Synthesis and Pharmacological Characterization of the Selective GluK1 Radioligand \((S)-2\text{-Amino-3-}(6-[^3]H\text{-2,4-dioxo-3,4-dihydrothieno[3,2-}
d[2\text{-pyrimidin-1(2H)-yl})propanoic\text{ acid}}\ (([^3]H\text{-NF608)}

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SUPPORTING INFORMATION

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**Attempted syntheses of [3H]-UBP310 from UBP310**

Bromination of commercially available UBP310 (Scheme S1, step a) was attempted using the reported conditions by Atlason *et al.*\(^1\) Despite the 3 to 4-fold excess of bromine only monobromination was observed after 3 days at rt. Increasing of reaction temperature, reaction time or concentration did not lead to a dibrominated product. From the crude reaction mixture two monobrominated products were isolated, and identified by LC-MS and \(^1\)H-NMR to be rotamers where the bromination had taken place at the uracil ring, compound **IH189**, rather than on the thiophene ring.

**Scheme S1.** Failed attempt for the one-step synthesis of dibromo-UBP310 from commercially available UBP310.

\[ \text{Reagents and conditions: a) 3 equiv. Br}_2, \text{AcOH, rt, 3-4 days.} \]

With this unexpected brominated analog of UBP310 in hand, compound **IH189**, we decided to explore and establish optimal conditions for incorporation of deuterium prior to the eventual tritiation reaction. Thus, **IH189** was dissolved in H\(_2\)O with a catalytic amount of Pd/C and stirred at rt under a D\(_2\) atmosphere. HPLC showed full conversion of **IH189**, however, \(^1\)H-NMR and LC-MS analysis confirmed full incorporation of hydrogen. We believed that the carboxylic acid

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Functionalities were the source of proton donation and consequently prepared the disodium salt of \textbf{IH189} and repeated the reaction. After 2.5 h at 40 °C full consumption of \textbf{IH189} was observed, however, the $^1$H-NMR spectrum of the crude product did not confirm deuterium or hydrogen incorporation. The molecular mass [M+1] was determined by LC-MS to 352, two protons less than the calculated molecular mass [M+1= 354] of \textbf{UBP310}. Based on these data, we concluded that an intramolecular cyclization had taken place to give lactone \textbf{IH249} (Scheme S2).

\textbf{Scheme S2:} Attempted deuteration of \textbf{IH189} provided lactone \textbf{IH249} instead of deuterium incorporation

$\text{IH189} \xrightarrow{a,b,c} \text{[2H]UBP310}$

\textit{Reagents and conditions:} a) Pd/C, H$_2$O, D$_2$ atm., rt; b) Pd/C, NaOH in D$_2$O, D$_2$ atm., rt; c) Pd/C, DMF, NEt$_3$, D$_2$ atm., rt.

\textbf{Synthesis of bromo precursors dimethyl ester \textbf{IH268} and dimethyl ester carbamate \textbf{IH274}}

To circumvent the basic conditions during the deuterium incorporation, we prepared the dimethyl ester of \textbf{IH189} (Scheme S3). Thus, \textbf{UBP310} was subjected to standard Fisher-esterification affording dimethyl ester \textbf{IH266} in quantitative yield. Bromination using excess bromine in AcOH at
rt afforded one monobrominated product which was confirmed by $^1$H-NMR and LCMS to be IH268.

Finally, IH268 was dissolved in MeOD and in the presence of Pd/C stirred under a D$_2$ atmosphere for 5 h. HPLC analysis showed full consumption of bromine IH268, however LC-MS and $^1$H-NMR showed incorporation of hydrogen rather than deuterium.

**Scheme S3:** Synthesis of dimethyl ester IH268 and attempted deuterium experiments

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UBP310       a          H266        b          H268        c,d          IH271
HHN          CO$_2$H    HN          CO$_2$Me    HN          CO$_2$Me    HN          CO$_2$Me
CO$_2$H      CO$_2$H    H266        H268        H271
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*Reagents and conditions:* a) MeOH, SOCl$_2$, 40 °C, 16 h, quant.; b) Br$_2$, AcOH, rt, 4d; c) Pd/C, MeOD, D$_2$, rt, 5h; d) Pd/C, DMF, NEt$_3$ (pH6-7), D$_2$, rt, 4h.

This was presumably due to the slightly acidic ammonium functionality (from the HCl salt) for which reason we next decided to protect the amine as its BOC carbamate (Scheme S4). Thus, dimethyl ester IH268 was treated with BOC$_2$O in the presence of DMAP to give IH273 in high yield. Bromination of IH273 by use of excess NBS in CDCl$_3$/AcOH (1:1) afforded a single monobrominated product, which was determined by $^1$H-NMR to be the uracil brominated analog IH274. Subsequently, IH274 was dissolved in THF with a catalytic amount of Pd/C and stirred at rt under a D$_2$ atmosphere. HPLC showed full consumption of IH274 after 3.5 h, however, again LC-MS and $^1$H-NMR analysis confirmed incorporation of hydrogen only.

**Scheme S4:** Synthesis of dimethyl ester carbamate IH274 and attempted incorporation of deuterium
Reagents and conditions: a) Boc₂O, DMAP, NEt₃, rt, 2d, 87% (crude); b) NBS, AcOH/CDCl₃ (1:1), rt, 3h, 39%; c) Pd/C, THF, D₂, rt, 3.5h; d) NaOMe or Cs₂CO₃, MeOD, rt, 30 min.

Conclusion

In summary, deuteriation of bromo analog IH189 without the use of base resulted in full hydrogen incorporation while addition of base resulted in an intramolecular cyclization to give the corresponding lactone IH249. To avoid basic conditions, dimethyl ester and carbamate protected analogs IH268 and IH274 were prepared. Unfortunately, these were also unsuccessful as starting points for incorporation of deuterium on the uracil ring. Regrettably, we conclude that uracil brominated UBP310 analogs are not feasible intermediates for deuterium incorporation and thus not a viable strategy for the synthesis of a tritium labeled analog of UBP310.
Experimental data

General remarks
All reactions involving dry solvents or sensitive agents were performed under a nitrogen or argon atmosphere, and glassware was dried prior to use. Commercially available chemicals were used without further purification. Solvents were dried prior to use with an SG water solvent purification system or dried by standard methods. Reactions were monitored by analytical thin-layer chromatography (TLC, Merck silica gel 60 F254 aluminum sheets), analytical HPLC or UPLC. Flash chromatography was carried out using the Merck silica gel 60 (15–40 μm) or Merck silica gel 60 (40–63 μm). $^1$H NMR spectra were recorded on a 400 MHz Bruker Avance III or 600 MHz Bruker Avance III HD, and $^{13}$C NMR spectra on a 101 MHz Bruker Avance III or 151 MHz Bruker Avance III HD. Chemical shifts are reported in δ (ppm) relative to the singlet at δ = 7.26 ppm of CDCl$_3$, the quintet at 2.50 ppm of DMSO-d$_6$, and the singlet at 4.79 ppm of D$_2$O for $^1$H NMR, and to the centre line of the triplet at δ = 77.16 ppm of CDCl$_3$, the heptuplet at 39.52 ppm of DMSO-d$_6$ for $^{13}$C-NMR. Analytical HPLC was performed using a Dionex Ultimate 3000 pump and Dionex Ultimate 3000 Diode Array Detector (200, 210, 225 and 254 nm) installed with a Phenomenex Gemini-NX 3μ C18 110A, 250 × 4.60 mm column. Solvent A: H$_2$O + 0.1% TFA; Solvent B: MeCN-H$_2$O 9:1 + 0.1% TFA. For HPLC control, data collection and data handling, Chromeleon software v. 6.80 was used. Preparative HPLC was carried out on an Ultimate 3000 Thermo SCIENTIFIC system with a Dionex Ultimate 3000 series pump, a Dionex Ultimate 3000 Diode Array Detector (200, 210, 225 and 254 nm), and a Phenomenex Gemini-NX 5μ C18 110A, 250 × 21.20 mm column for preparative purifications or a Phenomenex Gemini-NX 5μ C18 110A, 250 × 10.00 mm column for semi-preparative purifications. Solvent A: H$_2$O + 0.1% TFA; Solvent B: MeCN-H$_2$O 9:1 + 0.1% TFA. For HPLC control, data collection and data handling, Chromeleon software v. 6.80 was used. UPLC-MS spectra were recorded using an Acquity UPLC H-Class Waters series solvent delivery system equipped with an autoinjector coupled to an Acquity QDa and
TUV detectors installed with an Acquity UPLC® BECH C18 1.7 µm column. Solvent A: 5% aq MeCN + 0.1% HCO$_2$H: Solvent B: MeCN + 0.1% HCO$_2$H. Usually, gradients from A:B 1:0 to 0:1 (5 min) or A:B 1:0 to 0:50 (5 min), were performed depending on the polarity of the compounds. For data collection and data handling, MassLynx software was used. Compounds were dried under high vacuum or freeze dried using a ScanVac Cool Safe Freeze Drier.

