Electronic Supplementary Material (ESI) for Photochemical & Photobiological Science
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Determination of photostability and photodegradation products of moxifloxacin in the presence of metal ions in solutions and solid phase. Kinetics and identification of photoproducts.

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Supporting Information

2. Experimental section

Method validation.

The method was validated for specificity, linearity, precision, recovery, limit of detection and limit of determination according to ICH guidelines.[1] The specificity of the method is its ability to measure analyte response in the presence of potential impurities. In order to evaluate the specificity, 20 µL of moxifloxacin solutions without and with ions of studied metals, before and after UVA irradiation were spotted onto TLC plates. For linearity studies aliquots of 0.03 µg per band to 2.5 µg per band of standard solutions were applied on a TLC plate. Further analytical procedure was as described in 2.5. Determination of linearity was made in three replicate. Linearity was assessed as a relationship between mean peak area and concentration from 0.03 to 1.5 in µg per band. Linearity was reported as the linear calibration equations and the correlation coefficients (r). For precision, repeatability of sample application and determination of peak area were asessed by making 5 replicates of different amounts of standard solution (0.12, 0.6, 1.2 µg per band). Assay accuracy was determined by estimating recovery percentage for moxifloxacin. Known amount of standard substance of

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moxifloxacin (80%, 100%, 120%) were added to model solution of this compound. Recovery in percentage value was calculated on the basis of determined content of moxifloxacin to weighed amount. Each level was analyzed in triplicate and a mean value from 9 analyses was taken as a result. In order to asses limit of detection (LOD) and limit of determination (LOQ) decreasing volumes (5, 4, 3, 2, 1 μg per band) of standard solution of the concentration 6 μg · mL^{-1} were applied on a TLC sheet. Further analytical procedure was as described in 2.5. Peak area was measured and signal to noise ratio 3:1 was regarded as LOD whereas signal to noise ratio 10:1 was regarded as LOQ.

In a subsequent step, the influence on photodegradation of moxifloxacin concentration in range of 20 μg mL^{-1} to 100 μg mL^{-1}, at constant concentration of metal ions (0.025 mol/L) was tested. Samples were exposed to UVA radiation for 6 h and 72 h in the solid phase and solutions, respectively.

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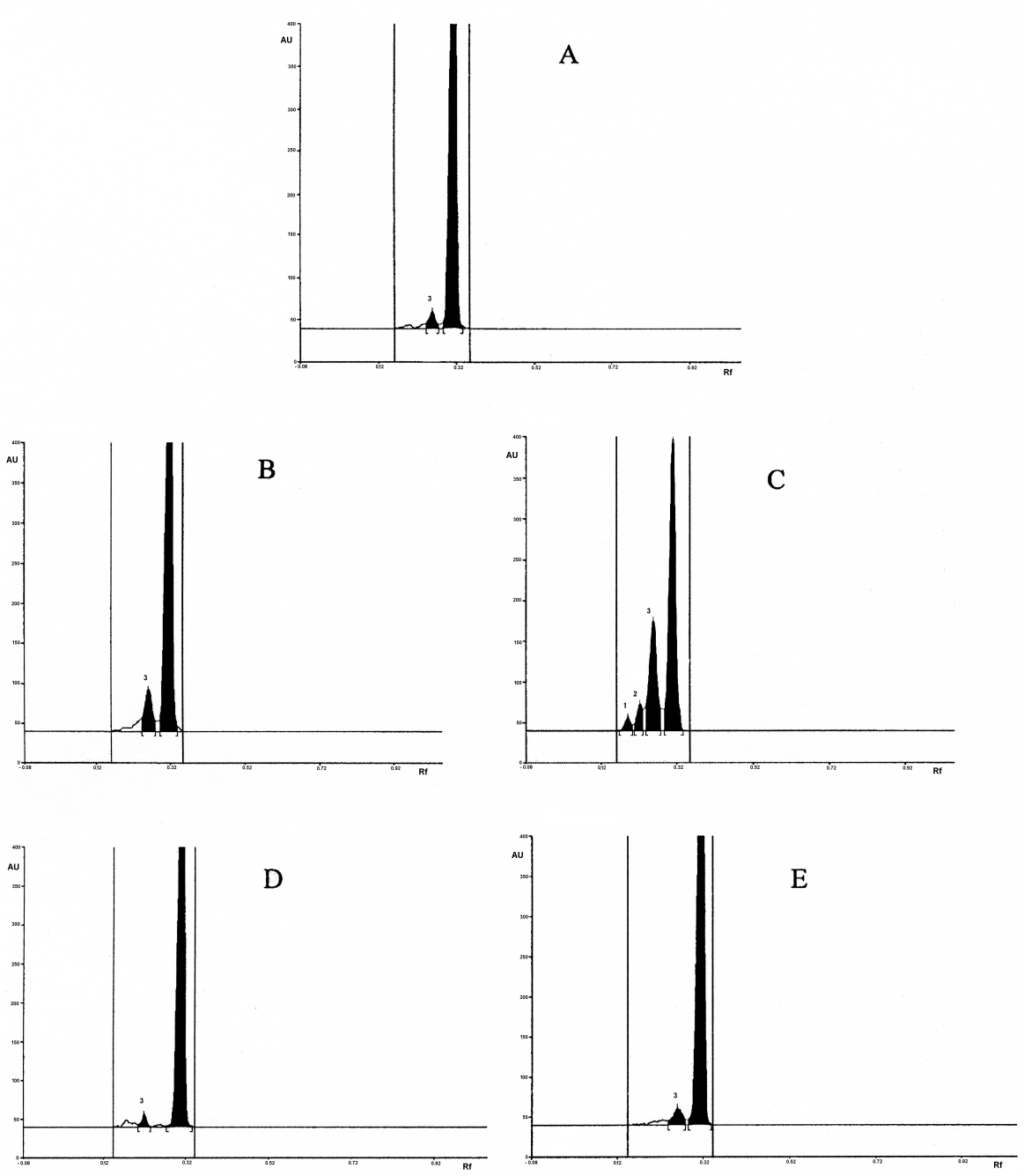

Figure S 1. Densitograms of MOXI after photodegradation in solutions for 72h: without metal ions (A) and in the presence of: Cu(II) (B), Fe(III) (C), Al(III) (D), Zn(II) (E)

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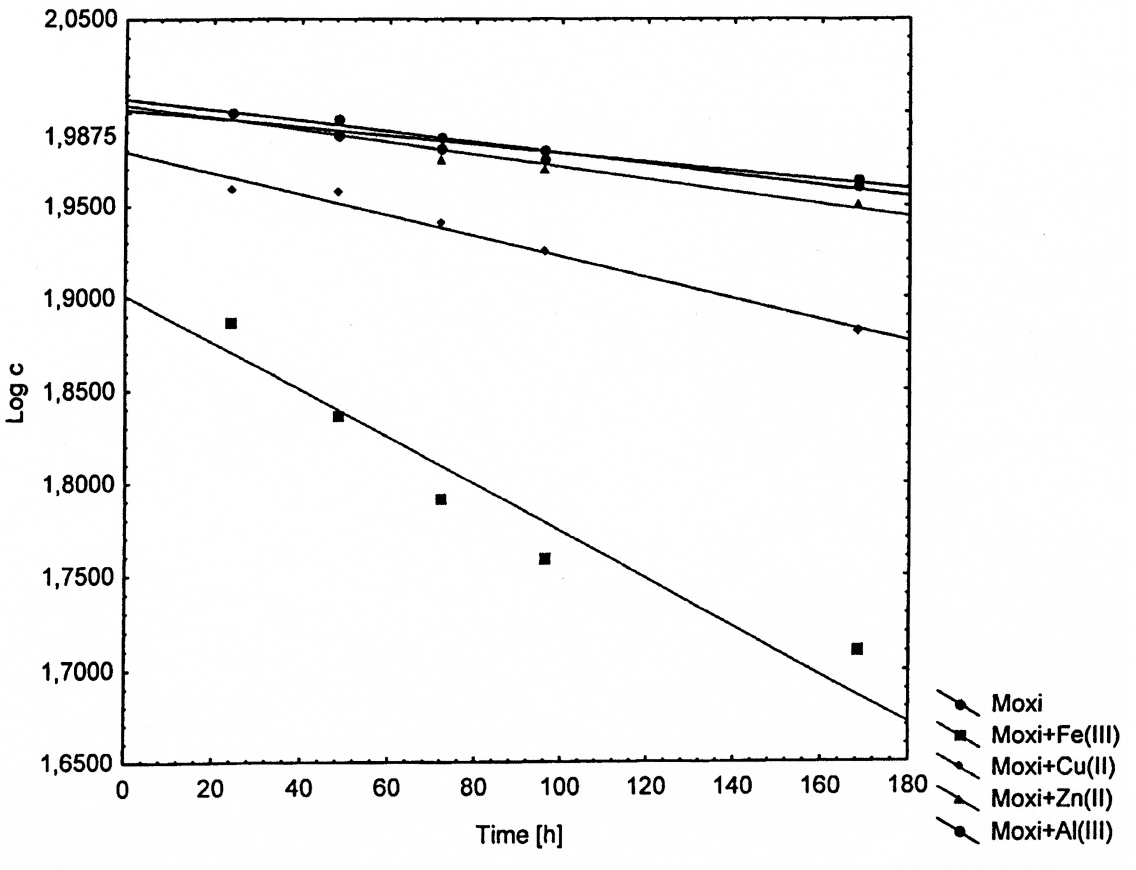

