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Persistency measurement of seven organic micropollutants in water

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Outline

- Background
- Experimental methods
- Results
- Conclusions



Background

Study asked for by Dutch water companies

Organic micropollutants (OMP) in the environment:

- Increasing numbers of compounds
- Increasing concentrations
- New compounds, little information available

Relevant question:

How persistent are some OMP?

Answer:

According to OECD Guideline 309



Experimental method

OECD Guideline 309: Aerobic Mineralisation in Surface Water – Simulation Biodegradation Test

- Determine the time course of aerobic primary and ultimate degradation in surface water
- Determine kinetic rate expressions
- Measurements based on ^{14}C labelled compounds

Disadvantage:

Poorly accessible and expensive procedure

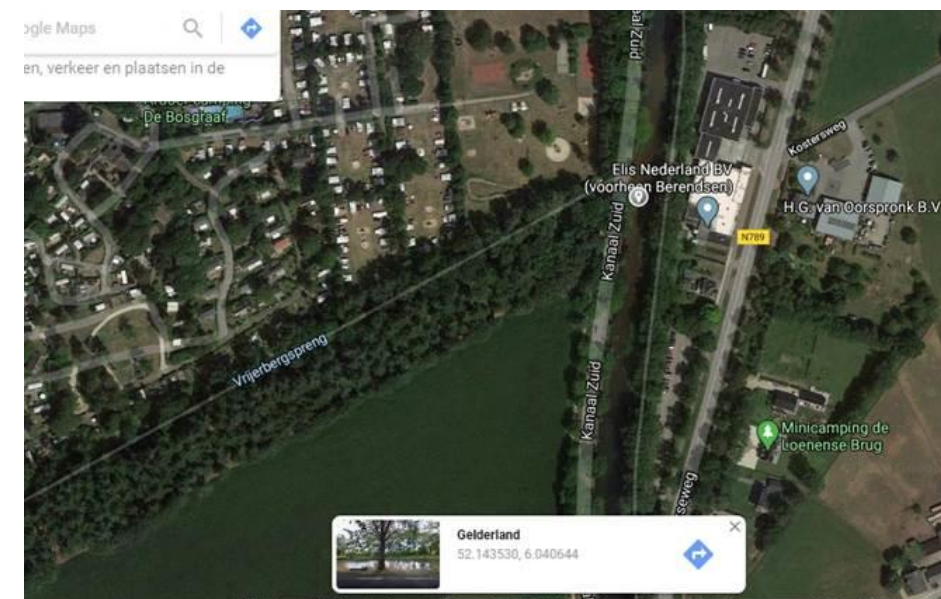
Question:

Can we apply OECD Guideline 309 in a less cumbersome, but still reliable way?

Experimental method

Surface water from site Schalterberg

- Originates from sub-surface springs
- No discharge of industrial or municipal wastewater
- Used as a source for drinking water
- Frequently analysed for anthropogenic compounds
- Samples taken at 0.5 m below water surface
- Kept at 13°C



Experimental method

Water quality

parameter	unit	Average value
EC	$\mu\text{S}/\text{cm}$	155
pH		7.3
Temperature (T)	$^{\circ}\text{C}$	13.2
Mass balance	g	0.0
Nitrate concentration ($\text{NO}_3\text{-N}$)	mg N /L	0.57
Turbidity	FNE	0.86
Oxygen	mg O_2 /L	7.5

