**SUPPORTING INFORMATION**

# Rapid On-demand Synthesis of Lomustine under Continuous Flow Conditions

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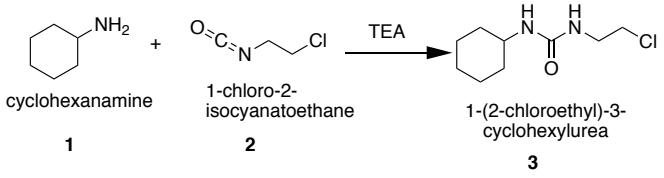
## General Information

### DESI-MS Analysis

The DESI-MS evaluation was done following the previously published method of Wleklinski *et al*[1] except that the density of reaction spots was 1536 spots/plate instead of 6144/plate using reagents that were pipetted into standard polypropylene 384-well plates using a liquid handling robot (Biomek i7; Beckman-Coulter, US). DESI-MS slides were fabricated from porous PTFE sheets (EMD, Millipore Fluoropore, Saint-Gobain) glued onto a glass support (Foxx Life Sciences). The PTFE sheet was cut with scissors and bonded to the glass slides using spray adhesive (Scotch Spray mount). No signs of interference from the glue was observed. The reagents were mixed at 1:1 stoichiometry in various solvents (EtOAc, THF, DMSO, Toluene, ACN, DCM, EtOH and MeOH) and rhodamine B dye was added to some wells of the plate as a fiducial marker. After the reagents were mixed, 50 nL of the reactions were deposited onto a porous PTFE surface at 1,536 spot density using a magnetic pin tool equipped with slotted transfer pins. DESI-MS data was acquired using a linear ion trap mass spectrometer (LTQ XL; Thermo Scientific, San Jose, CA) equipped with a commercial DESI-imaging source (DESI 2D source, Prosolia Inc., Indianapolis, IN). The instrument was controlled using Xcalibur v. 4.0 software to run worklists for DESI-MS data acquisition. The DESI spray angle was 55° using MeOH as spray solvent, and with an applied voltage of 5kV. Mass spectra were acquired at the positive ion mode over the *m/z* range of 50-500. The DESI-MS imaging lateral resolution was 350 µm. This was achieved using stage speed of 4,376 µm/sec and the instrument scan time of 80 ms. For data processing, data were visualized using an in-house software designed[1] to automatically search for the *m/z* values of reactants, intermediates, and lomustine fragments to generate a YES/NO visualization output for each spot in the PTFE plate imaged by DESI-MS. Data files also were combined into .img files using Firefly software (Prosolia Inc., Indianapolis, IN). Ion images were plotted using BioMAP (Novartis, freeware). The expected *m/z* values for the lomustine fragments were plotted and visualized using the BioMAP rainbow false color scale where the minimum and maximum ion intensity values were set to the best contrast for each ion.

### Carbamylation of Cyclohexylamine in flow




cyclohexanamine **1**

1-chloro-2-isocyanatoethane **2**

1-(2-chloroethyl)-3-cyclohexylurea **3**

Scheme S1: Synthesis of **3** in flow

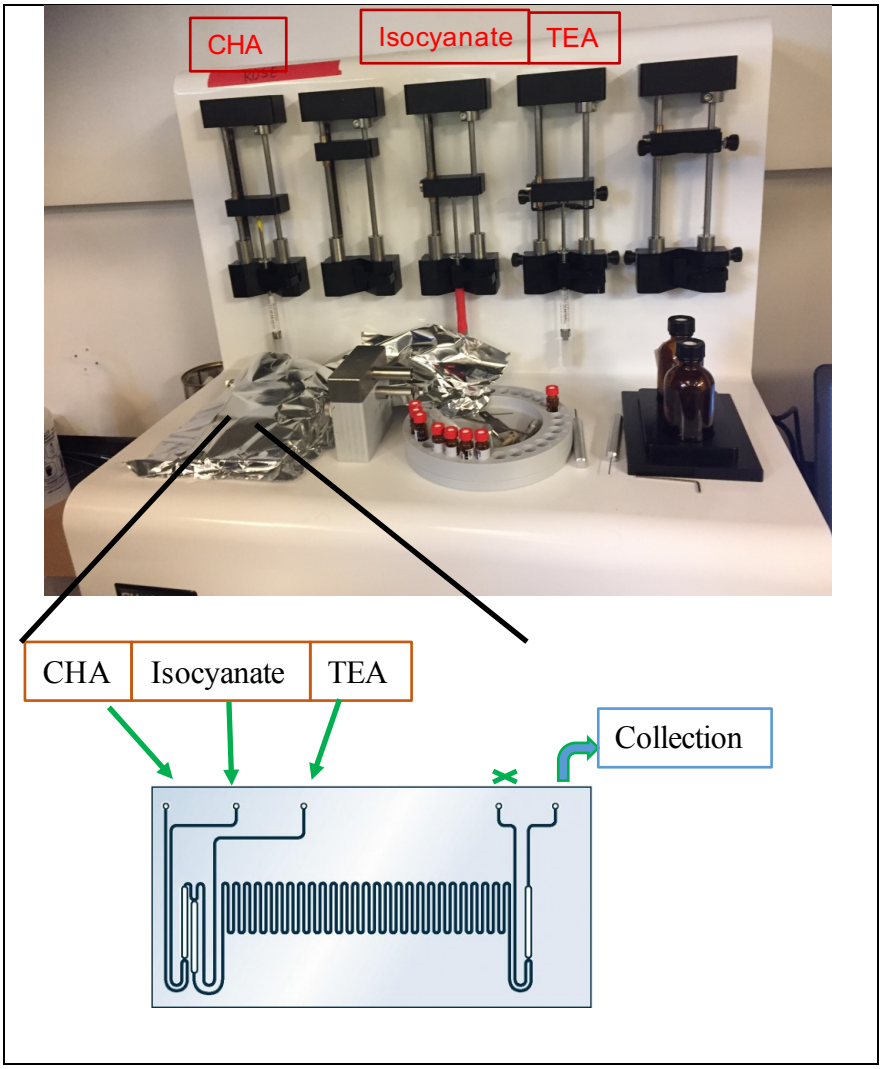



Figure S1: Set up of continuous flow synthesis of 3, **3** using chemtrix S1 system

**NMR**

$^{1}$H NMR (500 MHz, $CDCl_3$, ppm): $\delta_H$ = 4.84 (t, J= 5.85, 1 H), 4.42 (d, J = 7.35 Hz, 1 H), 3.62 (t, J=5.60Hz, 2 H), 3.54 (t, J=5.70Hz, 2 H), 3.51-3.45 (m, 1 H), 1.95-1.92 (m, 2 H), 1.72-1.67 (m, 2 H), 1.62-1.58 (m, 1 H), 1.39-1.30 (m, 2 H), 1.19-1.06 (m, 3 H); $^{13}$C NMR (500 MHz, $CDCl_3$, ppm): $\delta_C$= 157.04, 49.29, 45.25, 42.12, 33.88, 25.57, 24.9.

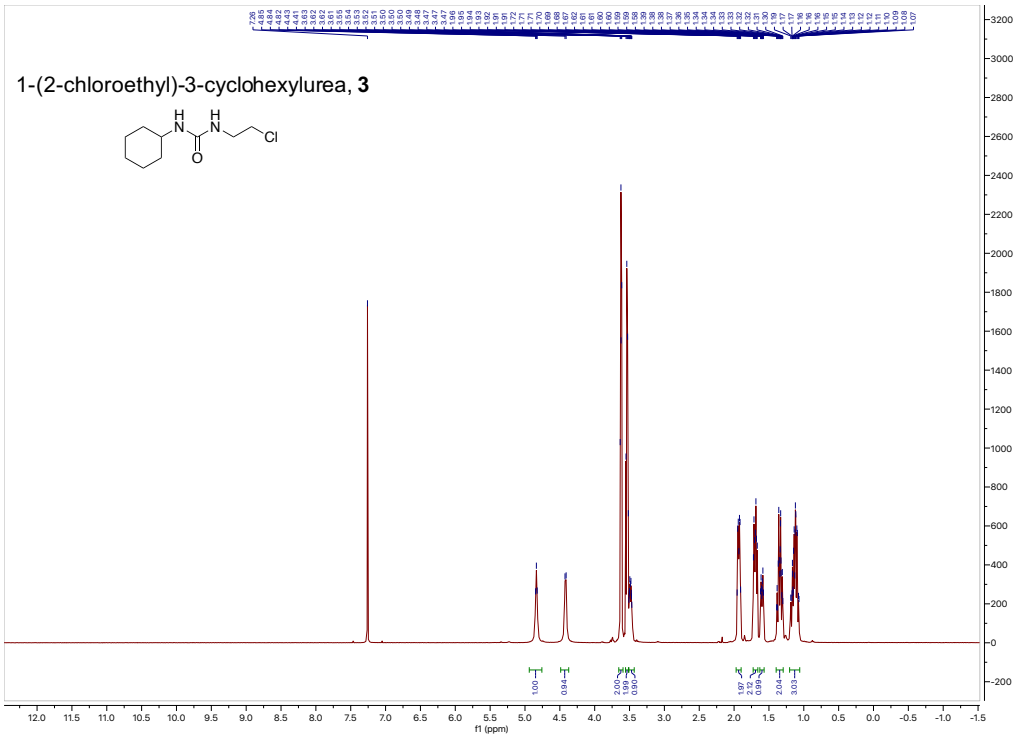



Figure S2: $^{1}$H NMR of 1-(2-chloroethyl)-3-cyclohexylurea, **3,** from flow synthesis

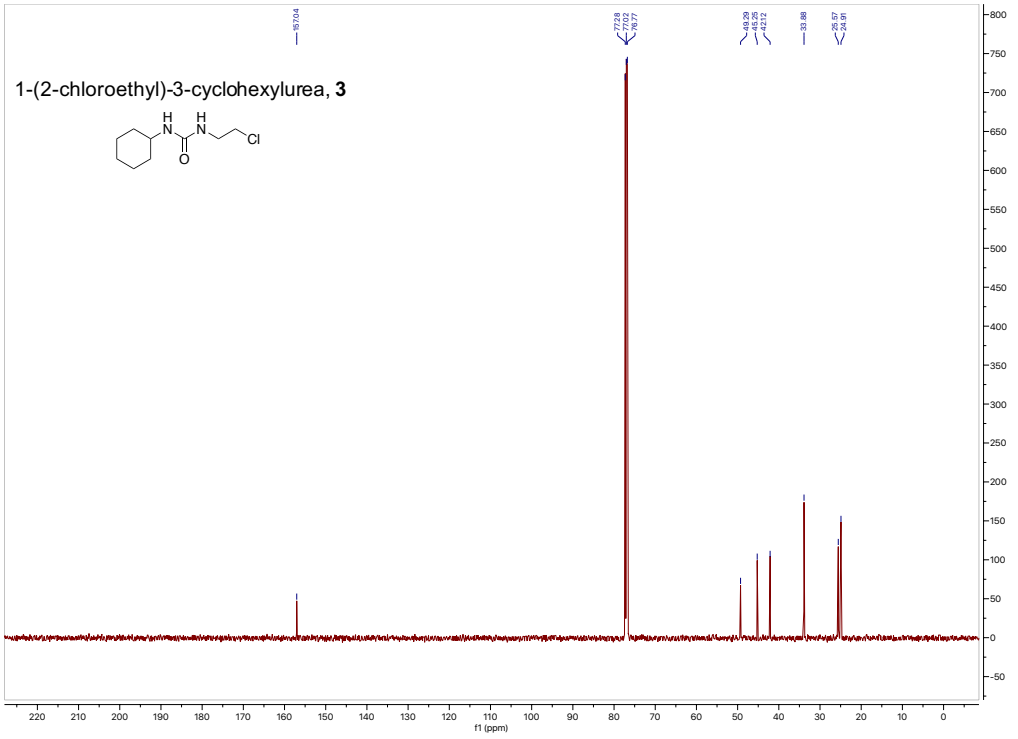



Figure S3: $^{13}$C NMR of 1-(2-chloroethyl)-3-cyclohexylurea, **3,** from flow synthesis

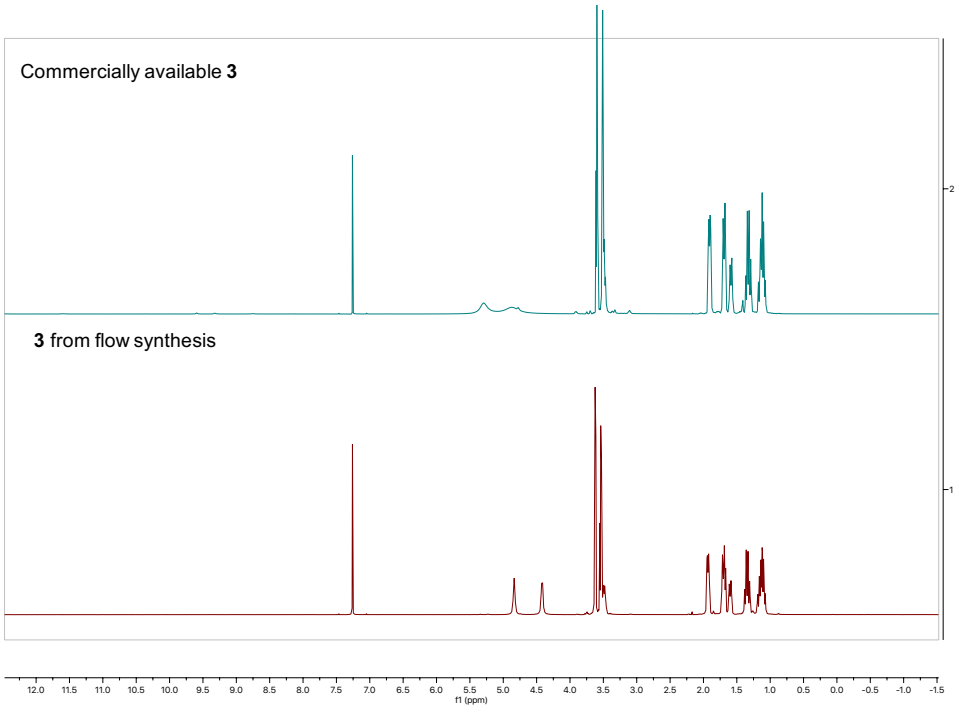



Figure S4: Comparison of $^{1}$H NMR of 1-(2-chloroethyl)-3-cyclohexylurea, **3**, derived from flow synthesis with commercially available **3.**

