

Supporting Information for:

Improved Synthesis of 1-(azidomethyl)-3,5-bis(trifluoromethyl)benzene:

Development of Batch and Microflow Azide Processes

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GC Release method for azide 1:

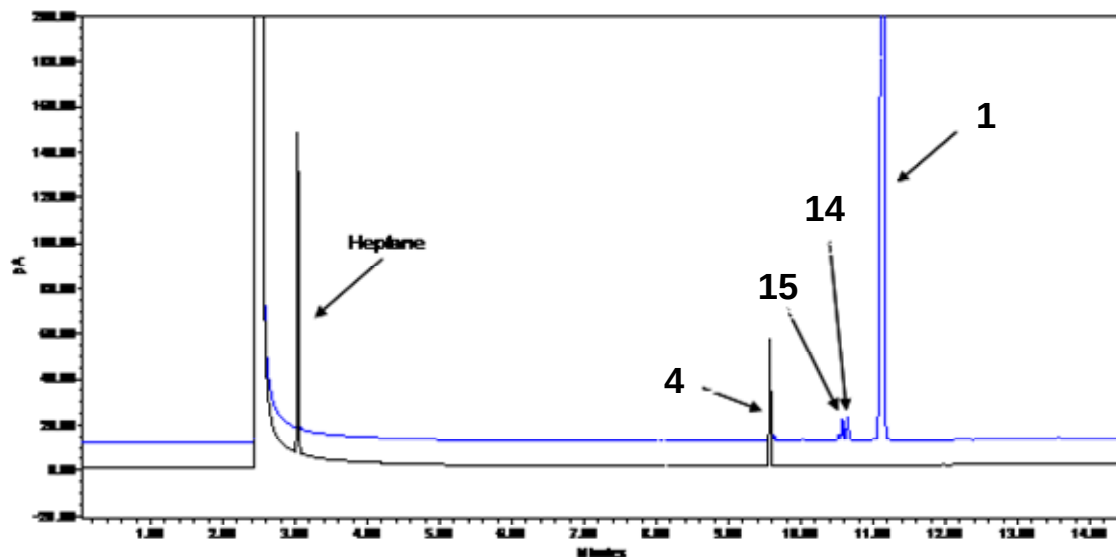


Figure 1. Specificity/Spiking Chromatograms Azide 1 by Gas Chromatography

Table 1. Gas Chromatography Method Conditions for Purity Determination of azide 1.

Column:	DB-210, 30M x 0.25mm, 0.25-micron film thickness
Head Pressure:	15.5 psi
Injection Mode:	Split with a split ratio of 50:1
Injection Temperature:	240 °C
Injection Volume:	1.0 µL
Detector:	FID @ 240 °C
Temperature Program:	35 °C hold for 1.5 min. ramp at 15 °C/min. to 200 °C, hold for 2 min.

In-Process HPLC method for Azide **1** reaction completion:

HPLC conditions (Normal Phase):

Column: Phenomenex Luna 5 micron Silica 25 cm x 4.6 mm

Flow rate: 0.5 ml/min

Wavelength: 250 nm

Temperature: 20 °C

Mobile phase: 1% acetonitrile/hexanes

Retention times:

Benzyl Chloride 4	8.11 min
Azide 1	8.99 min

In-Process Gas Chromatography method for Azide **1** reaction completion:

GC conditions: HP-1 Column, 30 m x 30 μ m x 320 μ m; inlet temperature 250°C, FID detector at 300°C., split ratio 25:1, 1.0 μ L injection

Initial Temp. (°C)	Rate (°C/min)	Final Temp. (°C)	Total Time (min)
35	20	175	8
175	50	300	11.5

Retention Times:

Azide 1: 5.84 min**4:** 5.18 min**7:** 4.39 min**6:** 5.89 min**5:** 5.73 min