Palladium-Catalyzed Asymmetric Heck-Matsuda Reaction of 1,4-Dihydroquinolines with Aryldiazonium Salts

Ze-Zhen Jiang,†‡ Yang-Jie Jiang,‡ Juan Du,‡ Di Chen,‡ Chang-Hua Ding,*, †‡ Bin Xu,*, †‡ Xue-Long Hou*,‡

†Department of Chemistry, Innovative Drug Research Center, Shanghai University, Shanghai, 200444, China
‡State Key Laboratory of Organometallic Chemistry, Shanghai-Hong Kong Joint Laboratory in Chemical Synthesis, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 345 Lingling Road, Shanghai 200032, China

*dingch@sioc.ac.cn; xubin@shu.edu.cn; xlhou@sioc.ac.cn

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1. General Methods

Commercially available reagents were used without further purification. Solvents were purified prior to use according to the standard methods. Column chromatography was performed on silica gel (300–400 mesh) using a forced flow of eluent. NMR spectra were recorded at room temperature on an NMR instrument operating at 400 MHz. Chemical shifts for $^1$H NMR are reported in parts per million with the solvent resonance as the internal standard ($7.26$ ppm for CHCl$_3$). The data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, sep = septet, bs = broad singlet, m = multiplet), coupling constants (Hz), and integration. $^{13}$C NMR spectra were recorded on an NMR instrument operating at 100 MHz with complete proton decoupling. Chemical shifts were reported in parts per million with the solvent resonance as the internal standard ($77.1$ ppm for CDCl$_3$). MS and HRMS were measured in EI or ESI mode, and the mass analysis mode of the HRMS was TOF. Infrared spectra were recorded from thin films of pure samples. Melting points were measured on an XT-4 micromelting point apparatus. Thin layer chromatography was performed on precoated glass-backed plates and visualized with UV light at 254 nm.

2. Screening of Reaction Conditions for Pd-catalyzed Asymmetric Heck-Matsuda Reaction of N-Boc-1,4-dihydroquinoline (1a) and $p$-methoxybenzenediazonium Tetrafluoroborate (2a)
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<sup>a</sup>molar ratio of 1a/2a/[Pd]/L/base = 100/150/10/20/300, c = 0.1 mol/L. <sup>b</sup>Isolated.
yield. *Determined by chiral HPLC. **Molar ratio of 1a/2a/[Pd]/L/base = 100/120/10/20/300. ***Molar ratio of 1a/2a/[Pd]/L/base = 100/100/10/20/200. DTBMP = 2,6-di-tert-butyl-4-methylpyridine. DABCO = 1,4-diazabicyclo[2.2.2]octane. DBU = 1,8-diazabicyclo[5.4.0]undec-7-ene.

3. Experimental Procedure for the Preparation of N-Boc-1,4-dihydroquinolines

3,4-dihydroquinolin-2(1H)-ones 5 (54.4 mmol, 1.0 equiv) and 160 mL CH₂Cl₂ were added to a flame dried and argon flushed 500 mL two-neck round-bottom flask. Then 7.5 mL Et₃N (54.4 mmol, 1.0 equiv), 14.2 g (Boc)₂O (65.3 mmol, 1.2 equiv) and 656 mg DMAP (4-dimethylaminopyridine) (5.4 mmol, 10 mol%) were added under the room temperature. The reaction was stirred until full conversion (monitored by TLC [petroleum ether (PE)/ethyl acetate (EA) = 5/1]). The reaction mixture was concentrated, then transferred to funnel. 200 mL water was added and the phases were separated. The aqueous phase was extracted with Et₂O three times. The combined organic phases were washed with saturated aqueous NaHCO₃ solution and saturated aqueous NaCl solution. Then the combined organic layers were dried over Na₂SO₄, filtered and the solvent was removed in vacuum. The resulting crude product was purified by flash column silica gel chromatography to yield the product 6.

N-Boc-3,4-dihydroquinolin-2(1H)-ones 6 (10.1 mmol, 1.0 equiv) and 40 mL THF were added to a flame dried and argon flushed 100 mL three-neck round-bottom flask. The solution was cooled to -78 °C and 12.2 mL DIBAL-H (1.0 M in hexane, 12.2 mmol, 1.2 equiv) were added dropwise in about 60 minutes. The reaction was stirred until full conversion [monitored by TLC (PE/EA = 5/1)] and then warm to -15 °C. 25 mL 30% H₂O₂ aqueous solution was added dropwise then the reaction mixture was warmed up to room temperature and the colourless precipitate was filtered over celite. The phases were separated and the aqueous phase was extracted with EtOAc (3×50 mL). The combined organic phases were washed with saturated
aqueous NaCl solution. Then the combined organic layers were dried over anhydrous Na₂SO₄, filtered and the solvent was removed in vacuum. The crude product was applied for the next step without further purification.