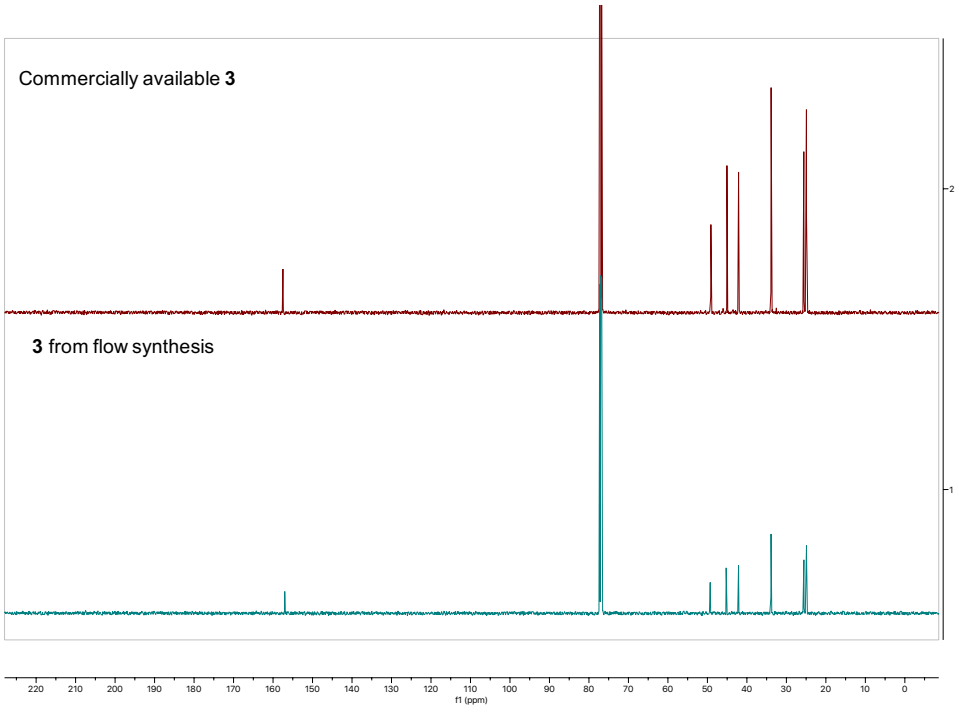
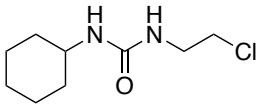


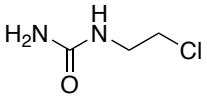

Figure S5: Comparison of $^{13}$C NMR of 1-(2-chloroethyl)-3-cyclohexylurea, **3**, derived from flow

synthesis with commercially available **3.**

**ESI-MS**

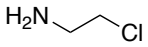


Chemical Formula: $C_9H_{17}ClN_2O$
Monoisotopic Exact Mass: 204.10



Chemical Formula: $C_3H_7ClN_2O$
Monoisotopic Exact Mass: 122.02

Chemical Formula: $C_6H_{11}^+$
Monoisotopic Exact Mass: 83.09



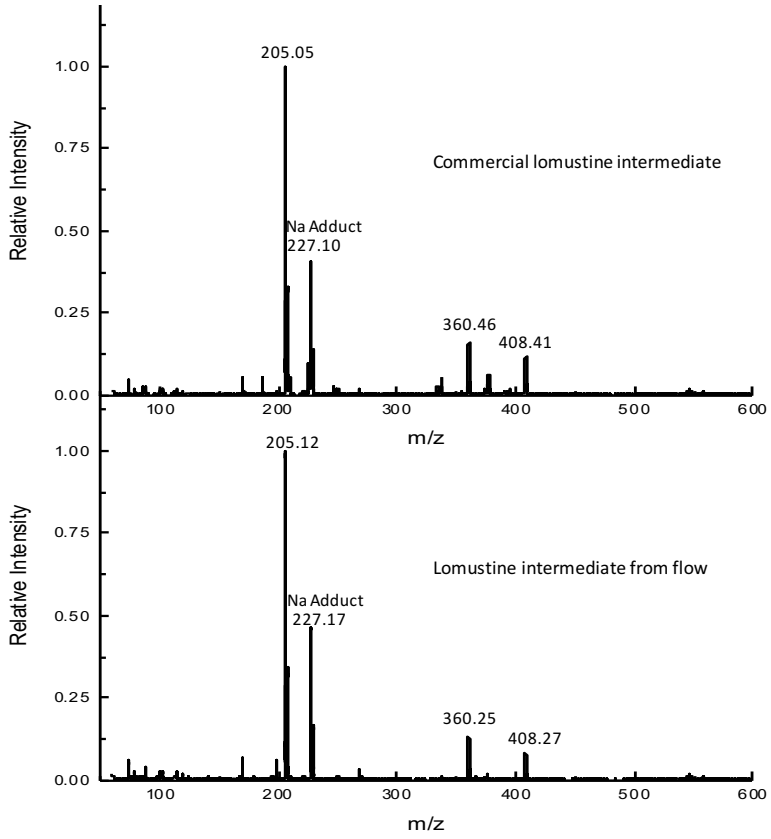
Chemical Formula: $C_2H_6ClN$
Monoisotopic Exact Mass: 79.02

Scheme S2: Fragments from 1-(2-chloroethyl)-3-cyclohexylurea, **3**

ESI-MS (*m/z*): 205/207 ($M+H^+$), 227/229 ($M+Na^+$).

ESI-MS/MS of *m/z* 205: 205 ($M+H^+$), 123 ($C_3H_7ClN_2O+H^+$), 83 ($C_6H_{11}^+$) (80 ($C_2H_6ClN^+$)

Full ESI-MS



MS/MS of *m/z*=205

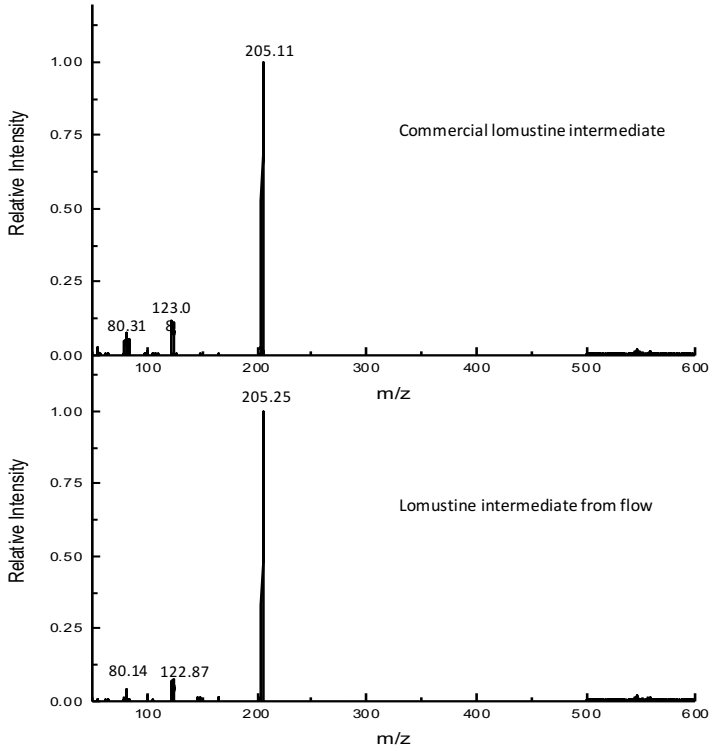



Figure S6: Full ESI-MS scan and MS/MS of commercially available **3** and synthesized **3** derived from flow under the reaction conditions of 50 °C, 1 min.

**DESI-MS Screening.**

**DESI-MS Outline.**

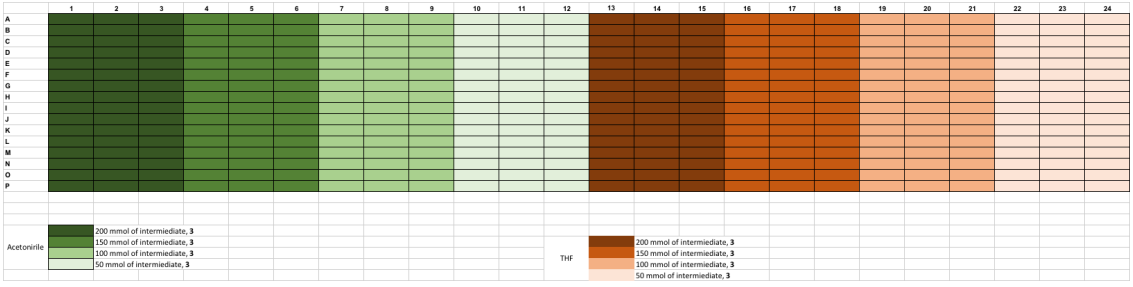



Figure S7: DESI master plate layout using four different concentrations in two solvents.

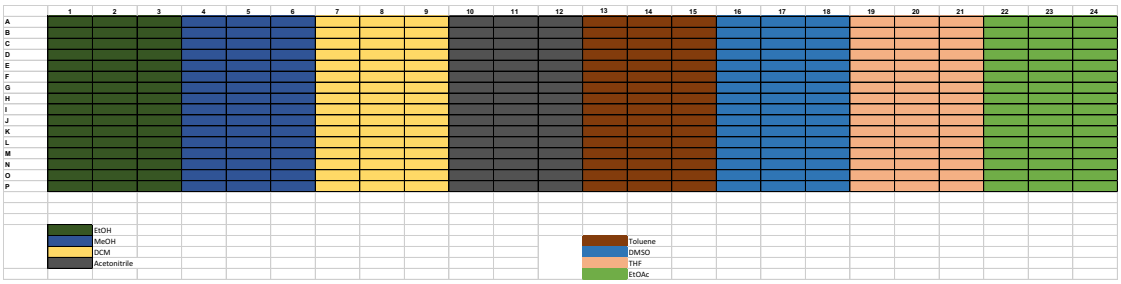



Figure S8: DESI master plate layout using eight different solvents.

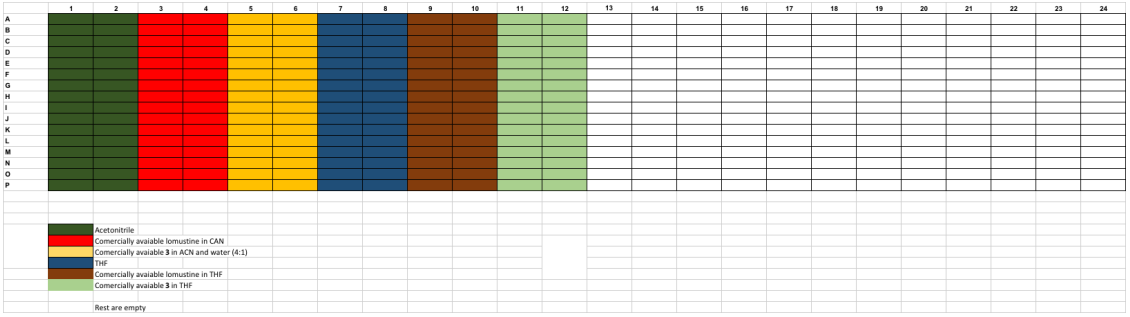



Figure S9: DESI master plate layout using only commercially available **3** and lomustine

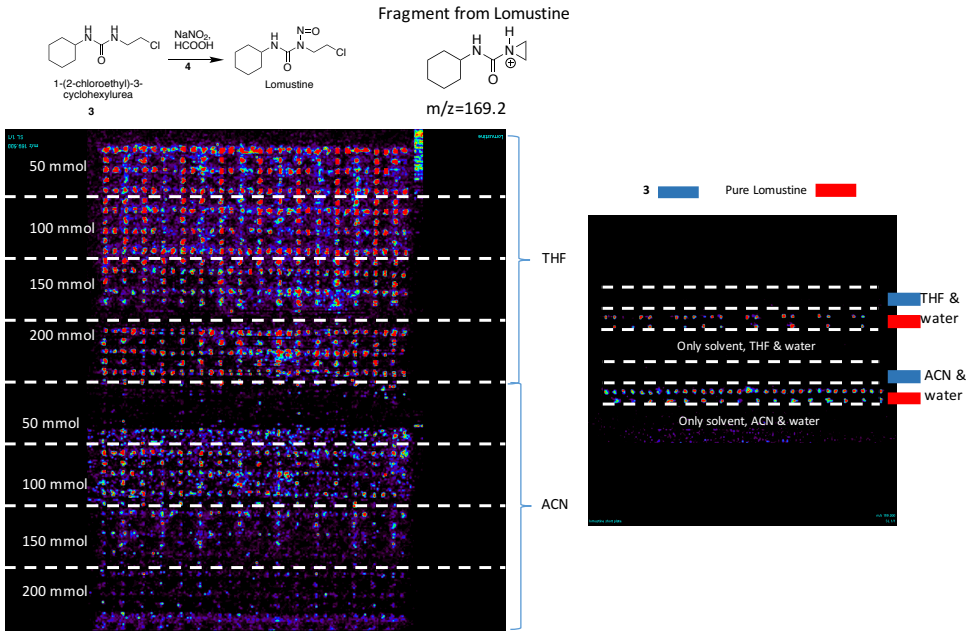



Figure S10: (left) Direct DESI-MS data comparison between the two nitrosation reactions in THF and ACN in different concentration. (right) DESI-MS data of commercially available **3** and lomustine standards. The data was analyzed using BioMAP imaging software.

