

Continuous flow synthesis of the iodination agent 1,3-diiodo-5,5-dimethylimidazolidine-2,4-dione telescoped with semi-continuous product isolation

Marta Ferreri,^{a,b} Audun Drageset,^a Cristian Gambarotti,^b and Hans-René Bjørsvik^{a,*}

^aDepartment of Chemistry, University of Bergen, Allégaten 41, N-5007 Bergen, Norway.

^bDepartment of Chemistry, Materials, and Chemical Engineering, Politecnico di Milano, Piazza Leonardo da Vinci 32, I-20133 Milan, Italy

Table of Content

TGA of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione	2
¹ H-NMR of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione	3
¹³ C-NMR of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione	4
ESI-MS of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione	5
FT-IR of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione	6

TGA of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione

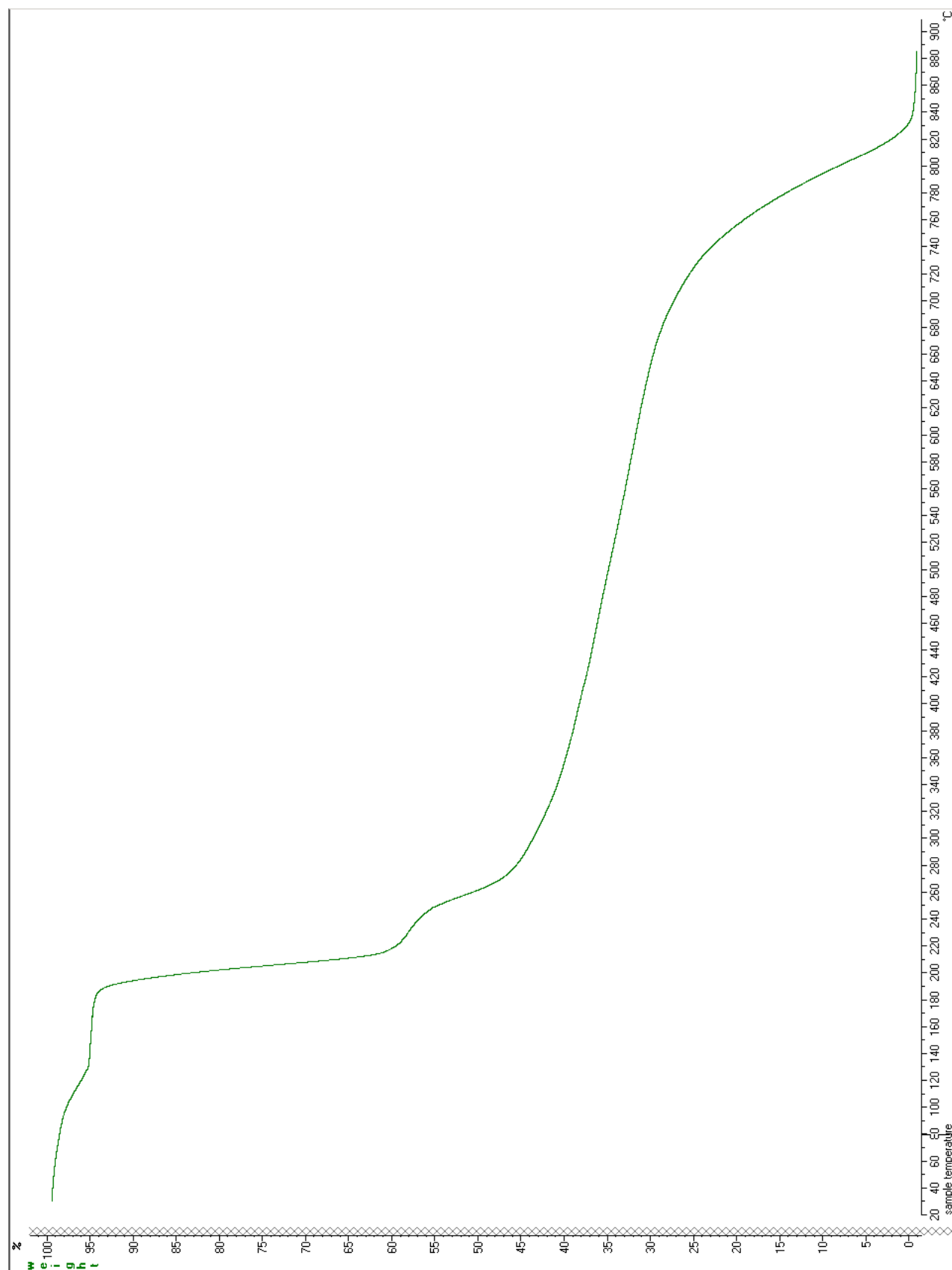
Thass TGA XP-10, Gravimetric Sensitivity: 4700.4 units/mg

Method: 2 steps.

