# *N*-Demethyl-*N*-nitrosolevofloxacin

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# 1. Spectral properties

## 1.1 NMR

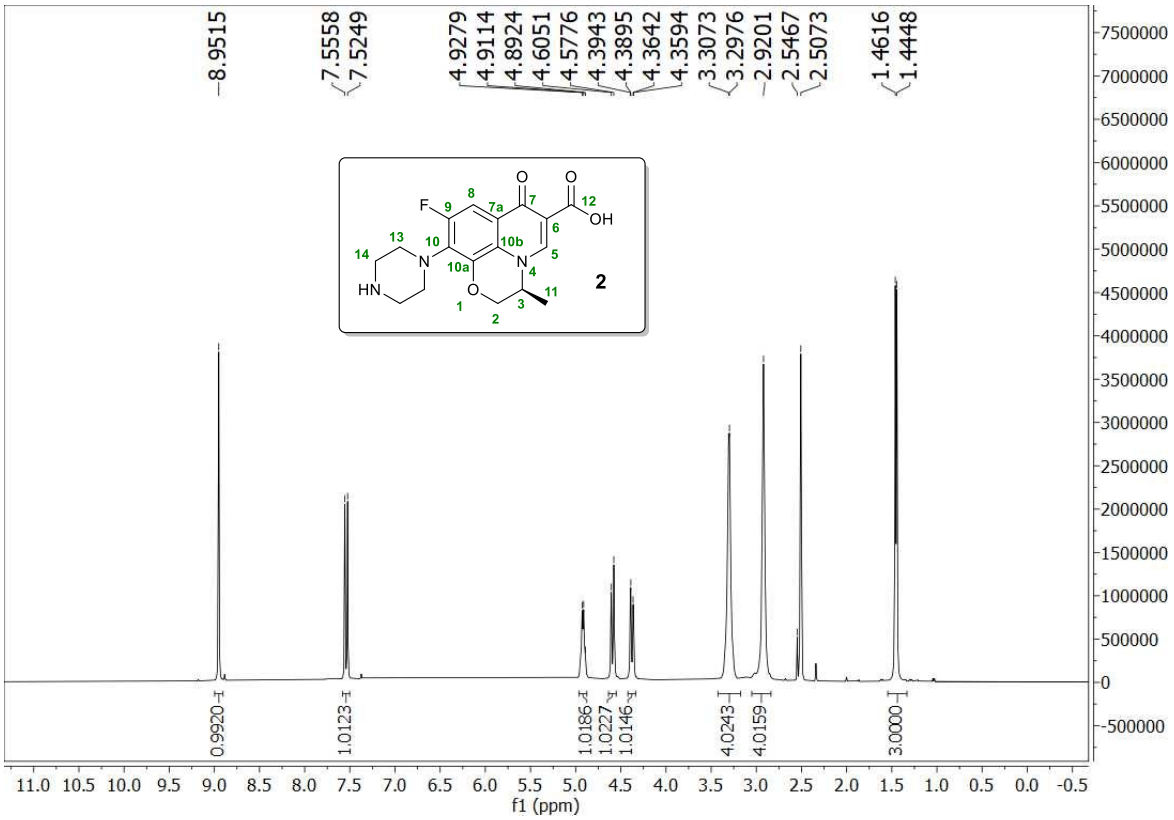



**Figure S1** - $^{1}$H NMR, compound **2** (DMSO-$d_6$, 400.2 MHz, 298K).

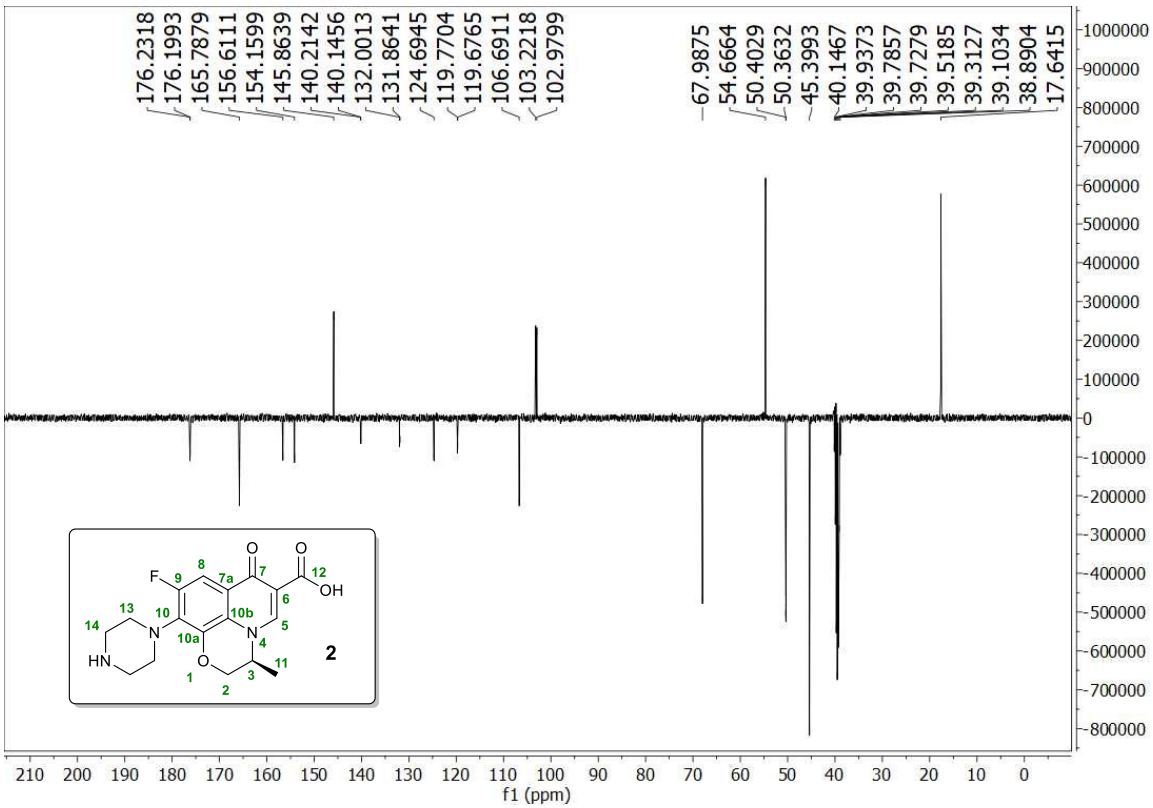



**Figure S2** – $^{13}$C APT NMR, compound **2** (DMSO-$d_6$, 100.6 MHz, 298K).

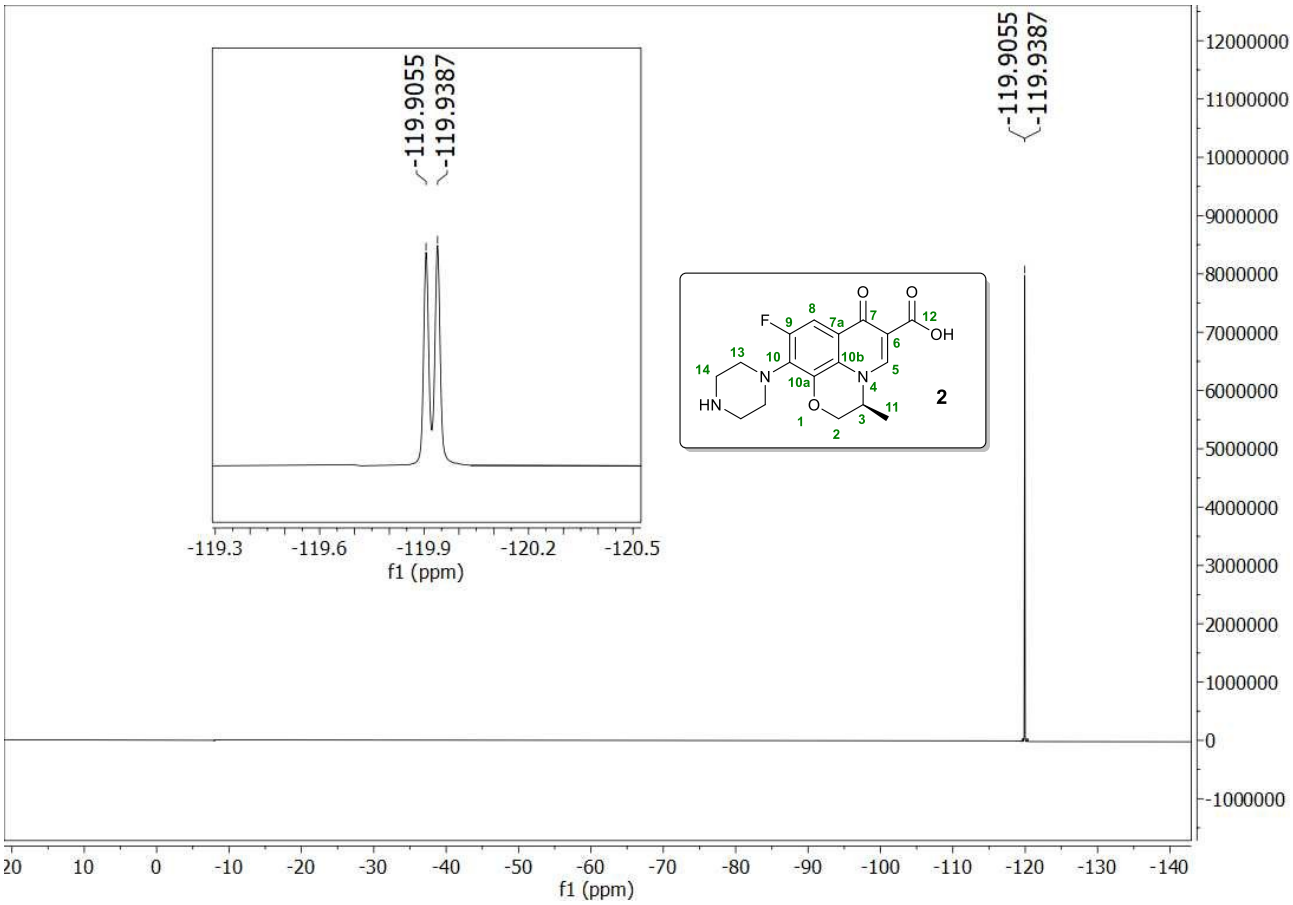



**Figure S3** – $^{19}F$ NMR, compound **2** (DMSO-$d_6$, 376.6 MHz, 298K).

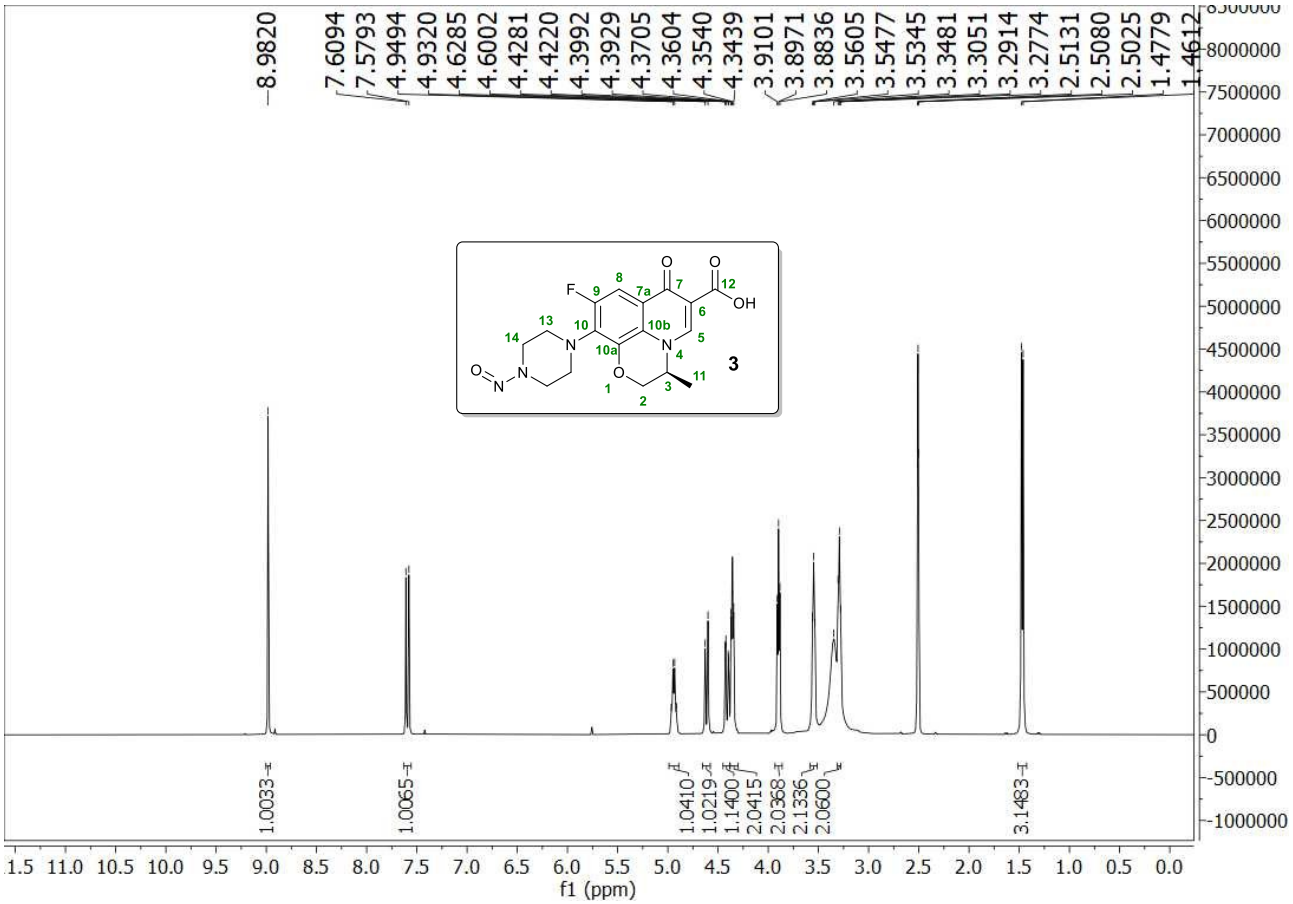



**Figure S4** - $^{1}$H NMR, compound **3** (DMSO-$d_6$, 400.2 MHz, 298K).

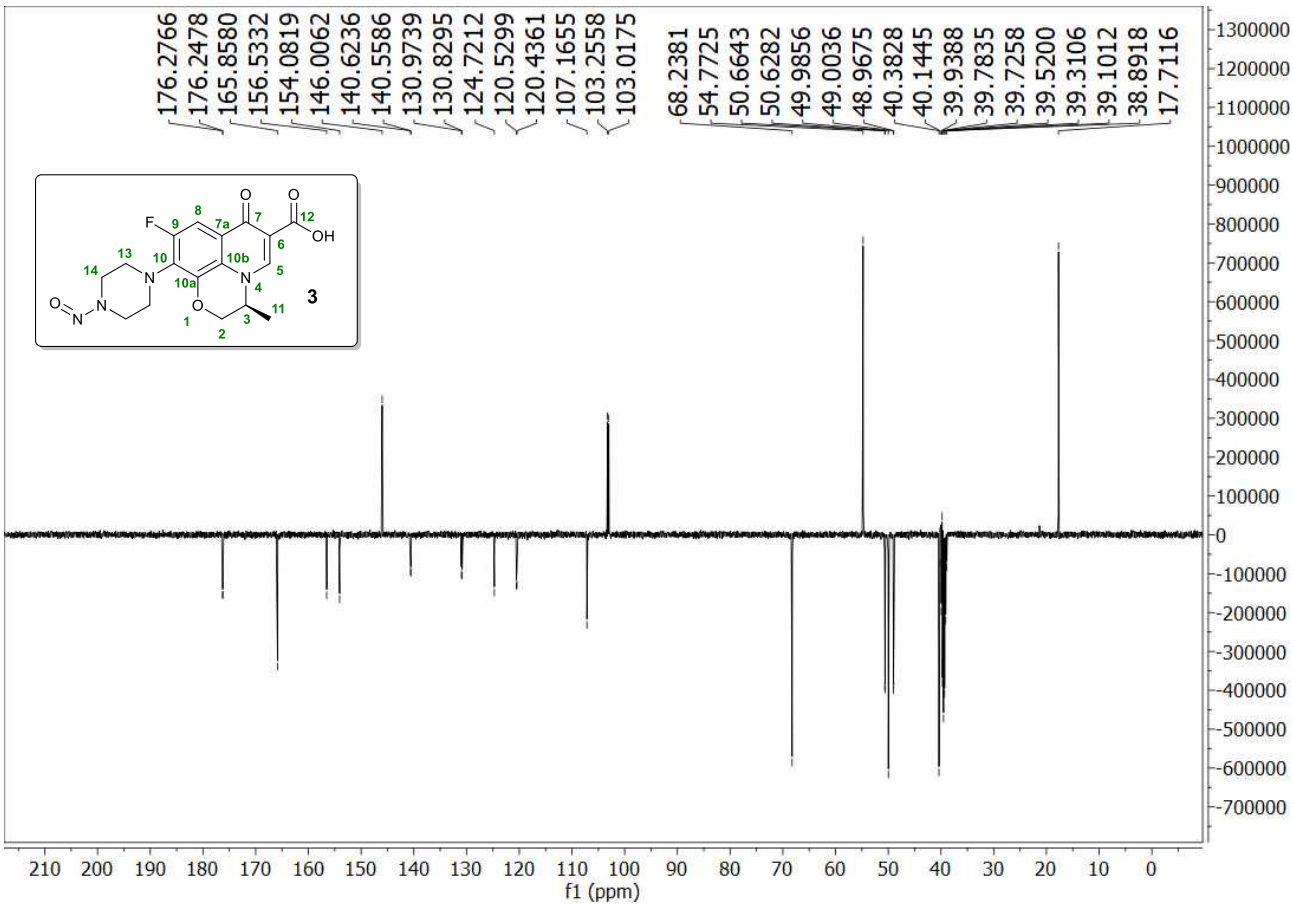



**Figure S5** – $^{13}$C APT NMR, compound **3** (DMSO-$d_6$, 100.6 MHz, 298K).