**(S)-3-((3-(2-Amino-2-carboxyethyl)-4-bromo-5-methyl-2,6-dioxo-3,6-dihydropyrimidin-1(2H)-yl)methyl)-thiophene-2-carboxylic acid (IH189).**

 commercially available **UBP310** (10.0 mg, 28.0 µmol) was stirred in glacial AcOH (2 mL) until dissolved. Then Br$_2$ in AcOH (1M solution, 85 µl, 85.0 µmol) was added and the reaction was stirred at r.t. for 3 d. The solvent was evaporated *in vacuo* and prep. HPLC ($R_t = 13.38$ and 13.81 min; Gradient run: A:B 100:0 to A:B 20:80 in 25 min) afforded the *title compound* as a mixture of rotamers as white solid (7.41 mg, 17.14 µmol, 61%). Rotamer 1: $^1$H NMR (600 MHz, TFA-d) $\delta$: 7.97 (d, $J = 5.1$ Hz, 1H), 7.36 (d, $J = 5.1$ Hz, 1H), 5.90 (d, $J = 17.3$ Hz, 1H), 5.72 (t, $J = 8.6$ Hz, 2H), 5.26 (s, 1H), 4.82 (dd, $J = 15.9$, 5.6 Hz, 1H), 4.73 (d, $J = 15.6$ Hz, 1H), 2.42 (s, 3H). $^{13}$C NMR (150 MHz, TFA-d) $\delta$: 170.3, 169.2, 168.1, 155.9, 145.6, 134.3, 127.2, 125.7, 87.2, 54.4, 51.9, 48.9, 42.0, 21.9. HPLC: $R_t = 1.37$ min. Rotamer 2: $^1$H NMR (600 MHz, TFA-d) $\delta$: 7.84 (d, $J = 5.1$ Hz, 1H), 7.26 (d, $J = 5.1$ Hz, 1H), 5.77 (d, $J = 17.2$ Hz, 1H), 5.57 – 5.50 (m, 2H), 5.10 (s, 1H), 4.95 (dd, $J = 15.9$, 2.7 Hz, 1H), 4.41 (dd, $J = 15.9$, 6.3 Hz, 1H), 2.28 (s, 3H). $^{13}$C NMR (150 MHz, TFA-d) $\delta$: 170.3, 169.1, 168.0, 154.8, 145.5, 134.3, 127.4, 125.7, 87.1, 54.1, 52.2, 49.7, 42.0, 21.7. HPLC: $R_t = 1.41$ min. LC-MS ($m/z$) calcd for C$_{14}$H$_{15}$BrN$_3$O$_6$S [M + H$^+$]: 432.0, 434.0; found: 431.9, 433.9.
Deuterium experiments (IH249):

1. Bromine IH189 (2.2 mg, 5.09 µmol) was dissolved in deionized H₂O (0.1 mL) and Pd/C (0.6 mg, 0.51 µmol) was added. The flask was evacuated and backfilled with argon gas (2x) and evacuated and backfilled with D₂ gas. The reaction was stirred under a D₂ atmosphere at 40 °C for 7 h. The reaction mixture was filtered through a PVD 0.45 µm filter, washed with deionized H₂O and the solvent was removed on the freeze-dryer. ¹H-NMR analysis of the crude reaction mixture only showed UBP310.

2. Bromine IH189 (0.57 mg, 1.18 µmol) was dissolved in dry DMSO (0.05 mL) and Pd/C (0.2 mg, 0.2 µmol) was added. The flask was evacuated and backfilled with argon gas (x2) and evacuated and backfilled with D₂ gas. The reaction was stirred under a D₂ atmosphere at 40 °C for 2.5 h. The reaction mixture was filtered through a PVD 0.45 µm filter, washed and the solvent was removed on the freeze-dryer. ¹H-NMR analysis of the crude reaction mixture only showed UBP310.

3. Bromine IH189 (0.3 mg, 0.69 µmol) was dissolved in a 0.1 M NaOH in D₂O sol. (14 µL, 1.39 µmol) and Pd/C (0.07 mg, 0.07 µmol) was added. The disodium salt was stirred under a D₂ atmosphere (1 atm.) at 40 °C for 2.5 h. The solution was cooled to r.t., filtered through a PVD 0.45 µm filter, washed with D₂O (0.1 ml) and the solvent was evaporated via freeze drying.

4. Bromine IH189 (1.94 mg, 3.55 µmol) was dissolved in 0.1 M NaOH in D₂O solution until the solution reached pH = 8. Pd/C (0.07 mg, 0.07 µmol) was added and the flask was evaporated.
and backfilled with D₂ gas (x3). The reaction mixture was stirred under D₂ atmosphere at r.t. for 2 h then filtered through a PVD 0.45 µm filter and the solvent was removed on the freeze-dryer.

5. Bromine **IH189** (0.70 mg, 1.49 µmol) was dissolved in dry, degassed DMF (30 µL) and NEt₃ (1 µl, 7.45 µmol) was added followed by Pd/C (0.01 mg, 0.15 µmol). The flask was evaporated and backfilled with D₂ gas (3 x) and the reaction was stirred under D₂ atmosphere at r.t. for 2 h. The mixture was diluted with H₂O (0.5 ml), filtered through a PVD 0.45 µm filter and the filtrate washed with H₂O (0.5 ml). The solution was frozen and evaporated on the freeze-dryer.

\(^1\)H NMR (600 MHz, TFA-d) δ: 7.60 (d, \(J = 5.1 \) Hz, 1H), 6.90 (d, \(J = 5.1 \) Hz, 1H), 5.57 (d, \(J = 17.3 \) Hz, 1H), 5.33-5.35 (m, 2H), 4.77-4.80 (m, 1H), 4.29 (dd, \(J = 10.8, 7.3 \) Hz, 1H), 1.80 (s, 3H).

HPLC: \(R_t = 1.14\)min; LC-MS (m/z) calcd for C\(_{14}\)H\(_{14}\)N\(_3\)O\(_6\)S [M + H\(^+\)]: 352.2; found: 352.1.

**Methyl 3-((3-(2-amino-3-methoxy-3-oxopropyl)-5-methyl-2,6-dioxo-3,6-dihydropyrimidin-1(2H)-yl)methyl)thiophene-2-carboxylate (IH266).**

![Chemical structure](image)

Commercially available **UBP310** (2.44 mg, 6.90 µmol) was added to a cooled solution of SOCl₂ in MeOH (10%, 0.2 mL) and then stirred at 40 °C for 16 h. The reaction was cooled to r.t., the solvent evaporated and the residue taken up in H₂O and evaporated on the freeze dryer to afford the **title compound** as a crude yellow solid (HCl salt, 3.05 mg, 7.30 µmol, quant.); \(^1\)H NMR (600 MHz, MeOD) δ: 7.56 (d, \(J = 3.7 \) Hz, 1H), 7.45 (s, 1H), 6.80 (d, \(J = 3.6 \) Hz, 1H), 5.53 – 5.39 (m, 2H), 4.47 (s, 1H), 4.32 (s, 1H), 4.28 (s, 1H), 3.87 (s, 3H), 3.82 (s, 3H), 1.93 (s, 3H); \(^{13}\)C NMR (150 MHz, CDCl₃) δ: 168.5, 165.2, 164.1, 153.7, 146.3, 141.3, 132.3, 128.9, 128.1, 111.5, 54.2, 53.4, 52.5,
Methyl (S)-3-((3-(2-amino-3-methoxy-3-oxopropyl)-4-bromo-5-methyl-2,6-dioxo-3,6-
dihydropyrimidin-1(2H)-yl)methyl)thiophene-2-carboxylate (IH268).

Dimethyl ester IH266 (3.05 mg, 7.30 µmol) was dissolved in AcOH (0.2 ml), Br₂ (1M in AcOH, 22 µl, 22 µmol) was added and the reaction stirred at r.t. for 4 d. The mixture was diluted with H₂O, frozen and the solvent was evaporated on the freeze-dryer. HPLC: Rᵣ = 1.55 and 1.61 min; ¹H NMR (600 MHz, MeOD) δ: 7.58 (d, J = 4.5 Hz, 1H), 6.96 (d, J = 4.9 Hz, 1H), 5.42 – 5.38 (m, 1H), 5.25 – 5.19 (m, 1H), 4.41 (s, 1H), 4.16-4.12 (m, 1H), 4.06 – 3.99 (m, 1H), 3.88 (s, 6H), 3.86 (s, 3H). LC-MS (m/z) calcd for C₁₆H₁₉BrN₃O₆S [M + H⁺]: 460.0, 462.0; found: 460.0, 462.0.