1: Iso at 27.9°C for 10 min, Sample period: 0.5s

2: Scan from 27.9 to 900°C at 5°C/min, Sample period: 0.5s

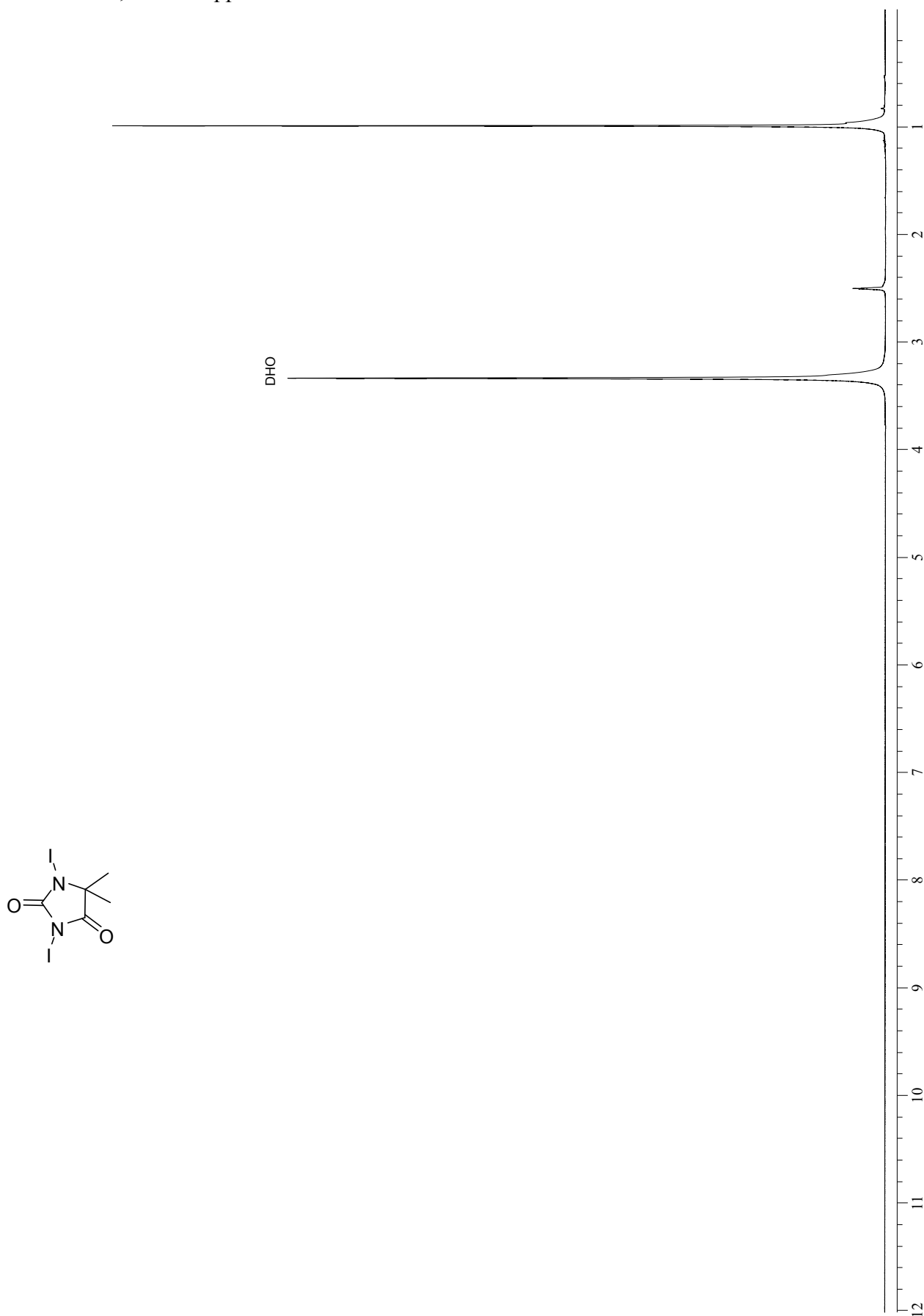
(These data are given in the "Information" menu of the TGA file editor)