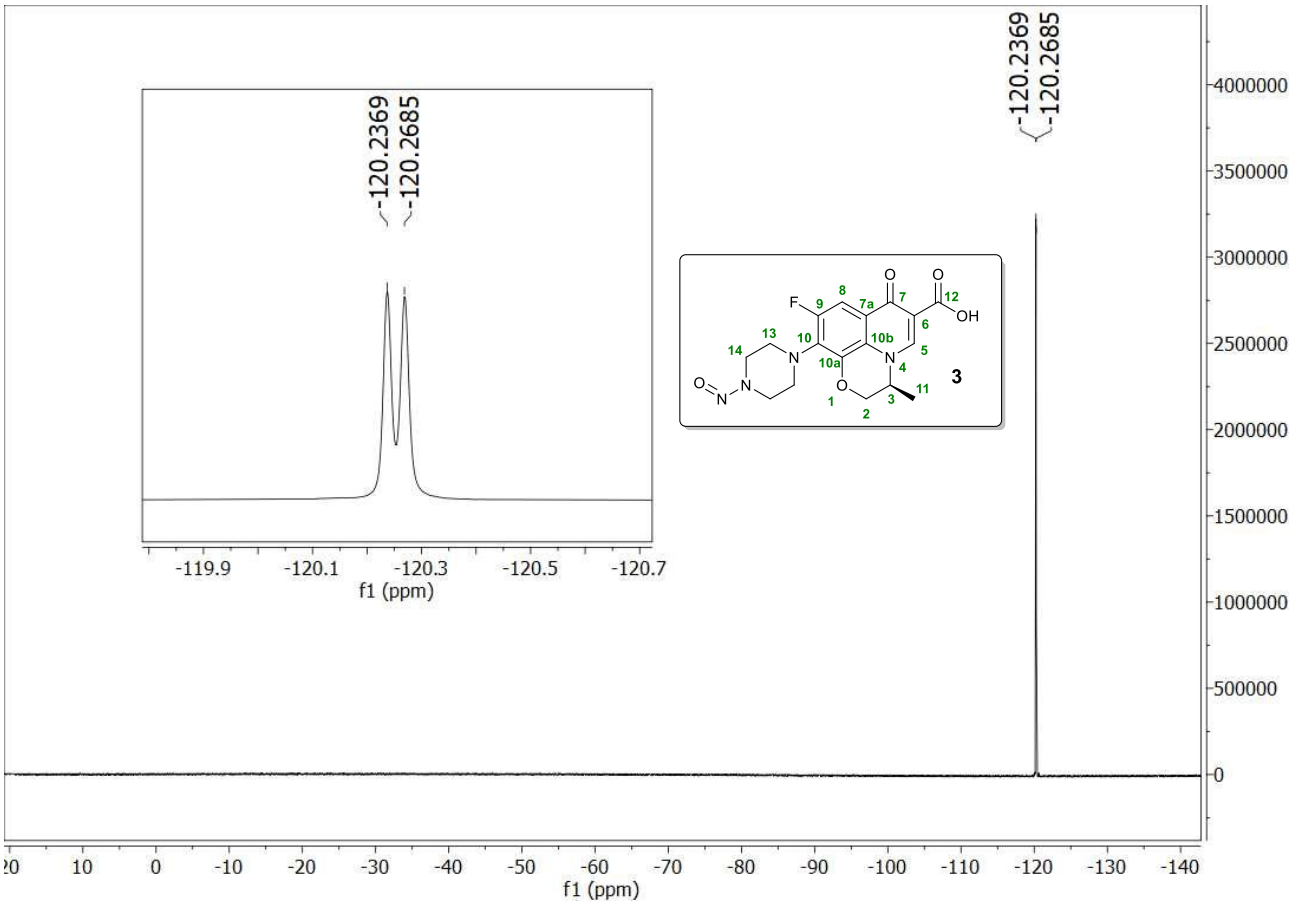



**Figure S6** – $^{19}F$ NMR, compound **3** (DMSO-$d_6$, 376.6 MHz, 298K).

## 1.2 HRMS

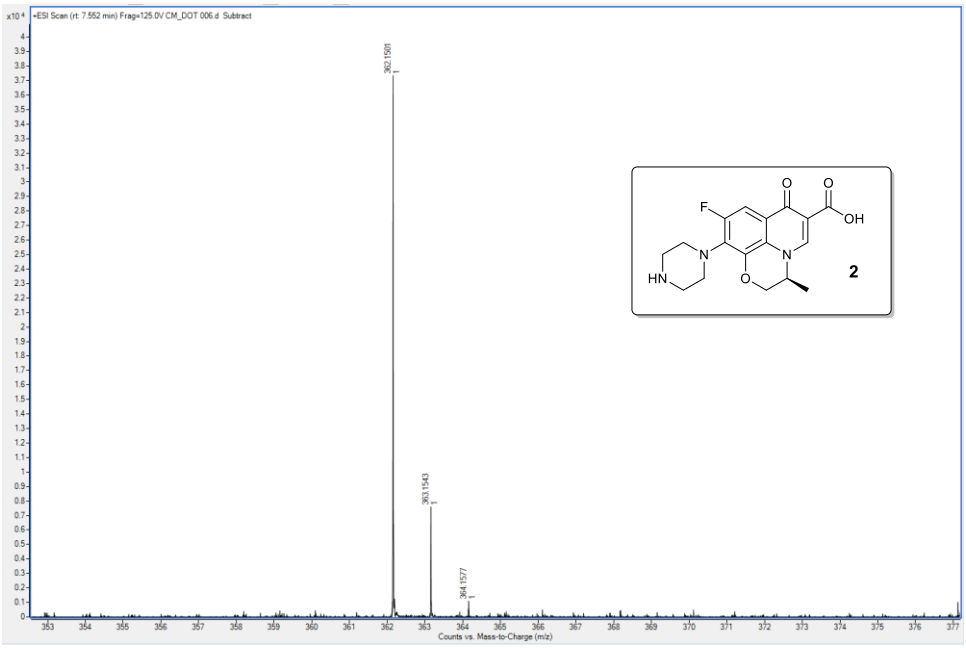



**Figure S7** – HRMS spectrum, compound **2**

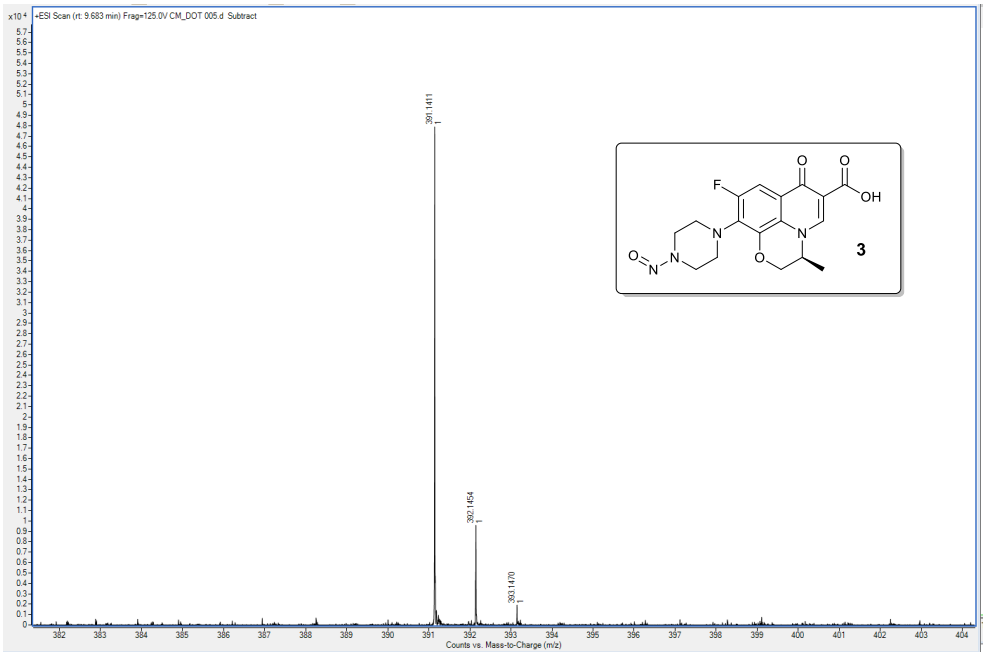



**Figure S8** – HRMS spectrum, compound **3**

## 1.3 IR

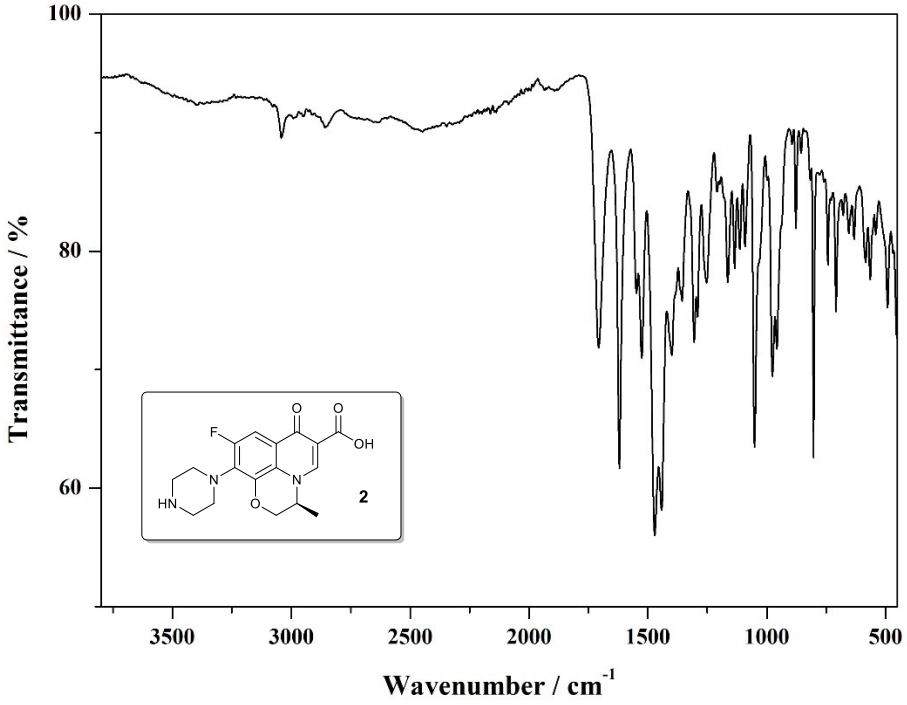



**Figure S9** – IR spectrum, compound **2**

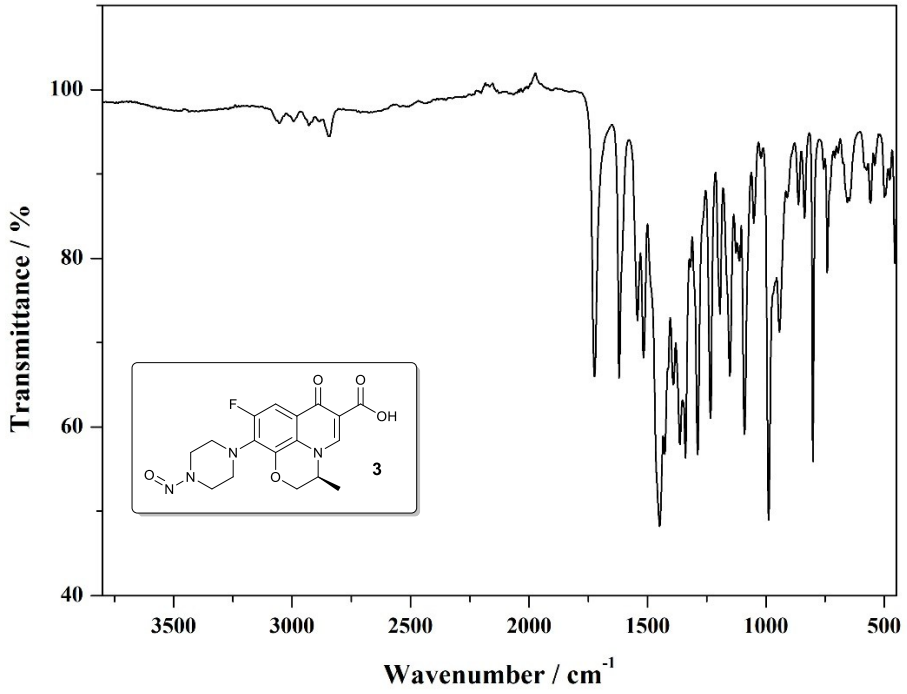
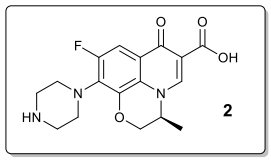



**Figure S10** – IR spectrum, compound **3**

## 1.4 Electronic Spectra



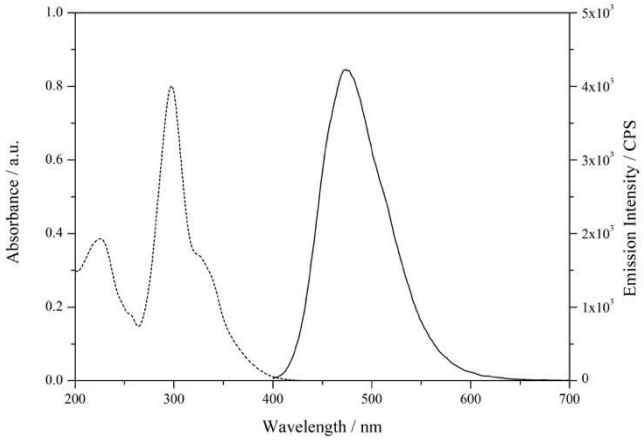
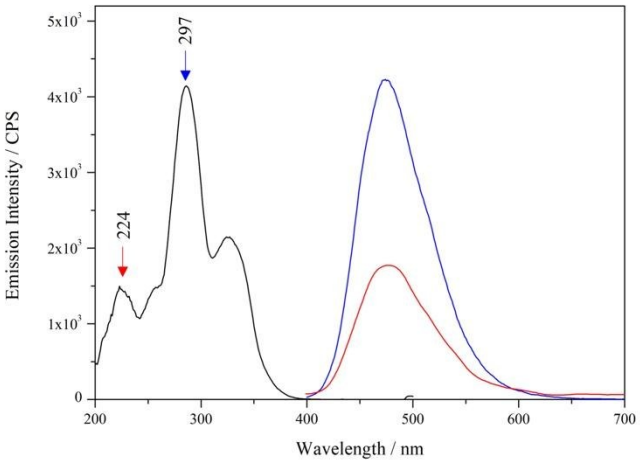




**Figure S11** - Compound **2**, absorption (dashed line) and emission (solid line) spectra. Emission spectrum is obtained upon excitation at 297 nm.



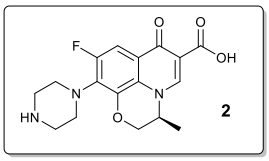




**Figure S12** – Compound **2**, excitation (black line) and emission spectra obtained upon excitation at 224 nm (red curve) and 297 nm (blue curve)

# 2. Crystal structure

## 2.1 Experimental details of XRD studies

X-Ray powder diffraction patterns were collected on a Bruker D8 Advance diffractometer equipped with a Cu Kα (λ = 1.5418 Å) radiation and a LynxEye XE-T silicon strip detector. During the screening by X-ray powder diffraction, small crystals were found in the sample recrystallized with 1-butanol. Single-crystal diffraction data were collected on such crystals using an Oxford Xcalibur CCD area detector diffractometer with graphite monochromator and Mo Kα (λ = 0.71069 Å) radiation. Other parameters and results of structure determination are shown in Table S1. Figure S13 shows the packing diagram of **3α** with the layered structure and the intramolecular hydrogen bond.

**Table S1.** Crystal data, data collection and structure refinement details of structures **3α** and **3β**.

| **Compound** | **3α** | **3β** |
|---|---|---|
| Empirical formula | $C_{17}H_{17}FN_4O_5$ | $C_{17}H_{17}FN_4O_5$ |
| Formula weight | 376.342 g $mol^{-1}$ | 376.342 g $mol^{-1}$ |
| Temperature | 298(2)K | 298(2)K |
| Wavelength | 1.54059Å | 0.71073Å |
| Crystal system | Monoclinic | Monoclinic |
| Space group | $P2_1$ (4) | $C2$ (5) |
| Unit cell dimension | $a$ = 13.145(6)Å<br>$b$ = 6.949(2)Å<br>$c$ = 9.890(3)Å<br>$\beta$ = 111.64(2)° | $a$ = 17.515(9)Å<br>$b$ = 7.116(3)Å<br>$c$ = 17.524(8)Å<br>$\beta$ = 118.31(6)° |
| Volume | 841.0(5)$Å^3$ | 1922.7(9)$Å^3$ |
| Z | 2 | 4 |
| Density (calculated) | 1.486g $cm^{-3}$ | 1.300g $cm^{-3}$ |
| Absorption coefficient | 1.011 $mm^{-1}$ | 0.104$mm^{-1}$ |
| Crystal habit | Powder | Acicular |
| Theta range for data collection | 1.00°-45.00° | 3.15°-20.80° |
| Independent reflections | - | 1762 [$R_{int}$ = 0.067] |
| Completeness to theta = 20.8° | - | 99.55% |
| Goodness-of-fit- on $F^2$ | - | 1.1224 |
| Final R indices | Rietveld:<br>R = 0.07657<br>$R_{wp}$ = 0.11371 | [I > 2σ (I)]:<br>R1 = 0.1220<br>wR2 = 0.2957<br><br>All data:<br>R1 = 0.1754<br>wR2 = 0.3496 |