In a flame dried and argon flushed 50 mL three-neck round-bottom flask, tert-butyl 2-hydroxy-3,4-dihydroquinoline-1(2H)-carboxylates 7 (2.0 mmol, 1.0 equiv), 0.7 mL Et₃N (5 mmol, 1.2 equiv) and 10 mL CH₂Cl₂ were added. The solution was cooled to 0 °C and 0.2 mL MsCl (2.4 mmol, 1.2 equiv) were added dropwise slowly and was stirred at 0 °C for 1 hour. Then the solution was stirred and refluxed at 50 °C. After full conversion [monitored by TLC (PE/EA = 5/1)], the reaction mixture was quenched by 1 mL H₂O. The phases were separated and the aqueous phase was extracted with CH₂Cl₂ (3×2 mL). The combined organic phases were washed with saturated aqueous NaCl solution. Then the combined organic layers were dried over anhydrous Na₂SO₄, filtered and the solvent was removed in vacuum. The resulting crude product was purified by flash column silica gel chromatography to yield the product 1.

![Chemical structure of tert-butyl quinoline-1(4H)-carboxylate 1a](image)

**tert-butyl quinoline-1(4H)-carboxylate 1a** Compound 1a was purified by flash chromatography on silica gel (PE/EA = 20/1). colourless oil (346.9 mg, total yield: 75%). ¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, J = 8.5 Hz, 1H), 7.23 – 7.15 (m, 1H), 7.07 (dd, J = 3.5, 2.1 Hz, 2H), 6.90 (d, J = 7.7 Hz, 1H), 5.27 (dt, J = 8.1, 4.1 Hz, 1H), 3.33 (d, J = 4.0 Hz, 2H), 1.57 (s, 9H); ¹³C NMR (101 MHz, CDCl₃) δ 151.63, 136.97, 128.15, 127.97, 127.29, 126.07, 124.47, 121.74, 108.58, 81.89, 28.30, 27.58; IR 2975, 1710, 1488, 1327, 1242, 1161, 1136, 1116, 1020, 855, 762, 724, 693; LRMS (ESI) m/z (M+Na)⁺: 254.0, (M+K)⁺: 260.0.
**tert-butyl 6-methoxyquinoline-1(4H)-carboxylate 1b.** Compound 1b was purified by flash chromatography on silica gel (PE/EA = 5/1). Light yellow oil (376.3 mg, total yield: 72%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.84 (d, $J$ = 9.1 Hz, 1H), 6.89 (d, $J$ = 7.7 Hz, 1H), 6.74 (dd, $J$ = 9.1, 2.6 Hz, 1H), 6.58 (d, $J$ = 2.4 Hz, 1H), 5.22 (dt, $J$ = 8.1, 4.1 Hz, 1H), 3.78 (s, 3H), 3.31 (d, $J$ = 3.7 Hz, 2H), 1.56 (s, 9H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 156.28, 151.65, 130.26, 129.28, 127.33, 122.78, 112.79, 111.66, 107.95, 81.69, 55.41, 28.31, 27.86; IR (film): 3008, 1694, 1498, 1299, 1256, 1160, 1117, 1016, 856, 838, 735, 688 cm$^{-1}$; LRMS (ESI) $m/z$ (M+Na)$^+$: 284.2; HRMS (ESI) $m/z$ Calcd for C$_{15}$H$_{20}$O$_3$N (M+H)$^+$: 262.1438, found 262.1438.

**tert-butyl 6-chloroquinoline-1(4H)-carboxylate 1c.** Compound 1c was purified by flash chromatography on silica gel (PE/EA = 30/1). Colorless oil (372.0 mg, total yield: 70%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.87 (d, $J$ = 8.9 Hz, 1H), 7.14 (dd, $J$ = 9.0, 2.5 Hz, 1H), 7.04 (d, $J$ = 2.5 Hz, 1H), 6.88 (dt, $J$ = 7.7, 1.4 Hz, 1H), 5.23 (dt, $J$ = 7.9, 4.1 Hz, 1H), 3.30 (d, $J$ = 4.0 Hz, 2H), 1.56 (s, 9H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 151.38, 135.56, 129.65, 129.47, 127.81, 127.21, 126.07, 122.96, 107.92, 82.26, 28.25, 27.41; IR (film): 2974, 1712, 1324, 1240, 1138, 1094, 1020, 814, 764 cm$^{-1}$; LRMS (ESI) $m/z$ (M+Na)$^+$: 288.1; HRMS (ESI) $m/z$ Calcd for C$_{14}$H$_{17}$O$_2$NCl (M+H)$^+$: 266.0942, found 266.0944.