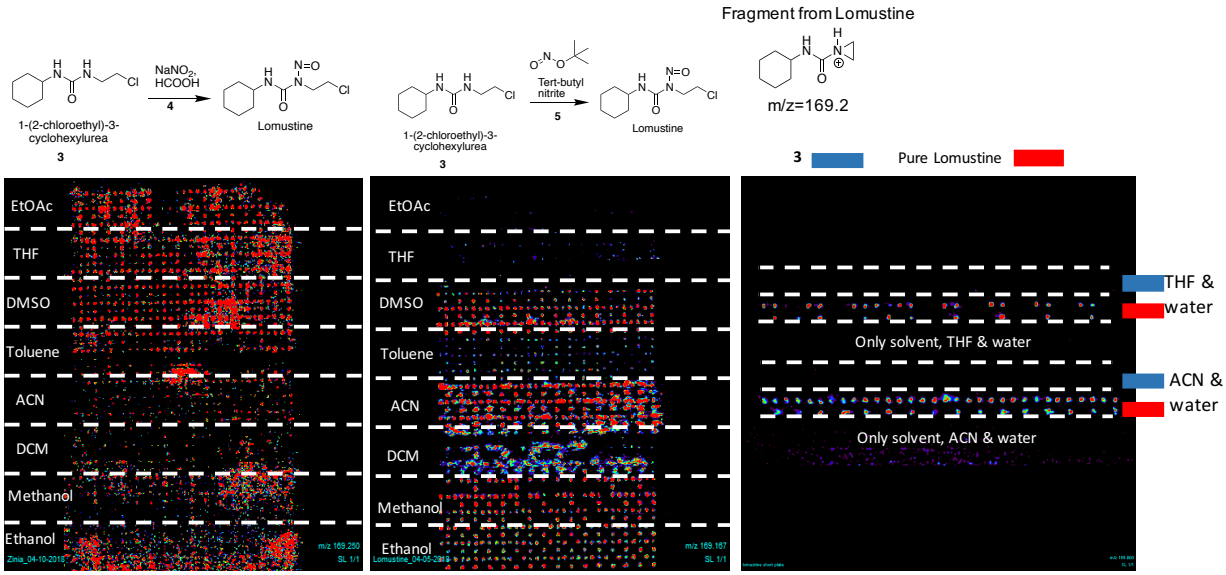
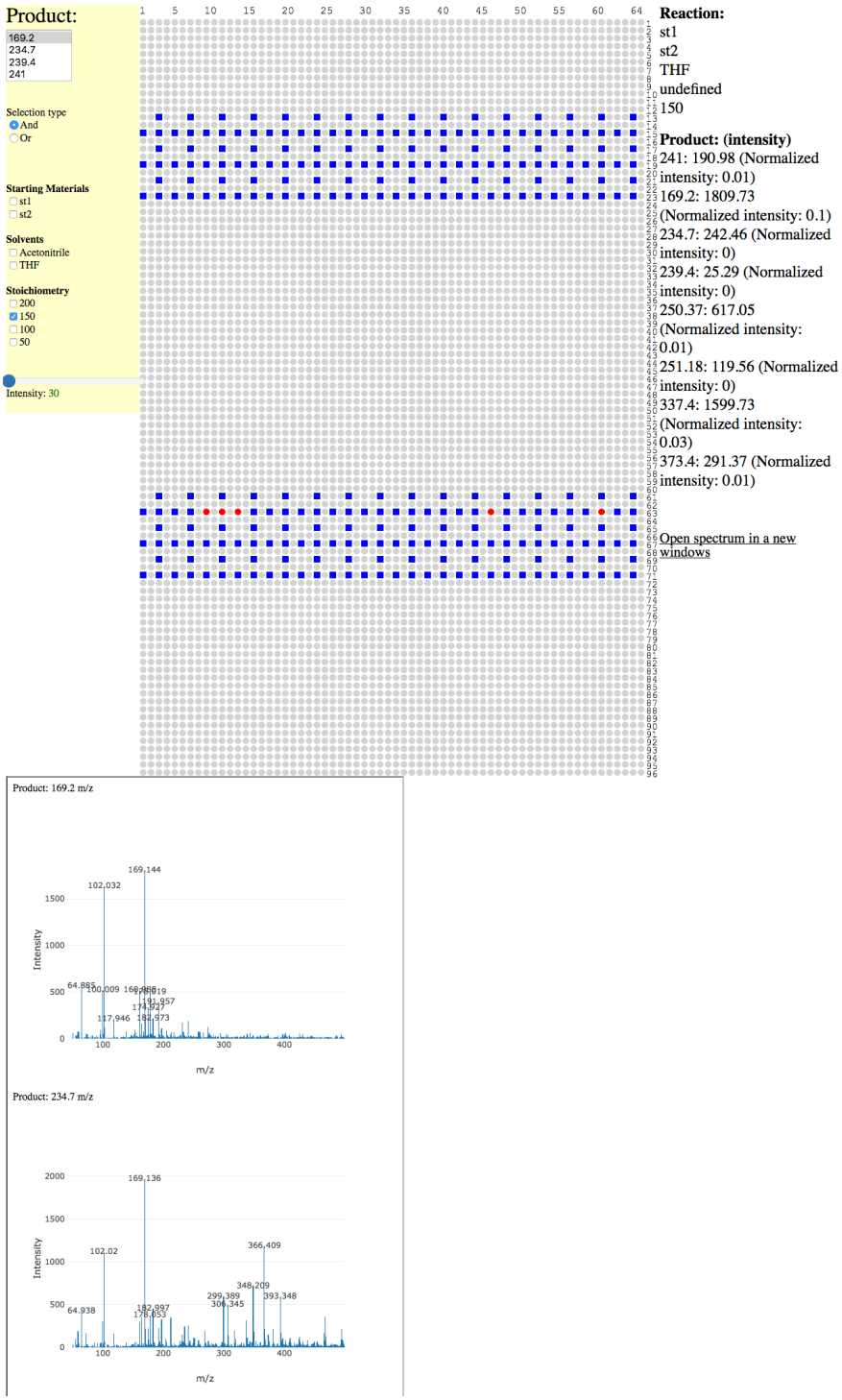
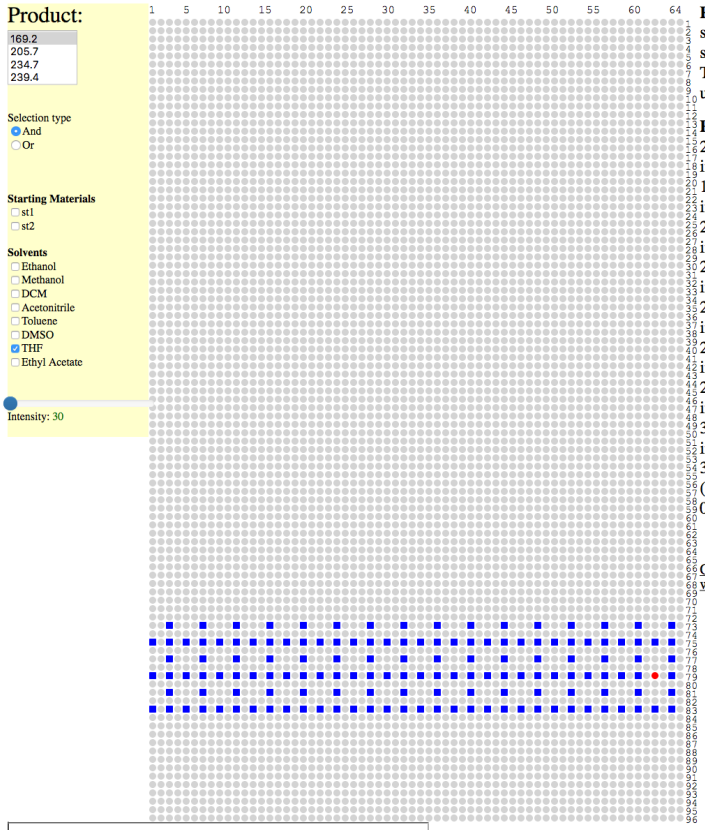



Figure S11: (left & center) Direct DESI-MS data comparison between the two nitrosation reactions in different solvents. (right) DESI-MS data of commercially available **3** and lomustine standards. The data was analyzed using BioMAP imaging software.

A


Product:
169.2
234.7
239.4
241
Selection type
And
Or
Starting Materials
st1
st2
Solvents
Acetonitrile
THF
Stoichiometry
200
150
100
50
Intensity: 30
Reaction:
st1
st2
THF
undefined
150
Product: (intensity)
241: 190.98 (Normalized intensity: 0.01)
169.2: 1809.73 (Normalized intensity: 0.1)
234.7: 242.46 (Normalized intensity: 0)
239.4: 25.29 (Normalized intensity: 0)
250.37: 617.05 (Normalized intensity: 0.01)
251.18: 119.56 (Normalized intensity: 0)
337.4: 1599.73 (Normalized intensity: 0.03)
373.4: 291.37 (Normalized intensity: 0.01)
Open spectrum in a new windows
Product: 169.2 m/z
169,144
102,032
Intensity
m/z
Product: 234.7 m/z
169,136
102,02
366,409
348,209
299,389
306,345
393,348
64,938
182,997
178,053

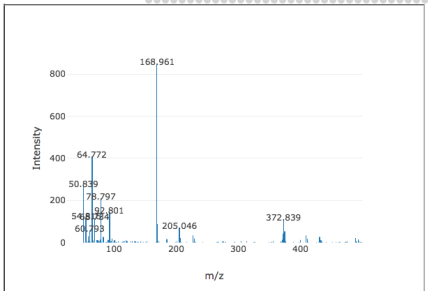
B



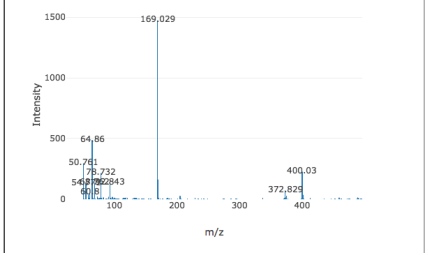

**Reaction:**
st1
st2
THF
undefined

**Product: (intensity)**
241: 5.07 (Normalized intensity: 0)
169.2: 849.03 (Normalized intensity: 0.22)
205.7: 3.13 (Normalized intensity: 0)
234.7: 0 (Normalized intensity: 0)
239.4: 71.86 (Normalized intensity: 0)
250.37: 0 (Normalized intensity: 0)
251.18: 86.73 (Normalized intensity: 0)
337.4: 136.38 (Normalized intensity: 0)
373.4: 1599.04 (Normalized intensity: 0.05)

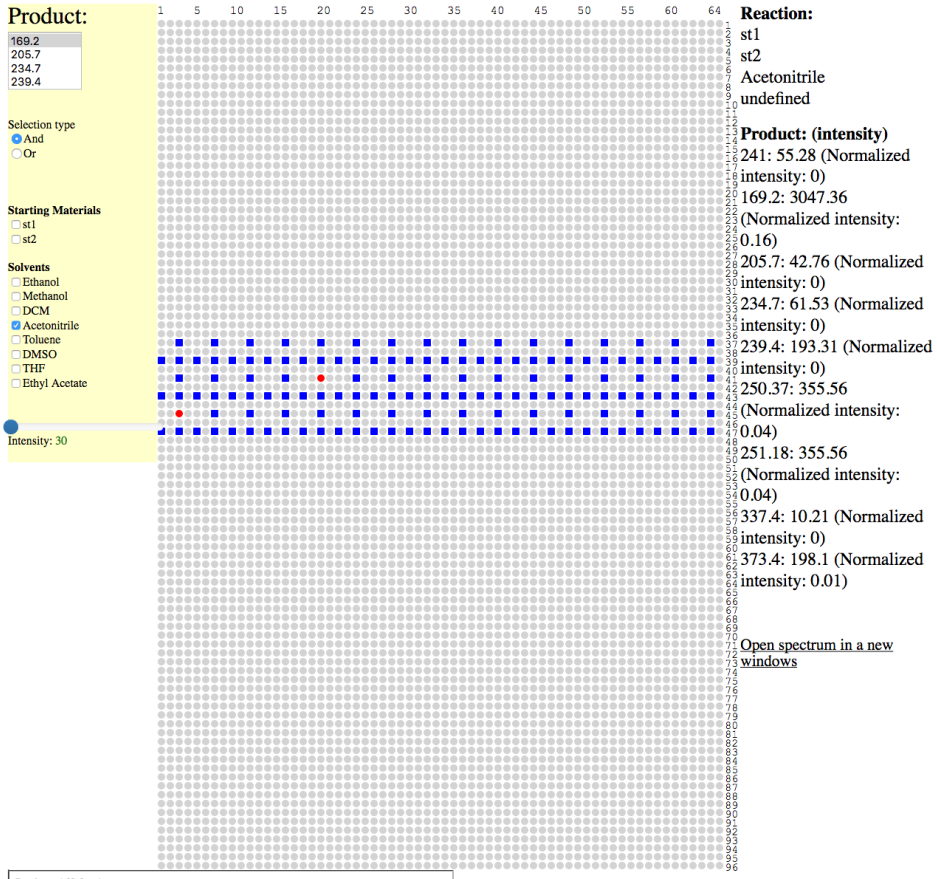
Open spectrum in a new windows




Product: 205.7 m/z



C


Product:
169.2
205.7
234.7
239.4
Selection type
And
Or
Starting Materials
st1
st2
Solvents
Ethanol
Methanol
DCM
Acetonitrile
Toluene
DMSO
THF
Ethyl Acetate
Intensity: 30
1 5 10 15 20 25 30 35 40 45 50 55 60 64
Reaction:
st1
st2
Acetonitrile
undefined
Product: (intensity)
241: 55.28 (Normalized intensity: 0)
169.2: 3047.36 (Normalized intensity: 0.16)
205.7: 42.76 (Normalized intensity: 0)
234.7: 61.53 (Normalized intensity: 0)
239.4: 193.31 (Normalized intensity: 0)
250.37: 355.56 (Normalized intensity: 0.04)
251.18: 355.56 (Normalized intensity: 0.04)
337.4: 10.21 (Normalized intensity: 0)
373.4: 198.1 (Normalized intensity: 0.01)
Open spectrum in a new windows

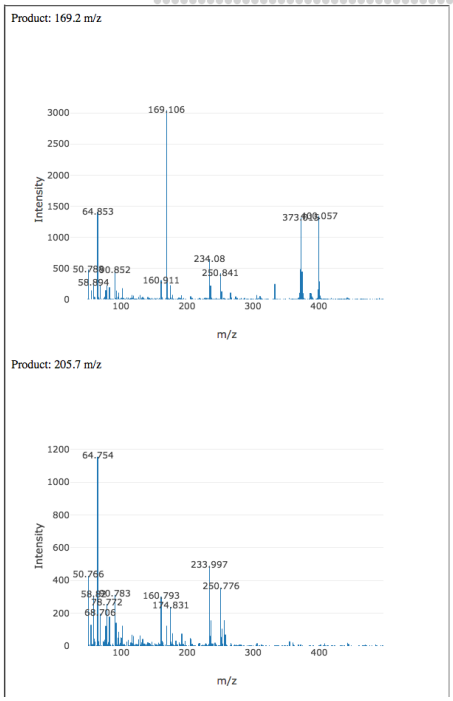

Product: 169.2 m/z
Intensity
m/z
169.106
64.853
373.405
373.057
234.08
250.841
160.911
Product: 205.7 m/z
Intensity
m/z
64.754
50.766
233.997
250.776
160.793
174.831

Figure S12: Map of the DESI-MS plates showing some of the expected ions where the nitrosation reaction using was screened using different stoichiometries as well as commercially available standards. Green dots indicate the presence of the *m/z* expected for the reaction product (successful reaction), Red dots indicate that the expected *m/z* for the reaction product was not present at the reaction spot (unsuccessful reaction condition). A: $NaNO_2$, concentration screening using the lomustine ion (*m/z* 169) intensity; B: $NaNO_2$, solvent screening using the lomustine ion (*m/z* 169) intensity; C: TBN, solvent screening using the lomustine ion (*m/z* 169) intensity

**Nitrosation of 1-(2-chloroethyl)-3-cyclohexylurea, 3 in flow.**

Three purification methods were examined to purify the compounds as described in the main manuscript. The NMR spectra for the different purification methods are shown here for comparison.

**NMR**

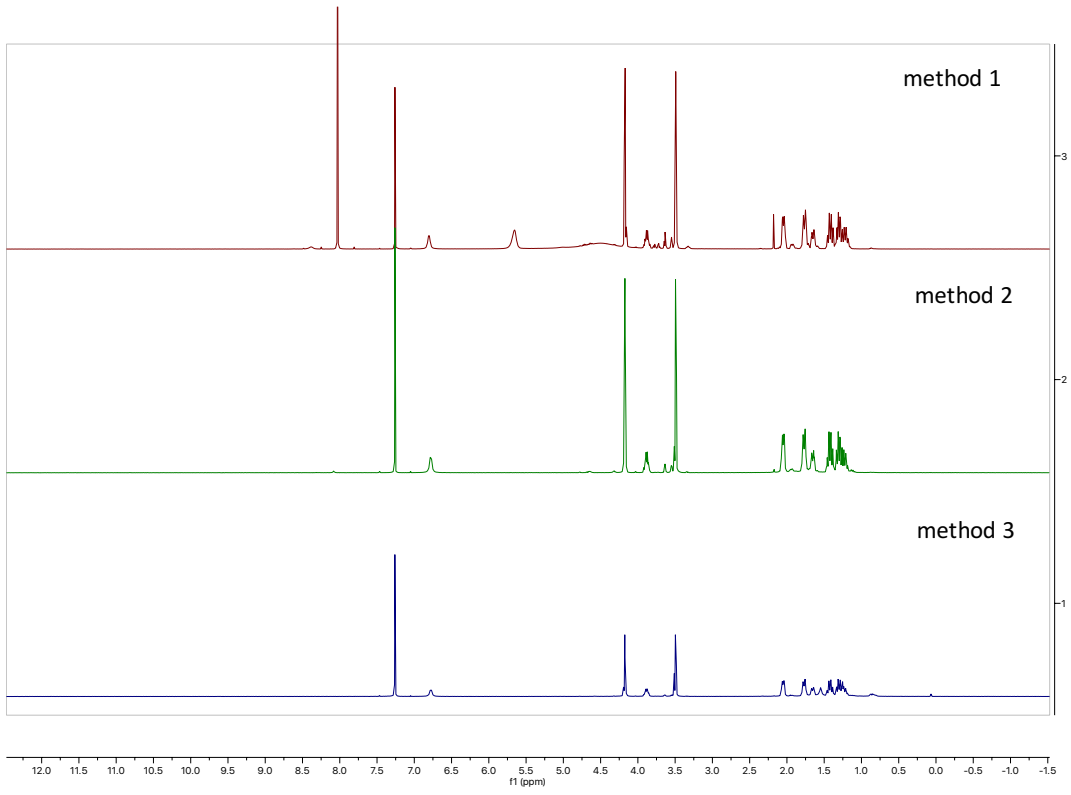
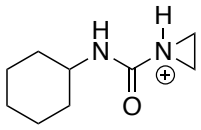
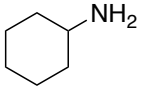



Figure S12: Comparison of $^1$H NMR of lomustine synthesized by continuous flow for different methods of purification.