Figure S 2. Chart of correlation of log c= f(t) for photodegradation of moxifloxacin in solutions with and without the presence of metal ions.

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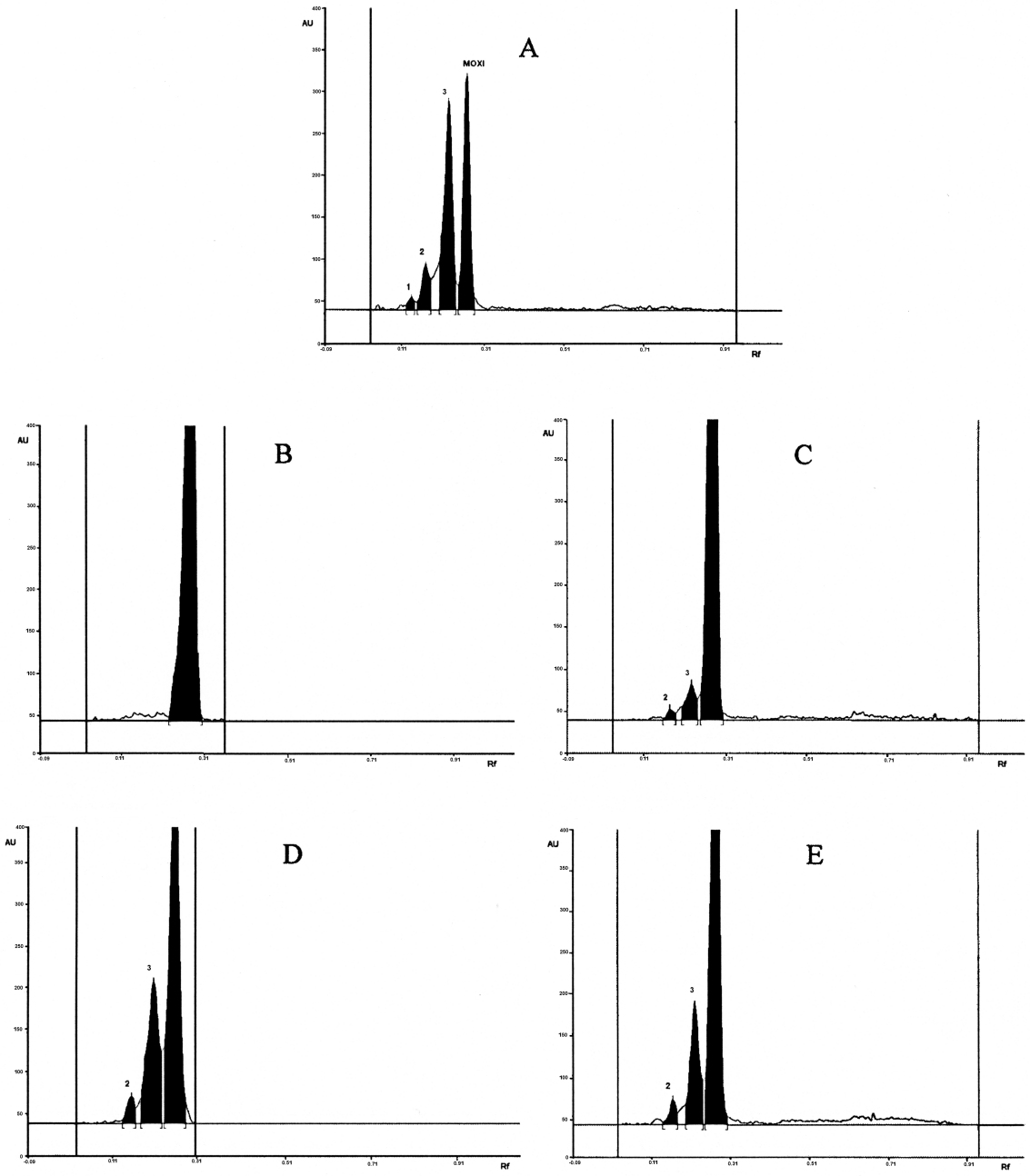

