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Supporting Information

for

Sequential two-step, one-pot microwave-assisted Urech synthesis of 5-monosubstituted hydantoins from L-amino acids in water

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NMR and MS spectra of synthesized compounds

Supporting Information

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1. General information

All chemicals were acquired from commercial sources (Merck or Thermo Fischer) at reagent grade and were used without further purification. The syntheses were carried out in an Anton-Parr Monowave 400 microwave reactor with the reaction temperature controlled using the on-broad infra-red sensor. The NMR spectra were acquired in a Bruker DMX 400 NMR instrument at a resolution of 400 MHz and 100 MHz, for ^{1}H and ^{13}C NMR respectively, using the residual solvent peak of DMSO-d_6 at 2.50 ppm (^{1}H NMR) and 39.5 ppm (^{13}C NMR) as reference. All chemical shifts (δ) were recorded in ppm unit and coupling frequency (*J*) in Hertz (Hz). Shorthand notations were used for the NMR characterization: s (singlet); d (doublet); q (quartet); dd (doublet of doublets); td (triplet of doublets); m (multiplet). The high resolution *m/z* values were measured in Dalton units (Da) using an Agilent 1290 Infinity LC system coupled to an Agilent 6520 Accurate-Mass Q-TOF mass spectrometer with the ESI source in positive mode. Shorthand notation: calcd (calculated). HPLC analyses were performed using a mobile phase of acetonitrile/ultrapure water (9:1) in an Agilent 1260 with a Zorbax C_{18} column (5 μm) and detected by diode array at 210 nm and 254 nm. All hydantoin compounds were reported to be >95% pure.

2. Synthesis and characterization of products

General procedure for the synthesis of (*S*)-5-benzylimidazolidine-2,4-dione (**H2a**):

A 30 mL microwave reactor vial was charged with L-phenylalanine (5 mmol), distilled water (7 mL), and potassium cyanate (25 mmol) and irradiated in an Anton–Paar Monowave 400 microwave reactor at 80 °C for 1 hour. Upon completion of the *N*-carbamylation reaction, as confirmed by TLC analysis, the reaction mixture was treated with concentrated hydrochloric acid (7 mL) and microwave irradiated at 80 °C for 15 min. The precipitates in the reaction mixture were filtered, washed with 1 M HCl solution (2 × 7 mL), distilled water (2 × 10 mL), and dried to afford **H2a** as a white solid. Yield 89%. $[\alpha]_D^{20}$ -100.9 (*c* 11, CH_3CN). ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.44 (s, 1H), 7.93 (s, 1H), 7.17 – 7.30 (m, 5H), 4.33 (td, *J* = 5, 1 Hz, 1H), 2.92 (m, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.2, 157.2, 135.6, 129.8, 128.1, 126.7, 58.4, 36.4. LC-MS (ESI) m/z: 191 $[M+H]^+$, 163, 120. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{10}H_{11}N_2O_2$ 191.0815; Found: 191.0815. The procedure was repeated by substituting L-phenylalanine with the corresponding amino acid to obtain **H2b–c** and **H2e–f**.

General procedure for the synthesis of (*S*)-5-((2,5-dioxoimidazolidin-4-yl)methyl)-1*H*-imidazol-3-ium chloride (**H2d**):

A 30 mL microwave reactor vial was charged with L-histidine (5 mmol), distilled water (7 mL), and potassium cyanate (25 mmol) and irradiated in an Anton-Paar Monowave 400 microwave reactor at 80 °C for 1 hour. Upon completion of the *N*-carbamylation reaction, as confirmed by TLC analysis, the

reaction mixture was treated with concentrated hydrochloric acid (7 mL) and microwave irradiated at 80 °C for 15 min. The reaction mixture was neutralized to pH 7 using saturated sodium bicarbonate solution and extracted with ethyl acetate multiple times. The organic extracts were combined, dried with anhydrous sodium sulfate, and concentrated under reduced pressure to afford **H2d** as a white solid. Yield 70%. ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.59 (s, 1H), 7.88 (s, 1H), 7.74 (s, 1H), 6.88 (s, 1H), 5.52 (s, 2H), 4.25 (t, *J* = 6 Hz, 1H), 2.95 (dd, *J* = 15, 4 Hz, 1H), 2.81 (dd, *J* = 15, 6 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.5, 157.4, 134.6, 131.6, 117.0, 57.6, 28.8. LC-MS (ESI) m/z: 181 $[M+H]^+$, 136. The procedure was repeated by substituting L-histidine with the corresponding amino acid to obtain **H2g–j**.

2.1 Characterization data

Carbamoyl-L-phenylalanine (**H1a**):

White solid. Yield 89%. $[\alpha]_D^{20}$ -33.4 (*c* 10, CH_3CN). ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.28 (t, *J* = 7 Hz, 2H), 7.20 (m, 3H), 6.18 (d, *J* = 8 Hz, 1H), 5.62 (s, 2H), 4.33 (dd, *J* = 13, 8 Hz, 1H), 2.99 (dd, *J* = 13, 5 Hz, 1H), 2.84 (dd, *J* = 13, 8 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 174.1, 158.4, 137.6, 129.4, 128.3, 126.6, 53.9, 37.7. LC-MS (ESI) m/z: 209 $[M+H]^+$, 166, 120. Data in accordance with literature [1].[20]

(*S*)-5-(4-Hydroxybenzyl)imidazolidine-2,4-dione (**H2b**):

White solid. Yield 84%. $[\alpha]_D^{20}$ -90.5 (*c* 10, acetone). ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.39 (1H, s), 9.27 (s, 1H), 7.87 (s, 1H), 6.96 (d, *J* = 8 Hz, 2H), 6.64 (d, *J* = 8 Hz, 2H), 4.23 (q, *J* = 5 Hz, 1H), 2.80 (d, *J* = 5 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.4, 157.3, 156.1, 130.8, 125.5, 114.9, 58.7, 35.6. LC-MS (ESI) m/z: 207 $[M+H]^+$, 179, 136, 107. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{10}H_{11}N_2O_3$ 207.0764; Found: 207.0759.

(*S*)-5-((1*H*-Indol-2-yl)methyl)imidazolidine-2,4-dione (**H2c**):

White solid. Yield 71%. $[\alpha]_D^{20}$ -97.1 (*c* 10, CH_3CN). ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.90 (s, 1H), 10.37 (s, 1H), 7.91 (s, 1H), 7.55 (d, *J* = 8 Hz, 1H), 7.32 (d, *J* = 8 Hz, 1H), 7.12 (d, *J* = 2 Hz, 1H), 7.05 (td, *J* = 7, 1 Hz, 1H), 6.97 (td, *J* = 7, 1 Hz, 1H), 4.30 (dd, *J* = 4, 1 Hz, 1H), 3.06 (d, *J* = 4 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.8, 157.5, 135.9, 127.5, 124.2, 120.9, 118.7, 118.4, 111.3, 108.0, 58.4, 26.5. LC-MS (ESI) m/z: 230 $[M+H]^+$, 130. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_{12}H_{12}N_3O_2$ 230.0924; Found: 230.0926.

(*S*)-5-Phenylimidazolidine-2,4-dione (**H2e**):

White solid. Yield 48%. $[\alpha]_D^{20}$ -136.0 (*c* 10, CH_3CN). ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.81 (s, 1H), 8.41 (s, 1H), 7.32 – 7.43 (m, 5H), 5.16 (s, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 174.3, 157.6, 136.1, 128.7, 128.3, 126.8, 61.3. LC-MS (ESI) m/z: 177 $[M+H]^+$, 149, 106. Data in accordance with literature [2].[21]

(*S*)-5-Isobutylimidazolidine-2,4-dione (**H2f**):

White solid. Yield 34%. $[\alpha]_D^{20}$ -78.0 (*c* 10, CH_3CN). ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.59 (s, 1H), 8.01 (s, 1H), 3.99 (m, 1H), 1.75 (m, 1H), 1.34 – 1.51 (m, 2H), 0.87 (t, *J* = 6 Hz, 6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 176.6, 157.5, 56.2, 40.8, 24.2, 23.2, 21.5. LC-MS (ESI) m/z: 157 $[M+H]^+$, 129. Data in accordance with literature [2].[21]

(*S*)-5-((*R*)-1-Hydroxyethyl)imidazolidine-2,4-dione (**H2g**):

White solid. Yield 78%. $[\alpha]_D^{20}$ -103.4 (*c* 10, CH_3CN). ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.89 (s, 1H), 5.48 (s, 1H), 4.93 (d, *J* = 6 Hz, 1H), 3.85 – 3.92 (m, 1H), 3.82 (dd, *J* = 3, 1 Hz, 1H), 1.12 (d, *J* = 6 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 174.8, 158.2, 65.2, 63.9, 20.3. LC-MS (ESI) m/z: 145 $[M+H]^+$, 101. Data in accordance with literature [3].[22]

(*S*)-5-Isopropylimidazolidine-2,4-dione (**H2h**):

White solid. Yield 86%. $[\alpha]_D^{20}$ -85.9 (*c* 10, CH_3CN). ^1H NMR (400 MHz, DMSO-d_6) δ 10.56 (s, 1H), 7.89 (s, 1H), 3.89 (dd, *J* = 4, 1 Hz, 1H), 1.94 – 2.02 (m, 1H), 0.93 (d, *J* = 7 Hz, 3H), 0.79 (d, *J* = 7 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.5, 157.9, 62.8, 29.6, 18.6, 15.9. LC-MS (ESI) m/z: 143 $[M+H]^+$, 115. HRMS (ESI) m/z: $[M+H]^+$ Calcd for $C_6H_{11}N_2O_2$ 143.0815; Found: 143.0814.

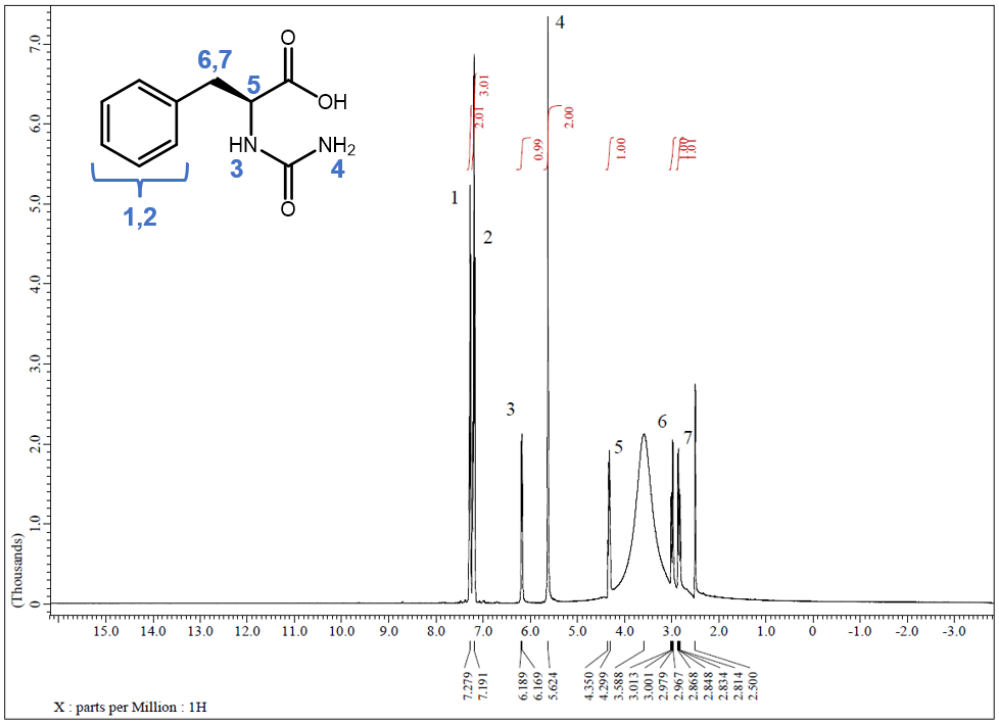
(*S*)-5-Methylimidazolidine-2,4-dione (**H2i**):

White solid. Yield 83%. $[\alpha]_D^{20}$ -23.2 (*c* 10, CH_3CN). ^1H NMR (400 MHz, DMSO-d_6) δ 10.58 (s, 1H), 7.88 (s, 1H), 4.02 (q, *J* = 7 Hz, 1H), 1.21 (d, *J* = 7 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 176.9, 157.2, 53.4, 17.3. LC-MS (ESI) m/z: 115 $[M+H]^+$. Data in accordance with literature [2].[21]

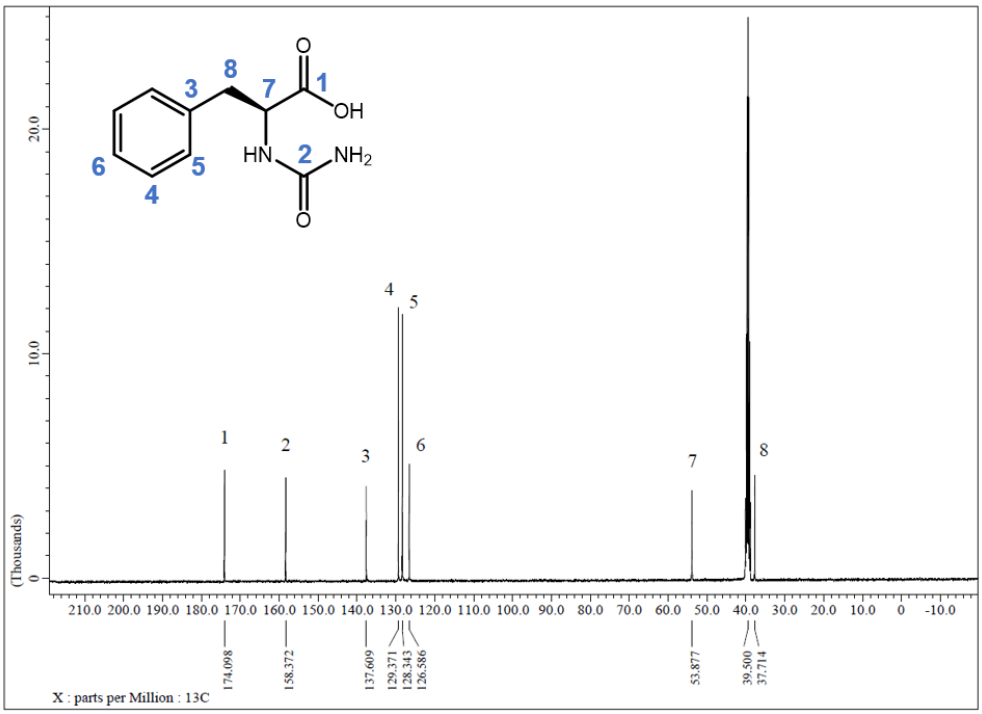
(*S*)-5-(2-(Methylthio)ethyl)imidazolidine-2,4-dione (**H2j**):

White solid. Yield 59%. $[\alpha]_D^{20}$ -49.9 (*c* 10, CH_3CN). ^1H NMR (400 MHz, DMSO-d_6) δ 10.66 (s, 1H), 8.02 (s, 1H), 4.10 (qd, *J* = 4, 1 Hz, 1H), 2.53 (m, 2H), 2.05 (s, 3H), 1.87 – 1.97 (m, 1H), 1.71 – 1.80 (m, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.9, 157.4, 56.6, 31.0, 28.8, 14.5. LC-MS (ESI) m/z: 175 $[M+H]^+$, 147, 127. Data in accordance with literature [2].[21]