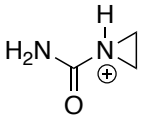
**ESI-MS**



Chemical Formula: $C_9H_{17}N_2O^+$
Monoisotopic Exact Mass: 169.13



Chemical Formula: $C_6H_{13}N$
Monoisotopic Exact Mass: 99.10



Chemical Formula: $C_3H_7N_2O^+$
Monoisotopic Exact Mass: 87.06

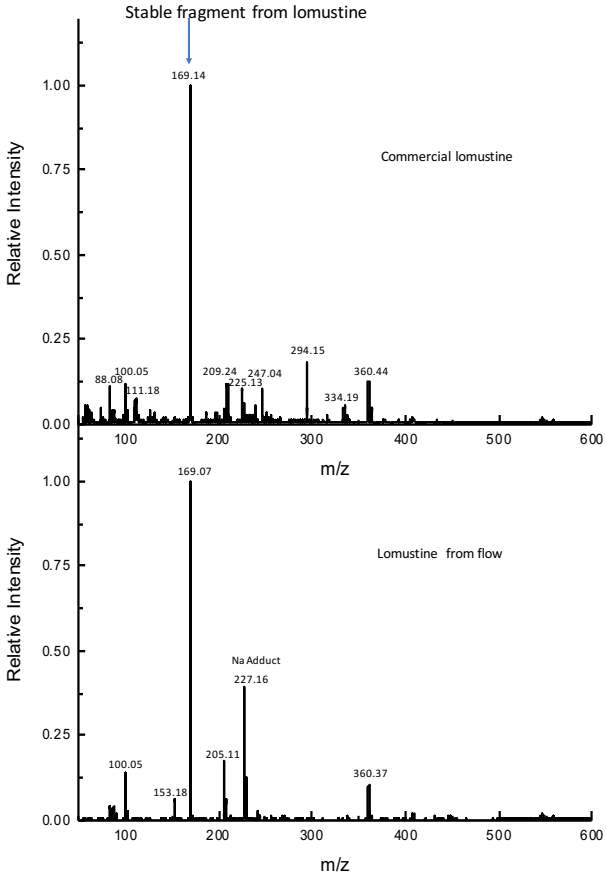
Chemical Formula: $C_6H_{11}^+$
Monoisotopic Exact Mass: 83.09

Scheme S3: Fragments of lomustine in MS

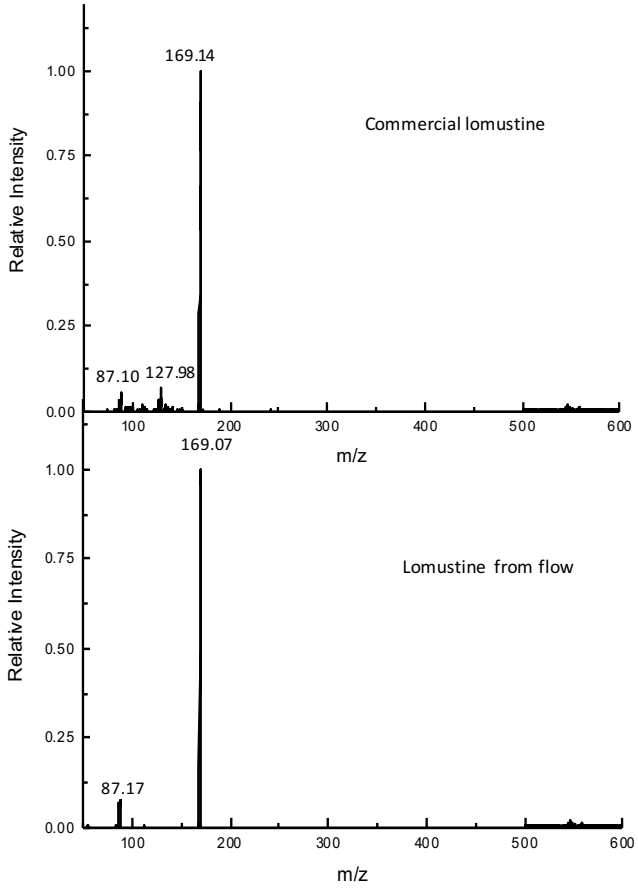
ESI-MS (*m/z*): 169 ($C_9H_{17}N_2O^+$), 100 ($C_6H_{13}N+H^+$), 87($C_3H_7N_2O^+$), 83 ($C_6H_{11}^+$)

ESI-MS/MS of *m/z* 169: 169 ($C_9H_{17}N_2O^+$), 100 ($C_6H_{13}N+H^+$), 87($C_3H_7N_2O^+$)

Full ESI-MS



MS/MS of *m/z*=169




The loss of a fragment in the MS/MS of mass 82 Dalton, corresponding to cyclohexene, is predicted and observed for the fragment ion *m/z* 169 with the structure indicated above. It is also worth noting that the lomustine sample generated by flow does not show the minor peak at *m/z* 127 which is observed in the MS/MS spectrum of the commercial lomustine sample, but is difficult to explain from its structure. Comparison of the mass spectra also suggests that the sample prepared in flow has fewer minor impurities.

Figure S13: Full MS scan and MS/MS of *m/z*=169 from commercially available lomustine and synthesized lomustine form flow. Reaction conditions were 25 °C, 8 min.

**Telescoped Synthesis of Lomustine**

**Reactors.**

For scale up and telescoping of the two steps, fluorinated ethylene propylene (FEP) tubing was used. The outer diameter of the FEP tube was 1/16 inches and the inner diameter is 0.8mm. The first reactor volume was 5 μL and the second reactor volume was 100 μL.

**Experimentation.**

Cyclohexaneamine, **1** (1 M, 1 equiv) and triethylamine (0.01 M, 0.01 equiv) were prepared in DCM separately. Next, the two separate solutions were mixed in a 1:1 (v:v) ratio and loaded into a 5 mL Hamilton gastight syringe. Then, a solution of 1-chloro-2-isocyanatoethane, **2** (0.7 M) was prepared in THF and loaded into a 5 mL Hamilton gastight syringe that was covered with

aluminum-tape for light protection since it is light sensitive. The two syringes were connected to a T-connection and outlet of the T-connector was connected to the first tubing reactor using micro-tubes, check valves and other connectors (Figure S14 & S15). The setup for producing **3** was assembled and placed in a heated $H_2O$ bath that was maintained at 50 °C. The outlet of the tube-reactor was connected to a four-way connector, where two of the outlets of the connectors were connected to a 10 mL Hamilton gastight syringe containing $H_2O$ and a 5 mL Hamilton gastight syringe containing DCM. The four-way connector provide sufficient mixing for the extraction of the triethylamine base in the aqueous phase and leaving **3** in the organic phase. The fourth outlet was connected to the liquid-liquid separator (SEP-10) in which the DCM passes through the membrane carrying with it **3** to the next reaction step. The outlet of the aqueous phase from the separator was connected to a waste vial. For using sodium nitrite as a nitrosation reagent, the outlet of the organic phase was connected to a four-way connector. Sodium nitrite, **4** (1.5 M, 3 equiv) solution in THF and formic acid were loaded into two separate 5 mL Hamilton gastight syringes and connected to the a four-way connector. The outlet of the four-way connector is connected to the second tubing reactor. When using TBN, **5** as a nitrosation reagent, we doubled the concentration of the starting material. The outlet of the organic phase from liquid-liquid extractor was connected to a T-connector, where one outlet was connected to a 5 mL Hamilton gastight syringe containing tert-Butyl nitrite, **5** (5 M) in ACN. The outlet of the T-connector was connected to the second tubing reactor. The second reactor was placed in a $H_2O$ bath with a constant temperature of 25 °C and the outlet of this reactor was connected to a collection vial. The reactions were monitored by TLC and ESI-MS. The purification and analyses were conducted as described above, except that HPLC-MS analysis was performed to evaluate the purity of the product. Mostly unreacted intermediate, **3** and cyclohexanamine, **1** were found after the reaction. We were able to remove intermediate **3** ,however, trace amounts of cyclohexanamine, **1**, were present sometimes that was confirmed by MS and HPLC-MS.

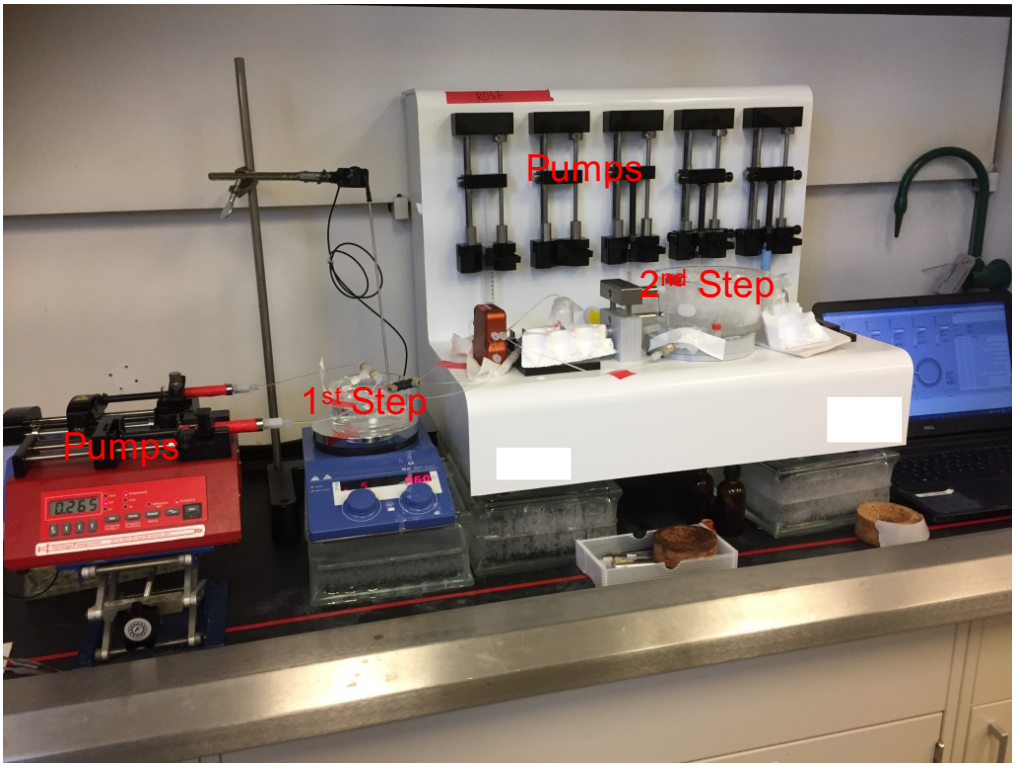



Figure S14: Telescoped two step synthesis of lomustine using sodium nitrite in the second step.

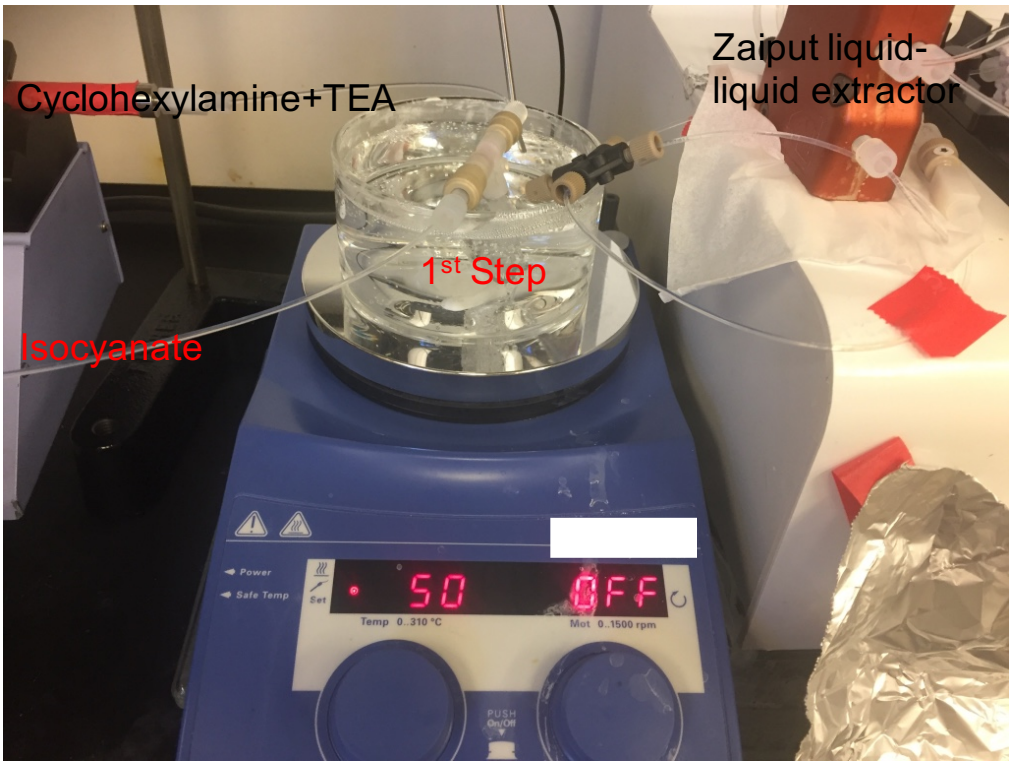



Figure S15: First step of the telescoped synthesis of lomustine.

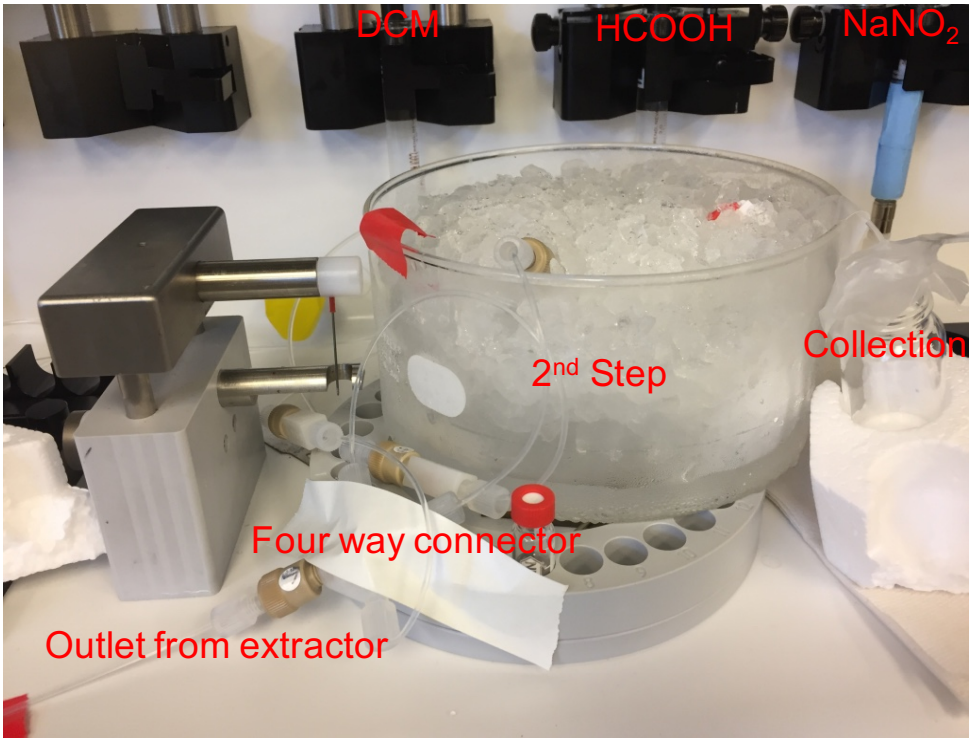



Figure S16: Second step of the telescoped synthesis of lomustine using $NaNO_2$ as a nitrosation reagent.

