

Supplementary Material

Quantification of grapiprant and its stability testing under changing environmental conditions by TLC-densitometry

Paweł Gumułka ^{1,2}, Monika Tarsa ³, Monika Dąbrowska ¹ and Małgorzata Starek ^{1,*}

¹ Department of Inorganic and Analytical Chemistry, Faculty of Pharmacy, Jagiellonian University Medical College, 9 Medyczna St, 30-688, Kraków, Poland

² Doctorial School of Medical and Health Sciences, Jagiellonian University Medical College, 16 Łazarza St, 31-530, Kraków, Poland

³ Department of Organic Chemistry, Faculty of Pharmacy, Jagiellonian University Medical College, 9 Medyczna St, 30-688, Kraków, Poland

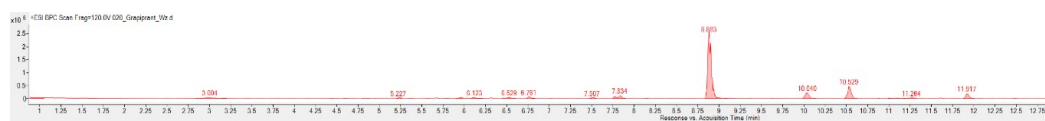
Table S1. Percentage of grapiprant content during degradation at room temperature.

| Environment | Incubation time [h] | | | | | | | | | | | |
|------------------|---------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | 0 | 24 | 48 | 96 | 192 | 384 | 528 | 720 | 888 | 1104 | 1272 | 1512 |
| 1MHCl | 100.00 | 93.69 | 83.21 | 78.74 | 77.64 | 75.21 | 72.95 | 72.68 | 68.53 | 66.26 | 56.76 | 38.91 |
| 0.5MHCl | 100.00 | 73.44 | 72.69 | 68.20 | 64.94 | 64.90 | 64.47 | 64.42 | 62.16 | 61.20 | 55.04 | 46.44 |
| 0.1MHCl | 100.00 | 88.97 | 83.79 | 81.86 | 81.84 | 80.50 | 78.83 | 76.84 | 69.79 | 67.13 | 58.51 | 54.70 |
| H ₂ O | 100.00 | 82.35 | 79.35 | 75.56 | 66.00 | 62.49 | 53.76 | 52.95 | 52.40 | 48.04 | 37.84 | 37.31 |
| 0.1MNaOH | 100.00 | 97.62 | 91.57 | 83.43 | 76.71 | 75.36 | 66.18 | 63.37 | 54.77 | 49.20 | 45.22 | 42.20 |
| 0.5MNaOH | 100.00 | 99.19 | 97.02 | 88.99 | 74.01 | 68.41 | 60.26 | 59.92 | 54.05 | 47.49 | 46.22 | 42.12 |
| 1MNaOH | 100.00 | 92.77 | 85.64 | 74.06 | 66.64 | 66.24 | 63.78 | 57.54 | 56.94 | 52.43 | 49.57 | 40.94 |
| buffer pH 2 | 100.00 | 86.30 | 83.55 | 71.67 | 68.04 | 63.99 | 58.67 | 52.18 | 50.15 | 42.56 | 40.87 | 31.30 |
| buffer pH 7 | 100.00 | 78.90 | 78.06 | 79.57 | 79.49 | 71.62 | 66.43 | 64.50 | 58.09 | 55.36 | 40.10 | 32.17 |
| buffer pH 8 | 100.00 | 86.65 | 84.30 | 82.15 | 79.95 | 76.82 | 66.67 | 64.60 | 63.03 | 60.86 | 50.61 | 42.13 |

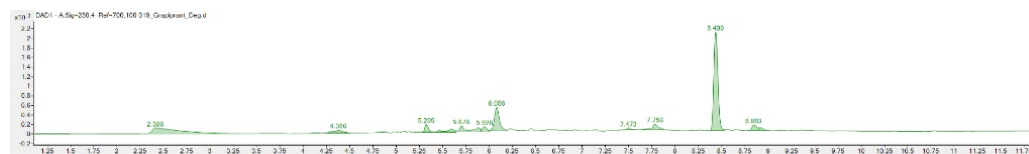
Table S2. Percentage of grapiprant content during degradation at 70°C.