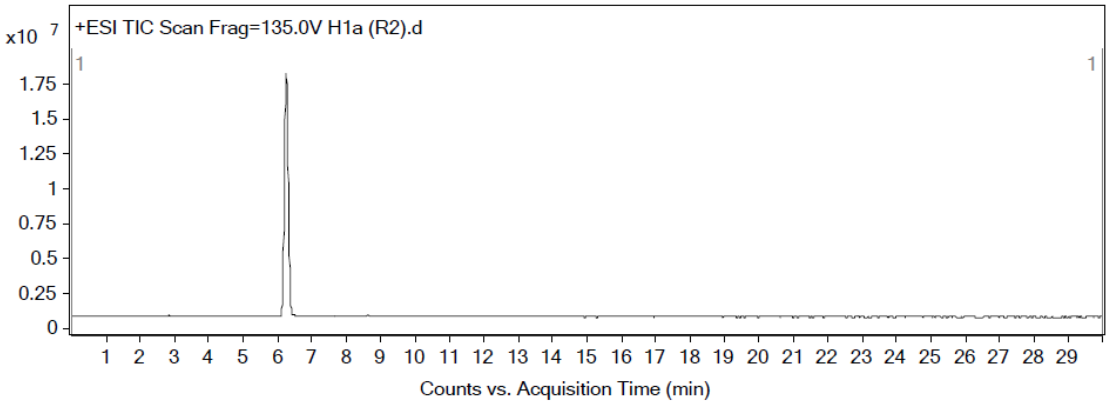
3. NMR and HRMS Spectra

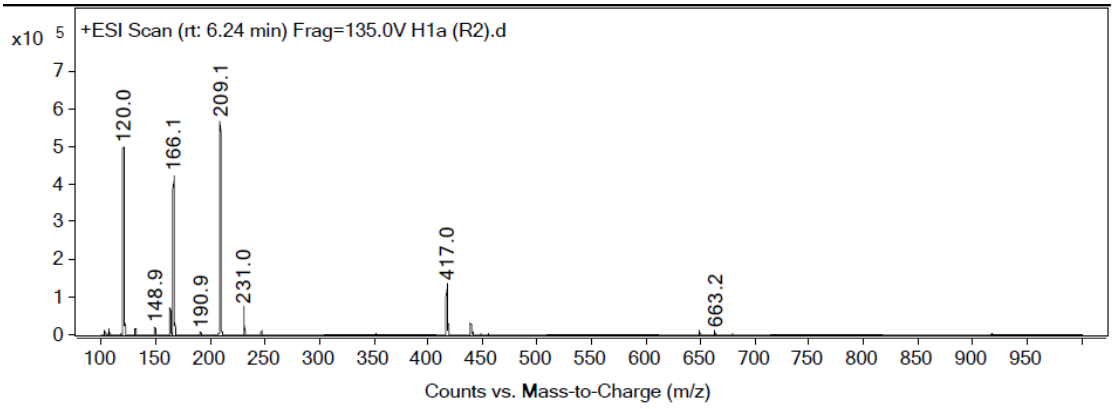


^1H NMR spectrum of carbamoyl-L-phenylalanine (**H1a**)

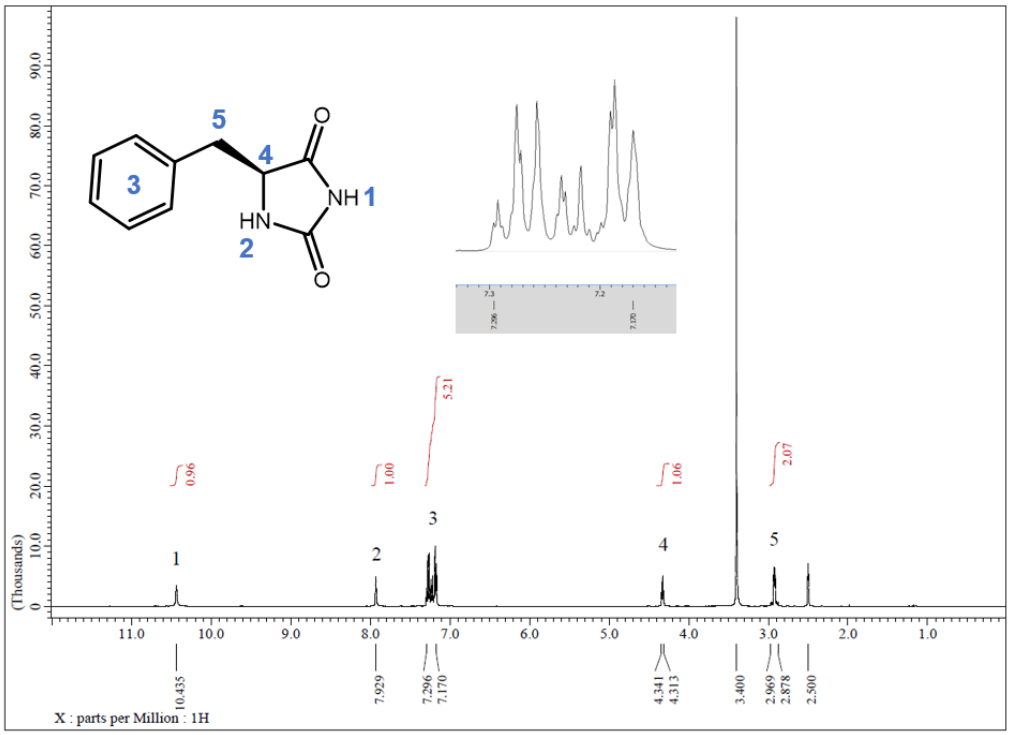


^{13}C NMR spectrum of carbamoyl-L-phenylalanine (**H1a**)

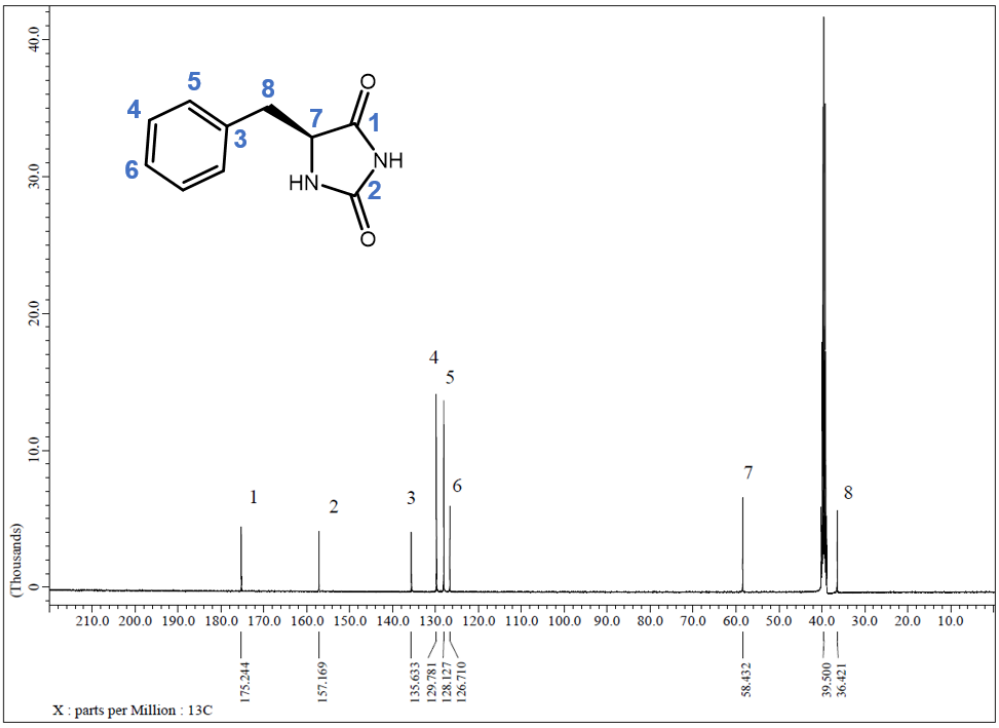




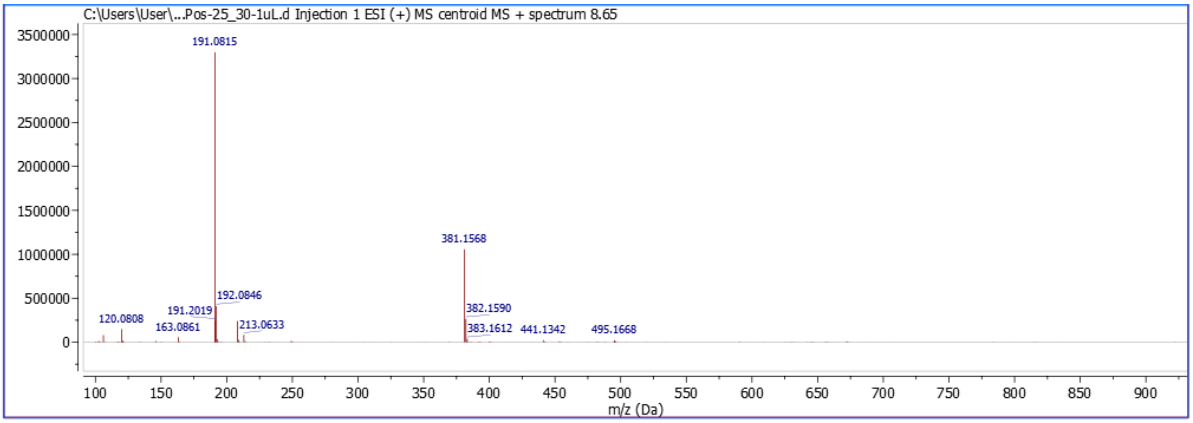
Mass spectrum of carbamoyl-L-phenylalanine (**H1a**)



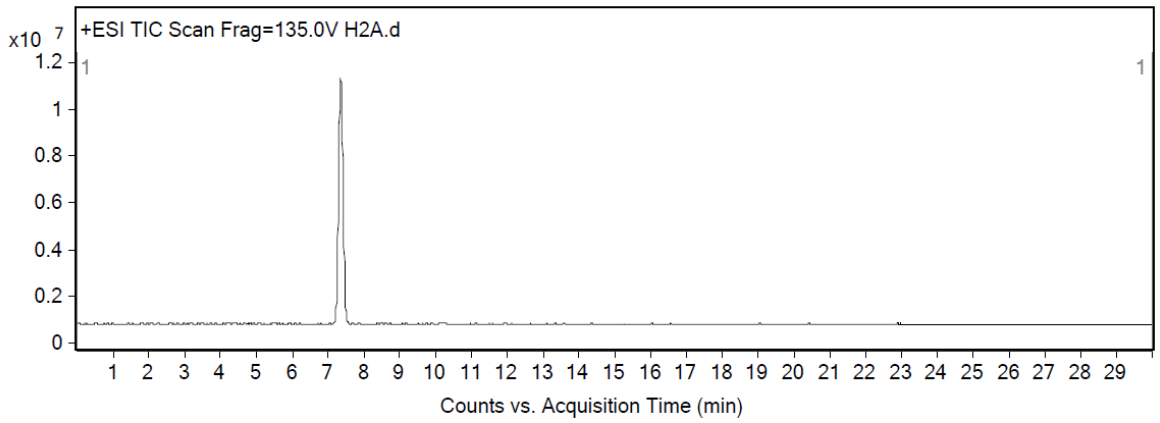
^{1}H NMR spectrum of (*S*)-5-benzylimidazolidine-2,4-dione (**H2a**)

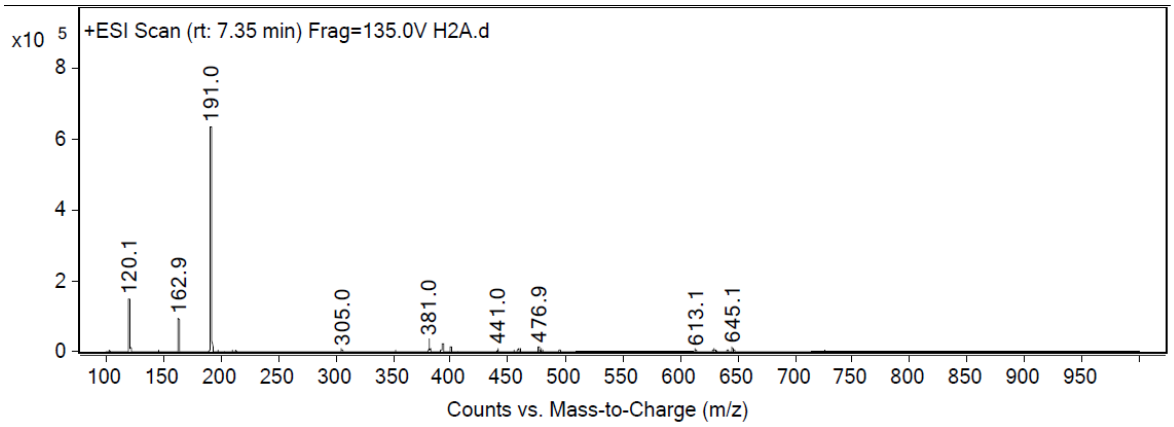


^{13}C NMR spectrum of (*S*)-5-benzylimidazolidine-2,4-dione (**H2a**)

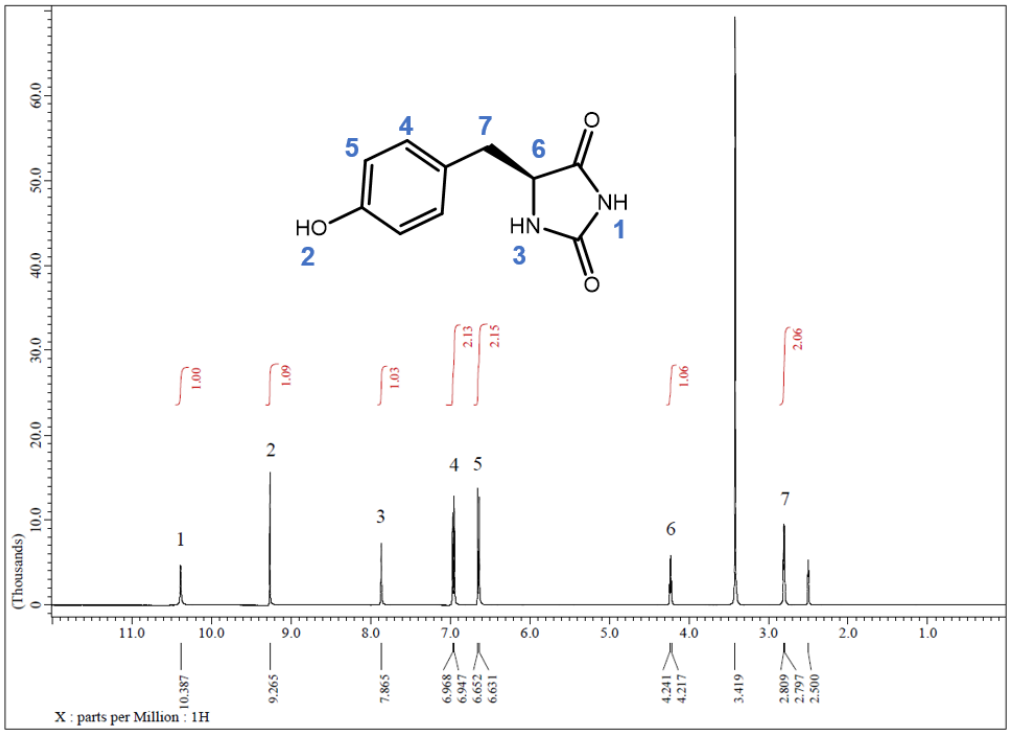


HRMS spectrum of (*S*)-5-benzylimidazolidine-2,4-dione (**H2a**)