Figure S 3. Densitograms of MOXI after photodegradation in the solid phase for 15 h: without metal ions (A) and in the presence of: Fe(III) (B), Cu(II) (C), Al(III) (D), Zn(II) (E)

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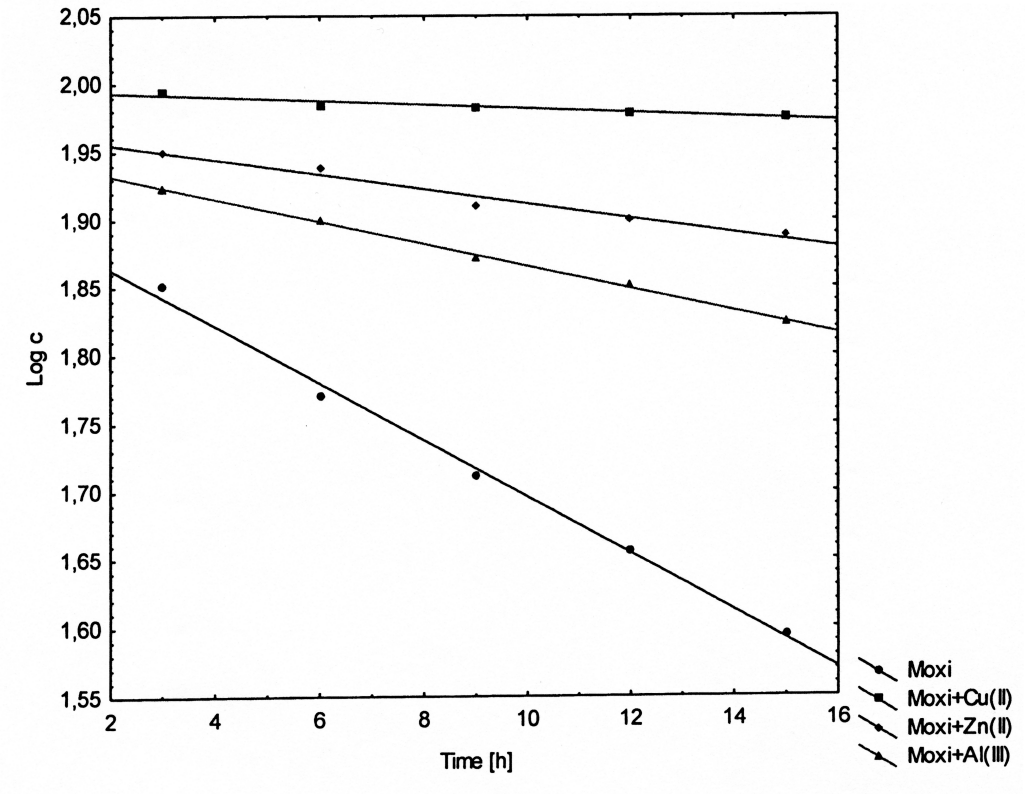

Figure S 4. Chart of correlation of log c= f(t) for photodegradation of MOXI in solid phase with and without the presence of metal ions.