Figure S17: Telescoped lomustine synthesis using TBN as a nitrosation reagent, before reaction initiation.

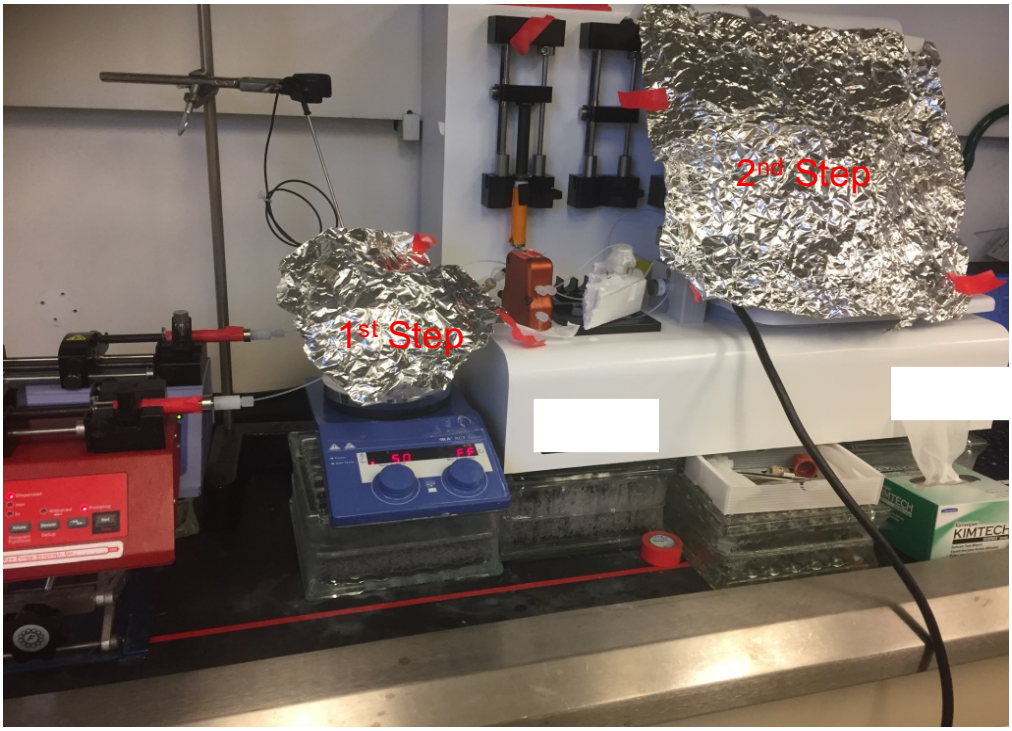



Figure S18: Telescoped lomustine synthesis using TBN (protected from light).

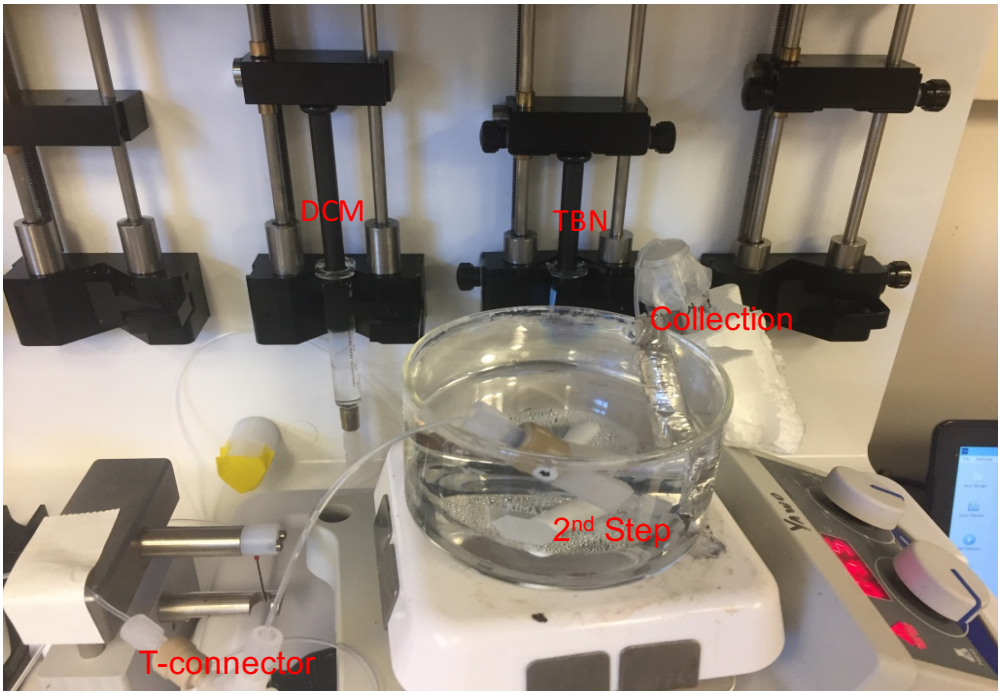


Figure S19: Second step of the telescoped synthesis of lomustine using TBN.

**TLC and Purification**. Reaction progress was monitored by TLC using 1:1 EtOAc:Hexanes as eluent. Lomustine was visualized under shortwave UV light (230 nm), while **3** was observed after staining with ninhydrin solution and heating. The extraction and purification was conducted by taking 500 µL from the collection vial and washing it with 2 mL of $H_2O$ and 2 mL of $Et_2O$ and extracted three times. The combined organic layers were dried using anhydrous $NaSO_4$. The $Et_2O$ was evaporated and the yellowish oil/solid was dissolved in hot petroleum ether, hot filtered, and the filtrate was removed under vacuum. The resulting solid was recrystallized from petroleum ether.

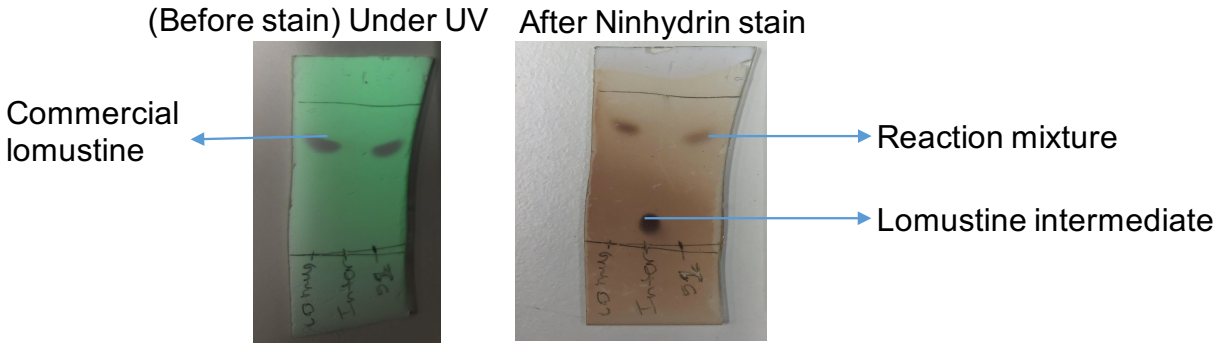



Figure S20: TLC monitoring during lomustine synthesis in flow

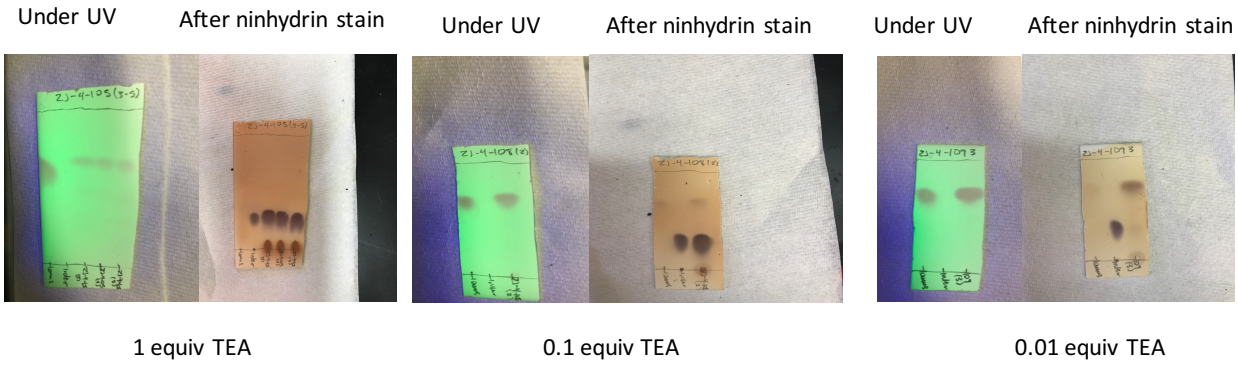



Figure S21: Comparison of TLC of telescoped lomustine synthesis using different equivalents of base

**NMR**

$^1$H NMR (500 MHz, $CDCl_3$, ppm): $\delta_H$ = 6.78 (s, 1H), 4.18 (t, J= 7.5, 2 H), 3.92-3.84 (m, 1H), 3.50

(t, J = 7.5 Hz, 2 H), 2.07-2.04 (m, 2 H), 1.79-1.75 (m, 2 H), 1.68-1.63 (m, 1 H), 1.45-1.39 (m, 2 H), 1.32-1.24 (m, 3 H); $^{13}$C NMR (500 MHz, $CDCl_3$, ppm): $\delta_C$= 151.78, 49.98, 40.03, 38.89, 33.09, 25.39, 24.76