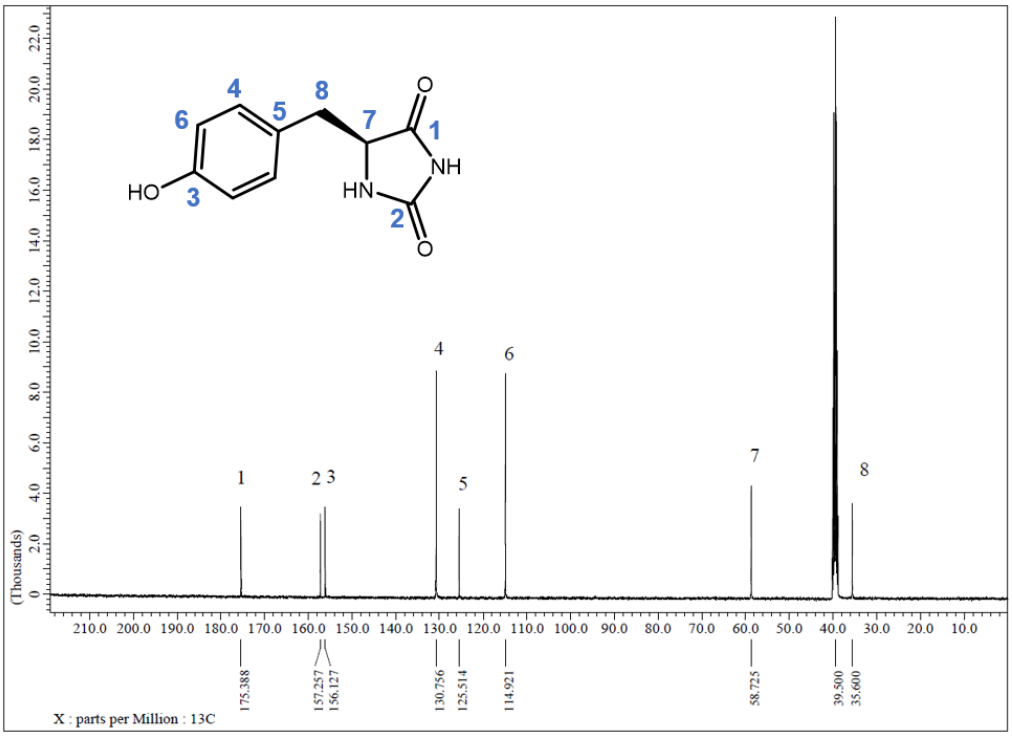




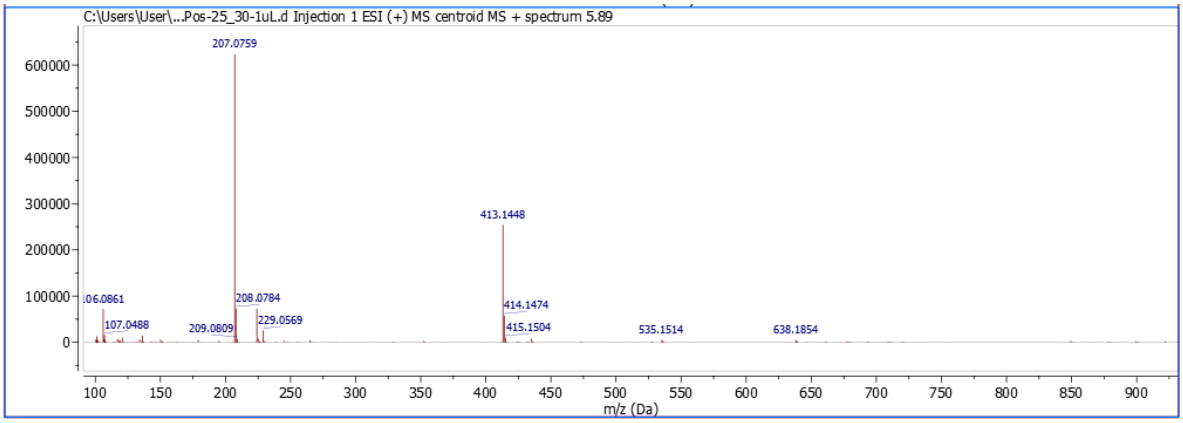
Mass spectrum of (*S*)-5-benzylimidazolidine-2,4-dione (**H2a**)



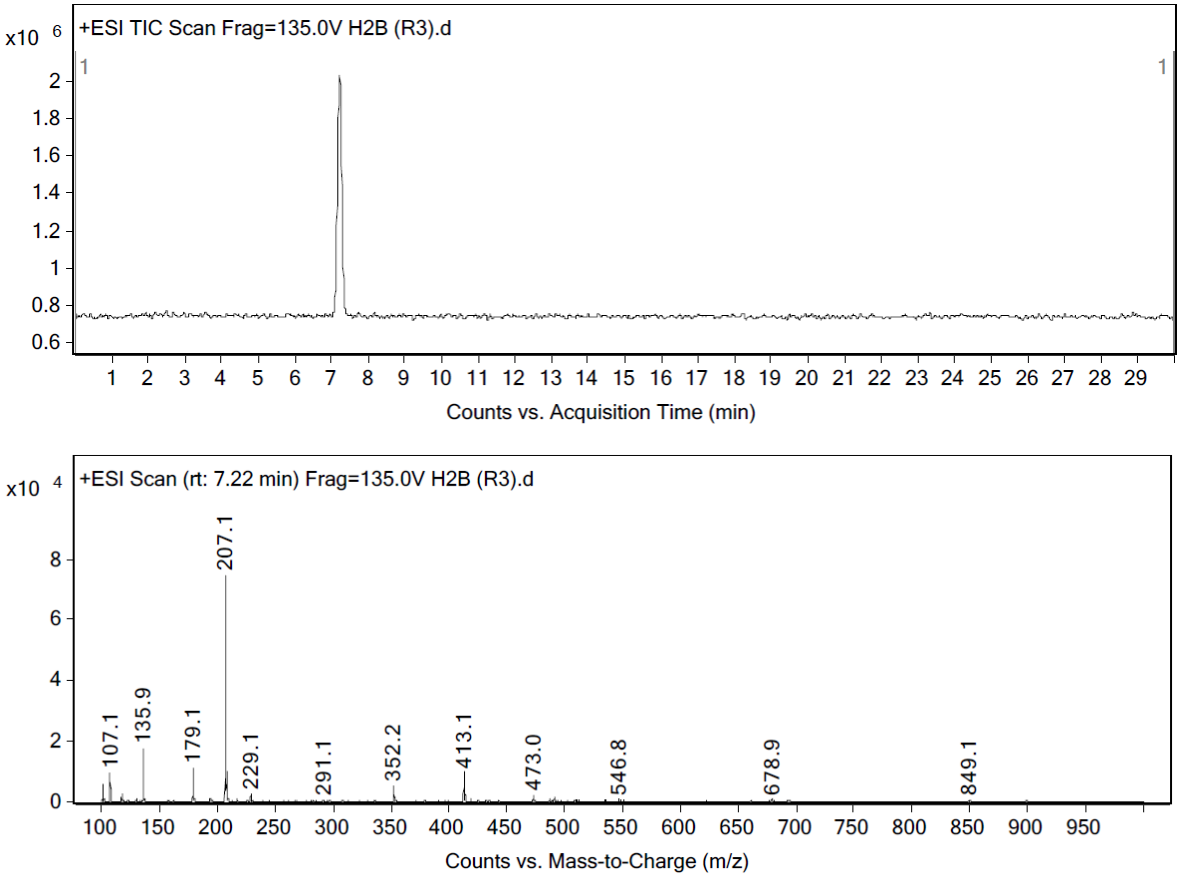
^{1}H NMR spectrum of (*S*)-5-(4-hydroxybenzyl)imidazolidine-2,4-dione (**H2b**)



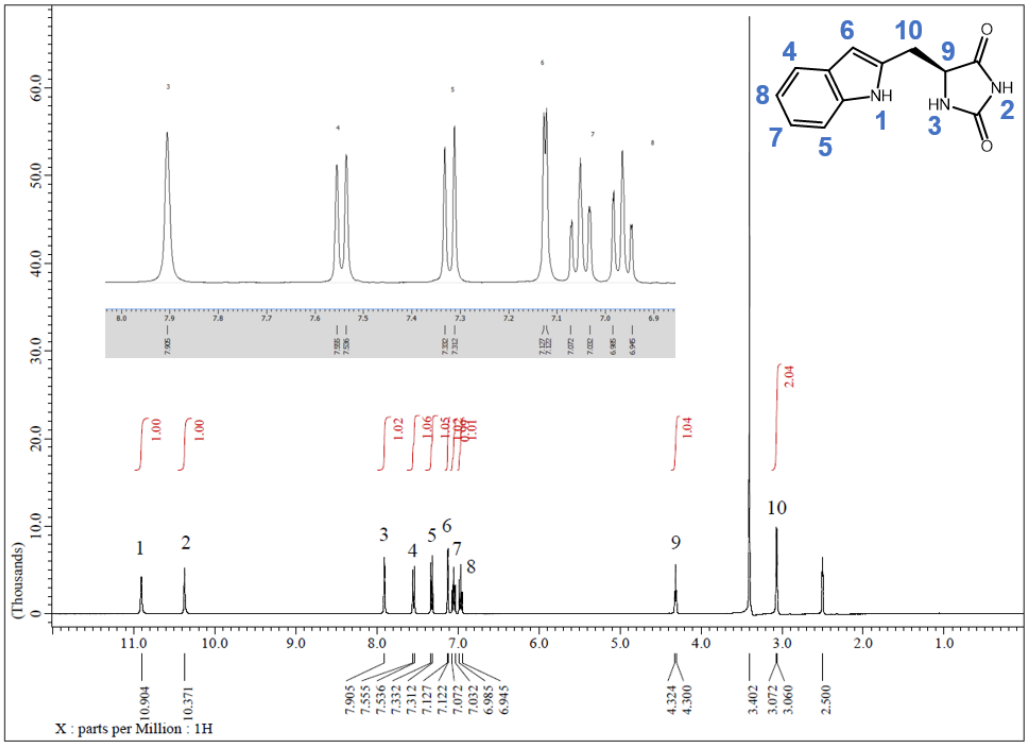
^{13}C NMR spectrum of (*S*)-5-(4-hydroxybenzyl)imidazolidine-2,4-dione (**H2b**)



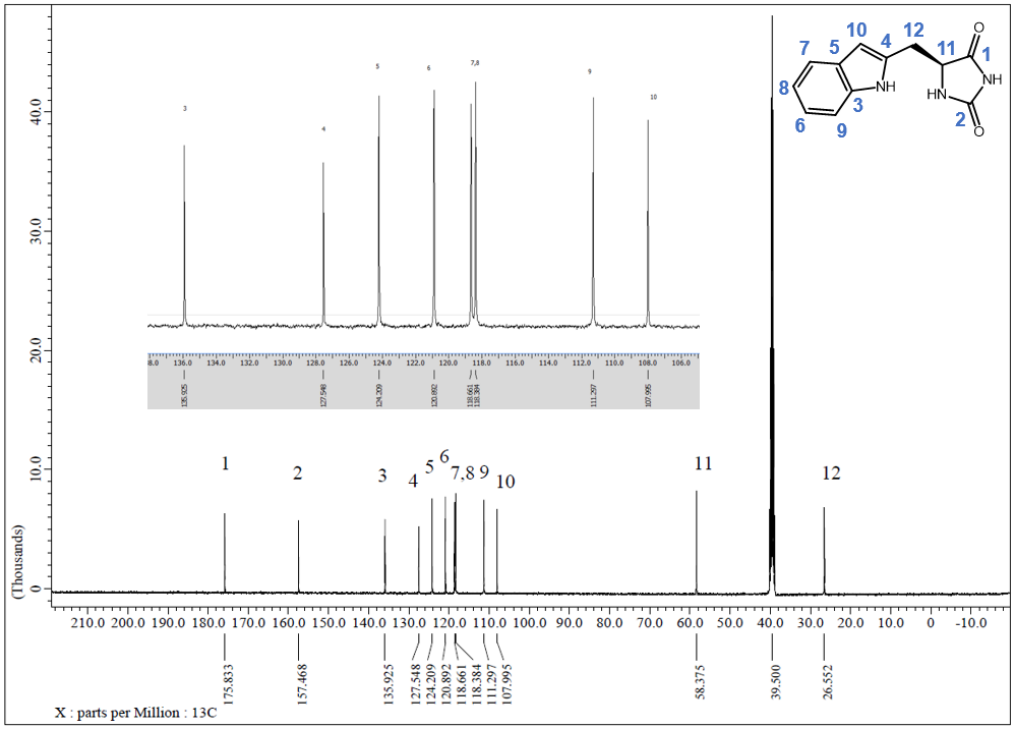
HRMS spectrum of (*S*)-5-(4-hydroxybenzyl)imidazolidine-2,4-dione (**H2b**)



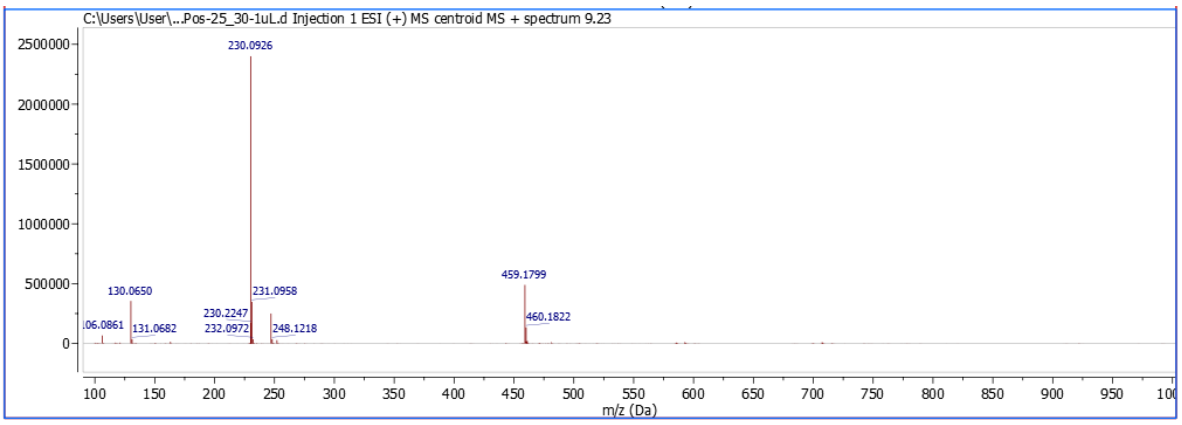
Mass spectrum of (*S*)-5-(4-hydroxybenzyl)imidazolidine-2,4-dione (**H2b**)



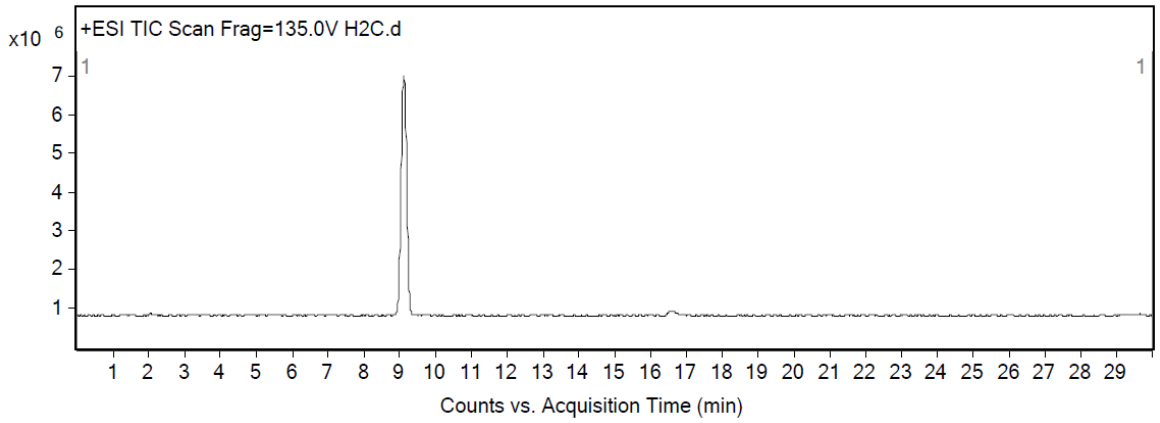
^{1}H NMR spectrum of (*S*)-5-((1*H*-indol-2-yl)methyl)imidazolidine-2,4-dione (**H2c**)