**tert-butyl 6-bromoquinoline-1(4H)-carboxylate 1d.** Compound 1d was purified by flash chromatography on silica gel (PE/EA = 20/1). Light yellow oil (496.3 mg, total yield: 80%). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.81 (d, $J$ = 8.9 Hz, 1H), 7.28 (dd, $J$ = 9.1, 1.8 Hz, 1H), 7.19 (s, 1H), 6.87 (d, $J$ = 7.7 Hz, 1H), 5.22 (dt, $J$ = 8.0, 4.0 Hz, 1H), 3.30
(d, J = 3.6 Hz, 2H), 1.56 (s, 9H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 151.32, 136.09, 130.75, 130.02, 128.97, 127.17, 123.28, 117.24, 107.94, 82.28, 28.23, 27.31; IR (film): 3067, 1713, 1660, 1478, 1334, 1241, 1138, 1020, 817, 763, 737 cm$^{-1}$; LRMS (ESI) $m/z$ (M+Na)$^+$: 332.1; HRMS (ESI) $m/z$ Calcd for C$_{14}$H$_{17}$O$_2$NBr (M+H)$^+$: 310.0437, found 310.0437.

**tert-butyl 6-nitroquinoline-1(4H)-carboxylate 1e.** Compound 1e was purified by flash chromatography on silica gel (PE/EA = 20/1). yellow oil (469.7 mg, total yield: 85%). $^1$H NMR (400 MHz, CDCl$_3$) δ 8.10 (d, J = 9.2 Hz, 1H), 8.03 (dd, J = 9.3, 2.4 Hz, 1H), 7.94 (s, 1H), 6.89 (d, J = 7.7 Hz, 1H), 5.27 (dt, J = 8.0, 4.1 Hz, 1H), 3.42 (d, J = 3.8 Hz, 2H), 1.58 (s, 9H); $^{13}$C NMR (101 MHz, CDCl$_3$) δ 150.99, 143.85, 142.71, 128.58, 126.86, 123.69, 121.83, 121.81, 107.73, 83.27, 28.14, 27.43; IR (film): 2971, 1712, 1691, 1508, 1303, 1235, 1146, 1018, 885, 727 cm$^{-1}$; LRMS (ESI) $m/z$ (M+Na)$^+$: 299.1; HRMS (ESI) $m/z$ Calcd for C$_{14}$H$_{17}$O$_4$N$_2$ (M+H)$^+$: 277.1183, found 277.1182.

### 4. Reference

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### Shimadzu LCsolution Analysis Report

- **Acquired by**: Admin
- **Sample Name**: jzz-9-22 AD-H
- **Sample ID**: AD-H,96/4,1,214
- **Injection Volume**: 2 uL
- **Data File Name**: jzz-9-22 AD-H.lcd
- **Method File Name**: 123.lcm
- **Report File Name**: Default.lcr
- **Data Acquired**: 2018-5-16 9:14:30
- **Data Processed**: 2018-5-25 0:34:12

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**<Chromatogram>**

![Chromatogram Image]

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==== Shimadzu LCsolution Analysis Report ====  

Acquired by: Admin  
Sample Name: jzz-8-39 AD-H  
Sample ID: AD-H,96/4,1,214  
Injection Volume: 2 uL  
Data File Name: jzz-8-39 AD-H.lcd  
Method File Name: 123.lcm  
Report File Name: Default.lcr  
Data Acquired: 2017-12-27 15:41:41  
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Detector A Ch1 214nm

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==== Shimadzu LCsolution Analysis Report ==== 

Acquired by: Admin
Sample Name: jzz-8-29 AD-H
Sample ID: AD-H,96/4,1.0,214
Vail #: 
Injection Volume: 2 uL
Data File Name: jzz-8-29 AD-H.lcd
Method File Name: 123.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 2017-12-21 22:20:07
Data Processed: 2017-12-21 22:31:51

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C:\LabSolutions\Data\Project1\Project1\jzz\work2\6-Br-p-OMe\jzz-8-29rac AD-H.lcd

