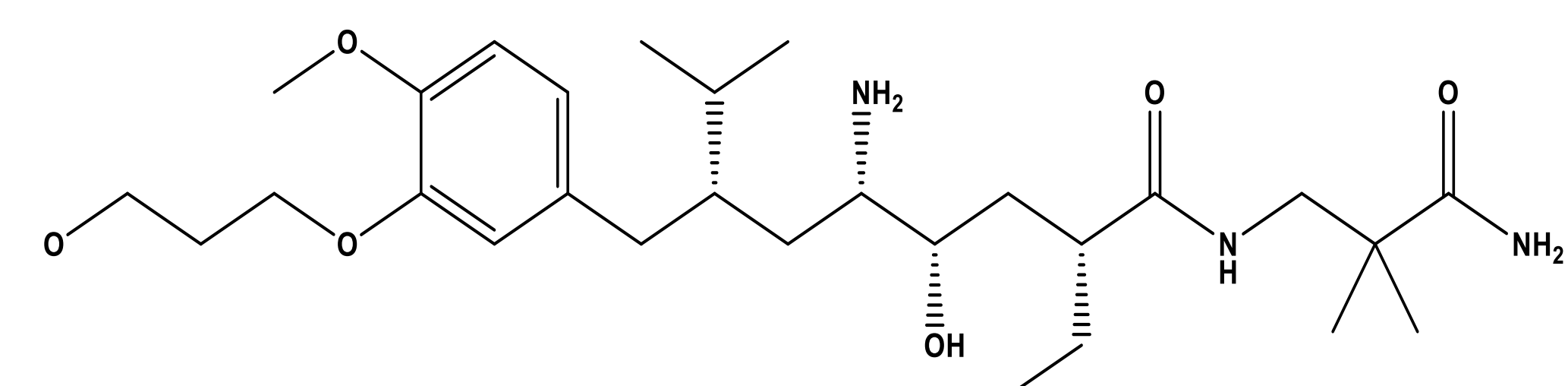


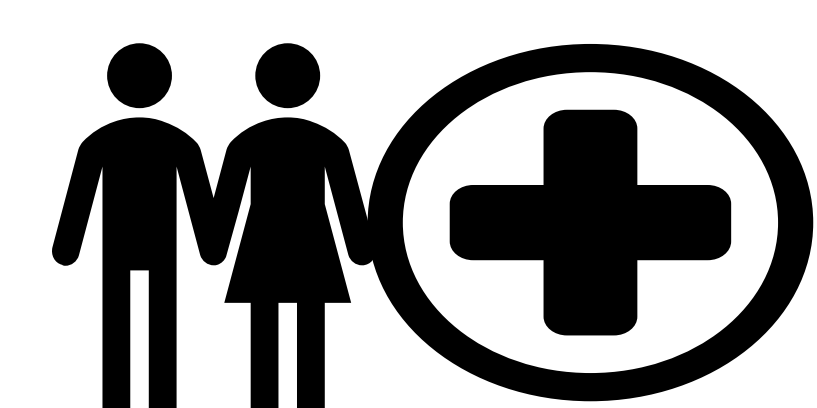


Introduction

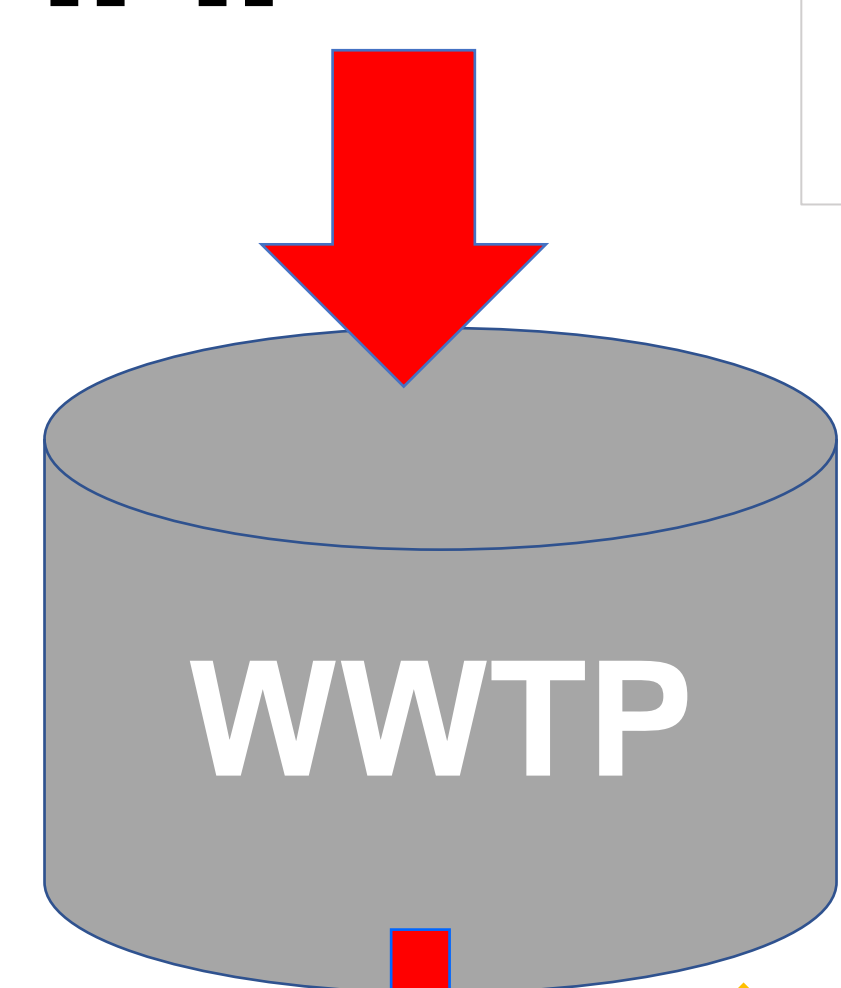
- Aliskiren (ALK) - treatment of hypertension



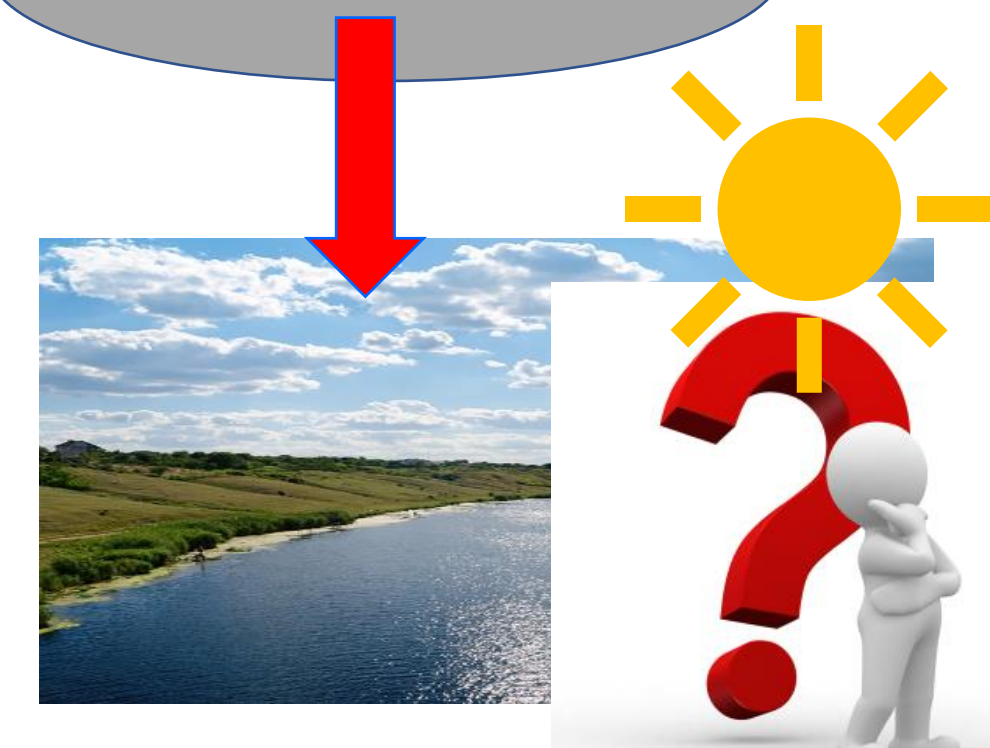
CAS: 173334-57-1
Chemical Formula: C₃₀H₅₃N₃O₆
Molecular Weight: 551.7583



- Mainly administered in hospitals
- Excretion via urine (up to 1.2%) and faeces (75%) of the parent drug (1-3)



- Aliskiren in WWTP effluents from EU: 0.4-1.9 µg/L (4)



- Fate of ALK in (natural) water?
- Photodegradation products (PDPs)? Degradation pathway?