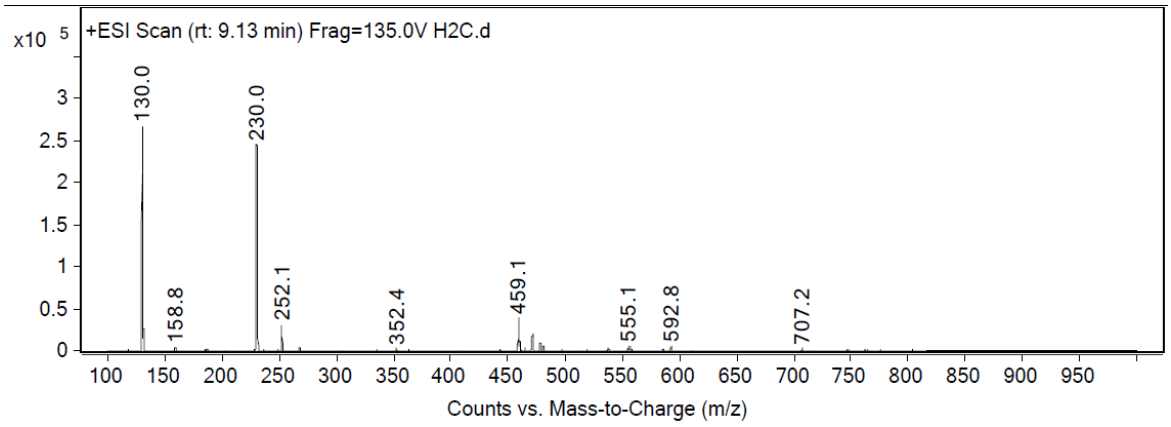


^{13}C NMR spectrum of (*S*)-5-((1*H*-indol-2-yl)methyl)imidazolidine-2,4-dione (**H2c**)

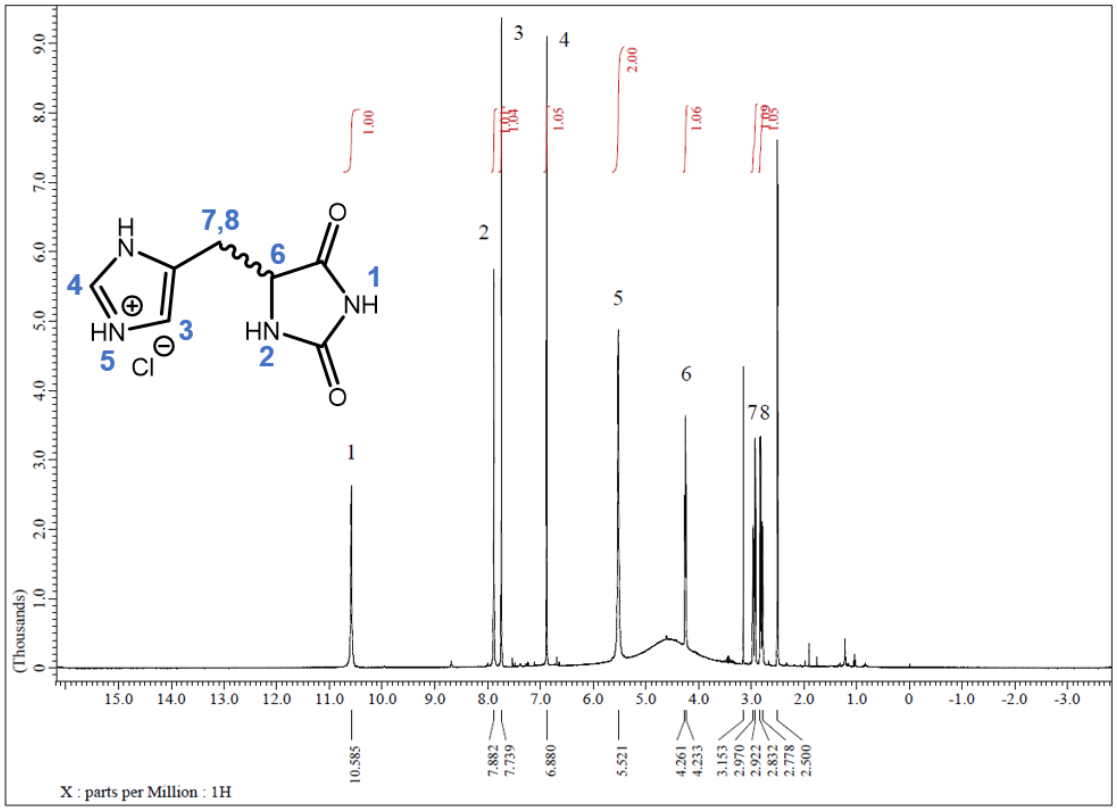


HRMS spectrum of (*S*)-5-((1*H*-indol-2-yl)methyl)imidazolidine-2,4-dione (**H2c**)

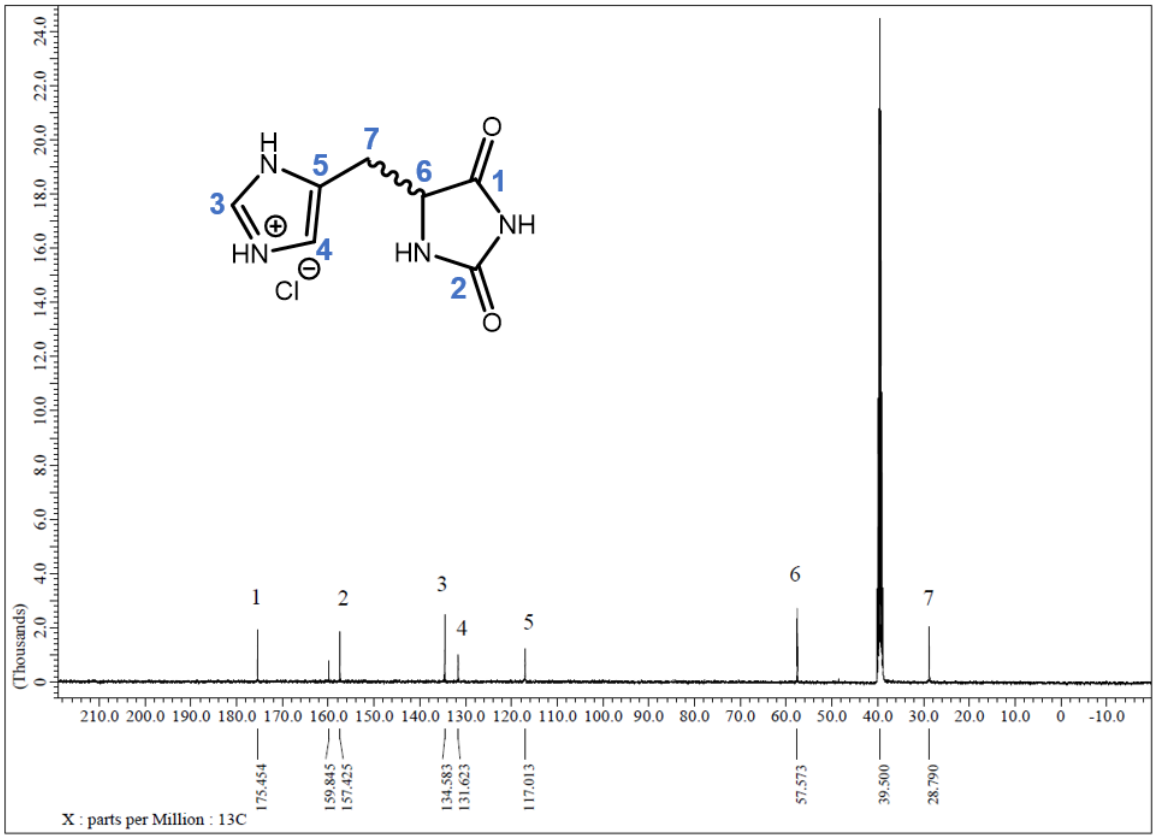




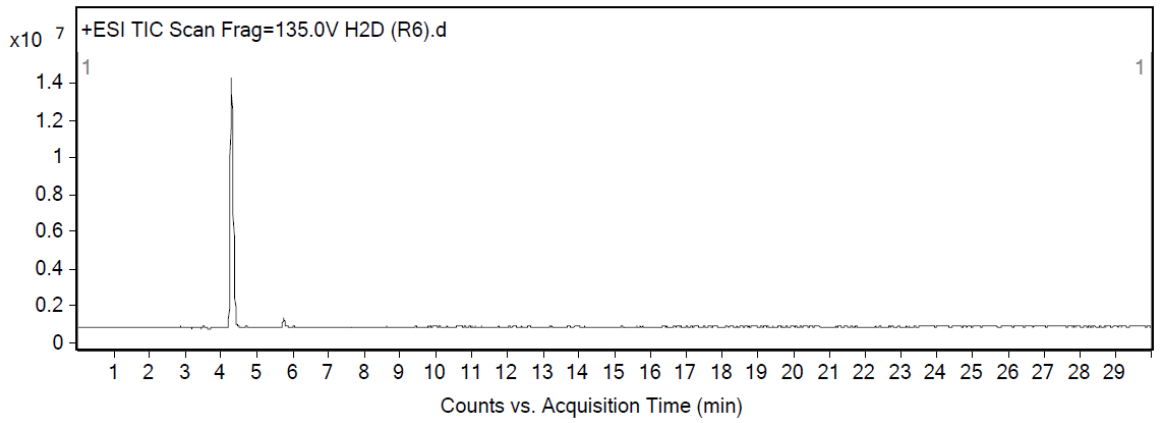
Mass spectrum of (*S*)-5-((1*H*-indol-2-yl)methyl)imidazolidine-2,4-dione (**H2c**)

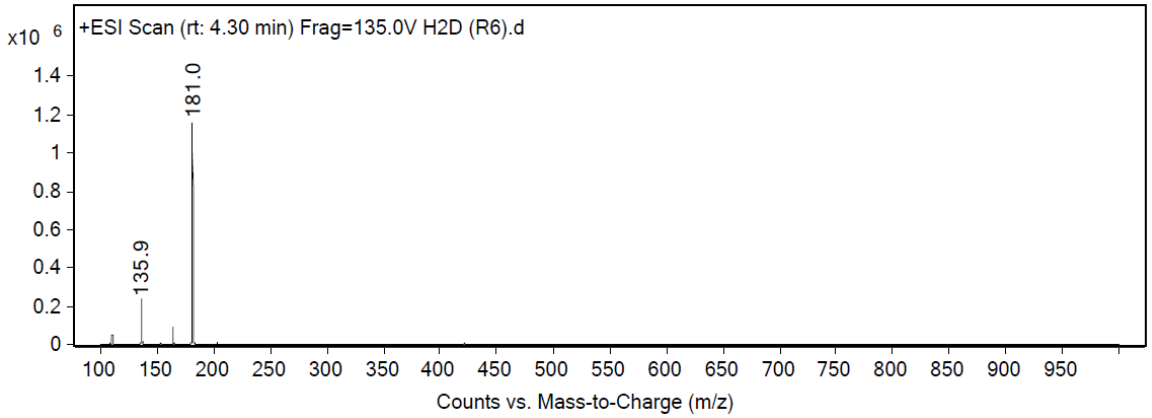


^{1}H NMR spectrum of (*S*)-5-((2,5-dioxoimidazolidin-4-yl)methyl)-1*H*-imidazol-3-ium chloride (**H2d**)

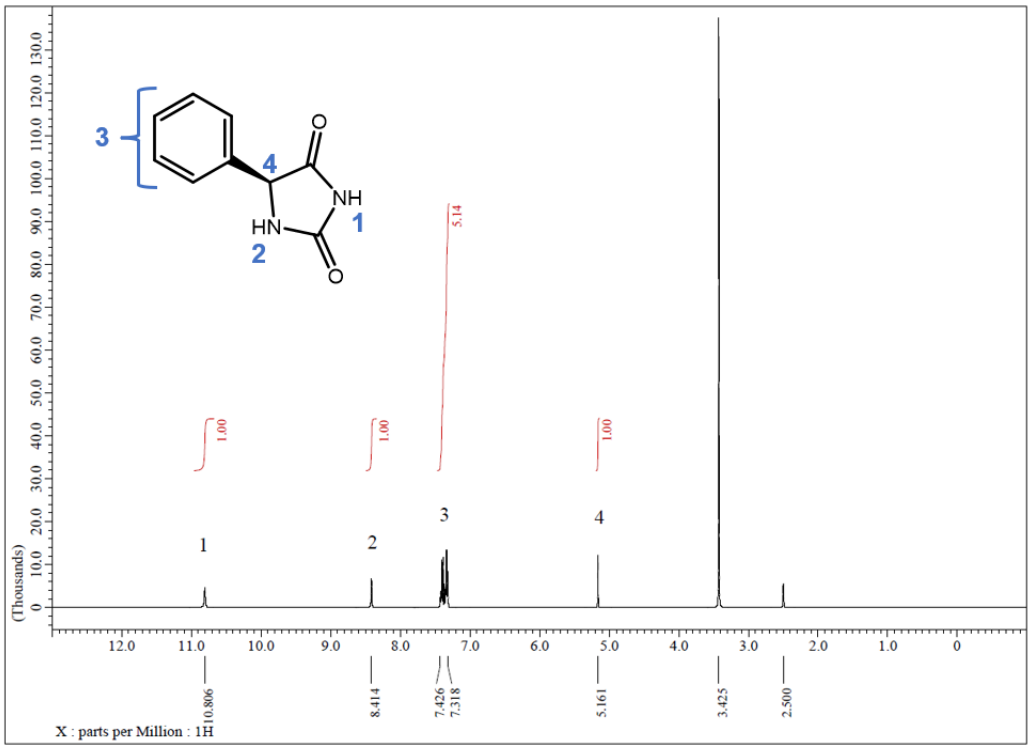


^{13}C NMR spectrum of (*S*)-5-((2,5-dioxoimidazolidin-4-yl)methyl)-1*H*-imidazol-3-ium chloride (**H2d**)

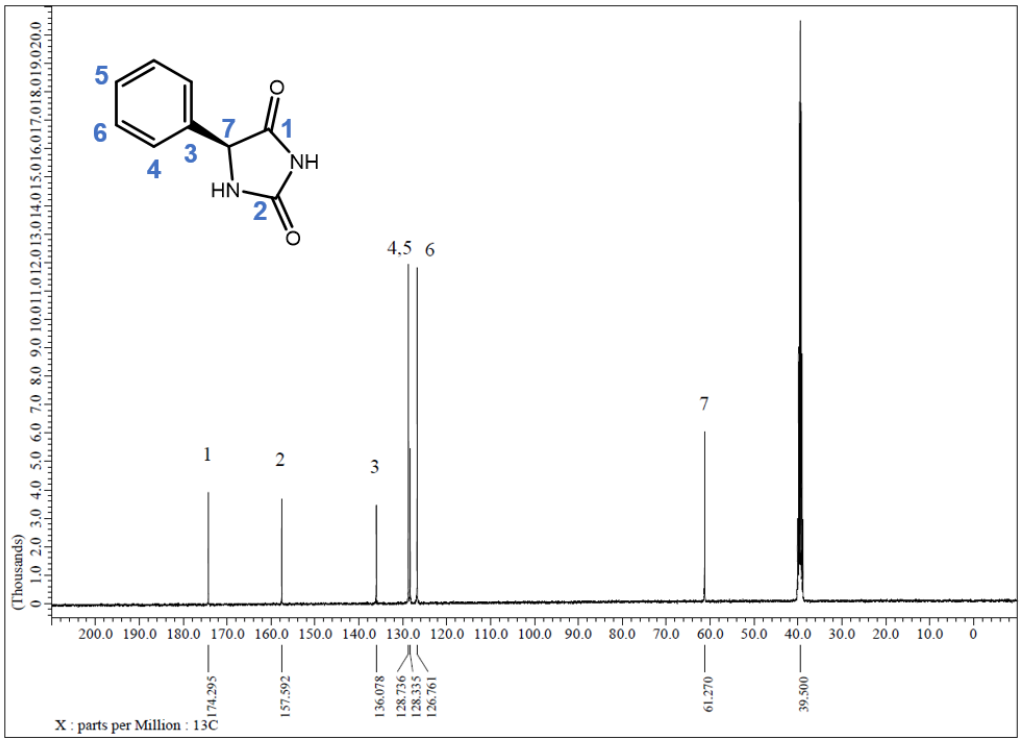




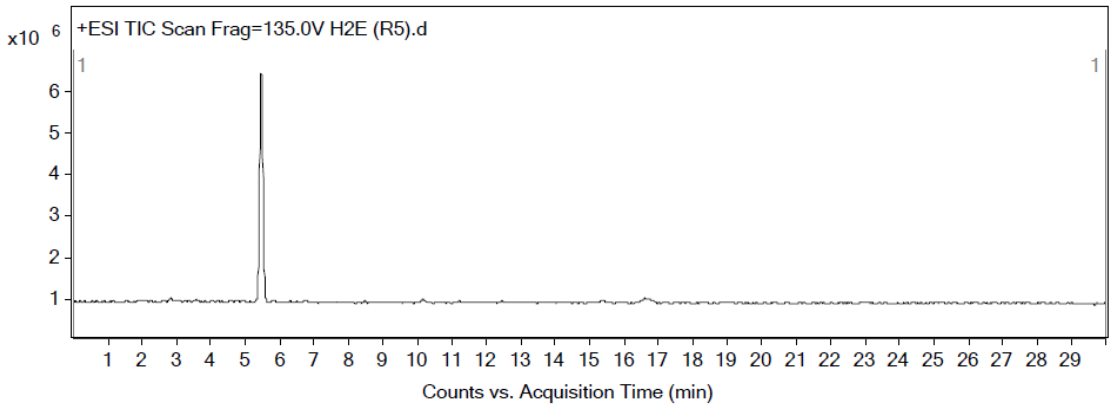
Mass spectrum of (*S*)-5-((2,5-dioxoimidazolidin-4-yl)methyl)-1*H*-imidazol-3-ium chloride (**H2d**)