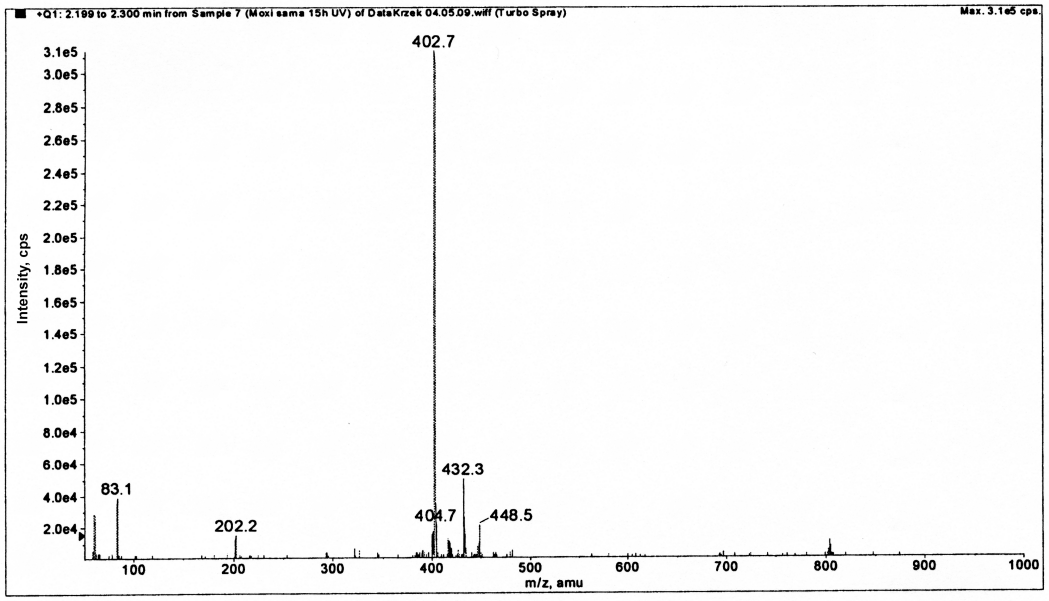

Figure S 5. Mass spectrum of MOXI at retention time of 2.21 min.

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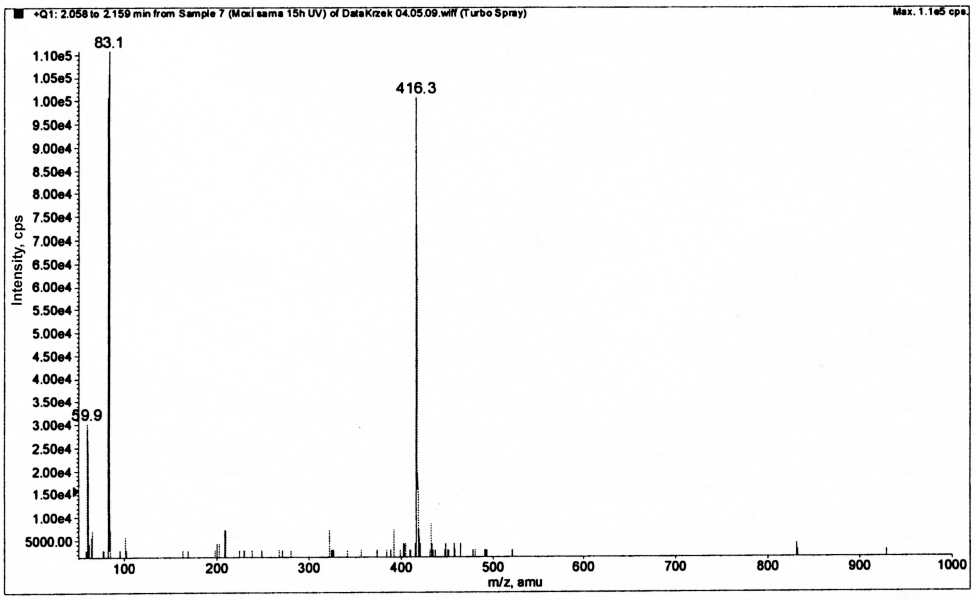

Figure S 6. Mass spectrum of degradation product P-II at retention time of 2.11 min.

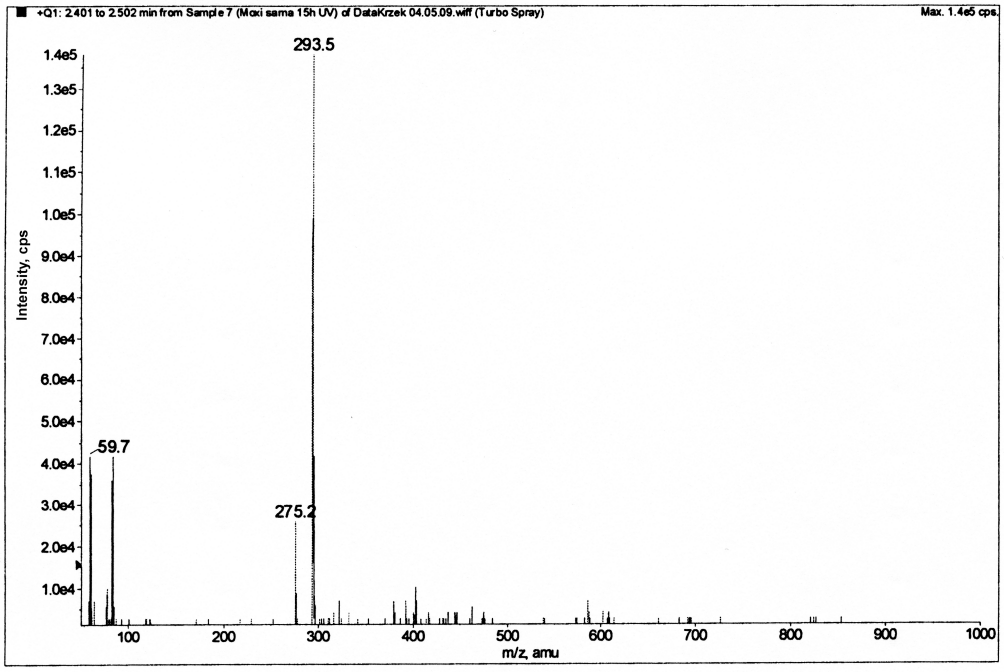

Figure S 7. Mass spectrum of degradation product P-I at retention time of 2.47 min.