| Environment | Incubation time [h] | | | | | | | | | | | | |
|------------------|---------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| | 0 | 1 | 2 | 4 | 24 | 72 | 96 | 144 | 192 | 312 | 384 | 504 | 696 |
| 1MHCl | 100.00 | 96.39 | 92.50 | 84.09 | 80.24 | 68.06 | 60.94 | 56.17 | 55.07 | 41.09 | 7.04 | 3.32 | 0.00 |
| 0.5M HCl | 100.00 | 93.08 | 84.81 | 75.69 | 65.88 | 58.42 | 52.52 | 43.39 | 38.25 | 25.35 | 8.51 | 3.76 | 0.00 |
| 0.1M HCl | 100.00 | 91.10 | 87.28 | 80.95 | 72.99 | 44.83 | 32.96 | 28.93 | 13.82 | 2.50 | 0.00 | 0.00 | 0.00 |
| H ₂ O | 100.00 | 99.09 | 96.82 | 78.67 | 75.19 | 75.18 | 73.25 | 66.60 | 48.03 | 38.97 | 25.65 | 11.64 | 5.31 |
| 0.1MNaOH | 100.00 | 99.41 | 98.09 | 86.70 | 83.53 | 81.56 | 73.95 | 65.19 | 59.53 | 57.05 | 50.68 | 47.26 | 43.74 |
| 0.5MNaOH | 100.00 | 99.80 | 95.42 | 88.18 | 87.79 | 82.84 | 76.40 | 65.21 | 61.47 | 60.66 | 41.83 | 33.43 | 28.87 |
| 1MNaOH | 100.00 | 97.18 | 94.44 | 88.25 | 84.98 | 78.77 | 75.50 | 73.39 | 71.08 | 69.73 | 41.89 | 31.59 | 30.27 |
| buffer pH 2 | 100.00 | 97.73 | 91.39 | 76.71 | 73.07 | 62.33 | 33.28 | 21.28 | 16.13 | 4.04 | 0.00 | 0.00 | 0.00 |
| buffer pH 7 | 100.00 | 91.79 | 85.50 | 78.65 | 74.08 | 68.63 | 65.54 | 65.13 | 55.25 | 48.08 | 24.16 | 15.95 | 3.56 |
| buffer pH 8 | 100.00 | 99.43 | 94.35 | 86.92 | 83.38 | 75.62 | 74.85 | 70.55 | 61.92 | 48.52 | 32.71 | 29.84 | 14.61 |

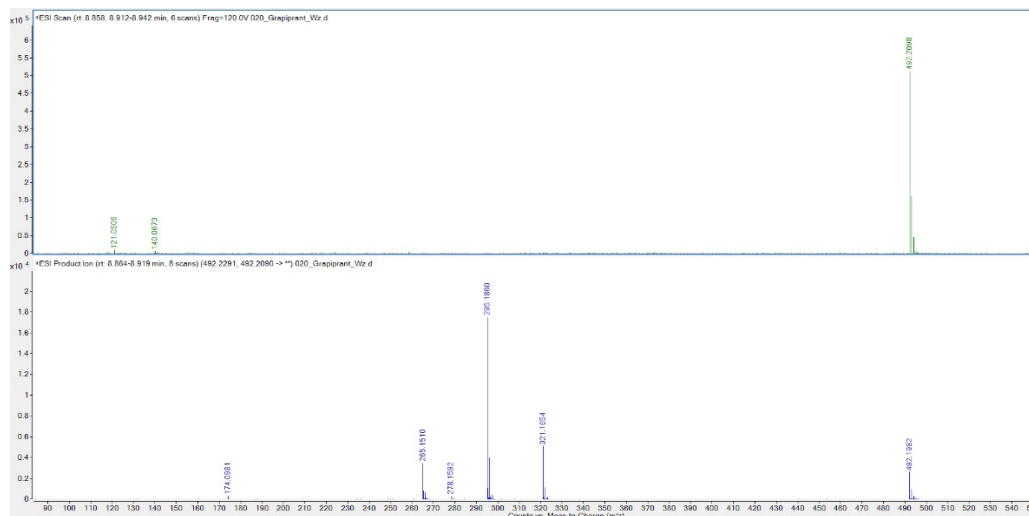
Grapiprant Standard Substance:



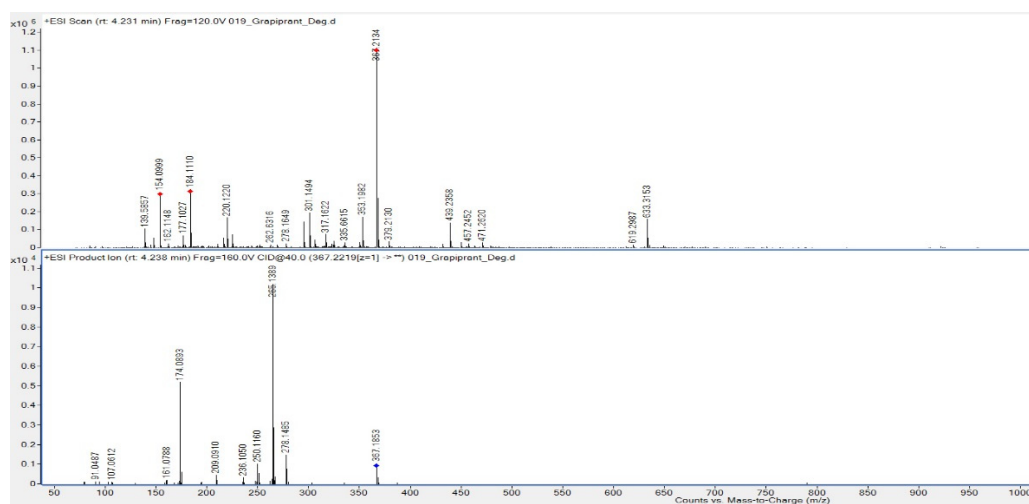
Grapiprant – acidic degradation:



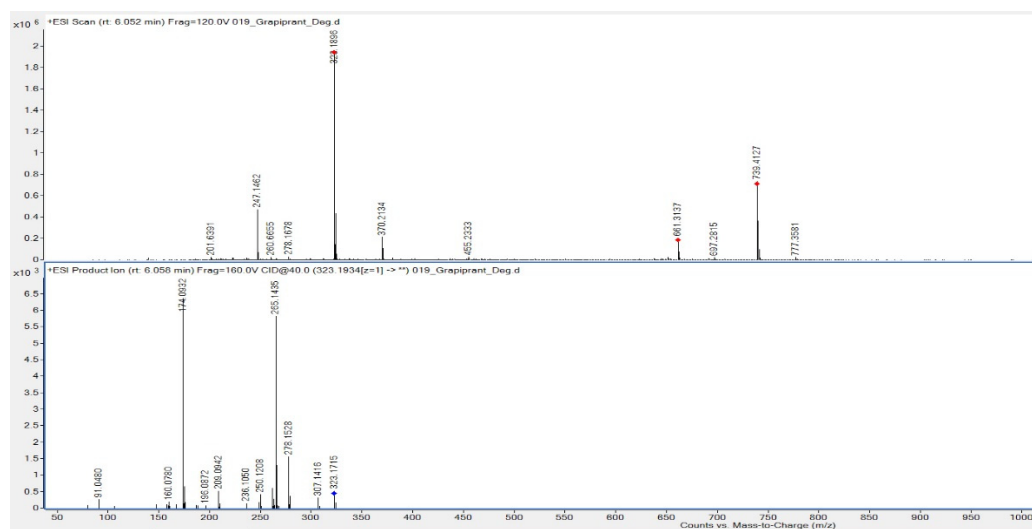
$t_r = 8.883$ min m/z 492:



$t_r = 4.36$ min m/z 367:



$t_r = 6.06$ min m/z 323:



$t_r = 2.4$ min m/z 295:

