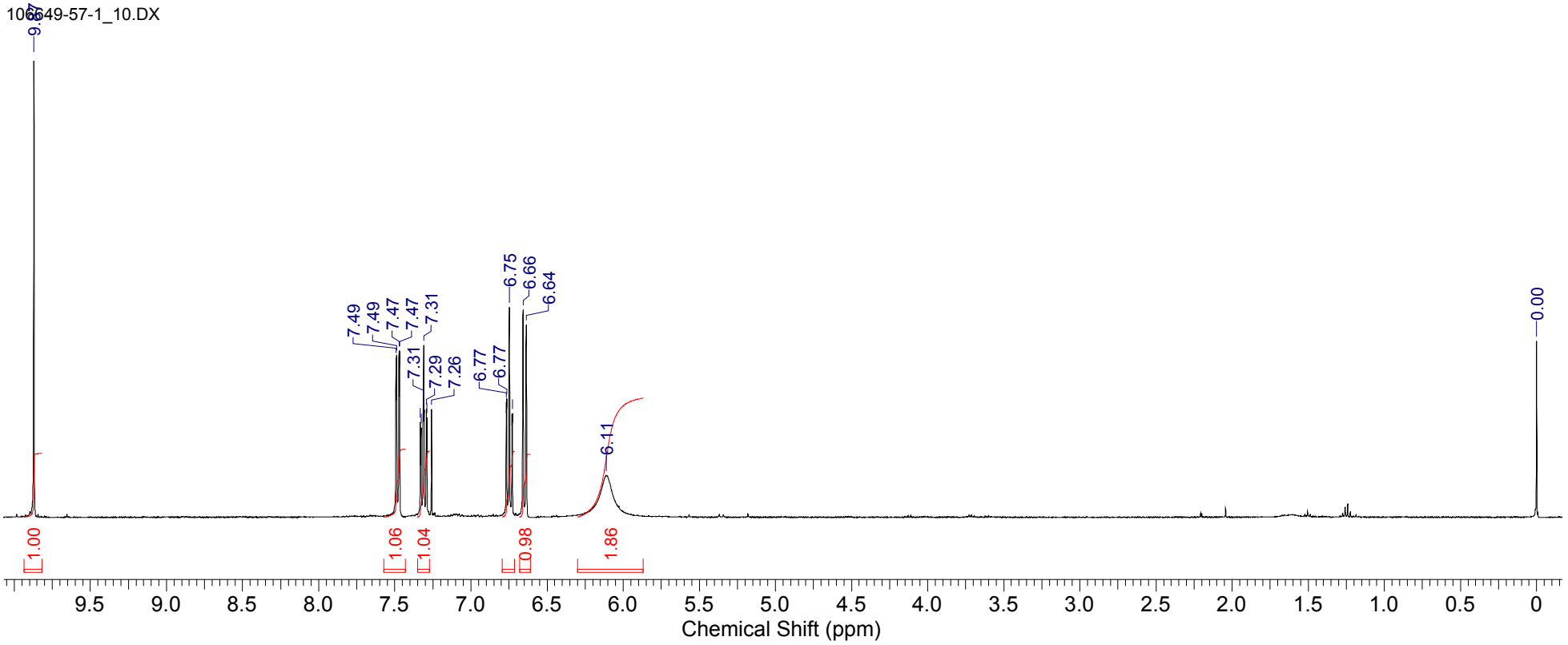

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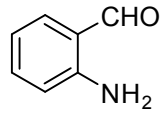

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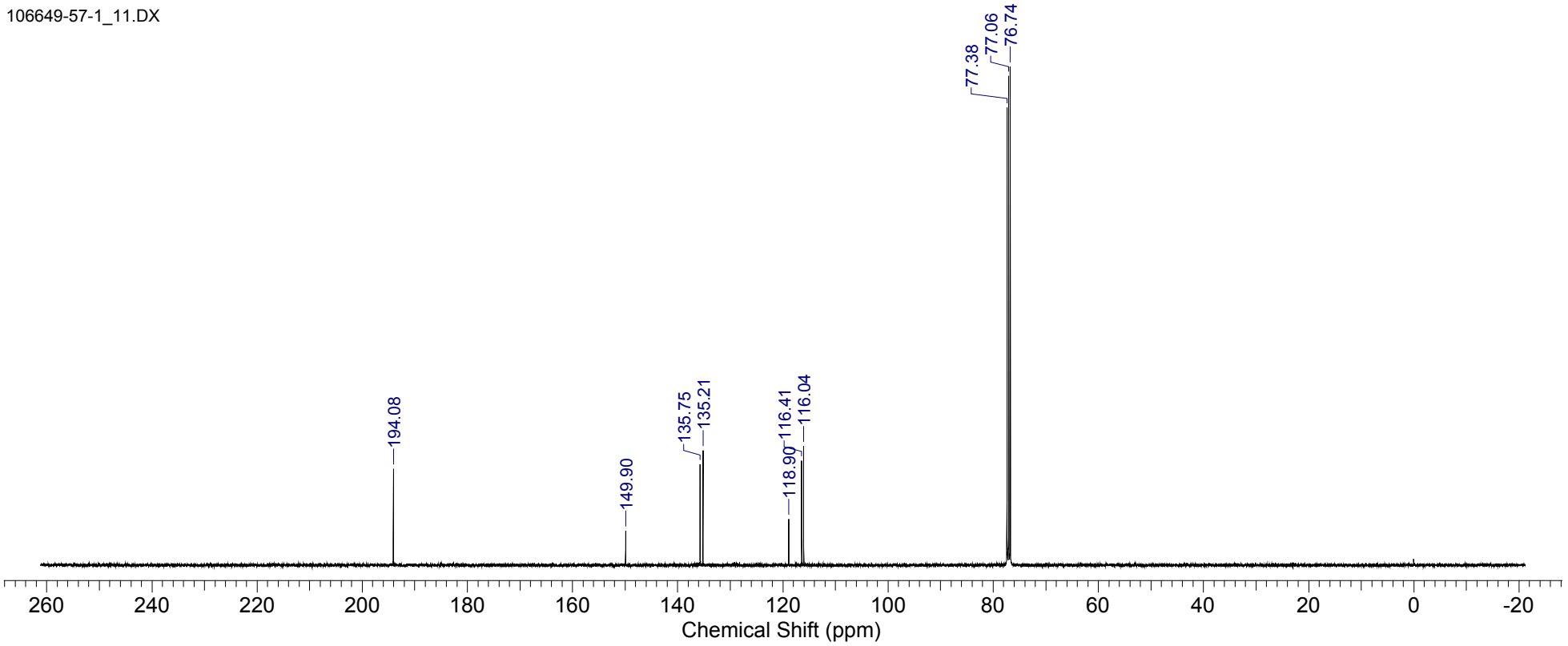

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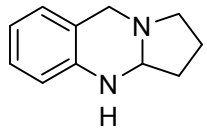

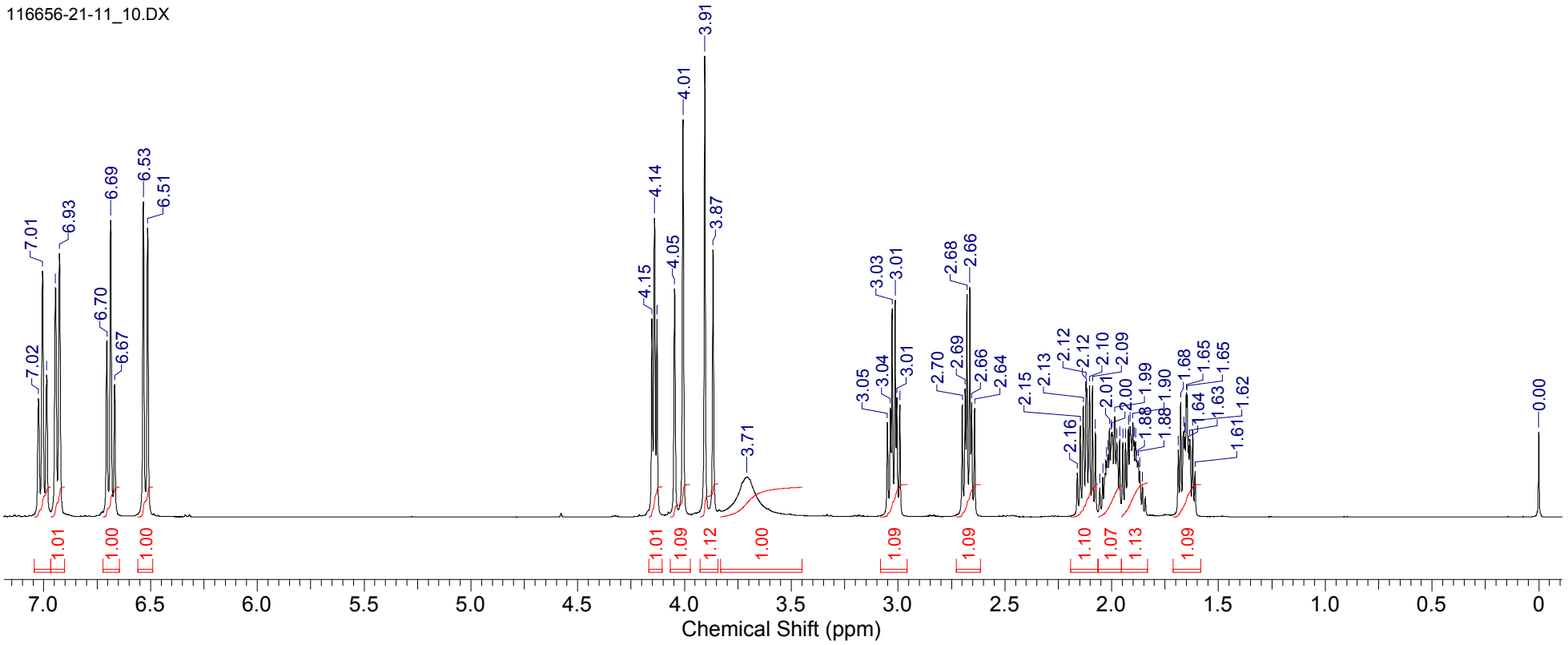

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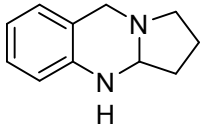
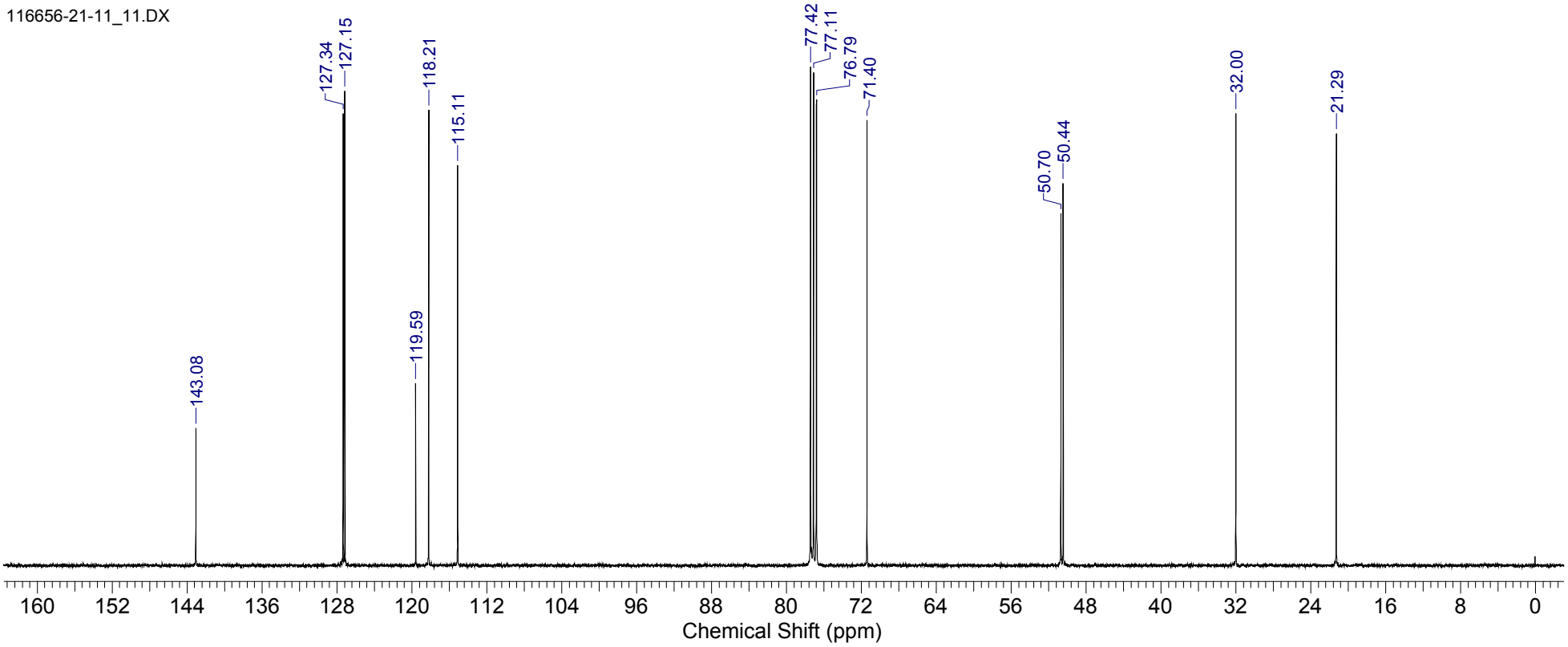
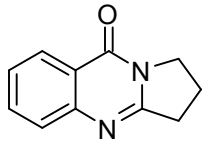
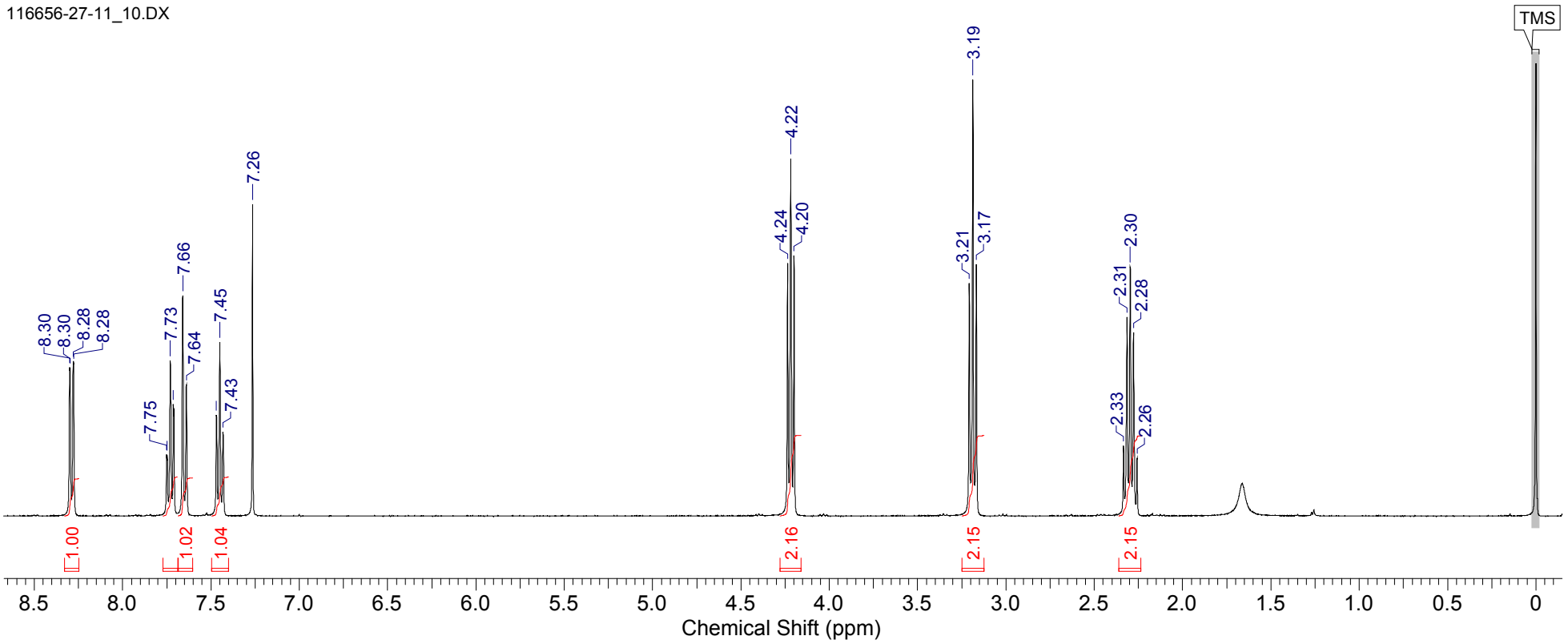