--- Shimadzu LCsolution Analysis Report ---

Acquired by: Admin
Sample Name: jzz-8-28-1 AD-H
Sample ID: AD-H,96/4,1.0,214
Vail #:
Injection Volume: 2 uL
Data File Name: jzz-8-29rac AD-H.lcd
Method File Name: 123.lcm
Batch File Name:
Report File Name: Default.lcr
Data Acquired: 2017-12-21 22:09:11
Data Processed: 2017-12-21 22:27:28

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Detector A Ch1 214nm

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==== Shimadzu LCsolution Analysis Report ==== 

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Sample Name: jzz-8-31 AD-H
Sample ID: AD-H,96/4,1.0,214
Injection Volume: 2 uL
Data File Name: jzz-8-31 AD-H.lcd
Method File Name: 123.lcm
Report File Name: Default.lcr
Data Acquired: 2017-12-21 22:53:27
Data Processed: 2017-12-21 23:14:11

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Acquired by: Admin
Sample Name: jzz-8-30-1 AD-H
Sample ID: AD-H,96/4,1.0,214
Vail #: 
Injection Volume: 2 uL
Data File Name: jzz-8-31rac AD-H.lcd
Method File Name: 123.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 2017-12-21 22:30:19
Data Processed: 2017-12-21 22:54:23

--- Chromatogram ---

Detector A Ch1 214nm

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==== Shimadzu LCsolution Analysis Report ====

Acquired by : Admin
Sample Name : jzz-8-66 AD-H
Sample ID : AD-H,96/4,1,214
Injection Volume : 2 μL
Data File Name : jzz-8-66 AD-H.lcd
Method File Name : 123.lcm
Report File Name : Default.lcr
Data Acquired : 2018-1-11 1:17:27
Data Processed : 2018-1-11 1:36:54

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DetA Ch1/214nm

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==== Shimadzu LCsolution Analysis Report ====

Acquired by: Admin
Sample Name: jzz-8-65 AD-H
Sample ID: AD-H_96/4_1_214
Vail #: 
Injection Volume: 2 μL
Data File Name: jzz-8-65 AD-H.lcd
Method File Name: 123.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 2018-1-11 0:58:33
Data Processed: 2018-1-11 1:18:59

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Detector A Ch1 214nm

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**** Shimadzu LCsolution Analysis Report ****

Acquired by: Admin
Sample Name: jzz-8-68 AD-H
Sample ID: AD-H,96/4,1,214
Vail #: 
Injection Volume: 2 uL
Data File Name: jzz-8-68 AD-H.lcd
Method File Name: 123.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 2018-1-12 23:03:13
Data Processed: 2018-1-12 23:17:36

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**** Shimadzu LCsolution Analysis Report ****

Acquired by : Admin
Sample Name : jzz-8-67 AD-H
Sample ID : AD-H,96/4,1,214
Vail # :
Injection Volume : 2 uL
Data File Name : jzz-8-67 AD-H.lcd
Method File Name : 123.lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 2018-1-12 22:49:42
Data Processed : 2018-1-12 23:04:09

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#### Shimadzu LCsolution Analysis Report ####

Acquired by: Admin
Sample Name: jzz-8-70 AD-H
Sample ID: AD-H,96/4,1,214
Vail #:
Injection Volume: 2 uL
Data File Name: jzz-8-70 AD-H.lcd
Method File Name: 123.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 2018-1-12 23:27:10
Data Processed: 2018-1-12 23:38:59

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![Chromatogram Image]

Detector A Ch1 214nm

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==== Shimadzu LCsolution Analysis Report ==== 

Acquired by: Admin  
Sample Name: jzz-8-69 AD-H  
Sample ID: AD-H,96/4,1,214  
Injection Volume: 2 µL  
Data File Name: jzz-8-69 AD-H.lcd  
Method File Name: 123.lcm  
Report File Name: Default.lcr  
Data Acquired: 2018-1-12 23:16:43  
Data Processed: 2018-1-12 23:38:06

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==== Shimadzu LCsolution Analysis Report ==== 

Acquired by: Admin
Sample Name: jzz-8-64 AD-H
Sample ID: AD-H, 96/4, 1, 214
Vail #:
Injection Volume: 2 uL
Data File Name: jzz-8-64 AD-H.lcd
Method File Name: 123.lcm
Batch File Name:
Report File Name: Default.lcr
Data Acquired: 2018-1-11 0:42:32
Data Processed: 2018-1-11 1:00:25