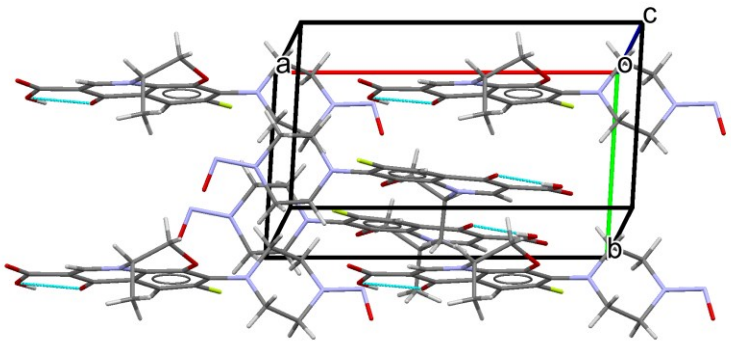



**Figure S13** – Packing diagram of compound **3α.**

## 2.2 Peak list of the solved crystal structures

**Table S2 –** d-spacings and squared structure factors for structure **3α**.

| d-spacing | $F^2$ | d-spacing | $F^2$ | d-spacing | $F^2$ | d-spacing | $F^2$ | d-spacing | $F^2$ |
|---|---|---|---|---|---|---|---|---|---|
| 12.2368 | 1086.32 | 3.12852 | 58.2942 | 2.42891 | 371.685 | 2.10988 | 1444.7 | 1.91193 | 24.1231 |
| 9.20669 | 6466.03 | 3.0689 | 163.99 | 2.42525 | 12.5079 | 2.10335 | 430.114 | 1.90795 | 911.922 |
| 9.13185 | 599.536 | 3.0592 | 262.834 | 2.41785 | 1937.97 | 2.10062 | 104.812 | 1.89825 | 350.554 |
| 6.32963 | 496.99 | 3.04579 | 279.911 | 2.41265 | 98.1333 | 2.09759 | 138.808 | 1.89752 | 108.051 |
| 6.25703 | 1073.53 | 3.04395 | 2323.68 | 2.39601 | 22.0613 | 2.09693 | 28.0692 | 1.89493 | 130.948 |
| 6.1184 | 37.5182 | 3.0376 | 591.715 | 2.38672 | 274.336 | 2.08953 | 130.306 | 1.89325 | 799.189 |
| 6.04264 | 769.524 | 3.02132 | 3123.96 | 2.38451 | 0.018743 | 2.08892 | 971.221 | 1.89289 | 280.373 |
| 5.54646 | 2167.11 | 2.98723 | 459.93 | 2.38204 | 466.583 | 2.08568 | 60.6381 | 1.89055 | 194.704 |
| 5.52996 | 2080.88 | 2.96928 | 883.682 | 2.37811 | 833.777 | 2.08285 | 1058.59 | 1.88859 | 5.82108 |
| 4.94466 | 2761.38 | 2.96028 | 62.2439 | 2.3397 | 95.0777 | 2.07666 | 179.625 | 1.87763 | 220.968 |
| 4.6794 | 1235.8 | 2.95275 | 1014.6 | 2.3293 | 1941.96 | 2.0755 | 205.313 | 1.86918 | 194.58 |
| 4.64984 | 545.019 | 2.88018 | 1241.5 | 2.32492 | 377.796 | 2.06915 | 354.917 | 1.86321 | 776.718 |
| 4.60335 | 3946.74 | 2.85274 | 433.736 | 2.30838 | 18.8215 | 2.06716 | 2878.82 | 1.86162 | 241.345 |
| 4.59209 | 118.457 | 2.84284 | 68.8541 | 2.30167 | 848.638 | 2.06572 | 176.068 | 1.86097 | 111.146 |
| 4.56592 | 3088.74 | 2.80732 | 321.581 | 2.30014 | 84.6926 | 2.05041 | 66.0596 | 1.85725 | 478.532 |
| 4.40743 | 872.444 | 2.79989 | 492.036 | 2.29605 | 192.876 | 2.04624 | 563.463 | 1.85427 | 0.744905 |
| 4.36645 | 671.092 | 2.78818 | 507.82 | 2.29288 | 62.0804 | 2.03947 | 581.681 | 1.85338 | 68.5875 |
| 4.07893 | 303.962 | 2.77323 | 50.6348 | 2.28958 | 113.357 | 2.03243 | 227.209 | 1.84882 | 13.3629 |
| 4.02881 | 142.978 | 2.76498 | 43.724 | 2.28357 | 1046.63 | 2.01887 | 2342.88 | 1.84858 | 282.479 |
| 3.86805 | 1213.43 | 2.7492 | 63.119 | 2.28296 | 1907.5 | 2.01441 | 545.157 | 1.84799 | 802.846 |
| 3.83767 | 1116.48 | 2.7286 | 116.981 | 2.27918 | 796.783 | 2.01421 | 140.42 | 1.84669 | 565.04 |
| 3.82387 | 521.361 | 2.71943 | 187.13 | 2.27592 | 934.038 | 2.01315 | 19.0638 | 1.84539 | 229.705 |
| 3.81591 | 4804.71 | 2.71879 | 98.2087 | 2.25542 | 133.829 | 2.00083 | 330.419 | 1.84425 | 150.575 |
| 3.72193 | 375.445 | 2.64499 | 1524.47 | 2.24633 | 122.337 | 1.99764 | 250.631 | 1.84343 | 144.563 |
| 3.69715 | 105.378 | 2.62988 | 27.0299 | 2.24523 | 834.492 | 1.99071 | 76.9246 | 1.84332 | 155.729 |
| 3.5177 | 116.93 | 2.61534 | 467.41 | 2.20372 | 1236.5 | 1.98726 | 157.061 | 1.84134 | 96.7164 |
| 3.4745 | 25683.9 | 2.61413 | 3042.91 | 2.18494 | 63.928 | 1.98461 | 740.14 | 1.83758 | 1284 |
| 3.37974 | 70.508 | 2.58798 | 297.627 | 2.18322 | 111.462 | 1.98173 | 132.493 | 1.83418 | 52.5633 |
| 3.35014 | 895.593 | 2.58482 | 342.713 | 2.1761 | 217.658 | 1.98119 | 447.075 | 1.82667 | 212.797 |
| 3.34238 | 2793.53 | 2.57151 | 551.762 | 2.17525 | 5.53784 | 1.9733 | 1.62096 | 1.82637 | 208.12 |
| 3.30853 | 1.0834 | 2.5564 | 653.193 | 2.17226 | 661.024 | 1.96958 | 778.756 | | |
| 3.28419 | 306.798 | 2.53241 | 735.374 | 2.16891 | 496.952 | 1.96605 | 660.479 | | |
| 3.27203 | 7.84348 | 2.47233 | 1178.53 | 2.16629 | 162.413 | 1.95693 | 178.979 | | |
| 3.26187 | 1680.88 | 2.45963 | 1748.27 | 2.16518 | 2536.95 | 1.93403 | 470.769 | | |
| 3.25072 | 4772.76 | 2.44772 | 914.426 | 2.15593 | 139.776 | 1.92737 | 60.696 | | |
| 3.24739 | 4406.49 | 2.44736 | 34.1937 | 2.14149 | 172.019 | 1.91884 | 103.793 | | |
| 3.16481 | 120.773 | 2.44673 | 1077.33 | 2.12537 | 938.862 | 1.91703 | 718.869 | | |