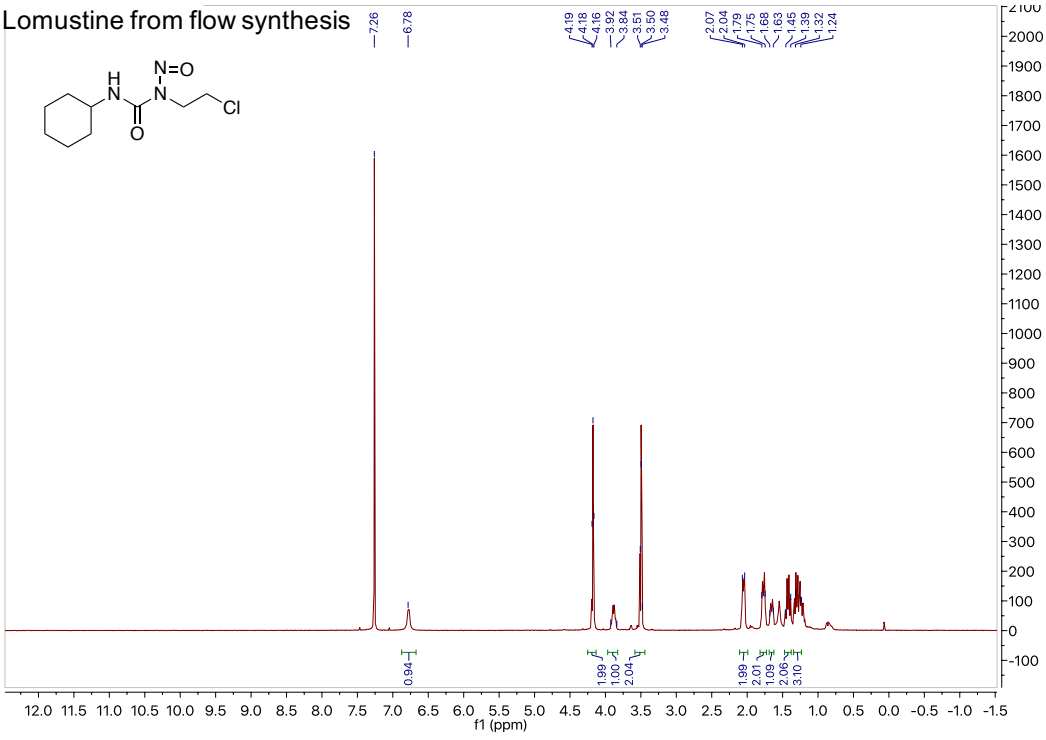



Figure S22: $^1$H NMR of lomustine from flow synthesis

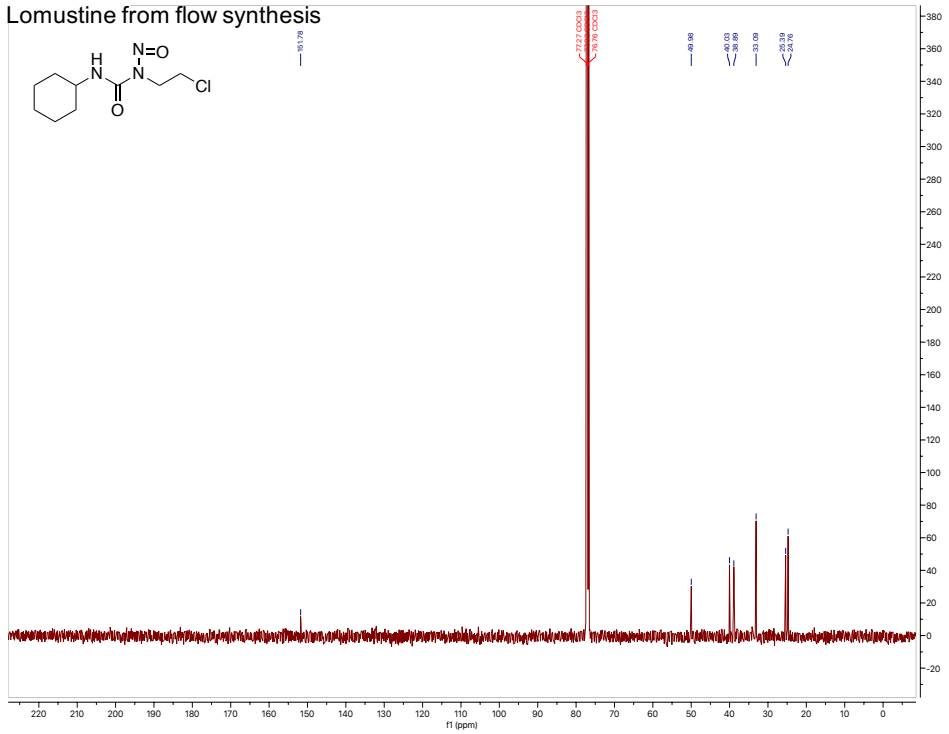



Figure S23: Carbon NMR of lomustine, **6** from flow synthesis

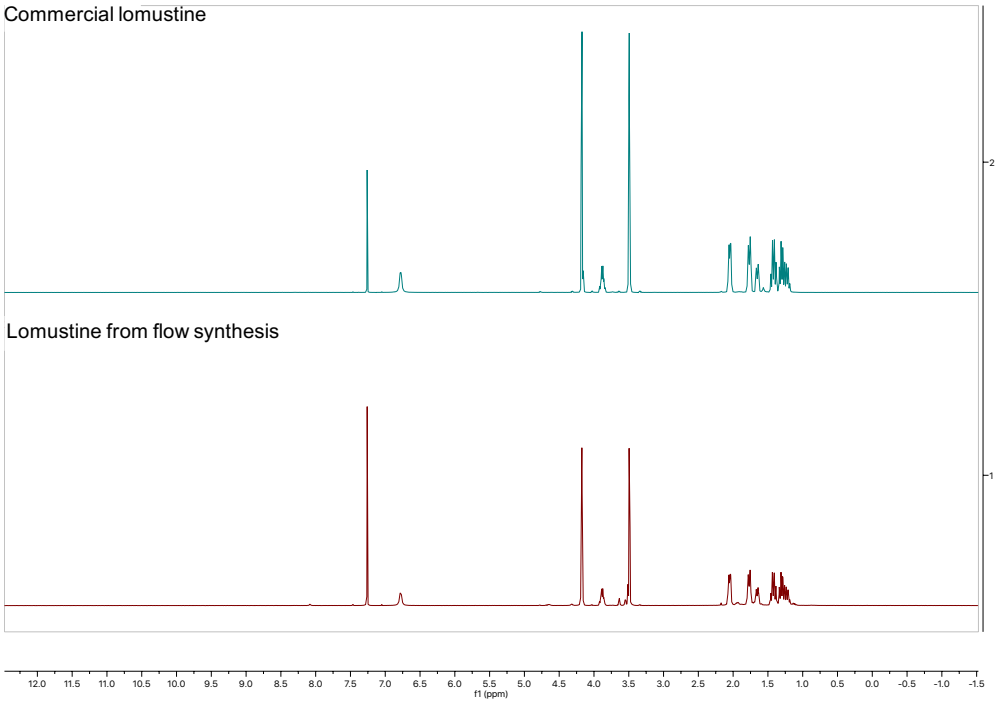



Figure S24: Comparison of Proton NMR of lomustine, **6** in flow synthesis with commercially

available lomustine, **6.**

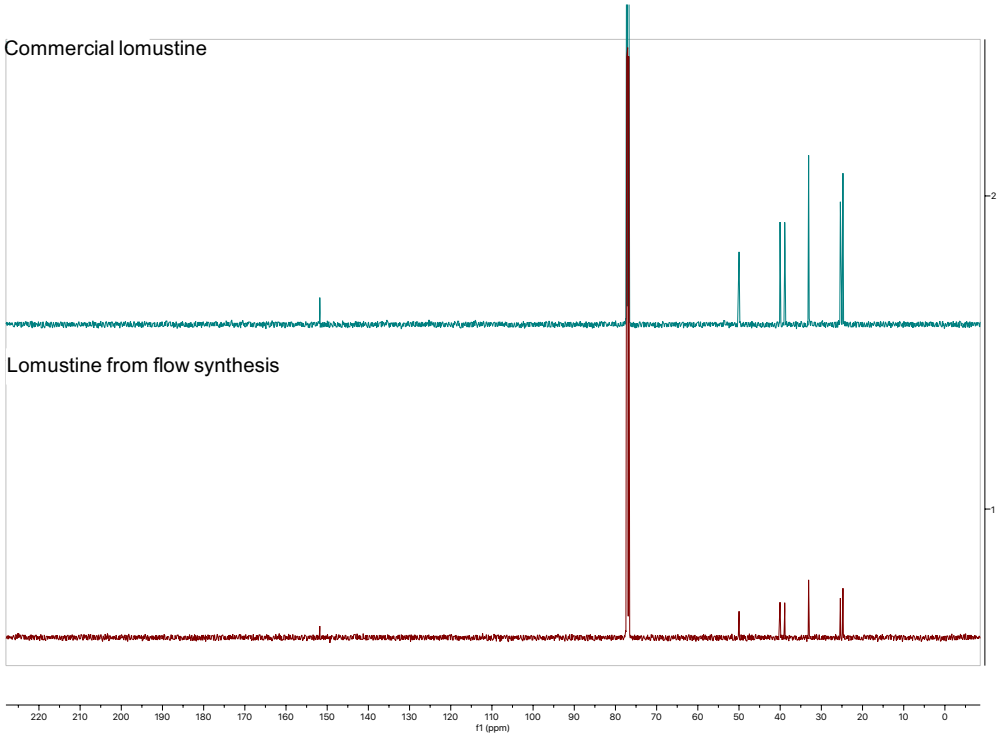



Figure S25: Comparison of carbon NMR of lomustine, **6** in flow synthesis with commercially available lomustine, **6.**

**HPLC/MS-MS analysis**

HPLC/MS analysis was performed on an Agilent 6545 UPLC/quadrupole time-of-flight (Q-TOF) mass spectrometer (Palo Alto, CA), with an Agilent XDB-C18 column (3.5 µm, 150 x 2.1 mm i.d) and 5 uL injection volume. A binary mobile phase consisting of solvent systems A and B were used. A was 0.1% formic acid (v/v) in dd$H_2O$ and B was 0.1% formic acid (v/v) in acetonitrile. Isocratic elution of A:B at 95:5 was used, with a column flow rate of 0.3 mL/min. Following the separation, the column effluent was introduced by positive mode electrospray ionization (ESI) into the mass spectrometer. High mass accuracy spectra was collected between 70 – 1000 m/z. Mass accuracy was improved by continuously infusing Agilent Reference Mass Correction Solution (G1969-85001). ESI capillary voltage was 3.5 kV, nebulizer gas pressure was 30 psig, gas temperature was 325 °C, drying gas flow rate was 8.0 L/min, fragmentor voltage was 130 V, skimmer was 45 V, and OCT RF V was 750 V.

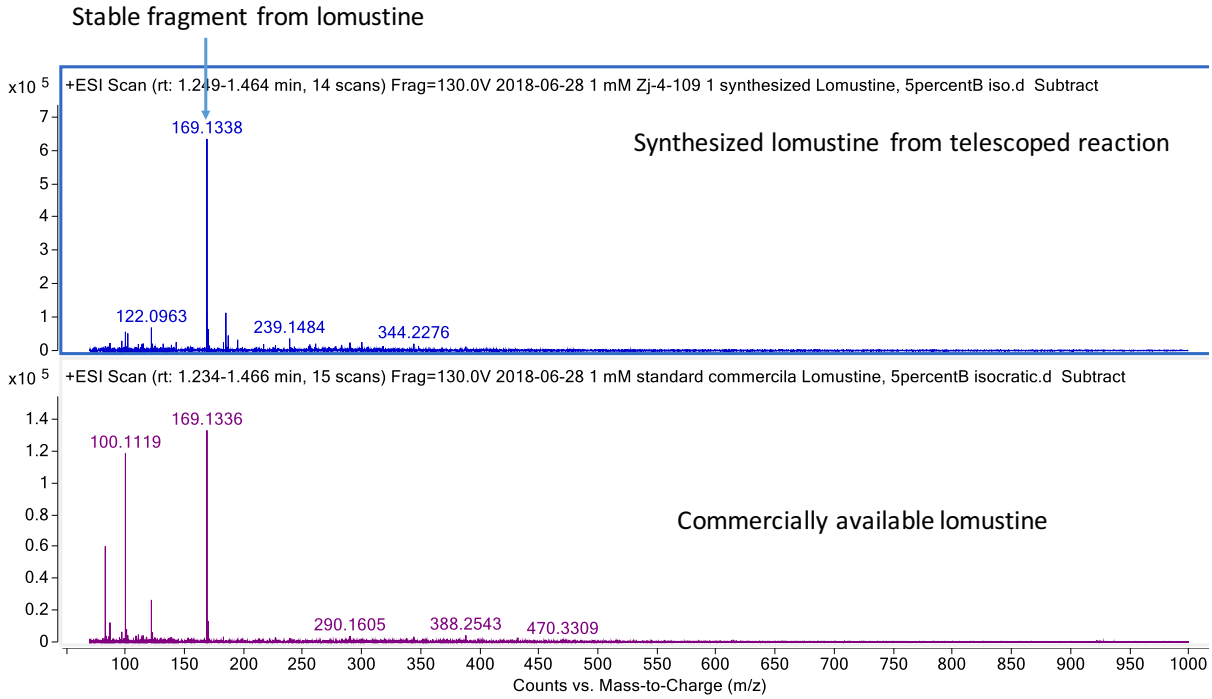



Figure S26: Full MS from HPLC-MS/MS and comparison between synthesized lomustine and commercially available lomustine.

## Flow Rates

### Flow Rates for 1st step Reaction in Labtrix S1 system

Chemtrix reactor chip: 3225, 10 μL, pressure: ambient pressure

| R1 Cyclohexane amine, **1** μL/min | R2 Triethylamine μL/min | R3 2-Chloroethyl isocyanate, **2** μL/min | Residence Time in min | Temperature °C |
|---|---|---|---|---|
| 20 | 20 | 20 | 0.167 | 50 |
| 6.67 | 6.67 | 6.67 | 0.5 | 50 |
| 3.33 | 3.33 | 3.33 | 1 | 50 |
| 1.11 | 1.11 | 1.11 | 3 | 50 |
| 0.67 | 0.67 | 0.67 | 5 | 50 |
| 0.417 | 0.417 | 0.417 | 8 | 50 |
| 0.333 | 0.333 | 0.333 | 10 | 50 |

**Flow Rates for 2nd step Reaction using $NaNO_2/HCO_2H$, 4 in Labtrix S1 system**

Chemtrix reactor chip: 3225, 10 μL, pressure: ambient pressure

| R1 **3** μL/min | R2 Sodium Nitrite μL/min | R3 Formic Acid μL/min | Residence Time in min | Temperature °C |
|---|---|---|---|---|
| 6.67 | 6.67 | 6.67 | 0.5 | 0 |
| 3.33 | 3.33 | 3.33 | 1 | 0 |
| 1.11 | 1.11 | 1.11 | 3 | 0 |
| 0.67 | 0.67 | 0.67 | 5 | 0 |
| 0.417 | 0.417 | 0.417 | 8 | 0 |
| 0.333 | 0.333 | 0.333 | 10 | 0 |

**Flow Rates for 2nd step Reaction using TBN, 5 in Labtrix S1 system.**

Chemtrix reactor chip: 3223, 10 μL, pressure: ambient pressure

| R1 **3** μL/min | R2 tert-Butyl nitrite μL/min | Residence Time in min | Temperature °C |
|---|---|---|---|
| 10 | 10 | 0.5 | 50 |
| 5 | 5 | 1 | 50 |
| 1.67 | 1.67 | 3 | 50 |
| 1 | 1 | 5 | 50 |
| 0.625 | 0.625 | 8 | 50 |
| 0.5 | 0.5 | 10 | 50 |
| 10 | 10 | 0.5 | 25 |
| 5 | 5 | 1 | 25 |
| 1.67 | 1.67 | 3 | 25 |
| 1 | 1 | 5 | 25 |
| 0.625 | 0.625 | 8 | 25 |
| 0.5 | 0.5 | 10 | 25 |

**Telescoped reaction in Tube**

Nitrosation Reagent: $NaNO_2/HCO_2H$, **4**

| R1 **1** + Triethyl amine µL/min | R2 **2** µL/min | Reactor volume, cm | Step 1 | Extraction step, $H_2O$ µL/min | Extraction step, DCM µL/min | R3 $NaNO_2$ µL/min | R3 $HCO_2H$ µL/min | Step 2 |
|---|---|---|---|---|---|---|---|---|
| 12.56 | 12.56 | Step 1: 5 Step 2: 100 | 1 min, 50 ℃ | 50.24 | 25.12 | 25.12 | 25.12 | 5 min, 0 ℃ |
| 12.56 | 12.56 | Step 1: 10 Step 2: 100 | 2 min, 50 ℃ | 50.24 | 25.12 | 25.12 | 25.12 | 5 min, 0 ℃ |
| 12.56 | 12.56 | Step 1: 50 Step 2: 100 | 10 min, 50 ℃ | 50.24 | 25.12 | 25.12 | 25.12 | 5 min, 0 ℃ |
| 12.56 | 12.56 | Step 1: 50 Step 2: 100 | 10 min, 50 ℃ | 50.24 | 25.12 | 50.24 | 67.02 | 3 min, 0 ℃ |

Nitrosation Reagent: TBN, **5**

Reactor volume: Step 1=5 cm; Step 2= 100 cm

| R1 **1** + Triethyl amine µL/min | R2 **2** µL/min | Step 1 | Extraction step, $H_2O$ µL/min | Extraction step, DCM µL/min | R3 TBN µL/min | Step 2 |
|---|---|---|---|---|---|---|
| 12.56 | 12.56 | 1 min, 50 ℃ | 50.24 | 25.12 | 50.2 | 5 min, 25/50 ℃ |
| 12.56 | 12.56 | 1 min, 50 ℃ | 50.24 | 25.12 | 12.56 | 8 min, 25 ℃ |