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Acquired by: Admin
Sample Name: jzz-8-63 AD-H
Sample ID: AD-H,96/4,1,214
Injection Volume: 2 μL
Data File Name: jzz-8-63 AD-H.lcd
Method File Name: 123.lcm
Report File Name: Default.lcr
Data Acquired: 2018-1-11 0:26:40
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Detector A Ch1 214nm

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### Shimadzu LCsolution Analysis Report

**Acquired by**: Admin  
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**Sample ID**: AD-H,96/4,1,214  
**Injection Volume**: 2 uL  
**Data File Name**: jzz-8-78 AD-H.lcd  
**Method File Name**: 123.lcm  
**Report File Name**: Default.lcr  
**Data Acquired**: 2018-1-18 0:16:41  
**Data Processed**: 2018-1-18 0:32:25

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Sample ID : AD-H,96/4,1,214  
Vail # :  
Injection Volume : 2 uL  
Data File Name : jzz-8-77 AD-H.lcd  
Method File Name : 123.lcm  
Batch File Name :  
Report File Name : Default.lcr  
Data Acquired : 2018-1-18 0:02:50  
Data Processed : 2018-1-18 0:17:52

<Chromatogram>

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==== Shimadzu LCsolution Analysis Report ====

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Sample ID: AD-H,96/4,1,214  
Injection Volume: 2 uL  
Data File Name: jzz-8-AD-H.lcd  
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![Chromatogram Image]

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### Shimadzu LCsolution Analysis Report

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**Sample Name:** jzz-8-83 AD-H  
**Sample ID:** AD-H,96/4,1,214  
**Injection Volume:** 2 uL  
**Data File Name:** jzz-8-83 AD-H.lcd  
**Method File Name:** 123.lcm  
**Report File Name:** Default.lcr  
**Data Acquired:** 2018-1-19 23:41:07  
**Data Processed:** 2018-1-20 0:03:49

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**<Chromatogram>**

![Chromatogram Image](C:\LabSolutions\Data\Project1\Project1\jzz\work2\6-NO2-p-Cl\jzz-8-83 AD-H.lcd)

**Detector A Ch1 214nm**

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== Shimadzu LCsolution Analysis Report ==

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Sample ID: AD-H,96/4,1.0,214
Vail #: 
Injection Volume: 2 uL
Data File Name: jzz-9-3l-racl AD-H.lcd
Method File Name: 123.lcm
Batch File Name:
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Data Acquired: 2018-3-30 20:59:23
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Detector A Ch1 214nm

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Sample ID: AD-H,96/4,1.0,214  
Vail #:  
Injection Volume: 2 uL  
Data File Name: jzz-9-3l-rac AD-H.lcd  
Method File Name: 123.lcm  
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Report File Name: Default.lcr  
Data Acquired: 2018-3-30 20:34:52  
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Detector A Ch1 214nm  

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Sample ID: AD-H,96/4,1,214 
Vial #: 
Injection Volume: 2 uL 
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Method File Name: 123.lcm 
Batch File Name: 
Report File Name: Default.lcr 
Data Acquired: 2018-4-2 23:39:22 
Data Processed: 2018-4-3 0:01:58 

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Detector A Ch1 214nm 

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Injection Volume: 2 μL  
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Method File Name: 123.lcm  
Batch File Name:  
Report File Name: Default.lcr  
Data Acquired: 2018-1-20 0:53:02  
Data Processed: 2018-1-20 1:20:45

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Detector A Ch1 214nm

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Injection Volume : 2 uL  
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Method File Name : 123.lcm  
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1 Det.A Ch1/214nm  

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==== Shimadzu LCsolution Analysis Report ====  

Acquired by: Admin  
Sample Name: jzz-8-98 AD-H  
Sample ID: AD-H,90/10,1,214  
Vail #:  
Injection Volume: 2 uL  
Data File Name: jzz-8-98 AD-H.lcd  
Method File Name: 123.lcm  
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1 Det.A Ch1/214nm
==== Shimadzu LCsolution Analysis Report ====

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Vial # :
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Detector A Ch1 214nm

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Sample ID: AD-H, 96/4, 1.0, 214  
Injection Volume: 2 uL  
Data File Name: jzz-9-27 AD-H.lcd  
Method File Name: 123.lcm  
Report File Name: Default.lcr  
Data Processed: 2018-5-25 0:29:33

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== Shimadzu LCsolution Analysis Report ==

Acquired by: Admin
Sample Name: jzz-9-27rac AD-H
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Vail #: 
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