**Table S3 –** d-spacings and squared structure factors for structure **3β**.

| d-spacing | $F^2$ | d-spacing | $F^2$ | d-spacing | $F^2$ | d-spacing | $F^2$ | d-spacing | $F^2$ |
|---|---|---|---|---|---|---|---|---|---|
| 15.428 | 26076.7 | 3.23089 | 2457.52 | 2.58447 | 3395.28 | 2.22548 | 453.051 | 2.03907 | 609.218 |
| 8.75383 | 24.5552 | 3.2306 | 1541.7 | 2.57134 | 2079 | 2.22536 | 708.464 | 2.03821 | 253.032 |
| 7.71402 | 999.37 | 3.21624 | 10141.8 | 2.57106 | 3038.41 | 2.22434 | 1157.6 | 2.03505 | 411.49 |
| 7.71006 | 1203.52 | 3.18911 | 871.256 | 2.57002 | 52.5926 | 2.204 | 458.809 | 2.01677 | 797.774 |
| 7.52075 | 47360.7 | 3.13383 | 8810.73 | 2.55703 | 12805.5 | 2.19943 | 99.6375 | 1.99302 | 3.29241 |
| 6.46119 | 12454.3 | 3.11851 | 567.536 | 2.52759 | 150.253 | 2.19375 | 879.665 | 1.99182 | 6396.91 |
| 6.43247 | 11717.2 | 3.11746 | 1033.66 | 2.51938 | 2497.66 | 2.19357 | 15.311 | 1.97523 | 101.918 |
| 5.87248 | 253.953 | 3.08561 | 915.069 | 2.50692 | 1.02114 | 2.19018 | 1066.87 | 1.9698 | 1073.82 |
| 5.70615 | 850.883 | 3.05536 | 935.112 | 2.4924 | 319.999 | 2.18954 | 5097.99 | 1.96901 | 142.291 |
| 5.57781 | 630.741 | 3.04304 | 4030 | 2.48822 | 1774.14 | 2.18846 | 2188.82 | 1.96678 | 42.9922 |
| 5.52283 | 4789.22 | 3.02821 | 2628.41 | 2.48665 | 45.6884 | 2.18417 | 214.123 | 1.96188 | 364.569 |
| 5.14268 | 6081.23 | 3.02677 | 1563.95 | 2.45123 | 1021.81 | 2.17869 | 779.374 | 1.9581 | 656.658 |
| 4.52992 | 861.058 | 3.01916 | 2123.06 | 2.44802 | 19.2353 | 2.17554 | 5060.57 | 1.95784 | 323.158 |
| 4.49128 | 1642.15 | 2.98315 | 2507.63 | 2.41419 | 387.981 | 2.1581 | 287.354 | 1.95763 | 478.242 |
| 4.48091 | 2998.3 | 2.9369 | 3215.04 | 2.4066 | 438.406 | 2.15373 | 345.034 | 1.95749 | 1942.04 |
| 4.47935 | 20940 | 2.93624 | 2059.81 | 2.36015 | 34.4348 | 2.15256 | 420.067 | 1.94601 | 11009.1 |
| 4.45073 | 2954.86 | 2.92598 | 4421.71 | 2.35876 | 3195.91 | 2.15211 | 346.332 | 1.94218 | 281.199 |
| 4.37909 | 4602.78 | 2.91794 | 2006 | 2.35762 | 4034.34 | 2.15142 | 378.294 | 1.93515 | 15.7474 |
| 4.37691 | 4983.7 | 2.88405 | 110.779 | 2.34785 | 273.844 | 2.14416 | 333.654 | 1.93477 | 466.518 |
| 4.24312 | 3906.69 | 2.88239 | 855.618 | 2.34687 | 121.247 | 2.1228 | 57.9148 | 1.92877 | 421.948 |
| 4.17909 | 3888.39 | 2.85308 | 7825.35 | 2.34443 | 38.616 | 2.12156 | 362.708 | 1.9285 | 383.071 |
| 4.16673 | 338.703 | 2.85207 | 454.515 | 2.34305 | 1434.76 | 2.12032 | 307.619 | 1.92751 | 0.321763 |
| 4.09864 | 1880.2 | 2.8297 | 1605.27 | 2.33111 | 423.261 | 2.11999 | 835.691 | 1.92323 | 28.7662 |
| 3.85701 | 178.804 | 2.78891 | 701.808 | 2.31366 | 2925.89 | 2.10435 | 645.739 | 1.9194 | 37.261 |
| 3.85503 | 1429.88 | 2.77087 | 121.852 | 2.29528 | 95.2237 | 2.0901 | 52.7823 | 1.91796 | 72.5039 |
| 3.76037 | 126.352 | 2.76142 | 42.6617 | 2.29339 | 488.972 | 2.08955 | 1918.29 | 1.90205 | 141.426 |
| 3.68291 | 54.1799 | 2.76087 | 1289.66 | 2.28952 | 1308.97 | 2.08407 | 1764.39 | 1.89903 | 8891.47 |
| 3.68204 | 395.998 | 2.75983 | 92.5021 | 2.2869 | 412.624 | 2.08336 | 40.9429 | 1.89491 | 408.154 |
| 3.64599 | 2701.91 | 2.75575 | 660.608 | 2.28628 | 815.191 | 2.08156 | 989.179 | 1.88778 | 241.192 |
| 3.60431 | 4230.83 | 2.72635 | 719.151 | 2.26496 | 45.4486 | 2.07743 | 18.8748 | 1.8802 | 168.21 |
| 3.56791 | 32.9419 | 2.70913 | 228.465 | 2.26466 | 57.4923 | 2.07727 | 776.931 | 1.87616 | 5944.83 |
| 3.558 | 99951.5 | 2.70343 | 227.956 | 2.25623 | 380.922 | 2.07642 | 3998.38 | 1.87365 | 1385.99 |
| 3.49247 | 2825.41 | 2.70093 | 1061.08 | 2.24564 | 15497 | 2.07073 | 104.283 | 1.87199 | 90.0338 |
| 3.467 | 36779.8 | 2.61521 | 1367 | 2.241 | 724.534 | 2.06595 | 382.887 | 1.87061 | 856.884 |
| 3.38182 | 851.297 | 2.6146 | 1501.36 | 2.24045 | 1794.01 | 2.06301 | 699.383 | 1.86982 | 2549.03 |
| 3.29614 | 12442.4 | 2.59134 | 43.3961 | 2.23968 | 708.289 | 2.05705 | 349.442 | 1.86474 | 2843.25 |
| 3.2865 | 1427.43 | 2.59044 | 4923.19 | 2.22584 | 1453.08 | 2.04932 | 257.414 | 1.86407 | 3056.42 |