## DESI-MS Data

## DESI-MS data of the nitrosation reaction using 4 in different stoichiometries

| Starting Material | Starting Material | Solvent | Stoichiometry | Product *m/z* | Intensity (Average) | Stdev | Intensity (max) | Normalized Intensity (Average) | Normalized Stdev | Normalized Intensity (max) | no of spots, n |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **3** | **4** | Acetonitrile | 150 | 169.2 | 213.3 | 191.6 | 934.1 | 0.025 | 0.030 | 0.166 | 144 |
| **3** | **4** | Acetonitrile | 150 | 234.7 | 8.0 | 6.7 | 45.1 | 0.001 | 0.001 | 0.007 | 144 |
| **3** | **4** | Acetonitrile | 150 | 239.4 | 1206.9 | 1349.7 | 6575.7 | 0.069 | 0.066 | 0.284 | 144 |
| **3** | **4** | Acetonitrile | 150 | 241.0 | 390.4 | 412.9 | 1980.9 | 0.022 | 0.020 | 0.088 | 144 |
| **3** | **4** | Acetonitrile | 150 | 250.4 | 143.7 | 184.3 | 923.0 | 0.008 | 0.005 | 0.024 | 144 |
| **3** | **4** | Acetonitrile | 150 | 251.2 | 34.7 | 40.6 | 205.3 | 0.002 | 0.001 | 0.006 | 144 |
| **3** | **4** | Acetonitrile | 150 | 337.4 | 831.0 | 707.6 | 4214.5 | 0.043 | 0.016 | 0.100 | 144 |
| **3** | **4** | Acetonitrile | 150 | 373.4 | 195.1 | 203.4 | 1017.1 | 0.010 | 0.006 | 0.026 | 144 |
| **3** | **4** | Acetonitrile | 100 | 169.2 | 428.3 | 487.1 | 3538.6 | 0.021 | 0.019 | 0.124 | 144 |
| **3** | **4** | Acetonitrile | 100 | 234.7 | 19.3 | 15.1 | 80.5 | 0.002 | 0.002 | 0.008 | 144 |
| **3** | **4** | Acetonitrile | 100 | 239.4 | 2066.8 | 2519.0 | 12435.6 | 0.061 | 0.049 | 0.220 | 144 |
| **3** | **4** | Acetonitrile | 100 | 241.0 | 664.8 | 776.1 | 3676.1 | 0.020 | 0.015 | 0.070 | 144 |
| **3** | **4** | Acetonitrile | 100 | 250.4 | 179.0 | 200.3 | 1072.1 | 0.006 | 0.003 | 0.016 | 144 |
| **3** | **4** | Acetonitrile | 100 | 251.2 | 50.4 | 57.4 | 348.6 | 0.002 | 0.001 | 0.005 | 144 |
| **3** | **4** | Acetonitrile | 100 | 337.4 | 1541.9 | 1537.4 | 9588.3 | 0.050 | 0.018 | 0.105 | 144 |
| **3** | **4** | Acetonitrile | 100 | 373.4 | 261.3 | 307.9 | 1751.0 | 0.008 | 0.004 | 0.022 | 144 |
| **3** | **4** | Acetonitrile | 50 | 169.2 | 53.9 | 58.1 | 322.0 | 0.018 | 0.019 | 0.105 | 144 |
| **3** | **4** | Acetonitrile | 50 | 234.7 | 11.7 | 16.2 | 177.5 | 0.004 | 0.002 | 0.015 | 144 |
| **3** | **4** | Acetonitrile | 50 | 239.4 | 382.9 | 825.0 | 5452.0 | 0.045 | 0.062 | 0.326 | 144 |
| **3** | **4** | Acetonitrile | 50 | 241.0 | 124.8 | 257.2 | 1692.1 | 0.016 | 0.019 | 0.101 | 144 |
| **3** | **4** | Acetonitrile | 50 | 250.4 | 21.3 | 24.1 | 145.3 | 0.005 | 0.002 | 0.012 | 144 |
| **3** | **4** | Acetonitrile | 50 | 251.2 | 6.3 | 6.7 | 37.3 | 0.001 | 0.001 | 0.004 | 144 |
| **3** | **4** | Acetonitrile | 50 | 337.4 | 116.2 | 130.7 | 681.0 | 0.020 | 0.015 | 0.075 | 144 |
| **3** | **4** | Acetonitrile | 50 | 373.4 | 21.0 | 24.5 | 133.5 | 0.004 | 0.003 | 0.014 | 144 |
| **3** | **4** | Acetonitrile | 200 | 169.2 | 134.4 | 122.3 | 642.8 | 0.007 | 0.002 | 0.012 | 144 |
| **3** | **4** | Acetonitrile | 200 | 234.7 | 7.7 | 8.7 | 57.9 | 0.001 | 0.001 | 0.003 | 144 |
| **3** | **4** | Acetonitrile | 200 | 239.4 | 2162.9 | 2123.7 | 15812.4 | 0.119 | 0.056 | 0.297 | 144 |
| **3** | **4** | Acetonitrile | 200 | 241.0 | 608.6 | 518.4 | 4106.0 | 0.035 | 0.017 | 0.090 | 144 |
| **3** | **4** | Acetonitrile | 200 | 250.4 | 39.5 | 26.3 | 138.1 | 0.003 | 0.001 | 0.006 | 144 |
| **3** | **4** | Acetonitrile | 200 | 251.2 | 20.5 | 49.7 | 382.9 | 0.001 | 0.000 | 0.002 | 144 |
| **3** | **4** | Acetonitrile | 200 | 337.4 | 485.0 | 296.1 | 1620.7 | 0.028 | 0.007 | 0.042 | 144 |
| **3** | **4** | Acetonitrile | 200 | 373.4 | 73.1 | 51.3 | 324.3 | 0.004 | 0.001 | 0.007 | 144 |
| **3** | **4** | THF | 200 | 169.2 | 1024.5 | 744.7 | 6746.3 | 0.041 | 0.059 | 0.443 | 144 |
| **3** | **4** | THF | 200 | 234.7 | 30.8 | 36.3 | 222.7 | 0.002 | 0.001 | 0.006 | 144 |
| **3** | **4** | THF | 200 | 239.4 | 1067.2 | 1527.8 | 7808.4 | 0.022 | 0.034 | 0.174 | 144 |
| **3** | **4** | THF | 200 | 241.0 | 333.9 | 480.7 | 2373.1 | 0.007 | 0.010 | 0.057 | 144 |
| **3** | **4** | THF | 200 | 250.4 | 749.2 | 367.0 | 2331.3 | 0.011 | 0.004 | 0.022 | 144 |
| **3** | **4** | THF | 200 | 251.2 | 237.1 | 227.8 | 1263.0 | 0.003 | 0.002 | 0.009 | 144 |
| **3** | **4** | THF | 200 | 337.4 | 13765.9 | 7680.8 | 50455.2 | 0.186 | 0.054 | 0.343 | 144 |
| **3** | **4** | THF | 200 | 373.4 | 1096.5 | 568.4 | 3314.2 | 0.016 | 0.005 | 0.038 | 144 |
| **3** | **4** | THF | 150 | 169.2 | 680.3 | 705.5 | 3251.7 | 0.036 | 0.033 | 0.202 | 144 |
| **3** | **4** | THF | 150 | 234.7 | 43.2 | 44.1 | 295.0 | 0.003 | 0.001 | 0.009 | 144 |
| **3** | **4** | THF | 150 | 239.4 | 1106.3 | 2107.4 | 12553.7 | 0.021 | 0.031 | 0.143 | 144 |
| **3** | **4** | THF | 150 | 241.0 | 366.5 | 601.2 | 2985.4 | 0.008 | 0.009 | 0.044 | 144 |
| **3** | **4** | THF | 150 | 250.4 | 349.0 | 338.2 | 1672.1 | 0.007 | 0.004 | 0.020 | 144 |
| **3** | **4** | THF | 150 | 251.2 | 92.1 | 92.6 | 353.0 | 0.002 | 0.001 | 0.008 | 144 |
| **3** | **4** | THF | 150 | 337.4 | 2590.0 | 3621.7 | 15618.2 | 0.044 | 0.051 | 0.185 | 144 |
| **3** | **4** | THF | 150 | 373.4 | 291.1 | 374.6 | 1736.1 | 0.006 | 0.006 | 0.024 | 144 |
| **3** | **4** | THF | 100 | 169.2 | 1152.9 | 849.2 | 4316.7 | 0.053 | 0.038 | 0.236 | 144 |
| **3** | **4** | THF | 100 | 234.7 | 40.4 | 42.7 | 243.0 | 0.002 | 0.001 | 0.014 | 144 |
| **3** | **4** | THF | 100 | 239.4 | 486.4 | 1259.8 | 8600.9 | 0.008 | 0.019 | 0.149 | 144 |
| **3** | **4** | THF | 100 | 241.0 | 166.2 | 407.2 | 2879.3 | 0.003 | 0.006 | 0.044 | 144 |
| **3** | **4** | THF | 100 | 250.4 | 1070.5 | 451.4 | 2449.5 | 0.013 | 0.004 | 0.020 | 144 |
| **3** | **4** | THF | 100 | 251.2 | 260.9 | 172.7 | 1492.2 | 0.003 | 0.001 | 0.011 | 144 |
| **3** | **4** | THF | 100 | 337.4 | 14213.4 | 9456.4 | 39713.7 | 0.157 | 0.076 | 0.339 | 144 |
| **3** | **4** | THF | 100 | 373.4 | 1262.2 | 675.0 | 3233.6 | 0.016 | 0.006 | 0.030 | 144 |
| **3** | **4** | THF | 50 | 169.2 | 708.7 | 560.9 | 3726.2 | 0.060 | 0.034 | 0.211 | 144 |
| **3** | **4** | THF | 50 | 234.7 | 46.1 | 56.3 | 341.8 | 0.003 | 0.002 | 0.020 | 144 |
| **3** | **4** | THF | 50 | 239.4 | 91.6 | 164.1 | 938.4 | 0.003 | 0.003 | 0.023 | 144 |
| **3** | **4** | THF | 50 | 241.0 | 54.0 | 70.6 | 369.5 | 0.003 | 0.003 | 0.015 | 144 |
| **3** | **4** | THF | 50 | 250.4 | 783.5 | 422.1 | 2172.0 | 0.012 | 0.003 | 0.018 | 144 |
| **3** | **4** | THF | 50 | 251.2 | 227.3 | 310.1 | 2172.0 | 0.003 | 0.002 | 0.014 | 144 |
| **3** | **4** | THF | 50 | 337.4 | 10050.7 | 9141.9 | 52775.8 | 0.125 | 0.074 | 0.268 | 144 |
| **3** | **4** | THF | 50 | 373.4 | 911.9 | 744.6 | 4270.9 | 0.012 | 0.006 | 0.022 | 144 |
| Rhodamine | | | | 443.3 | 12213.1 | 8739.2 | 70545.6 | 0.491 | 0.101 | 0.690 | 384 |

## DESI-MS data of the nitrosation reaction using 4 as a nitrosation reagent

| Starting Material | Starting Material | Solvent | Product *m/z* | Intensity (Average) | Stdev | Intensity (max) | Normalized Intensity (Average) | Normalized Stdev | Normalized Intensity (max) | No of spots, n |
|---|---|---|---|---|---|---|---|---|---|---|
| **3** | **4** | Toluene | 169.2 | 2554.4 | 5564.3 | 36332.5 | 0.406 | 0.173 | 0.719 | 144 |
| **3** | **4** | Toluene | 205.7 | 7.2 | 10.4 | 63.4 | 0.003 | 0.003 | 0.015 | 144 |
| **3** | **4** | Toluene | 234.7 | 2.8 | 4.5 | 24.2 | 0.001 | 0.001 | 0.004 | 144 |
| **3** | **4** | Toluene | 239.4 | 2.7 | 5.8 | 38.3 | 0.000 | 0.001 | 0.007 | 144 |
| **3** | **4** | Toluene | 241.0 | 2.5 | 5.8 | 38.1 | 0.000 | 0.001 | 0.004 | 144 |
| **3** | **4** | Toluene | 250.4 | 1.4 | 4.4 | 43.3 | 0.000 | 0.001 | 0.004 | 144 |
| **3** | **4** | Toluene | 251.2 | 30.9 | 134.5 | 1279.6 | 0.002 | 0.006 | 0.039 | 144 |
| **3** | **4** | Toluene | 337.4 | 100.7 | 267.5 | 1413.6 | 0.007 | 0.018 | 0.146 | 144 |
| **3** | **4** | Toluene | 373.4 | 440.4 | 1209.9 | 8746.2 | 0.026 | 0.043 | 0.215 | 144 |
| **3** | **4** | Acetonitrile | 169.2 | 861.8 | 2266.6 | 24796.5 | 0.471 | 0.136 | 0.774 | 144 |
| **3** | **4** | Acetonitrile | 205.7 | 6.4 | 12.7 | 84.5 | 0.003 | 0.004 | 0.026 | 144 |
| **3** | **4** | Acetonitrile | 234.7 | 0.8 | 2.0 | 15.5 | 0.000 | 0.001 | 0.007 | 144 |
| **3** | **4** | Acetonitrile | 239.4 | 1.1 | 2.4 | 17.8 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Acetonitrile | 241.0 | 6.9 | 18.6 | 151.1 | 0.001 | 0.002 | 0.008 | 144 |
| **3** | **4** | Acetonitrile | 250.4 | 5.3 | 13.5 | 106.8 | 0.001 | 0.002 | 0.012 | 144 |
| **3** | **4** | Acetonitrile | 251.2 | 330.2 | 517.7 | 2676.7 | 0.039 | 0.035 | 0.213 | 144 |
| **3** | **4** | Acetonitrile | 337.4 | 46.4 | 73.3 | 493.7 | 0.005 | 0.006 | 0.033 | 144 |
| **3** | **4** | Acetonitrile | 373.4 | 651.5 | 1237.0 | 9488.0 | 0.065 | 0.035 | 0.190 | 144 |
| **3** | **4** | DMSO | 169.2 | 4904.5 | 8789.6 | 53558.7 | 0.355 | 0.137 | 0.642 | 144 |
| **3** | **4** | DMSO | 205.7 | 25.7 | 26.7 | 151.4 | 0.002 | 0.002 | 0.012 | 144 |
| **3** | **4** | DMSO | 234.7 | 1.8 | 2.3 | 13.1 | 0.000 | 0.001 | 0.002 | 144 |
| **3** | **4** | DMSO | 239.4 | 17.1 | 26.6 | 228.2 | 0.001 | 0.000 | 0.002 | 144 |
| **3** | **4** | DMSO | 241.0 | 5.2 | 6.1 | 26.9 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | DMSO | 250.4 | 1.6 | 9.6 | 110.6 | 0.000 | 0.000 | 0.001 | 144 |
| **3** | **4** | DMSO | 251.2 | 101.2 | 179.2 | 1054.9 | 0.002 | 0.003 | 0.021 | 144 |
| **3** | **4** | DMSO | 337.4 | 443.8 | 791.7 | 4937.7 | 0.009 | 0.011 | 0.053 | 144 |
| **3** | **4** | DMSO | 373.4 | 2602.8 | 3506.2 | 30323.8 | 0.049 | 0.033 | 0.232 | 144 |
| **3** | **4** | THF | 169.2 | 1604.6 | 1828.4 | 9837.0 | 0.343 | 0.165 | 0.671 | 144 |
| **3** | **4** | THF | 205.7 | 14.3 | 16.2 | 107.3 | 0.002 | 0.003 | 0.014 | 144 |
| **3** | **4** | THF | 234.7 | 9.2 | 20.8 | 141.7 | 0.000 | 0.001 | 0.003 | 144 |
| **3** | **4** | THF | 239.4 | 68.5 | 72.4 | 381.6 | 0.002 | 0.002 | 0.007 | 144 |
| **3** | **4** | THF | 241.0 | 22.7 | 22.2 | 99.5 | 0.001 | 0.000 | 0.004 | 144 |
| **3** | **4** | THF | 250.4 | 4.1 | 7.3 | 32.2 | 0.000 | 0.000 | 0.001 | 144 |
| **3** | **4** | THF | 251.2 | 254.1 | 351.0 | 1963.4 | 0.008 | 0.008 | 0.040 | 144 |
| **3** | **4** | THF | 337.4 | 122.6 | 116.9 | 840.1 | 0.003 | 0.002 | 0.019 | 144 |
| **3** | **4** | THF | 373.4 | 3643.6 | 3629.0 | 24476.2 | 0.079 | 0.036 | 0.222 | 144 |
| **3** | **4** | Ethanol | 169.2 | 1939.9 | 2663.0 | 23988.5 | 0.315 | 0.159 | 0.644 | 144 |
| **3** | **4** | Ethanol | 205.7 | 18.0 | 22.0 | 108.9 | 0.003 | 0.003 | 0.017 | 144 |
| **3** | **4** | Ethanol | 234.7 | 9.7 | 28.9 | 313.9 | 0.001 | 0.001 | 0.003 | 144 |
| **3** | **4** | Ethanol | 239.4 | 3.1 | 3.4 | 20.9 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Ethanol | 241.0 | 4.1 | 3.4 | 17.3 | 0.000 | 0.000 | 0.003 | 144 |
| **3** | **4** | Ethanol | 250.4 | 3.5 | 3.4 | 17.0 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Ethanol | 251.2 | 53.0 | 37.6 | 152.7 | 0.002 | 0.001 | 0.006 | 144 |
| **3** | **4** | Ethanol | 337.4 | 263.7 | 193.3 | 1112.6 | 0.009 | 0.005 | 0.049 | 144 |
| **3** | **4** | Ethanol | 373.4 | 553.9 | 508.5 | 2842.8 | 0.024 | 0.010 | 0.074 | 144 |
| **3** | **4** | DCM | 169.2 | 463.4 | 643.7 | 3574.7 | 0.429 | 0.135 | 0.733 | 144 |
| **3** | **4** | DCM | 205.7 | 6.8 | 10.5 | 72.0 | 0.005 | 0.008 | 0.056 | 144 |
| **3** | **4** | DCM | 234.7 | 1.8 | 9.0 | 77.5 | 0.000 | 0.001 | 0.006 | 144 |
| **3** | **4** | DCM | 239.4 | 1.6 | 3.8 | 22.9 | 0.000 | 0.001 | 0.005 | 144 |
| **3** | **4** | DCM | 241.0 | 6.9 | 17.3 | 111.6 | 0.001 | 0.003 | 0.027 | 144 |
| **3** | **4** | DCM | 250.4 | 4.4 | 11.7 | 91.1 | 0.001 | 0.002 | 0.009 | 144 |
| **3** | **4** | DCM | 251.2 | 68.1 | 211.5 | 1683.3 | 0.009 | 0.014 | 0.103 | 144 |
| **3** | **4** | DCM | 337.4 | 32.4 | 77.4 | 572.0 | 0.007 | 0.013 | 0.060 | 144 |
| **3** | **4** | DCM | 373.4 | 381.0 | 586.2 | 3159.2 | 0.072 | 0.062 | 0.206 | 144 |
| **3** | **4** | Ethyl Acetate | 169.2 | 2710.5 | 3037.9 | 15437.7 | 0.325 | 0.124 | 0.586 | 144 |
| **3** | **4** | Ethyl Acetate | 205.7 | 23.4 | 33.0 | 173.7 | 0.005 | 0.006 | 0.042 | 144 |
| **3** | **4** | Ethyl Acetate | 234.7 | 7.6 | 29.1 | 260.3 | 0.001 | 0.002 | 0.017 | 144 |
| **3** | **4** | Ethyl Acetate | 239.4 | 2.0 | 3.6 | 16.7 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Ethyl Acetate | 241.0 | 4.7 | 9.6 | 73.9 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Ethyl Acetate | 250.4 | 3.5 | 7.4 | 58.3 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Ethyl Acetate | 251.2 | 115.4 | 304.2 | 2298.6 | 0.005 | 0.009 | 0.058 | 144 |
| **3** | **4** | Ethyl Acetate | 337.4 | 34.3 | 65.0 | 512.9 | 0.002 | 0.002 | 0.012 | 144 |
| **3** | **4** | Ethyl Acetate | 373.4 | 2627.7 | 3123.4 | 13965.7 | 0.104 | 0.081 | 0.247 | 144 |
| **3** | **4** | Methanol | 169.2 | 1516.5 | 3046.6 | 34891.7 | 0.369 | 0.172 | 0.780 | 144 |
| **3** | **4** | Methanol | 205.7 | 15.8 | 21.8 | 167.1 | 0.003 | 0.004 | 0.028 | 144 |
| **3** | **4** | Methanol | 234.7 | 1.3 | 1.6 | 7.2 | 0.000 | 0.001 | 0.002 | 144 |
| **3** | **4** | Methanol | 239.4 | 1.6 | 2.1 | 15.3 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Methanol | 241.0 | 2.7 | 4.0 | 22.3 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Methanol | 250.4 | 1.3 | 2.3 | 14.7 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **4** | Methanol | 251.2 | 19.6 | 25.3 | 173.7 | 0.001 | 0.001 | 0.006 | 144 |
| **3** | **4** | Methanol | 337.4 | 288.8 | 315.4 | 1590.6 | 0.013 | 0.010 | 0.052 | 144 |
| **3** | **4** | Methanol | 373.4 | 510.8 | 507.8 | 2698.7 | 0.027 | 0.014 | 0.072 | 144 |
| **3** | | | 443.3 | 12333.9 | 13328.7 | 384.0 | 0.354 | 0.083 | 0.646 | 384 |