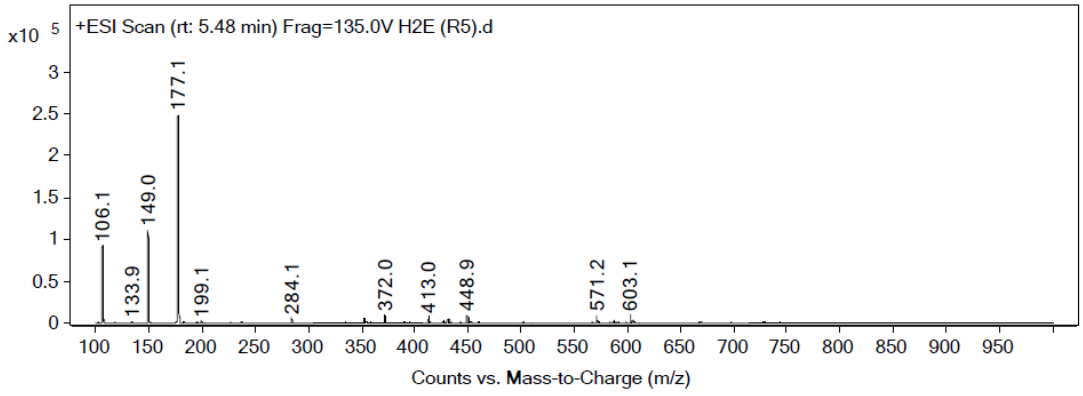


^{1}H NMR spectrum of (*S*)-5-phenylimidazolidine-2,4-dione (**H2e**)



^{13}C NMR spectrum of (*S*)-5-phenylimidazolidine-2,4-dione (**H2e**)





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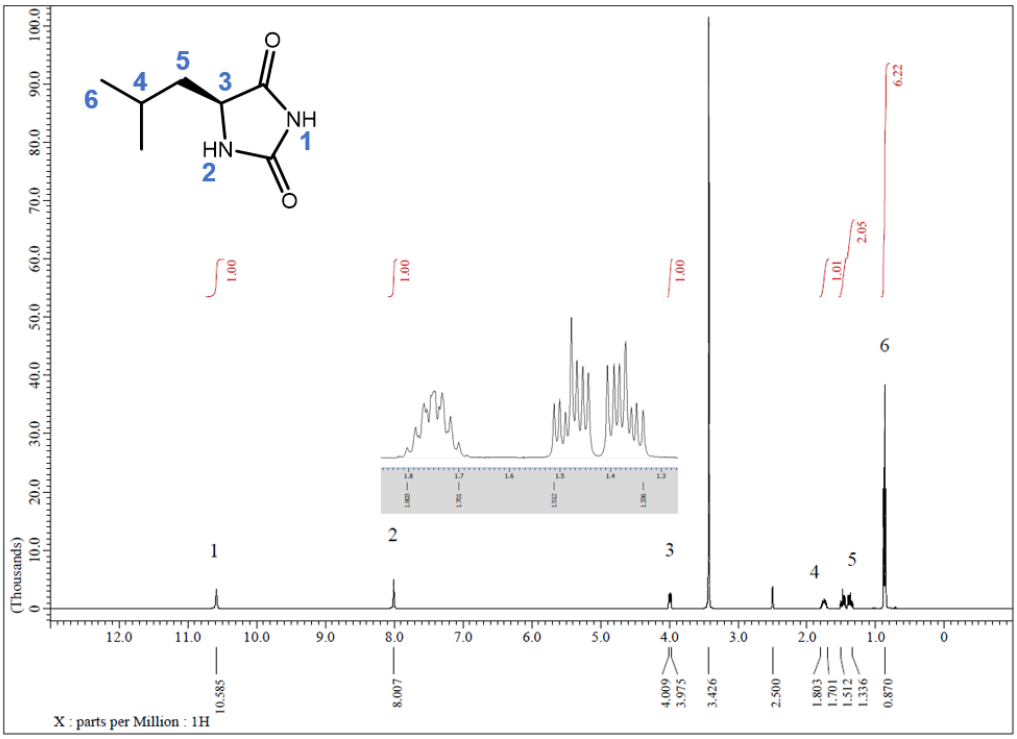

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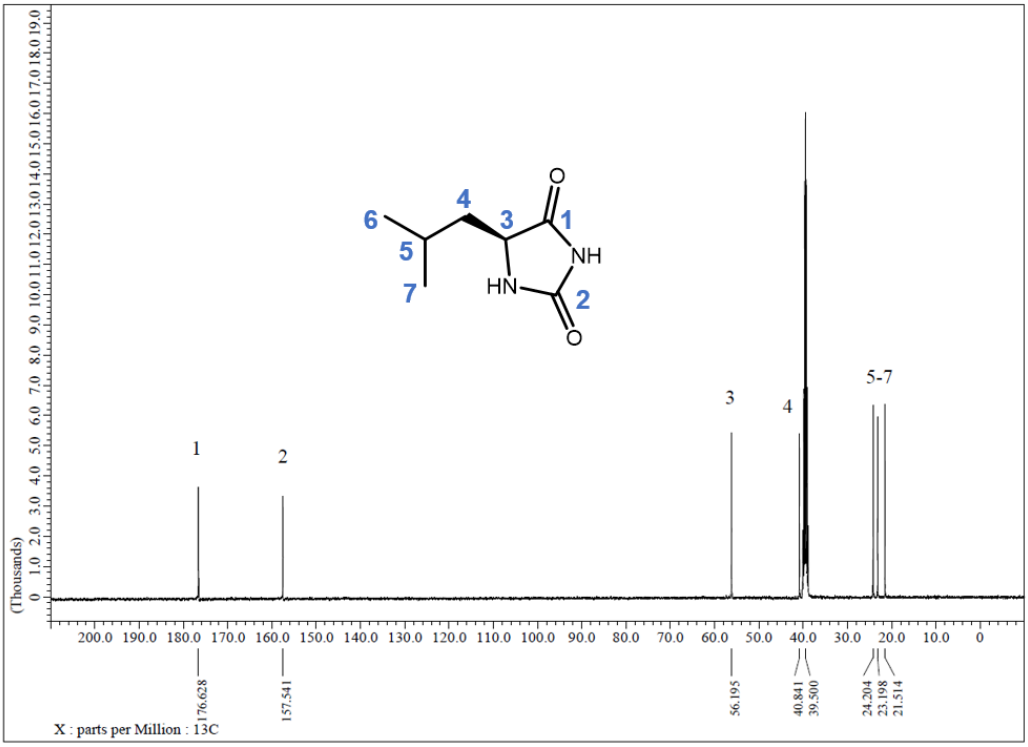
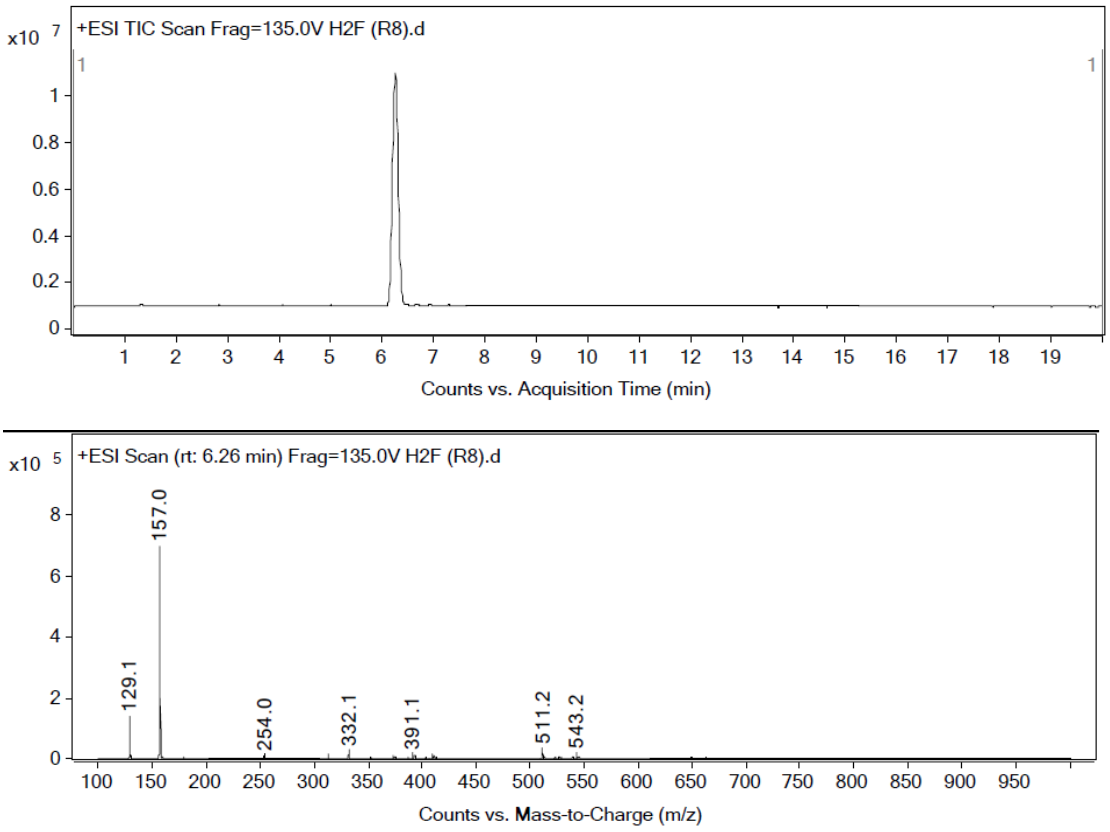
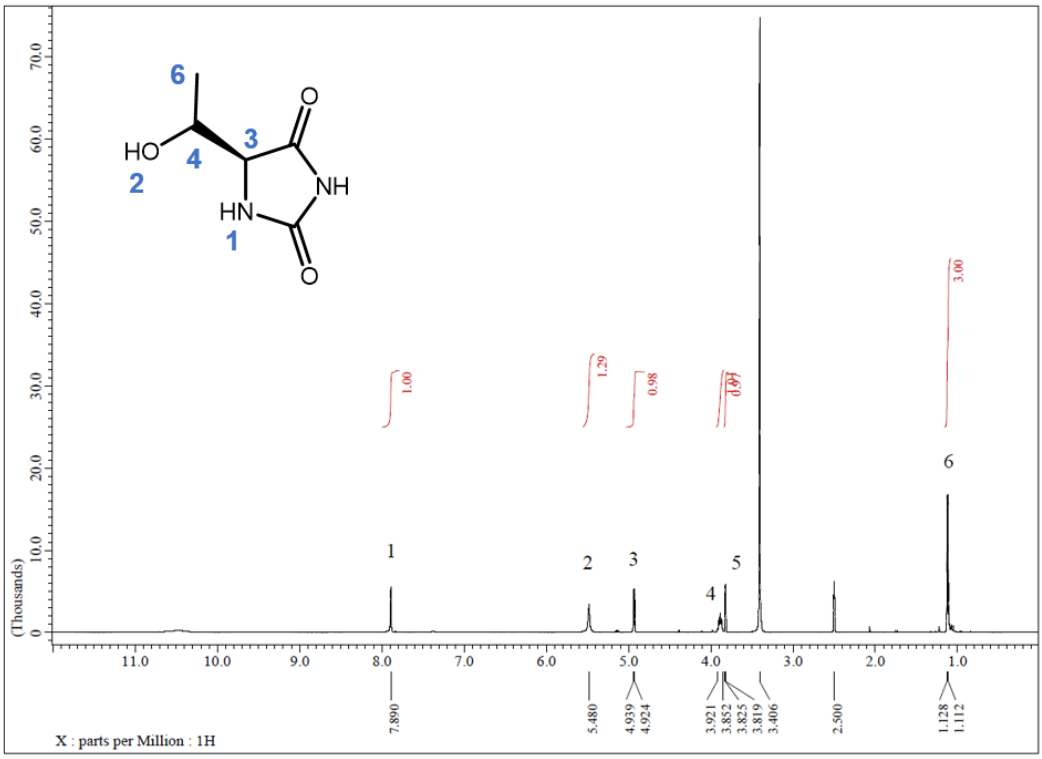


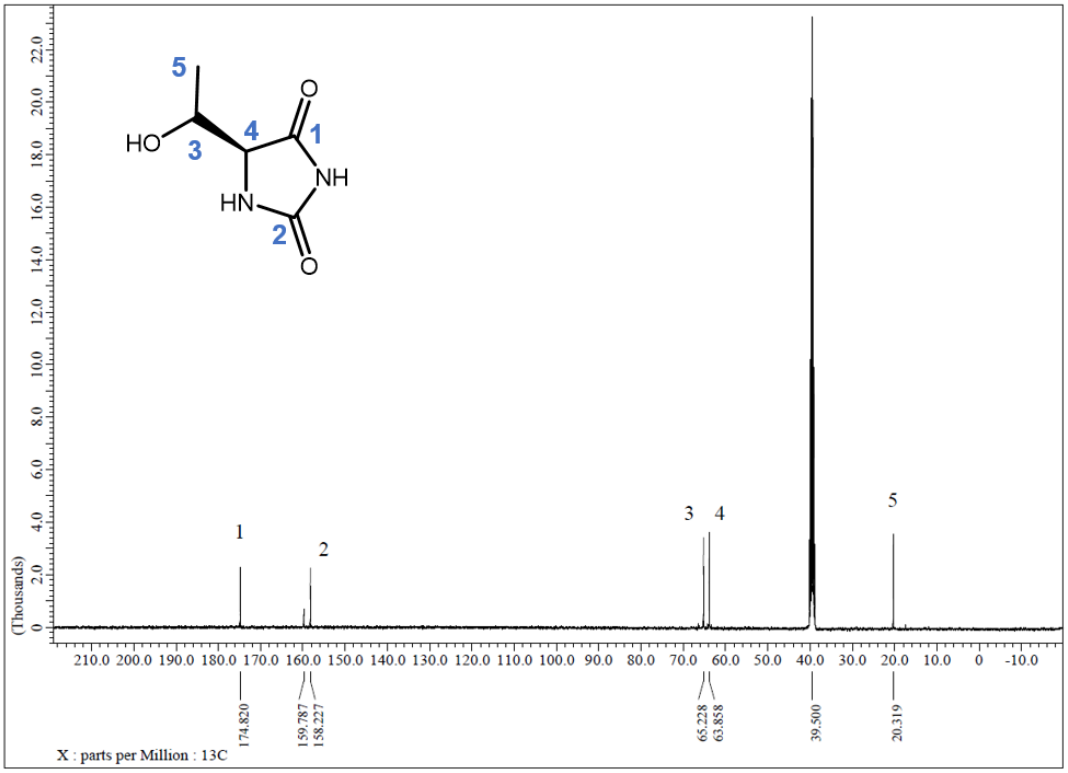
^{13}C NMR spectrum of (*S*)-5-isobutylimidazolidine-2,4-dione (**H2f**)