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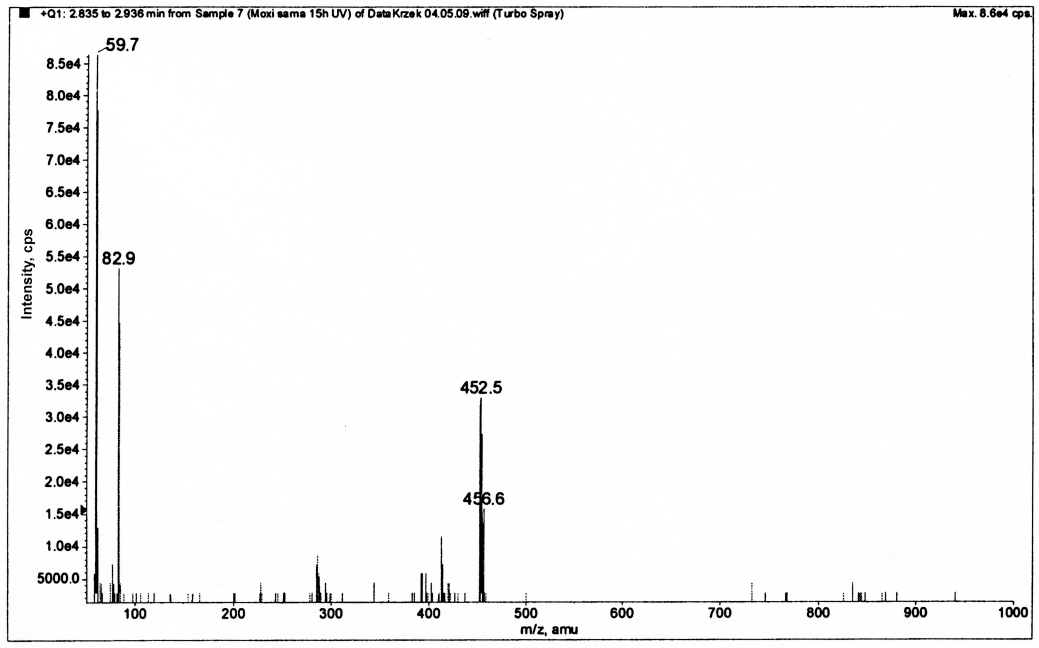

Figure S 8. Mass spectrum of degradation product P-III at retention time of 2.88 min.