Experimental method: Biodegradation potential of water

Degradation of reference substance

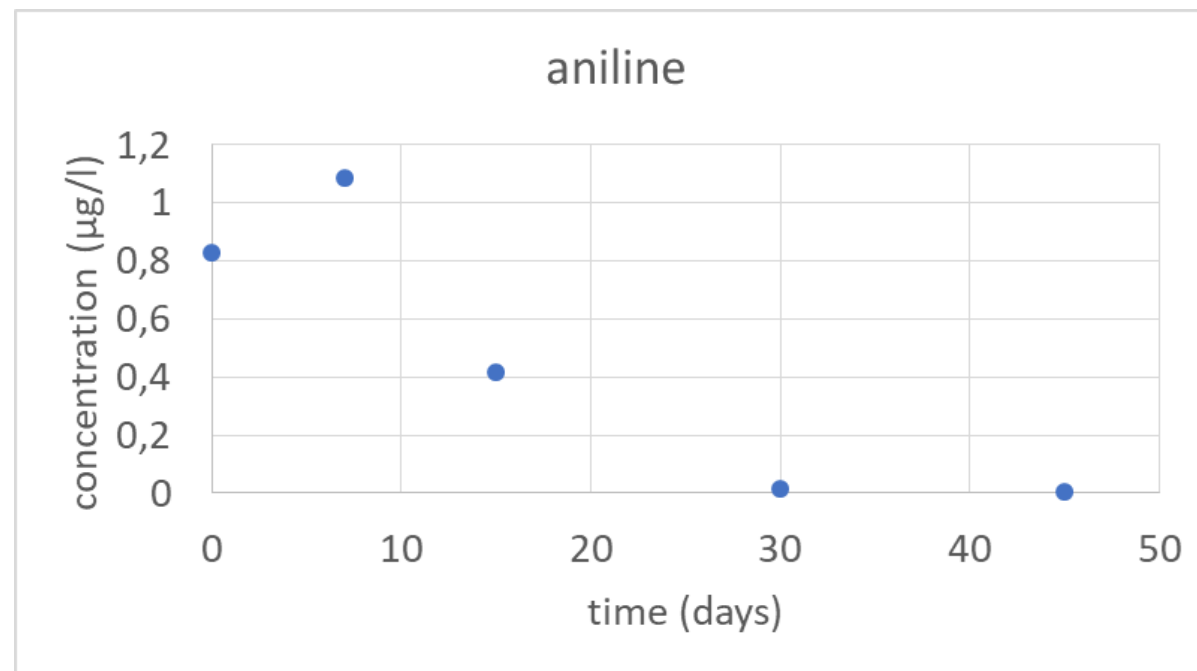
aniline

Water spiked with 1 µg/L

Analyses on day 0, 7, 15, 30, 45 and 60

Result:

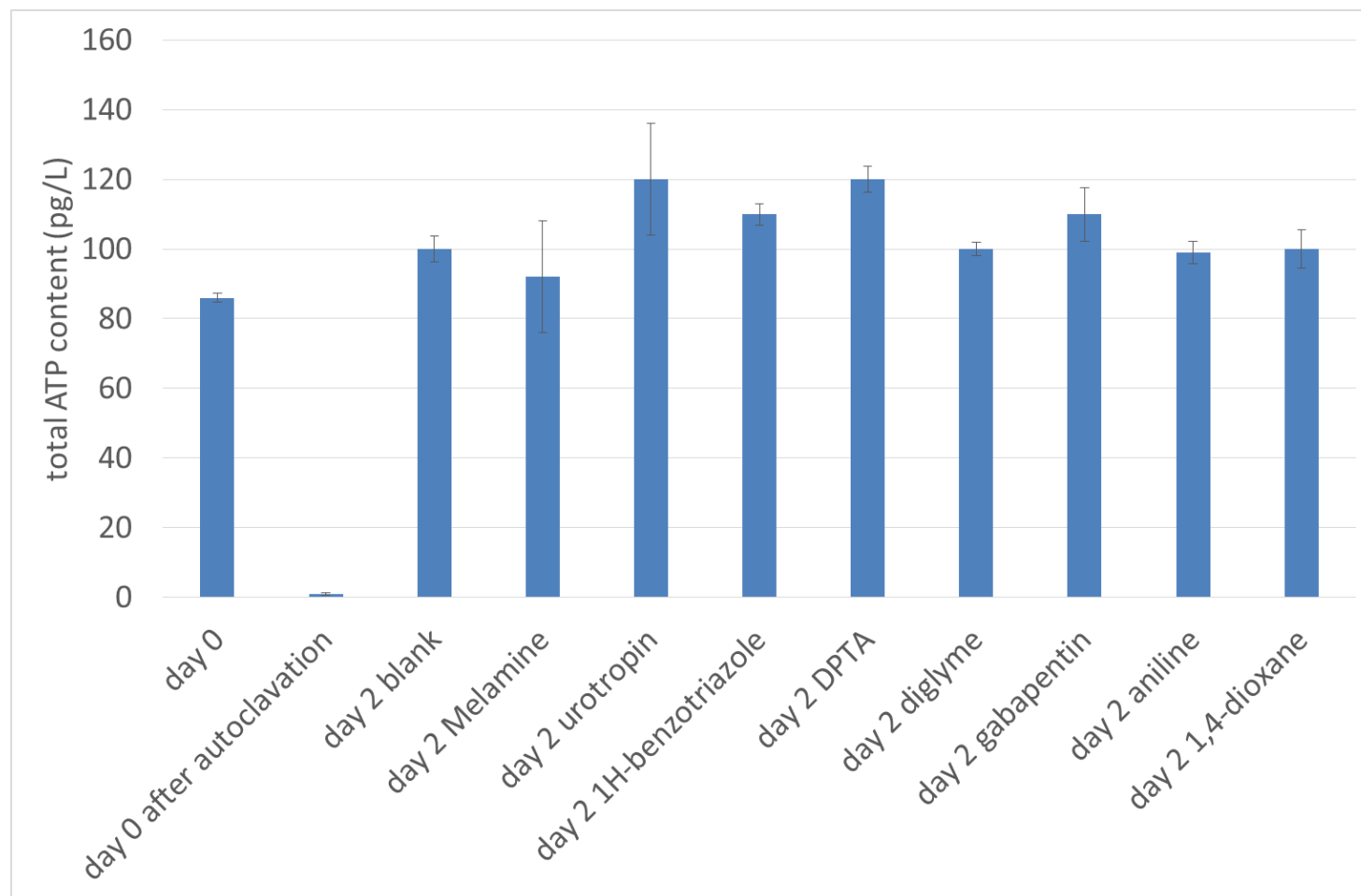
>98% degradation after 30 days of incubation.