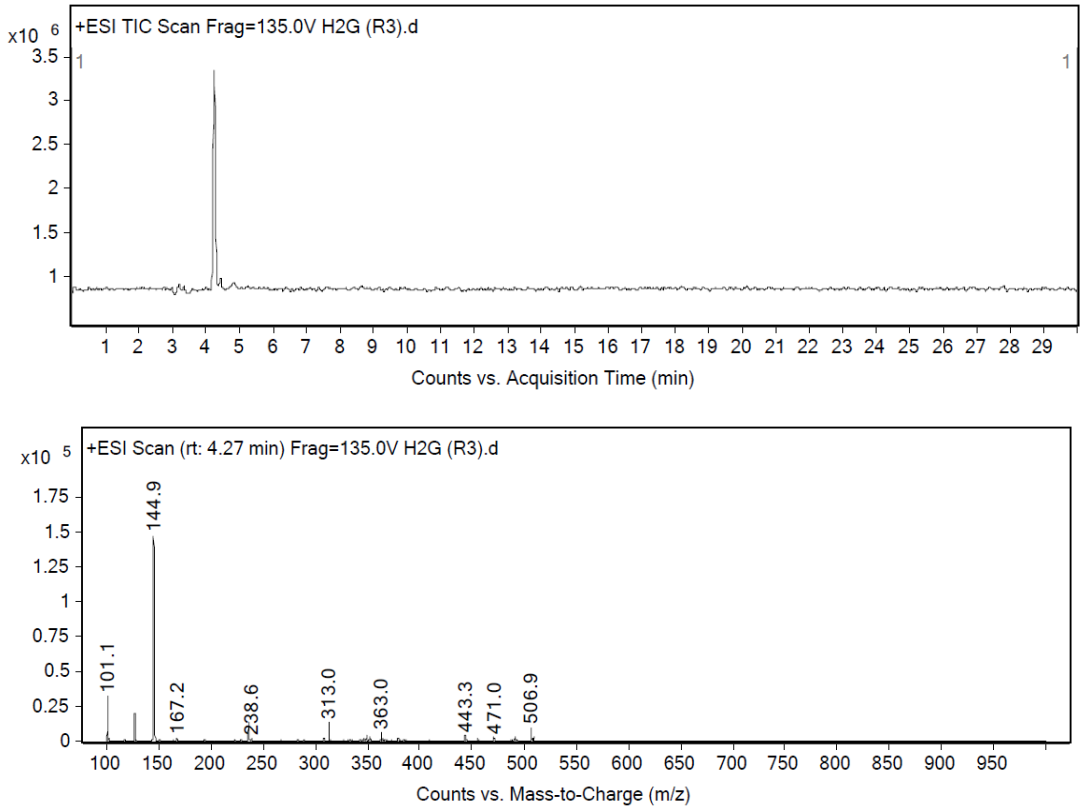
Mass spectrum of (*S*)-5-isobutylimidazolidine-2,4-dione (**H2f**)



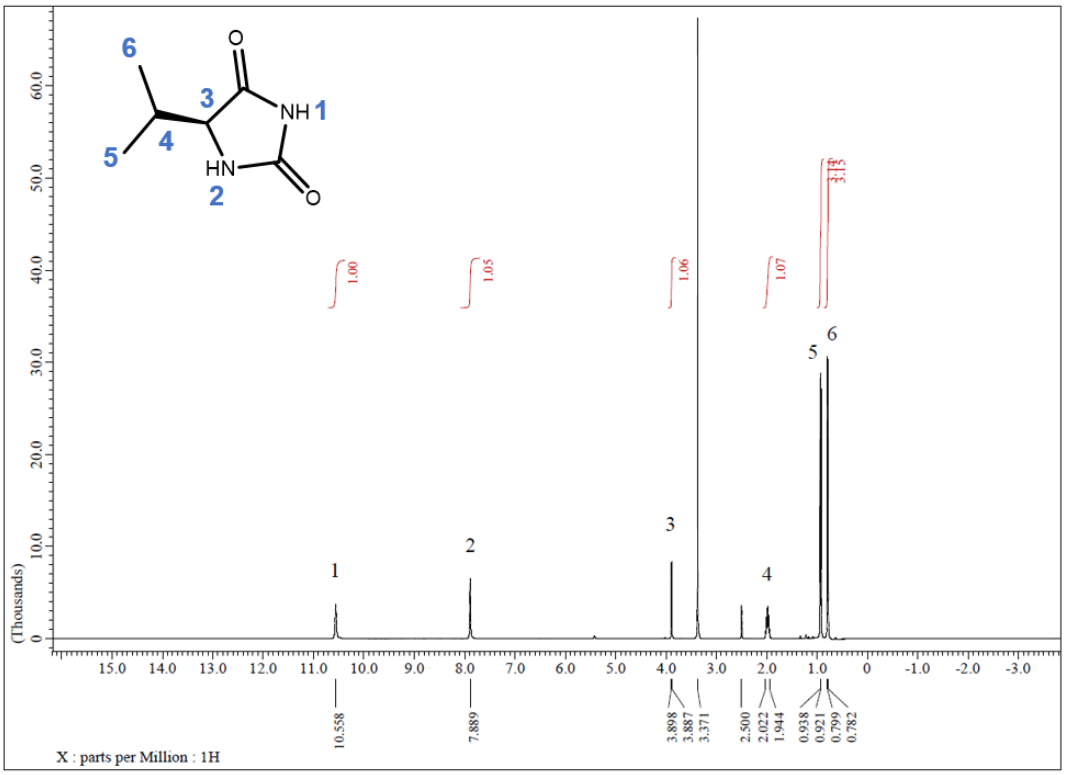
^{1}H NMR spectrum of (*S*)-5-((*R*)-1-hydroxyethyl)imidazolidine-2,4-dione (**H2g**)



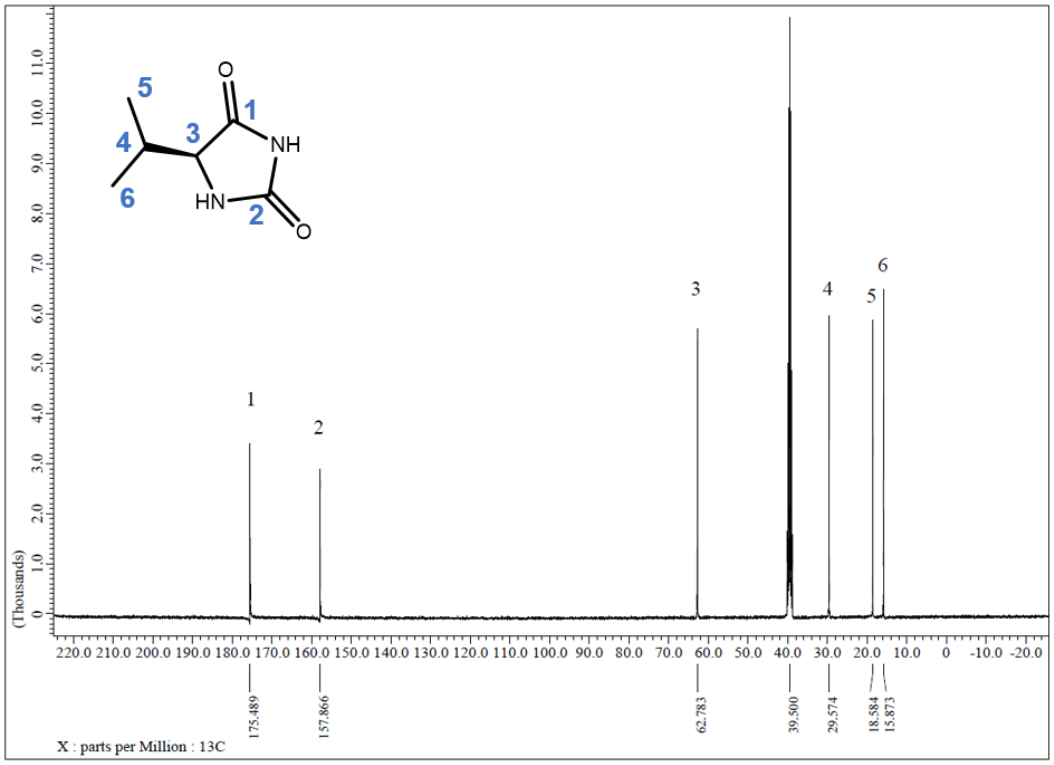
^{13}C NMR spectrum of (*S*)-5-((*R*)-1-hydroxyethyl)imidazolidine-2,4-dione (**H2g**)



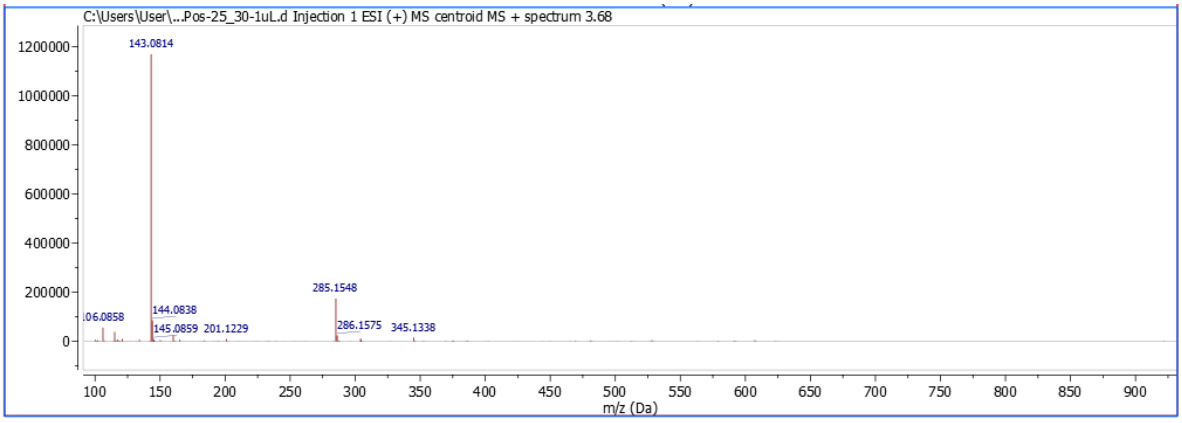
Mass spectrum of (*S*)-5-((*R*)-1-hydroxyethyl)imidazolidine-2,4-dione (**H2g**)



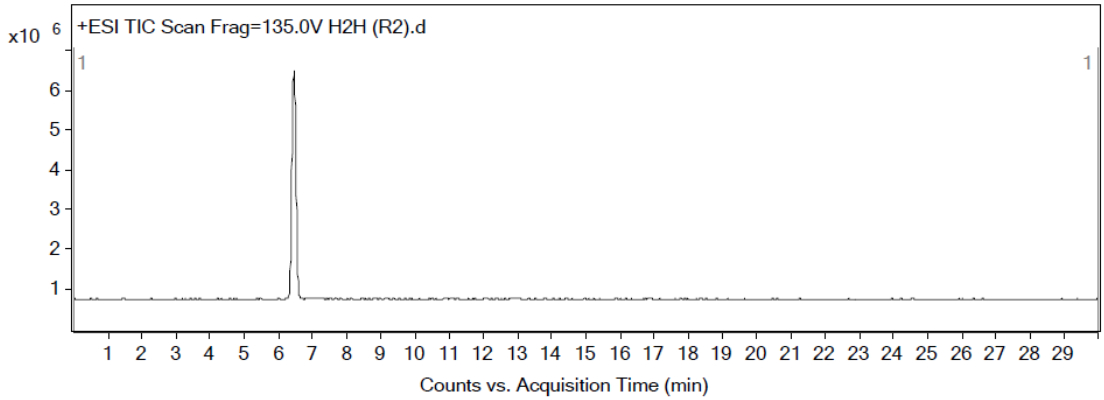
^{1}H NMR spectrum of (*S*)-5-isopropylimidazolidine-2,4-dione (**H2h**)

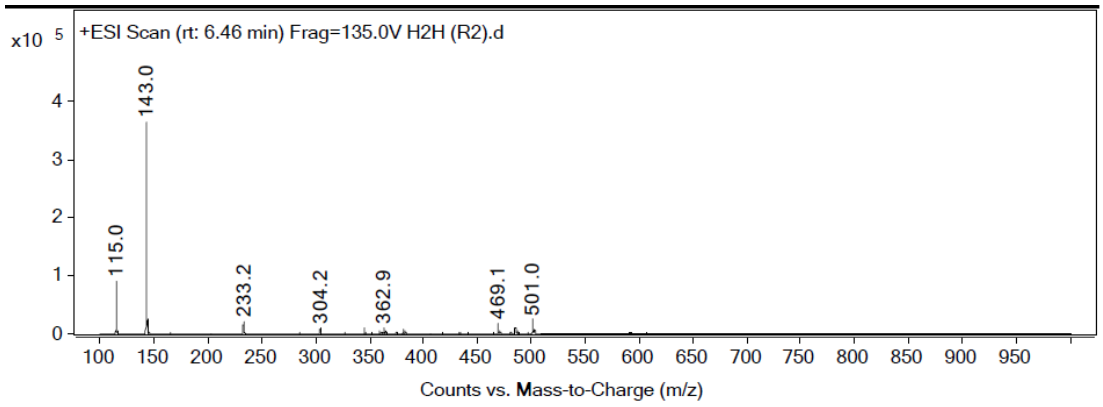


^{13}C NMR spectrum of (*S*)-5-isopropylimidazolidine-2,4-dione (**H2h**)

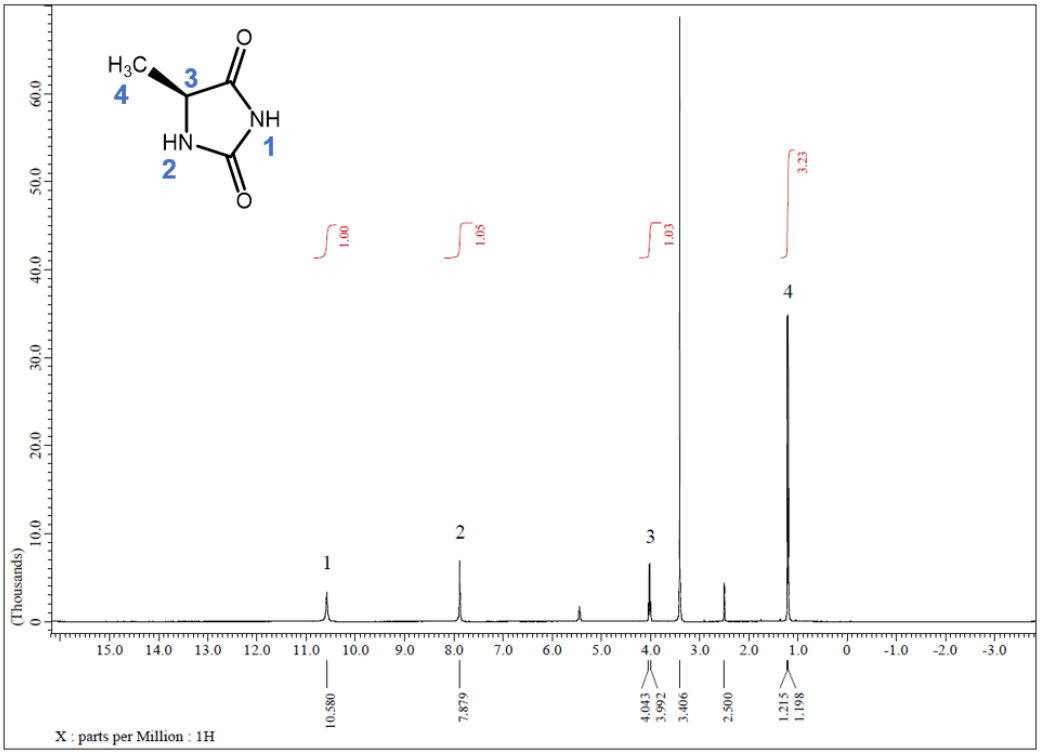


HRMS spectrum of (*S*)-5-isopropylimidazolidine-2,4-dione (**H2h**)

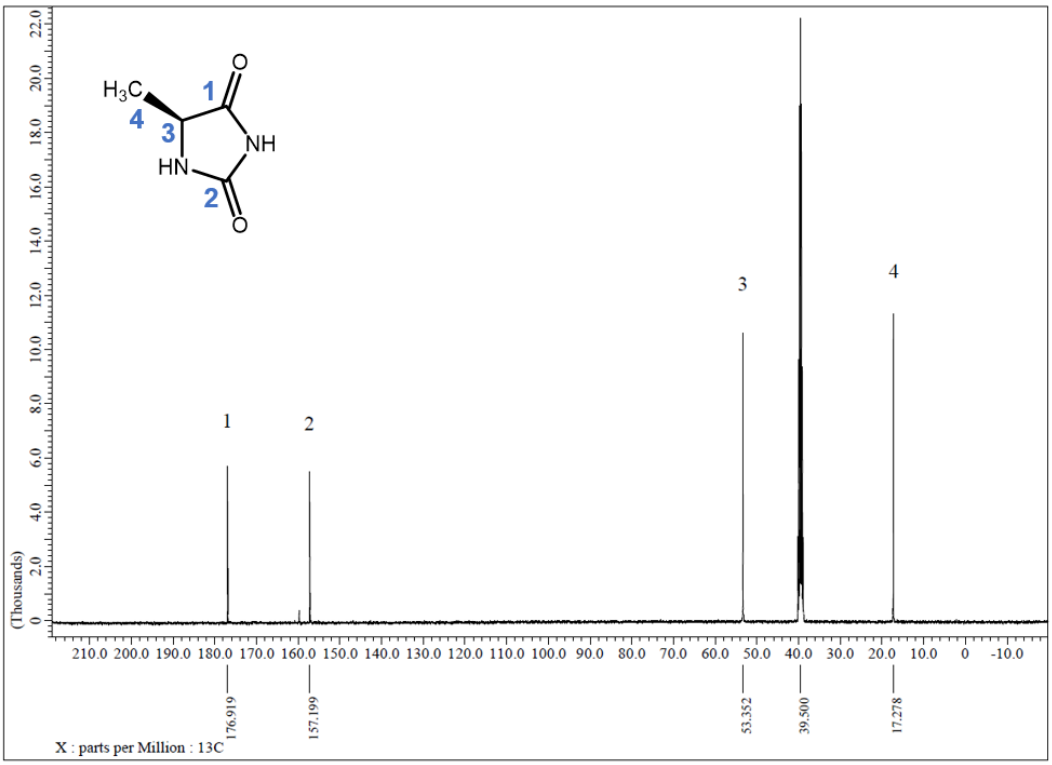




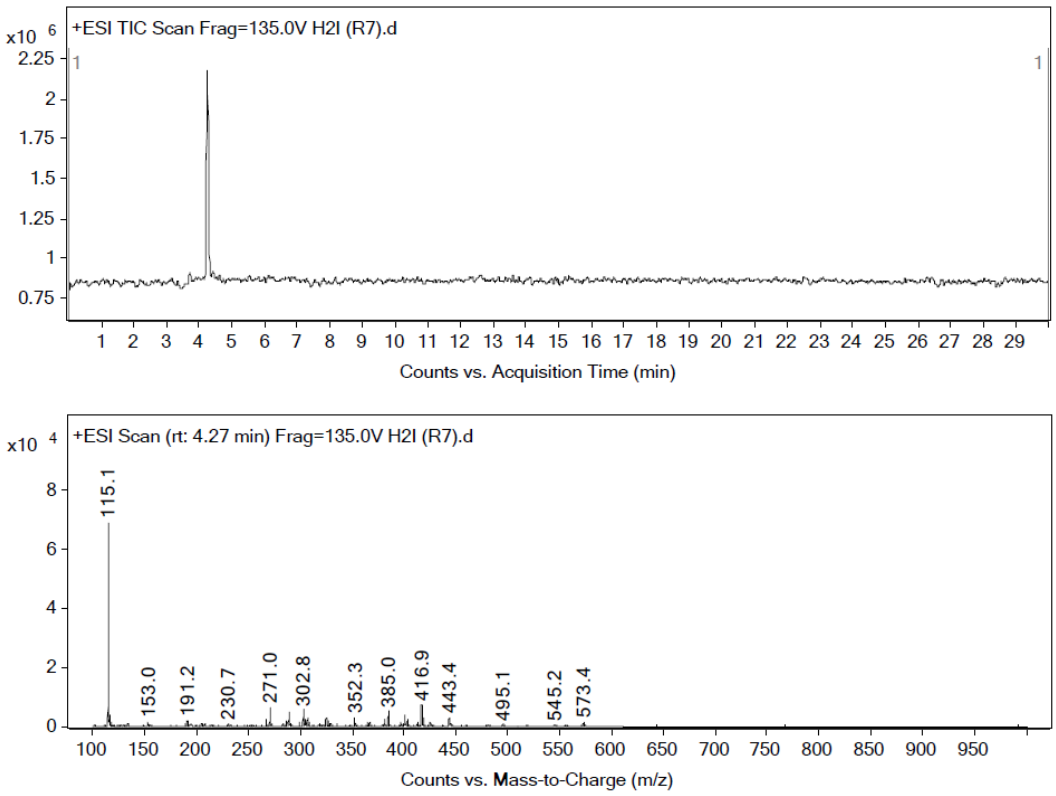
Mass spectrum of (*S*)-5-isopropylimidazolidine-2,4-dione (**H2h**)



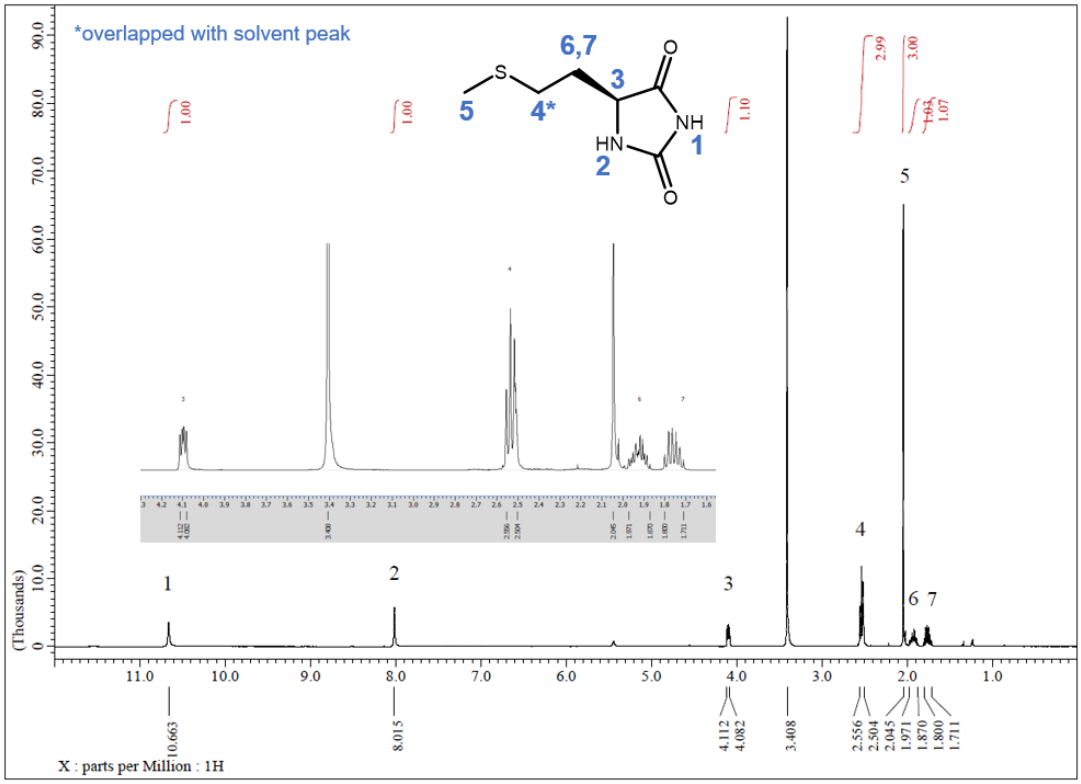
^{1}H NMR spectrum of (*S*)-5-methylimidazolidine-2,4-dione (**H2i**)



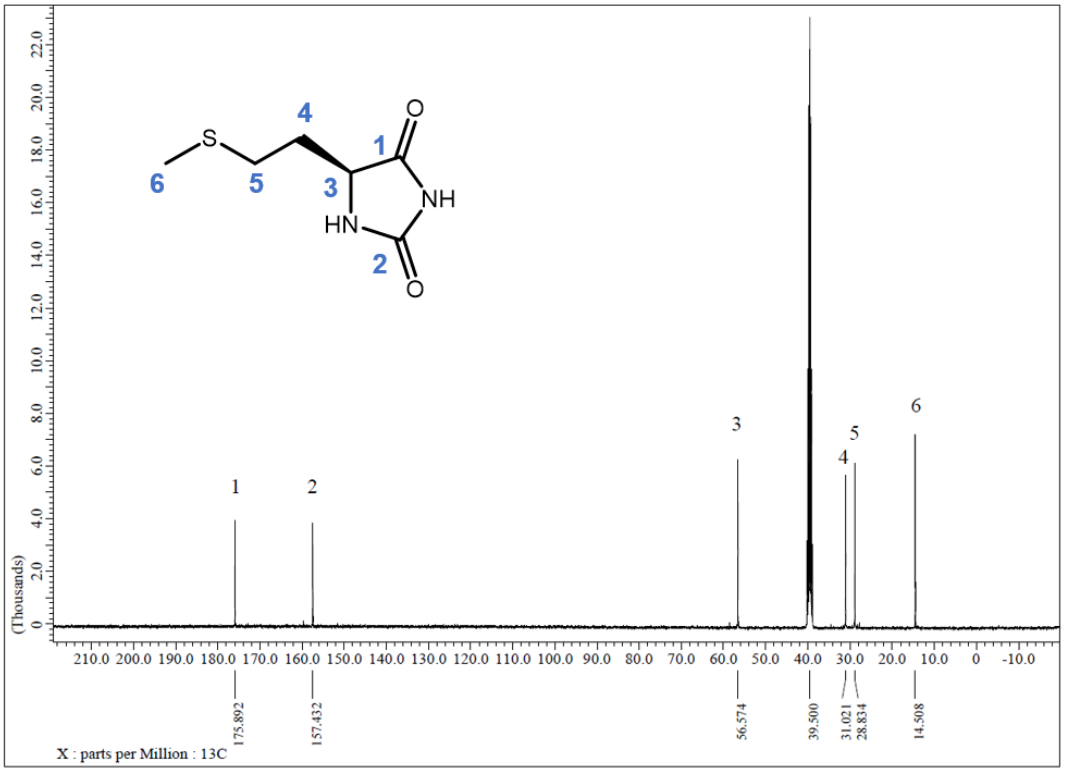
^{13}C NMR spectrum of (*S*)-5-methylimidazolidine-2,4-dione (**H2i**)



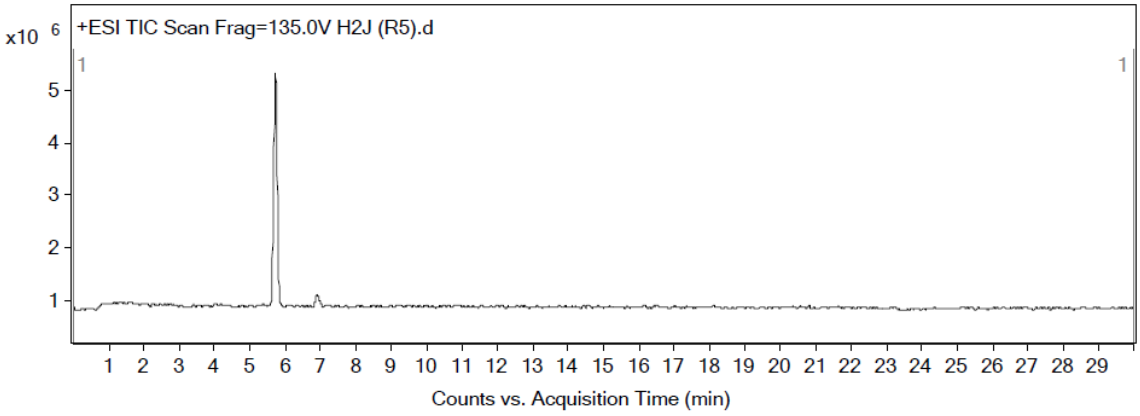
Mass spectrum of (*S*)-5-methylimidazolidine-2,4-dione (**H2i**)

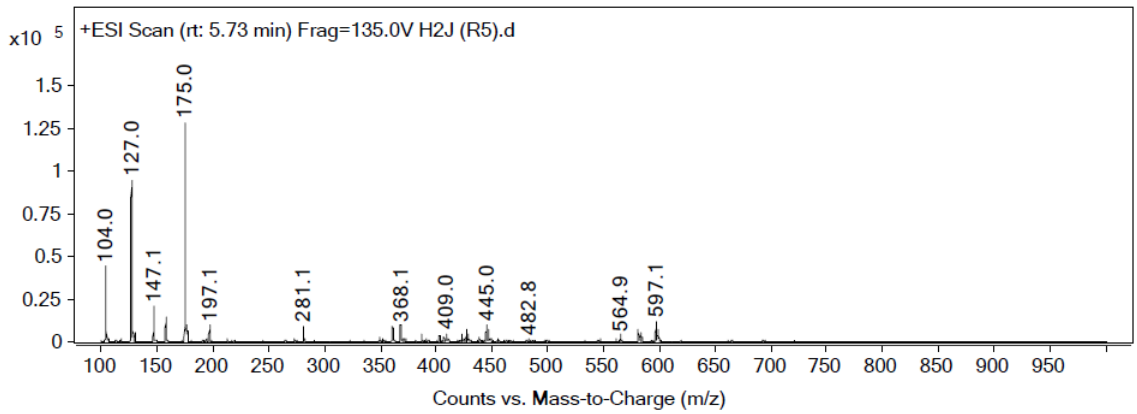


^{1}H NMR spectrum of (*S*)-5-(2-(methylthio)ethyl)imidazolidine-2,4-dione (**H2j**)



^{13}C NMR spectrum of (*S*)-5-(2-(methylthio)ethyl)imidazolidine-2,4-dione (**H2j**)





Mass spectrum of (*S*)-5-(2-(methylthio)ethyl)imidazolidine-2,4-dione (**H2j**)