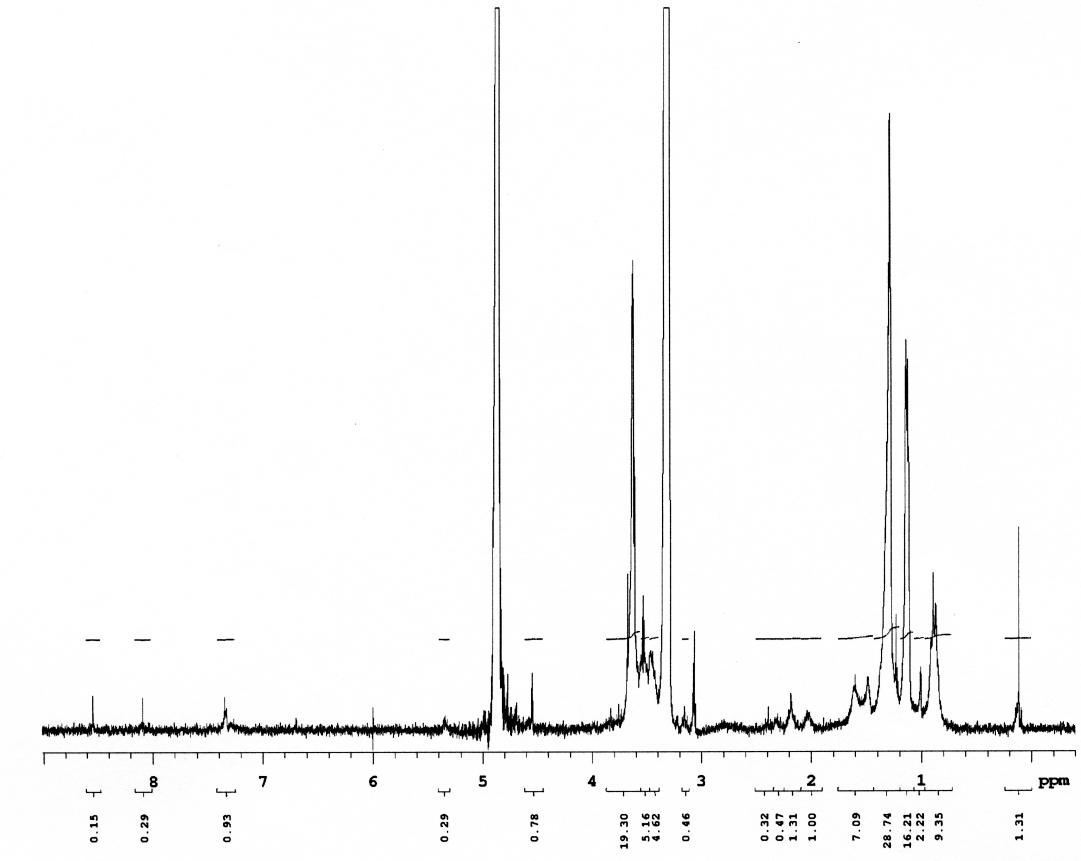

Figure S 9. ^{1}H NMR spectrum of degradation product P-I.

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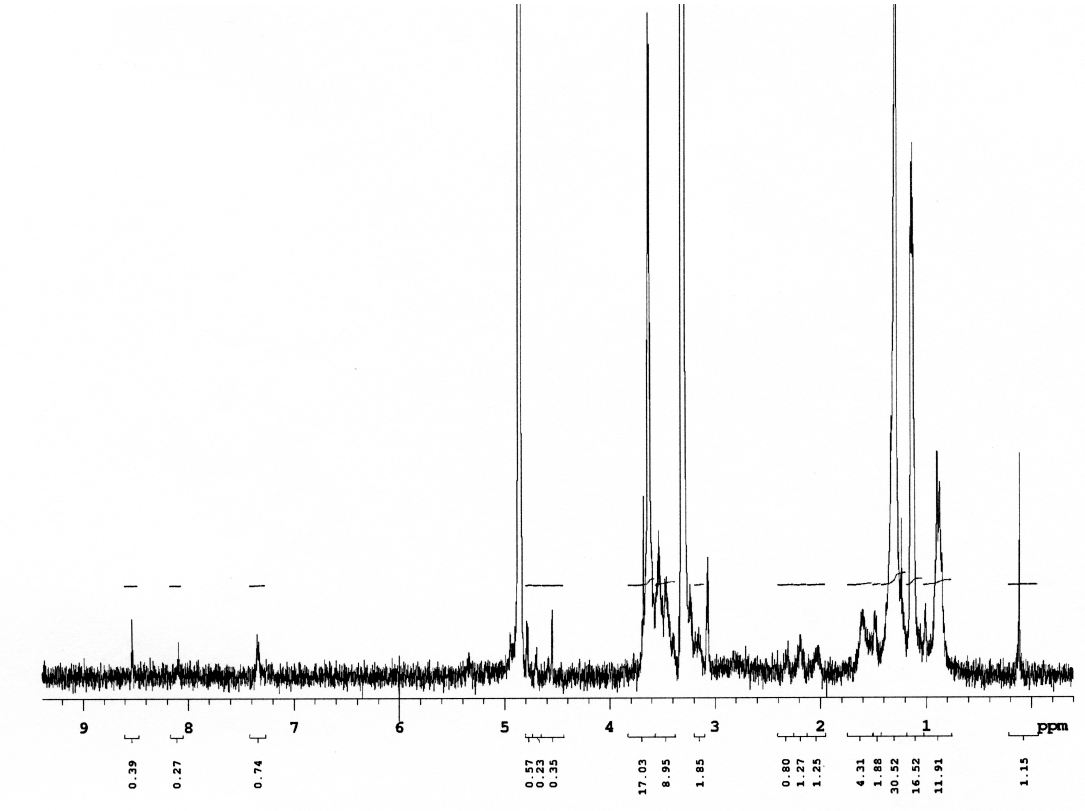

Figure S 10. ^{1}H NMR spectrum of degradation product P-II.