Goals

- To identify the photodegradation products of aliskiren in water using UHPLC-MS/MS
- To propose the possible chemical structures of the PDPs and the degradation pathway

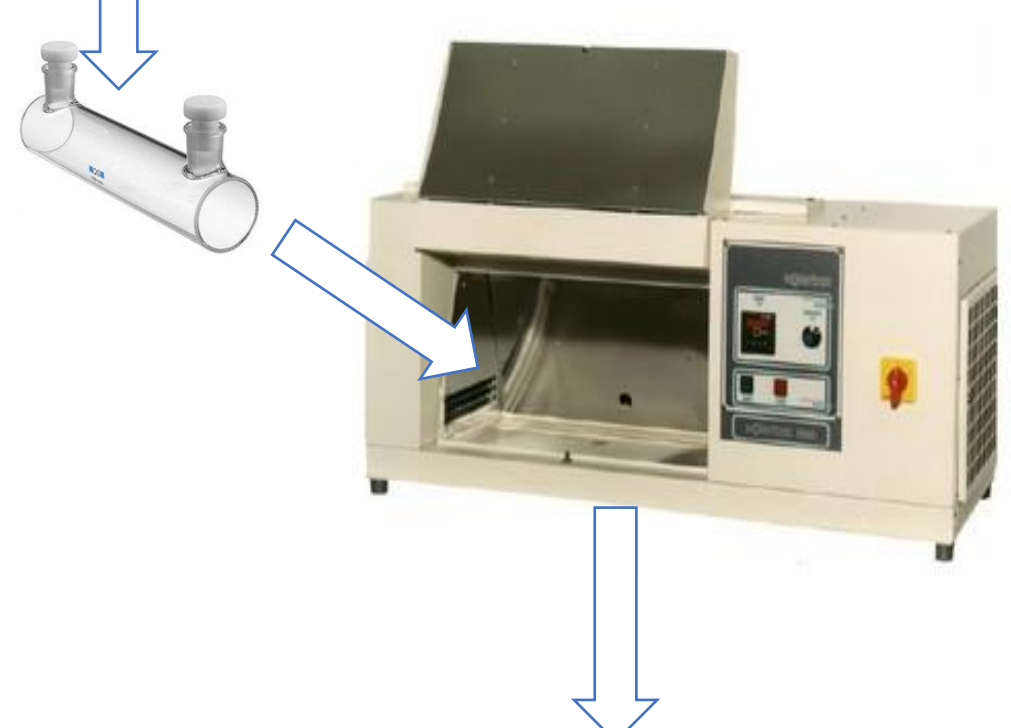
Instrumentation and Methods

10.0 mg/L of ALK

Simulated solar irradiation

(Solarbox 3000e, Cofomegra, Milan)

- Irr. 600 W/m², Temp. 30±2 °C
- Xe lamp, outdoor UV filter



Instrumentation and Methods

Withdrawing samples at prefixed irradiation times and recording their **UV-Vis spectrums**