Experimental method: Biodegradation potential of water

ATP measurements

- Directly after sampling (day 0)
- After autoclavation (day 0)
- After 2 days blank
- After 2 days incubation with OMP





Experimental method: Biodegradation potential of water

Conclusions Aniline and ATP experiments:

- Surface water showed sufficient microbial activity for biodegradation of the selected OMP
- OMP weren't toxic to the microorganisms present in the surface water

Experimental method: 7 OMP

- Gabapentin (60142-96-3)
- 1H-benzotriazole (95-14-7)
- Diglyme (111-96-6)
- DTPA (67-43-6)
- 1,4-dioxane (123-91-1)
- Melamine (108-78-1)
- Urotropin (100-97-0)

- Relevant for drinking water production
- Frequently observed OMP
- Not regulated yet

Stock solutions made in Milli-Q water

Experimental method: test system

- 1 L green bottles
- 300 g of water and air
- Air tight sealing
- 13 ± 1 °C in darkness
- Gentle agitation (20 rpm)
- Samples after 0, 7, 15, 30, 45 and 60 days of incubation
- Direct analyses using HPLC and GC-MS
- Duplicate samples

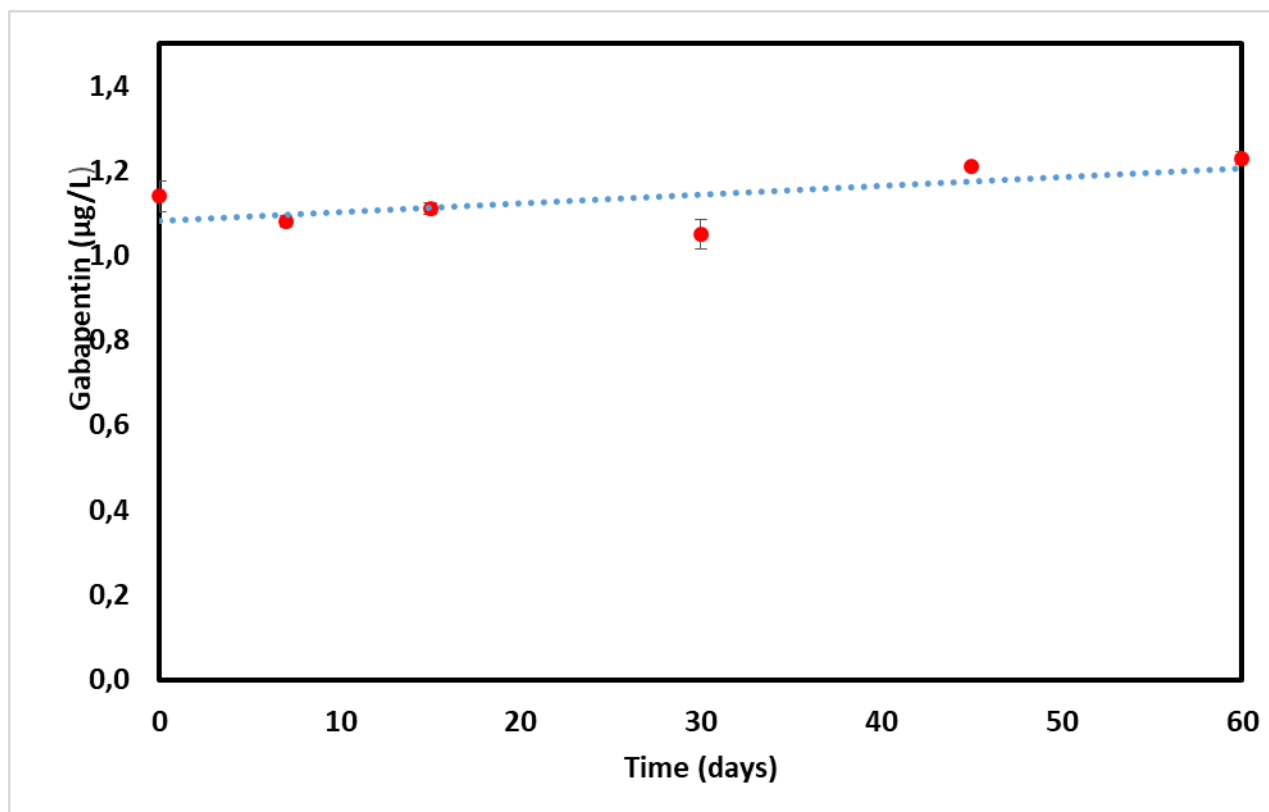


Compound	Dosed concentration (µg/L)
Gabapentin	1
1H-benzotriazole	1
Diglyme	20
DTPA	100
1,4-dioxane	100
Melamine	5
Urotropin	5
Aniline (reference)	1

Results

Gabapentin

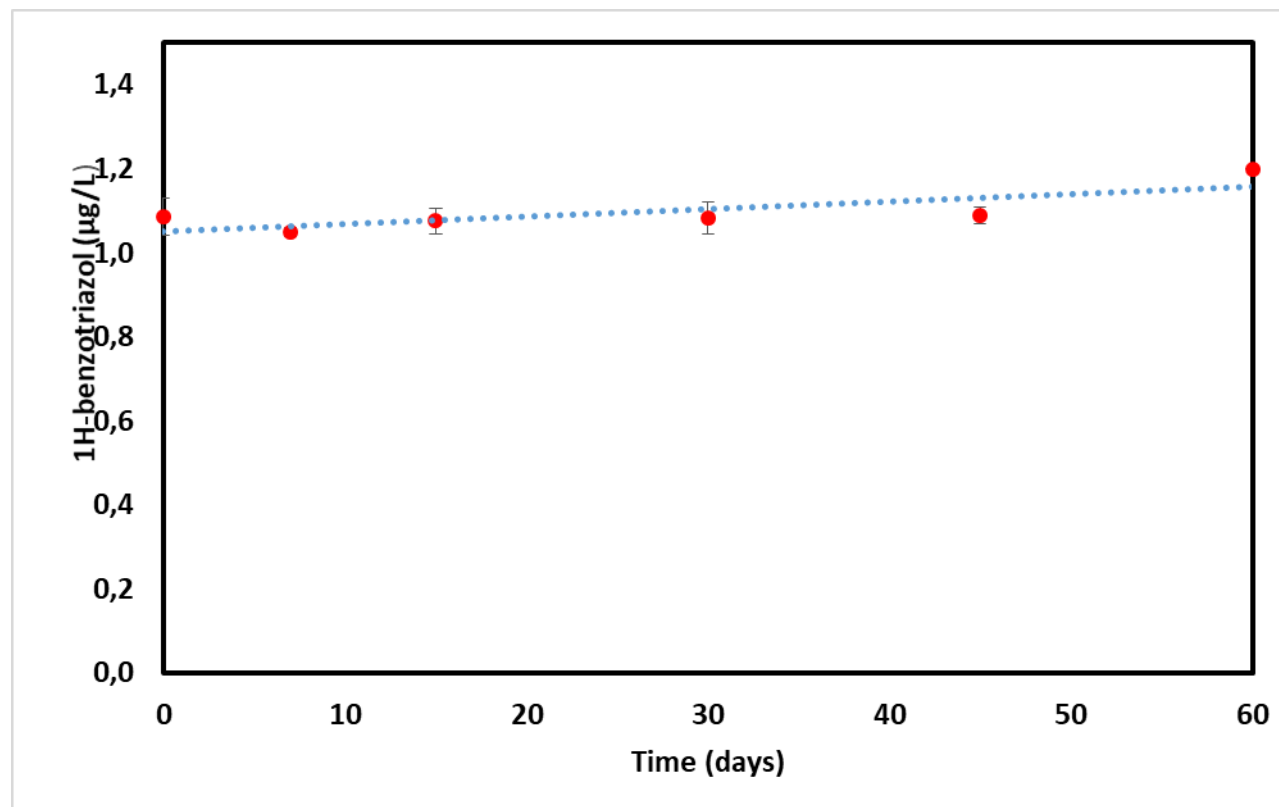
- Concentration remained stable in course of time
- Not primarily degraded in surface water
- DT50 calculated based on concentration in aqueous phase
- DT50 > 10,000 days