¹H-NMR of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione

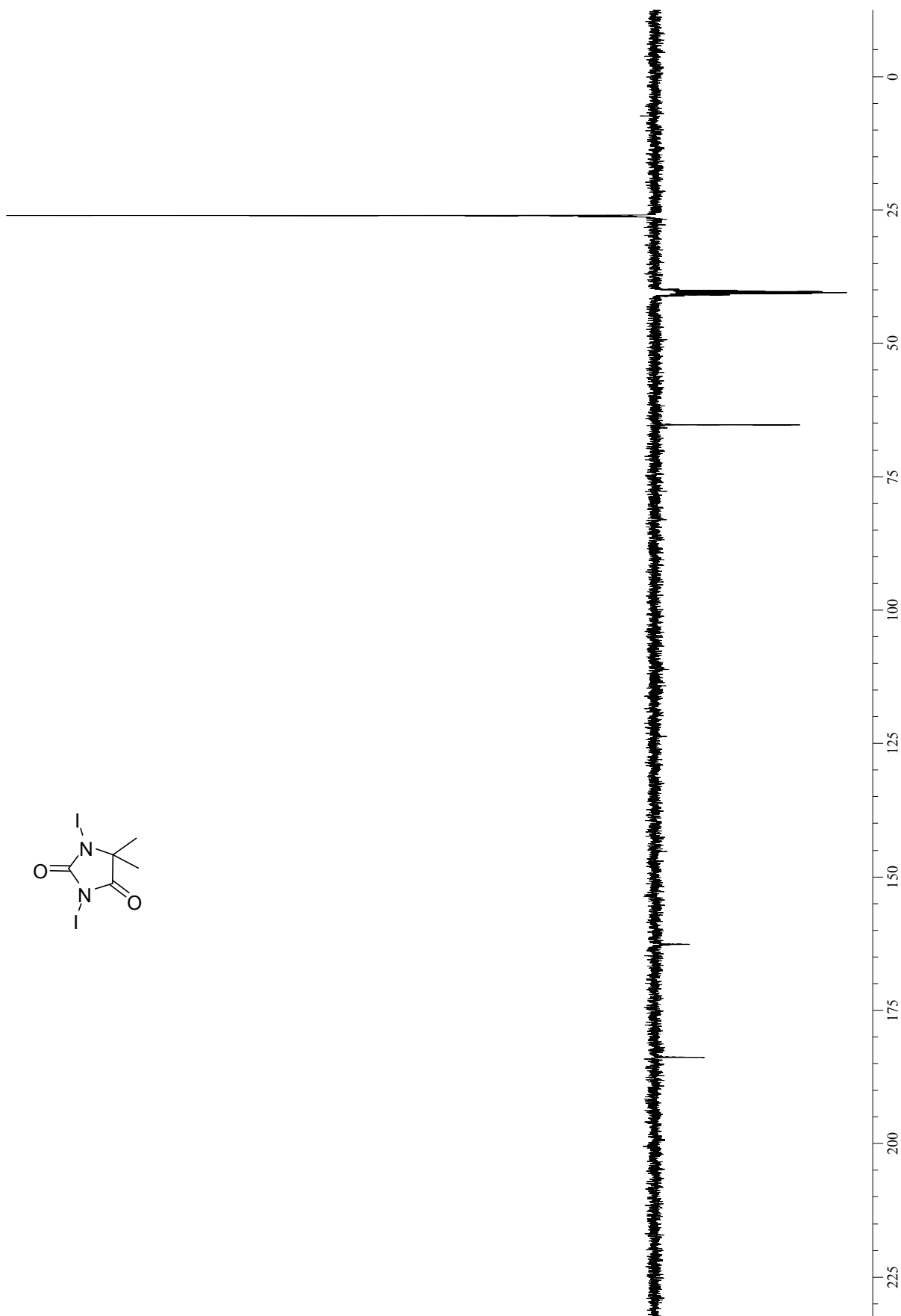
Bruker AV 400 (400 MHz) equipped with a 5 mm multinuclear probe with reverse detection was used to record ¹H-NMR spectra. 32 scans were acquired with an acquiring time of 5 seconds.