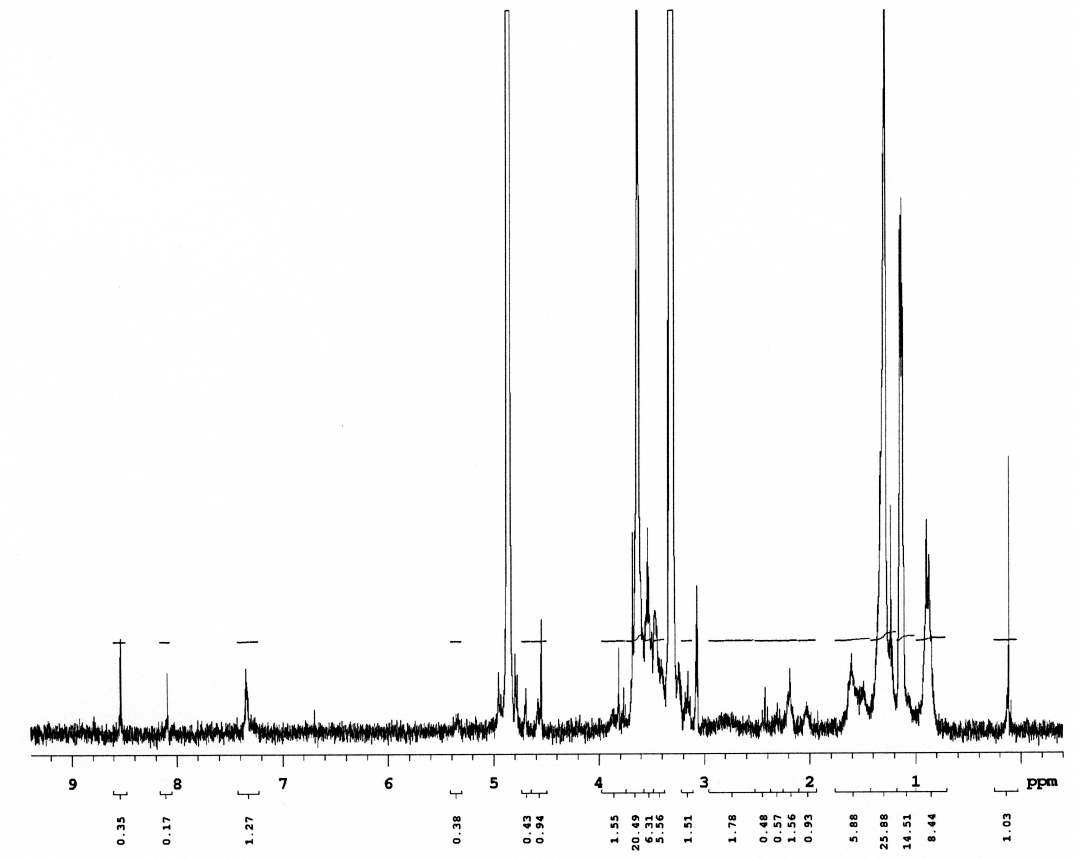

Figure S 11. ^{1}H NMR spectrum of degradation product P-III.

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References

1. ICH-Q2 (R1) Validation and Analytical Procedures: Text and Methodology, International Conference on Harmonization, Geneva, November 2005, http://www.ich.org

Table S 1. Validation of the method

Parameter	**MOXI**
R_F	0.27±0.03
Limit of detection, [ng/band]	12
Limit of quantitation, [ng/band]	30
Linearity range, [μg/band]	0.03 – 1.5
Regression coefficients P = a c+b±S_e[a]	a = 9354.5 b = 2766.4±808.2
Standard deviation of the regression coefficients	S_a = 596.0 S_b = 502.1
Correlation coefficients, r	r= 0.9900
Precision	level 1: x_m=3882.5 RSD=0.38% level 2: x_m=9047.4 RSD=0.31% level 3: x_m=14907.4 RSD=1.07%
Indirect precision	level 1: x_m=4185.0 RSD=1.08% level 2: x_m=8959.9 RSD=0.64% level 3: x_m=14712.2 RSD=0.44%
Recovery, [%] n=3	level 80%: 101.50% level 100%: 101.34% level 120%: 100.34%

[a] P = peak area; c = concentration; a and b = regression coefficients, S_e = standard error of the estimate, S_a = standard deviation of the regression coefficient a, S_b = standard deviation of the regression coefficient b, RSD = relative standard deviation