4. Chromatograms of compounds

```
Acq. Operator   : SYSTEM                        Seq. Line :   1
Acq. Instrument : HPLC 1260                     Location : Vial 1
Injection Date  : 4/7/2024 3:32:06 PM               Inj :   1
                                               Inj Volume : 10.000 µl
Method          : C:\CHEM32\1\DATA\CHANG WEI JIN H2A 2024-07-04 15-30-36\HYDANTOIN.M (
                  Sequence Method)
Last changed    : 4/7/2024 3:30:45 PM by SYSTEM
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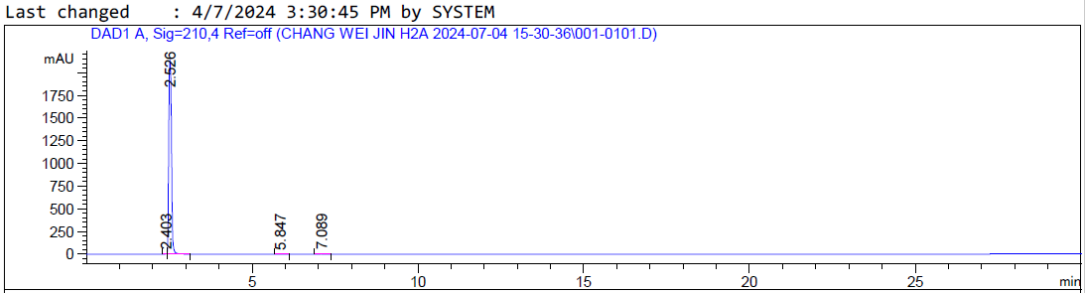
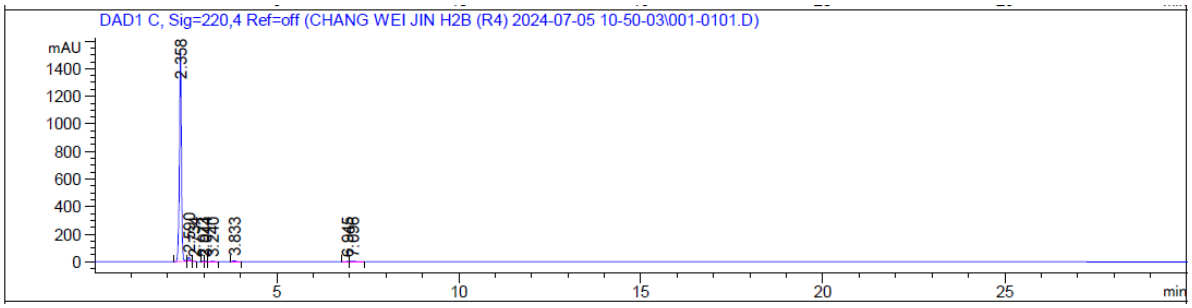


Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.403	BV	0.0480	20.58212	6.67029	0.1715
2	2.526	VB	0.0913	1.19494e4	2133.56299	99.5478
3	5.847	BB	0.1119	7.91664	1.07548	0.0660
4	7.089	BB	0.1312	25.78545	3.03190	0.2148
Totals :				1.20037e4	2144.34066	

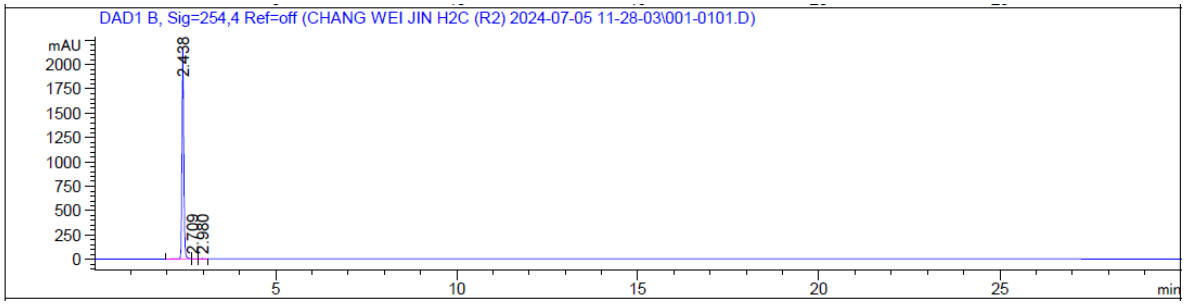
Chromatogram and HPLC integrations of **H2a**.



Signal 3: DAD1 C, Sig=220,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.358	BV	0.0581	5826.54150	1543.05322	95.9929
2	2.590	VB	0.0581	118.87553	31.47240	1.9585
3	2.734	BB	0.0524	5.85003	1.77786	0.0964
4	2.973	BV	0.0584	9.89534	2.59918	0.1630
5	3.044	VV	0.0557	5.55367	1.48331	0.0915
6	3.240	VB	0.1110	14.20711	1.95022	0.2341
7	3.833	BB	0.0822	37.24251	6.98077	0.6136
8	6.945	BV	0.1061	15.36413	2.18387	0.2531
9	7.096	VB	0.1371	36.23384	3.94834	0.5970
Totals :				6069.76367	1595.44917	

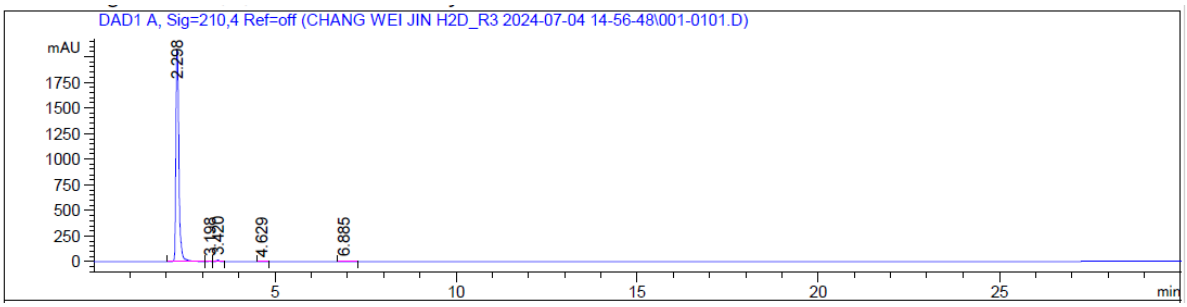
Chromatogram and HPLC integrations of **H2b**.



Signal 2: DAD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.438	BV	0.0583	8261.82910	2176.68433	99.6956
2	2.709	VB	0.0679	9.51642	2.05883	0.1148
3	2.980	BB	0.0703	15.70722	3.37788	0.1895
Totals :				8287.05273	2182.12103	

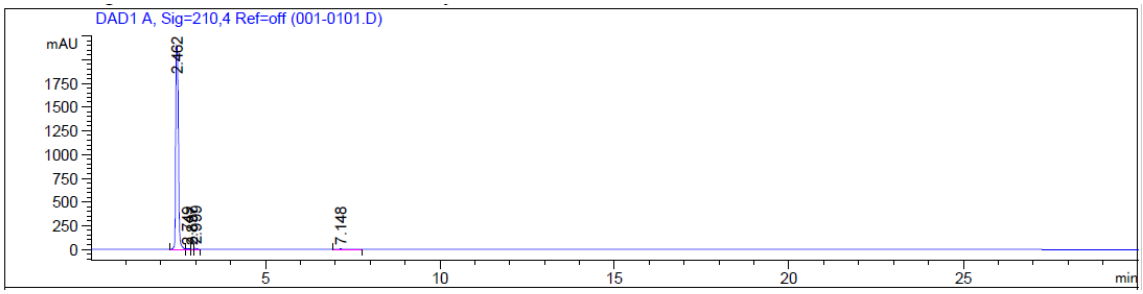
Chromatogram and HPLC integrations of **H2c**.



Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.298	BB	0.0909	1.19004e4	2074.88818	99.1798
2	3.198	BV	0.0828	9.44985	1.65101	0.0788
3	3.420	VB	0.0741	65.08628	13.05972	0.5424
4	4.629	BB	0.0908	6.53019	1.10785	0.0544
5	6.885	BB	0.1238	17.34209	2.15695	0.1445
Totals :				1.19988e4	2092.86372	

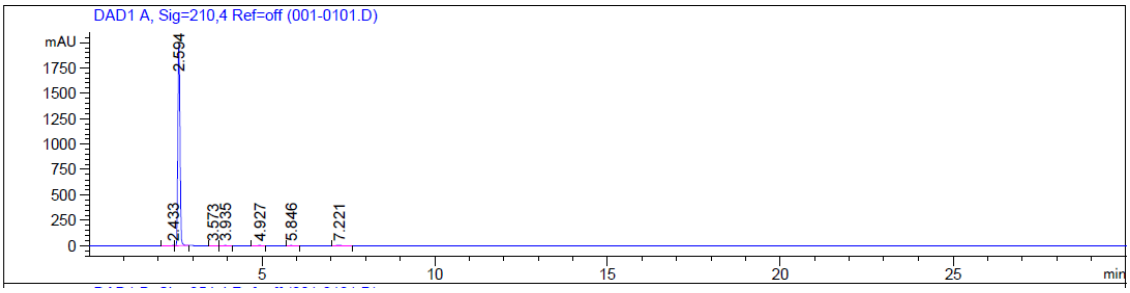
Chromatogram and HPLC integrations of **H2d**.



Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.462	BV	0.0866	1.15082e4	2145.84326	98.9389
2	2.749	VV	0.0870	36.11869	5.93891	0.3105
3	2.897	VV	0.0545	7.37558	1.93413	0.0634
4	2.999	VB	0.0673	49.22173	11.20341	0.4232
5	7.148	BB	0.1362	30.71308	3.43865	0.2640
Totals :				1.16317e4	2168.35836	

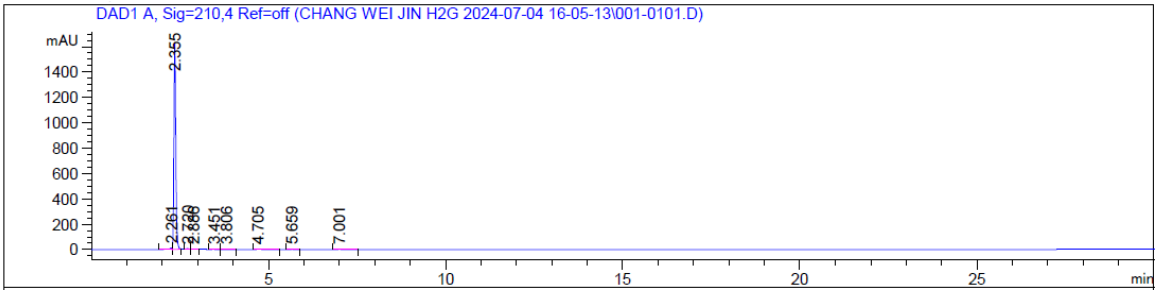
Chromatogram and HPLC integrations of **H2e**.



Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.433	BB	0.0740	15.73734	2.96331	0.1829
2	2.594	BB	0.0675	8455.26465	1993.84448	98.2673
3	3.573	VB	0.1246	21.32673	2.33320	0.2479
4	3.935	BB	0.1093	22.54987	3.01346	0.2621
5	4.927	BB	0.1084	21.42057	2.82658	0.2490
6	5.846	BB	0.1085	16.58592	2.40312	0.1928
7	7.221	BB	0.1300	51.46523	6.12673	0.5981
Totals :				8604.35031	2013.51088	

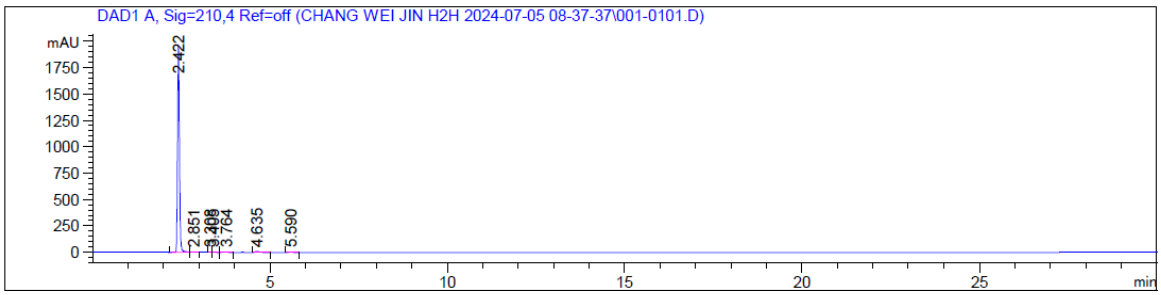
Chromatogram and HPLC integrations of **H2f**.



Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.261	BV	0.0692	55.54348	11.03689	0.8953
2	2.355	VV	0.0588	6015.70117	1639.38928	96.9619
3	2.720	BV	0.0812	16.12285	2.97426	0.2599
4	2.886	VB	0.0849	9.75303	1.65237	0.1572
5	3.451	BB	0.1131	13.81607	1.69416	0.2227
6	3.806	BB	0.1069	15.91802	2.13667	0.2566
7	4.705	BB	0.1313	36.14483	3.65674	0.5826
8	5.659	BB	0.1050	16.47449	2.43191	0.2655
9	7.001	BB	0.1359	24.71371	2.77591	0.3983
Totals :				6204.18764	1667.74819	

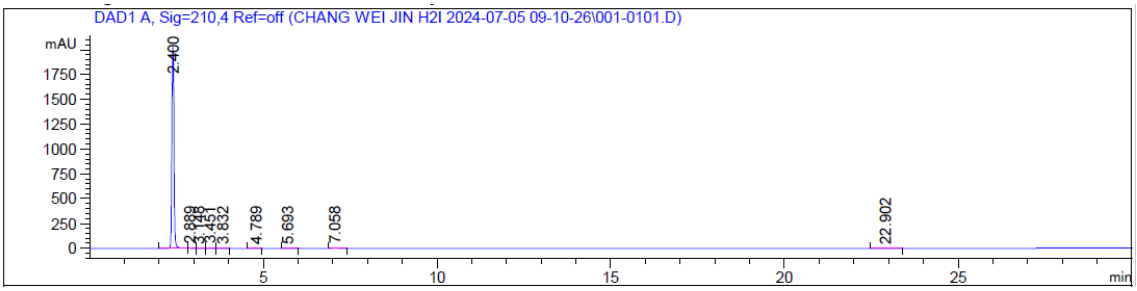
Chromatogram and HPLC integrations of **H2g**.



Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.422	BV	0.0646	7871.98828	1968.86609	98.3380
2	2.851	VB	0.1019	23.49626	3.19237	0.2935
3	3.308	BV	0.0706	8.47418	1.81192	0.1059
4	3.405	VB	0.0874	11.54817	1.88816	0.1443
5	3.764	BB	0.1185	15.21869	1.80299	0.1901
6	4.635	BB	0.1022	61.07473	8.66549	0.7630
7	5.590	BB	0.1048	13.23519	1.96022	0.1653
Totals :				8005.03551	1988.18724	

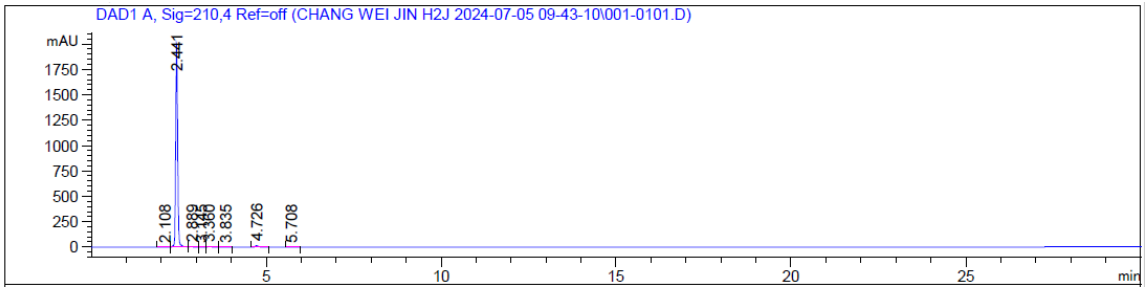
Chromatogram and HPLC integrations of **H2h**.



Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.400	BV	0.0699	8741.37305	2044.75537	97.5539
2	2.889	VV	0.1088	30.63241	3.76871	0.3419
3	3.148	VV	0.1633	28.19417	2.23343	0.3146
4	3.451	VB	0.1410	32.44205	3.07816	0.3621
5	3.832	BB	0.1322	16.16709	1.71199	0.1804
6	4.789	BB	0.1154	16.98713	2.07756	0.1896
7	5.693	BB	0.1082	14.48612	2.05597	0.1617
8	7.058	BB	0.1308	49.59040	5.97726	0.5534
9	22.902	BB	0.3420	30.68933	1.32613	0.3425
Totals :				8960.56175	2066.98460	

Chromatogram and HPLC integrations of **H2i**.



Signal 1: DAD1 A, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.108	BV	0.2010	21.04398	1.64822	0.2402
2	2.441	VV	0.0691	8506.89258	2020.25354	97.0935
3	2.889	VV	0.1384	45.47044	4.26387	0.5190
4	3.145	VV	0.1424	23.84449	2.23623	0.2721
5	3.360	VB	0.1439	49.40802	4.57715	0.5639
6	3.835	BB	0.1245	15.77759	1.79521	0.1801
7	4.726	BB	0.1003	84.84641	12.33343	0.9684
8	5.708	BB	0.1068	14.26201	2.05921	0.1628
Totals :				8761.54551	2049.16687	

Chromatogram and HPLC integrations of **H2j**.

5. References

1. Chin, E.-Z.; Tan, S.-P.; Liew, S.-Y.; Kurz, T. *Malaysian J. Chem.* **2021,** *23,* 19–25. doi:10.1055/a-1330-4198
2. Wang, M. X.; Lin, S. J. *J. Org. Chem.* **2002,** *67,* 6542–6545. doi:10.1021/jo0256282
3. Harada, K. *Nature* **1963,** *4912,* 1201. doi:10.1038/2001201a0