Deuterium experiments with bromine IH268:

1. Bromine IH268 (1.0 mg, 2.17 µmol) was dissolved in MeOD (0.4 ml), Pd/C (0.1 mg, 0.1 µmol) was added and the flask was evaporated and backfilled with D₂ gas (3x). The reaction mixture was stirred under D₂ atmosphere at r.t. for 5 h then filtered through a PVD 0.45 µm filter and the solvent was removed on the freeze-dryer. Crude ¹H-NMR was identical with IH266.

2. Bromine IH268 (1.0 mg, 2.17 µmol) was dissolved in degassed DMF (0.5 ml) and the solution was neutralized to pH=7 with NEt₃. Pd/C (0.1 mg, 0.1 µmol) was added and the flask was
evaporated and backfilled with D$_2$ gas (3x). The reaction mixture was stirred under D$_2$ atmosphere at r.t. for 4 h then filtered through a PVD 0.45 µm filter and the solvent was removed on the freeze-dryer. Compound could not be identified as IH266 or the lactone. LC-MS found: 398.1.

**Methyl (S)-3-((3-(2-((tert-butoxycarbonyl)amino)-3-methoxy-3-oxopropyl)-5-methyl-2,6-dioxo-3,6-dihydropyrimidin-1(2H)-yl)methyl)thiophene-2-carboxylate (IH273).**

IH266 (10.0 mg, 23.93 µmol) was dissolved in MeOH (0.5 mL) and cooled to 0 °C. NEt$_3$ (3.3 µL, 23.93 µmol) followed by BOC$_2$O (5.8 mg, 26.57 µmol) were added and the reaction was stirred at r.t. for 2 d. The solvent was evaporated and the residue dissolved in EtOAC (1 mL) and washed with H$_2$O (2 x 0.5 mL). The organic phase was dried over MgSO$_4$, filtered and evaporated to afford the title compound as a crude off-white solid (10 mg, 20.77 µmol, 87%); $R_f$ = 0.50 (DCM/EtOAc 2:1); $^1$H NMR (600 MHz, MeOD) δ: 7.54 (d, $J = 5.2$ Hz, 1H), 7.38 (s, 1H), 6.94 (d, $J = 5.1$ Hz, 1H), 5.50 (d, $J = 16.4$ Hz, 1H), 5.41 (d, $J = 16.5$ Hz, 1H), 4.67 (dd, $J = 9.6$, 4.5 Hz, 1H), 4.45 (dd, $J = 13.8$, 4.6 Hz, 1H), 3.89 (s, 3H), 3.74 (s, 3H), 3.69 (d, $J = 9.9$ Hz, 1H), 1.90 (s, 3H), 1.39 (s, 9H). LC-MS (m/z) calcd for C$_{21}$H$_{28}$N$_3$O$_8$S [M + H$^+$], 482.1; found; 482.1.

**Methyl (S)-3-((4-bromo-3-((tert-butoxycarbonyl)amino)-3-methoxy-3-oxopropyl)-5-methyl-2,6-dioxo-3,6-dihydropyrimidin-1(2H)-yl)methyl)thiophene-2-carboxylate (IH274).**

Methyl (S)-3-((4-bromo-3-((tert-butoxycarbonyl)amino)-3-methoxy-3-oxopropyl)-5-methyl-2,6-dioxo-3,6-dihydropyrimidin-1(2H)-yl)methyl)thiophene-2-carboxylate (IH274).
Dimethyl ester carbamate **IH273** (34.58 mg, 71.82 µmol) was dissolved in CDCl₃/AcOH (1:1, 1 mL) and NBS (33.4 mg, 187.66 µmol) was added. The reaction was stirred at r.t. under N₂ atmosphere for 3 h. The reaction was diluted with H₂O (1 ml) and the phases were separated. The organic phase was washed with H₂O (1 ml), dried over MgSO₄, filtered and evaporated. Prep. TLC (heptane/EtOAc 1:1) afforded the title compound as a clear oil (15.68 mg, 27.98 µmol, 39%); Rᵣ = 0.16 (heptane/EtOAc 2:1); ¹H NMR (400 MHz, CDCl₃) δ: 7.34 (d, J = 5.1 Hz, 1H), 6.93 (d, J = 5.1 Hz, 1H), 6.13 (s, 1H), 5.50 (d, J = 16.9 Hz, 1H), 5.19 (d, J = 16.9 Hz, 1H), 4.67 (t, J = 8.1 Hz, 1H), 4.32 (dd, J = 14.4, 10.7 Hz, 1H), 3.88 (s, 3H), 3.81 (s, 3H), 3.49 (dd, J = 14.4, 3.6 Hz, 1H), 2.04 (s, 3H), 1.39 (s, 9H). LC-MS (m/z) calcd. for C₂₁H₂₇BrN₃O₈S [M + H⁺]: 560.1, 562.1; found: 460.1, 462.1 [M + H⁺ – Boc].