Acquisition Time (sec)	5.1120	***Comment***	liangh
Date	16 Sep 2011 14:34:35		
Date Stamp	16 Sep 2011 14:34:35		
File Name	\\scone\NMR-Archive\jcamp\liangh\2011\116656-27-11_10.DX		
Frequency (MHz)	400.13	***Nucleus***	1H
Number of Transients	16	***Origin***	Bruker BioSpin GmbH
Original Points Count	32768	***Owner***	shr-ato-nmr1
Points Count	32768	***SW(cyclical) (Hz)***	6410.06
Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	2393.0459	***Sweep Width (Hz)***	6409.86
Temperature (degree C)	25.000		





Acquisition Time (sec)	1.1535	***Comment***	liangh
Date	28 Oct 2011 01:52:52		
Date Stamp	28 Oct 2011 01:52:52		
File Name	\\scone\NMR-Archive\jcamp\liangh\2011\116656-27-13_11.DX		
Frequency (MHz)	100.62	***Nucleus***	13C
Number of Transients	2048	***Origin***	Bruker BioSpin GmbH
Original Points Count	32768	***Owner***	shr-ato-nmr1
Points Count	32768	***SW(cyclical) (Hz)***	28408.22
Solvent	CHLOROFORM-d		
Spectrum Offset (Hz)	12076.6396	***Sweep Width (Hz)***	28407.36
Temperature (degree C)	26.400