## DESI-MS data of the nitrosation reaction using 5 as a nitrosation reagent

| Starting Material | Starting Material | Solvent | Product*m/z* | Intensity (Average) | Stdev | Intensity (max) | Normalized Intensity (Average) | Normalized Stdev | Normalized Intensity (max) | No of spots, n |
|---|---|---|---|---|---|---|---|---|---|---|
| **3** | **5** | Ethyl Acetate | 169.2 | 41.1 | 47.5 | 326.1 | 0.006 | 0.005 | 0.028 | 144 |
| **3** | **5** | Ethyl Acetate | 205.7 | 26.2 | 10.1 | 59.8 | 0.004 | 0.001 | 0.011 | 144 |
| **3** | **5** | Ethyl Acetate | 234.7 | 46.6 | 22.4 | 225.0 | 0.007 | 0.002 | 0.026 | 144 |
| **3** | **5** | Ethyl Acetate | 239.4 | 54.1 | 121.6 | 768.5 | 0.005 | 0.006 | 0.027 | 144 |
| **3** | **5** | Ethyl Acetate | 241.0 | 18.6 | 34.1 | 215.5 | 0.002 | 0.002 | 0.010 | 144 |
| **3** | **5** | Ethyl Acetate | 250.4 | 243.1 | 89.4 | 605.5 | 0.038 | 0.008 | 0.076 | 144 |
| **3** | **5** | Ethyl Acetate | 251.2 | 239.7 | 89.6 | 605.5 | 0.039 | 0.010 | 0.112 | 144 |
| **3** | **5** | Ethyl Acetate | 337.4 | 6.5 | 4.5 | 38.0 | 0.001 | 0.001 | 0.004 | 144 |
| **3** | **5** | Ethyl Acetate | 373.4 | 19.5 | 23.7 | 107.1 | 0.003 | 0.004 | 0.022 | 144 |
| **3** | **5** | Ethanol | 169.2 | 1059.5 | 1113.0 | 5710.9 | 0.047 | 0.027 | 0.128 | 144 |
| **3** | **5** | Ethanol | 205.7 | 44.2 | 47.6 | 224.7 | 0.002 | 0.001 | 0.006 | 144 |
| **3** | **5** | Ethanol | 234.7 | 22.5 | 14.2 | 73.3 | 0.001 | 0.000 | 0.002 | 144 |
| **3** | **5** | Ethanol | 239.4 | 437.1 | 485.3 | 2453.7 | 0.016 | 0.012 | 0.091 | 144 |
| **3** | **5** | Ethanol | 241.0 | 155.0 | 199.6 | 1496.5 | 0.005 | 0.004 | 0.027 | 144 |
| **3** | **5** | Ethanol | 250.4 | 75.4 | 57.5 | 339.3 | 0.003 | 0.002 | 0.009 | 144 |
| **3** | **5** | Ethanol | 251.2 | 33.5 | 21.6 | 123.8 | 0.001 | 0.000 | 0.003 | 144 |
| **3** | **5** | Ethanol | 337.4 | 28.4 | 16.3 | 85.8 | 0.001 | 0.000 | 0.003 | 144 |
| **3** | **5** | Ethanol | 373.4 | 547.1 | 727.2 | 4628.6 | 0.021 | 0.014 | 0.065 | 144 |
| **3** | **5** | THF | 169.2 | 77.4 | 147.1 | 824.4 | 0.008 | 0.009 | 0.066 | 144 |
| **3** | **5** | THF | 205.7 | 23.6 | 18.8 | 81.5 | 0.004 | 0.002 | 0.009 | 144 |
| **3** | **5** | THF | 234.7 | 55.6 | 74.9 | 771.9 | 0.008 | 0.004 | 0.020 | 144 |
| **3** | **5** | THF | 239.4 | 98.5 | 215.8 | 1279.6 | 0.008 | 0.010 | 0.046 | 144 |
| **3** | **5** | THF | 241.0 | 31.2 | 63.7 | 401.6 | 0.002 | 0.003 | 0.013 | 144 |
| **3** | **5** | THF | 250.4 | 278.7 | 221.3 | 1061.8 | 0.046 | 0.021 | 0.088 | 144 |
| **3** | **5** | THF | 251.2 | 285.5 | 222.9 | 1061.8 | 0.048 | 0.020 | 0.088 | 144 |
| **3** | **5** | THF | 337.4 | 5.7 | 5.9 | 46.3 | 0.001 | 0.001 | 0.007 | 144 |
| **3** | **5** | THF | 373.4 | 23.1 | 41.3 | 258.5 | 0.003 | 0.004 | 0.021 | 144 |
| **3** | **5** | DCM | 169.2 | 357.7 | 408.6 | 2428.0 | 0.028 | 0.018 | 0.076 | 144 |
| **3** | **5** | DCM | 205.7 | 56.1 | 22.9 | 159.1 | 0.005 | 0.002 | 0.011 | 144 |
| **3** | **5** | DCM | 234.7 | 111.7 | 69.4 | 561.9 | 0.010 | 0.003 | 0.017 | 144 |
| **3** | **5** | DCM | 239.4 | 255.9 | 372.5 | 1692.9 | 0.019 | 0.016 | 0.055 | 144 |
| **3** | **5** | DCM | 241.0 | 76.3 | 115.2 | 670.7 | 0.006 | 0.005 | 0.018 | 144 |
| **3** | **5** | DCM | 250.4 | 538.9 | 212.4 | 1632.1 | 0.057 | 0.017 | 0.086 | 144 |
| **3** | **5** | DCM | 251.2 | 551.4 | 202.1 | 1632.1 | 0.058 | 0.015 | 0.086 | 144 |
| **3** | **5** | DCM | 337.4 | 3.3 | 3.5 | 28.0 | 0.000 | 0.000 | 0.002 | 144 |
| **3** | **5** | DCM | 373.4 | 102.8 | 212.9 | 2118.2 | 0.007 | 0.007 | 0.038 | 144 |
| **3** | **5** | Toluene | 169.2 | 292.1 | 271.9 | 1570.8 | 0.025 | 0.016 | 0.098 | 144 |
| **3** | **5** | Toluene | 205.7 | 25.0 | 15.0 | 107.4 | 0.003 | 0.001 | 0.011 | 144 |
| **3** | **5** | Toluene | 234.7 | 37.9 | 32.5 | 268.9 | 0.005 | 0.001 | 0.012 | 144 |
| **3** | **5** | Toluene | 239.4 | 179.6 | 173.3 | 1095.6 | 0.012 | 0.007 | 0.034 | 144 |
| **3** | **5** | Toluene | 241.0 | 57.9 | 59.1 | 394.2 | 0.004 | 0.002 | 0.011 | 144 |
| **3** | **5** | Toluene | 250.4 | 257.8 | 80.3 | 515.9 | 0.040 | 0.006 | 0.054 | 144 |
| **3** | **5** | Toluene | 251.2 | 257.5 | 78.3 | 463.2 | 0.040 | 0.006 | 0.054 | 144 |
| **3** | **5** | Toluene | 337.4 | 5.5 | 4.5 | 24.3 | 0.001 | 0.000 | 0.002 | 144 |
| **3** | **5** | Toluene | 373.4 | 136.8 | 119.1 | 632.3 | 0.012 | 0.008 | 0.045 | 144 |
| **3** | **5** | DMSO | 169.2 | 3063.0 | 4126.1 | 20263.6 | 0.192 | 0.113 | 0.754 | 144 |
| **3** | **5** | DMSO | 205.7 | 28.5 | 52.8 | 347.2 | 0.003 | 0.002 | 0.014 | 144 |
| **3** | **5** | DMSO | 234.7 | 19.5 | 14.6 | 117.7 | 0.004 | 0.003 | 0.033 | 144 |
| **3** | **5** | DMSO | 239.4 | 30.2 | 77.6 | 745.0 | 0.002 | 0.003 | 0.016 | 144 |
| **3** | **5** | DMSO | 241.0 | 10.1 | 24.1 | 233.9 | 0.001 | 0.001 | 0.005 | 144 |
| **3** | **5** | DMSO | 250.4 | 108.8 | 94.5 | 872.6 | 0.020 | 0.008 | 0.039 | 144 |
| **3** | **5** | DMSO | 251.2 | 105.8 | 71.3 | 325.7 | 0.022 | 0.007 | 0.040 | 144 |
| **3** | **5** | DMSO | 337.4 | 5.5 | 8.9 | 57.0 | 0.001 | 0.001 | 0.007 | 144 |
| **3** | **5** | DMSO | 373.4 | 2038.4 | 3628.2 | 27327.2 | 0.095 | 0.060 | 0.239 | 144 |
| **3** | **5** | Methanol | 169.2 | 1526.0 | 1662.0 | 6465.5 | 0.070 | 0.055 | 0.179 | 144 |
| **3** | **5** | Methanol | 205.7 | 46.2 | 46.0 | 249.9 | 0.002 | 0.001 | 0.006 | 144 |
| **3** | **5** | Methanol | 234.7 | 24.3 | 25.1 | 113.3 | 0.001 | 0.000 | 0.003 | 144 |
| **3** | **5** | Methanol | 239.4 | 483.9 | 642.3 | 3403.1 | 0.019 | 0.017 | 0.090 | 144 |
| **3** | **5** | Methanol | 241.0 | 194.0 | 382.7 | 3346.8 | 0.006 | 0.006 | 0.029 | 144 |
| **3** | **5** | Methanol | 250.4 | 71.5 | 104.6 | 744.7 | 0.003 | 0.002 | 0.010 | 144 |
| **3** | **5** | Methanol | 251.2 | 43.1 | 65.4 | 548.5 | 0.002 | 0.001 | 0.006 | 144 |
| **3** | **5** | Methanol | 337.4 | 26.3 | 29.5 | 167.7 | 0.001 | 0.001 | 0.004 | 144 |
| **3** | **5** | Methanol | 373.4 | 1043.9 | 1687.0 | 11607.0 | 0.039 | 0.035 | 0.128 | 144 |
| **3** | **5** | Acetonitrile | 169.2 | 3290.0 | 3100.9 | 15073.0 | 0.150 | 0.056 | 0.313 | 144 |
| **3** | **5** | Acetonitrile | 205.7 | 33.4 | 12.5 | 102.0 | 0.004 | 0.001 | 0.008 | 144 |
| **3** | **5** | Acetonitrile | 234.7 | 57.2 | 44.7 | 317.8 | 0.006 | 0.002 | 0.012 | 144 |
| **3** | **5** | Acetonitrile | 239.4 | 187.1 | 221.0 | 1440.7 | 0.008 | 0.004 | 0.025 | 144 |
| **3** | **5** | Acetonitrile | 241.0 | 64.9 | 87.3 | 669.7 | 0.003 | 0.001 | 0.008 | 144 |
| **3** | **5** | Acetonitrile | 250.4 | 299.6 | 100.6 | 629.8 | 0.038 | 0.010 | 0.063 | 144 |
| **3** | **5** | Acetonitrile | 251.2 | 311.5 | 96.9 | 629.8 | 0.040 | 0.009 | 0.063 | 144 |
| **3** | **5** | Acetonitrile | 337.4 | 8.2 | 9.1 | 50.7 | 0.000 | 0.000 | 0.005 | 144 |
| **3** | **5** | Acetonitrile | 373.4 | 1274.8 | 1544.7 | 12113.3 | 0.049 | 0.025 | 0.120 | 144 |
| **3** | | | 443.3 | 40997.0 | 24891.7 | 384.0 | 0.654 | 0.118 | 0.807 | 384 |

**References.**

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