**Deuterium experiments with bromine IH274:**

1. Bromine **IH274** (1.36 mg, 2.43 µmol) was dissolved in dry THF (0.1 mL) and Pd/C (0.3 mg, 0.28 µmol) was added. The vial was evacuated and backfilled with D₂ gas. The reaction was stirred at r.t. for 20 h. The reaction was filtered and the solvent was evaporated to afford a crude residue; Rᵣ = 0.29 (heptane/EtOAc 1:1); LC-MS (m/z) calcd for C₂₁H₂₈N₃O₈S [M + H⁺], 482.1; found: 482.2.

2. An oven-dried vial was evacuated and backfilled with N₂ (3x) and then filled with Pd/C (10 wt%, 0.2 mg, 0.19 µmol). The vial was again evacuated and backfilled with N₂ (2x) before evacuated and backfilled with D₂. Bromine **IH274** (0.4 mg, 0.17 µmol), dissolved in dry THF (0.05 mL), was added via a syringe and the reaction mixture was stirred at r.t. for 3.5 h. LC-MS (m/z) calcd for C₂₁H₂₈N₃O₈S [M + H⁺], 482.1; found: 482.2.
HRMS spectrum (negative mode) of $^{3}$H-NF608
HPLC prep trace of [3H]NF608

Sample Name: PREP_AM15335; Date Acquired: 12/2/2015 10:33:15 AM CET; Channel: UV 215 nm; Inject.Vol.: 700.00; Instrument Method: Prep_3H_Synergy 4u POLAR RP80
Sample Name: PREP_AM15335; Date Acquired: 12/2/2015 10:33:15 AM CET; Channel: UV 245 nm; Inject.Vol.: 700.00; Instrument Method: Prep_3H_Synergy 4u POLAR RP80
Sample Name: PREP_AM15335; Date Acquired: 12/2/2015 10:33:15 AM CET; Channel: RAD (LSC 500 ul, 2.8ml QSF 302/min); Inject.Vol.: 700.00; Instrument Method: Prep_3H_Synergy 4u POLAR RP80

Error Log
All Peaks Table group contains information that doesn’t match the data being reported.
HPLC analytical trace of [3H]NF608

Project Name: glutamate NF6081

Acquired: 12/1/2015 6:28:08 PM CET

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Sample Name: HOT_AM15335_crude after lyo; Date Acquired: 12/1/2015 6:28:08 PM CET; Channel: UV 215 nm; Inject Vol.: 10.00; Instrument Method: ANAL_3H_Synergi 4u POLAR RP80

Sample Name: HOT_AM15335_crude after lyo; Date Acquired: 12/1/2015 6:28:08 PM CET; Channel: UV 245 nm; Inject Vol.: 10.00; Instrument Method: ANAL_3H_Synergi 4u POLAR RP80

Sample Name: HOT_AM15335_crude after lyo; Date Acquired: 12/1/2015 6:28:08 PM CET; Channel: RAD (LSC 500 ul, 2.8ml QSF 302/min); Inject Vol.: 10.00; Instrument Method: ANAL_3H_Synergi 4u POLAR RP80

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Error Log
All Peaks Table group contains information that doesn’t match the data being reported.
HPLC analytical trace of NF608 (cold)

Sample Name: STD_NF608; Date Acquired: 11/17/2015 11:32:26 AM CET; Channel: UV 215 nm; Inject.Vol.: 10.00; Instrument Method: ANAL_3H_Synergi 4u POLAR RP80

Sample Name: STD_NF608; Date Acquired: 11/17/2015 11:32:26 AM CET; Channel: UV 245 nm; Inject.Vol.: 10.00; Instrument Method: ANAL_3H_Synergi 4u POLAR RP80

Error Log
All Peaks Table group contains information that doesn't match the data being reported.
$^1$H NMR of IH189
$^1$H NMR of IH268
$^1$H NMR of **IH274**