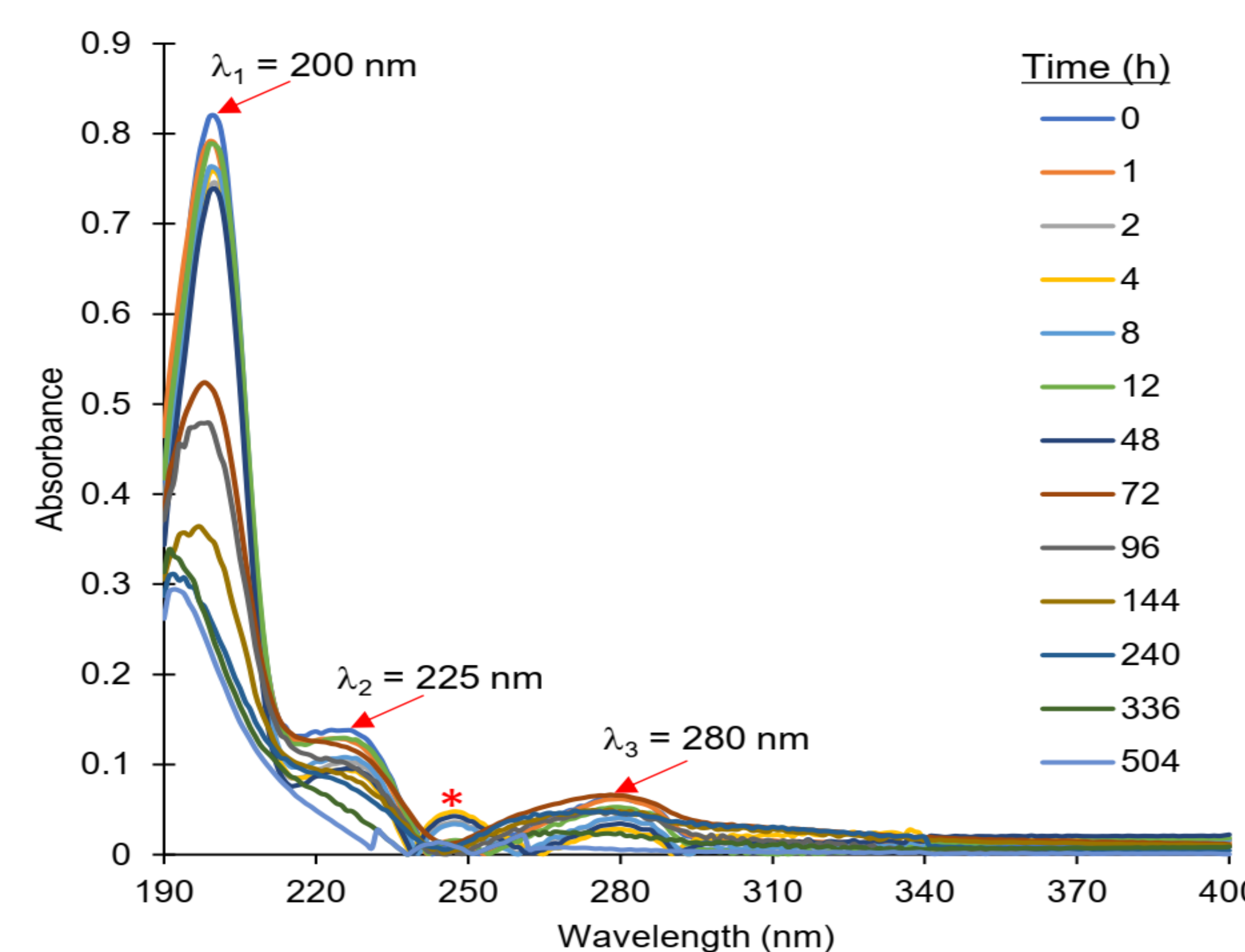
UHPLC-MS/MS Analysis

- Nexera UHPLC (Shimadzu) coupled with 3200 QTRAP LC-MS/MS system (Sciex)
- Details of the method development and validation workflow (5)