Solvent DMSO-d₆; δ = 0.99 ppm



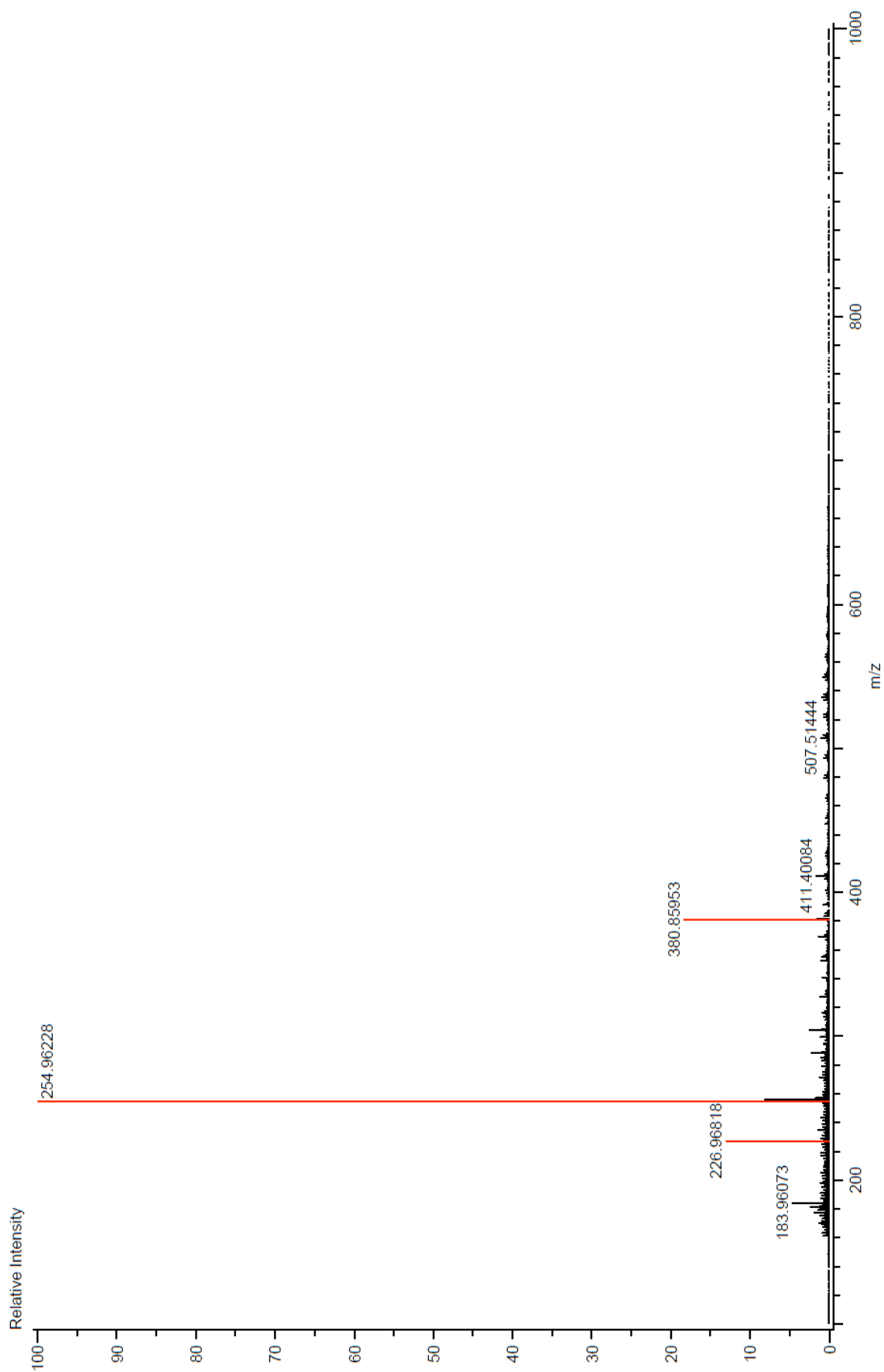
¹³C-NMR of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione

Bruker AV 400 (100 MHz for ¹³C) equipped with a 5 mm multinuclear probe with reverse detection was used to record ¹³C-NMR spectra. 256 scans were acquired with an acquiring time of 5 seconds. Solvent DMSO-d₆; δ = 183.8, 162.6, 65.2, 26.0 ppm



ESI-MS of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione

Internal Sample Id: Orifice1 Volt Sweep: 40V Spec. Record Interval: 0.8[s]
Ionization Mode: ESI+ Acquired m/z Range: 100.0..1000.0 Ring Lens Volt: 20[V]
MS Calibration Name: PEG_ESI+_1000 Time of Maximum: 0.192[min]
Reduction History: Determine m/z[Peak Detect[Centroid,50,Area];Correct Base[5.0%]];Average(MS[1] 0.187..0.210)-1.0*Average(MS[1] 0.090..0.133);Correct Base[5.0%]



FT-IR of 1,3-Diiodo-5,5-dimethylimidazolidine-2,4-dione

Thermo Scientific Nicolet™ iS™50 FT-IR Spectrometer – ATR module