Results and Discussion

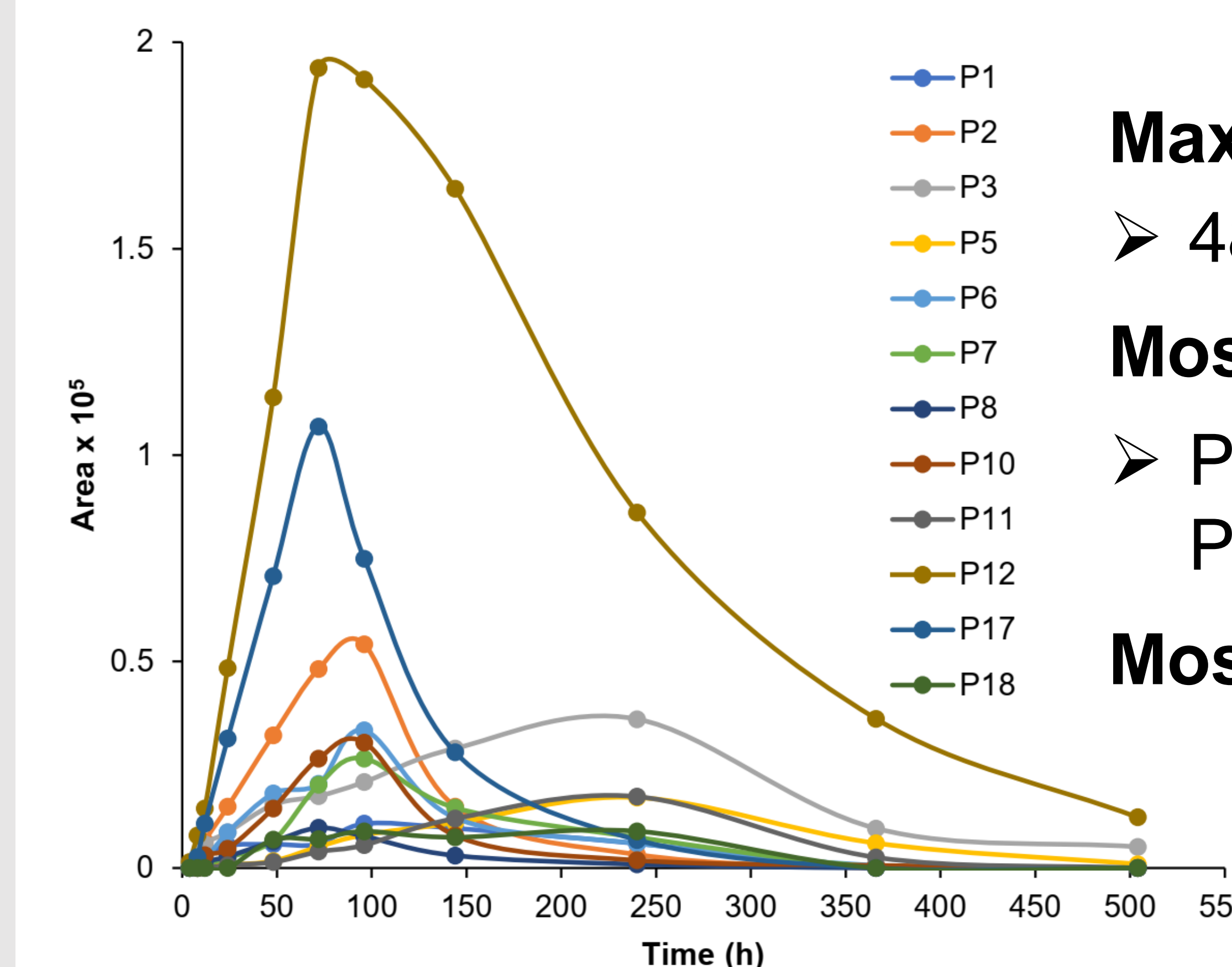
The **degradation progress** was first monitored via UV-Vis absorption measurements



- Significant absorbance decreases @ λ_1 , λ_2 , λ_3
- New absorption peak appeared (indicated by *)
- Rapid degradations at 48-72 h and 96-240 h
- A total of **18 PDPs** identified in MilliQ water
- 7** of them detected in spiked river water

Results and Discussion

LC-MS/MS results: Profiles of 11 abundant PDPs



Max. intensities:

- 48–96 h and 96–240 h

Most abundant:

- P12 (m/z 306.2) and P2 (m/z 382.3)

Most persistent: P12

- 4 PDPs** still present after 21 days of irradiation
- 7 PDPs found in this study were previously found in biological samples and in water (1, 3)
- Validation of the method: LOD, LOQ, precision, recovery and matrix effects evaluated

Conclusions and Future Directions

- A UHPLC-MS/MS method developed & validated for the identification of PDPs of ALK in water
- Structures of 11 PDPs proposed for the first time, based on MS and MS/MS data
- A complete degradation pathway will be proposed based on the current findings

References

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- [2] Burckhardt *et al.* (2014). *Biomed. Chromatogr.*, 28, 1679-1691.
- [3] Waldmeier *et al.* (2007). *Drug Metab. Dispos.*, 35, 1418-1428.
- [4] Singer *et al.* (2016). *Environ. Sci. Technol.*, 50, 6698-6707.
- [5] Gosetti *et al.* (2020). *Environ. Pollut.*, 256, 113370.