Results

1H-benzotriazole

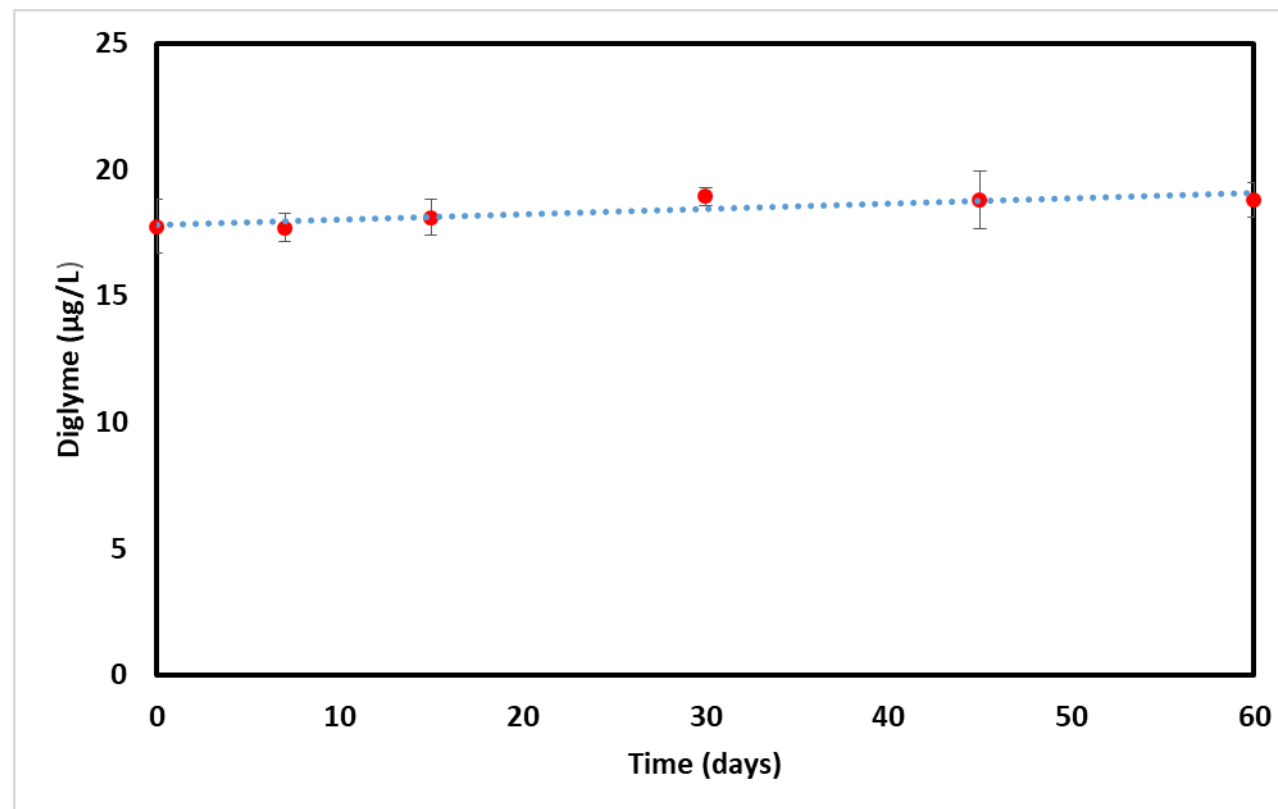
- Concentration remained stable in course of time
- Not primarily degraded in surface water
- DT50 calculated based on concentration in aqueous phase
- DT50 > 10,000 days



Results

Diglyme

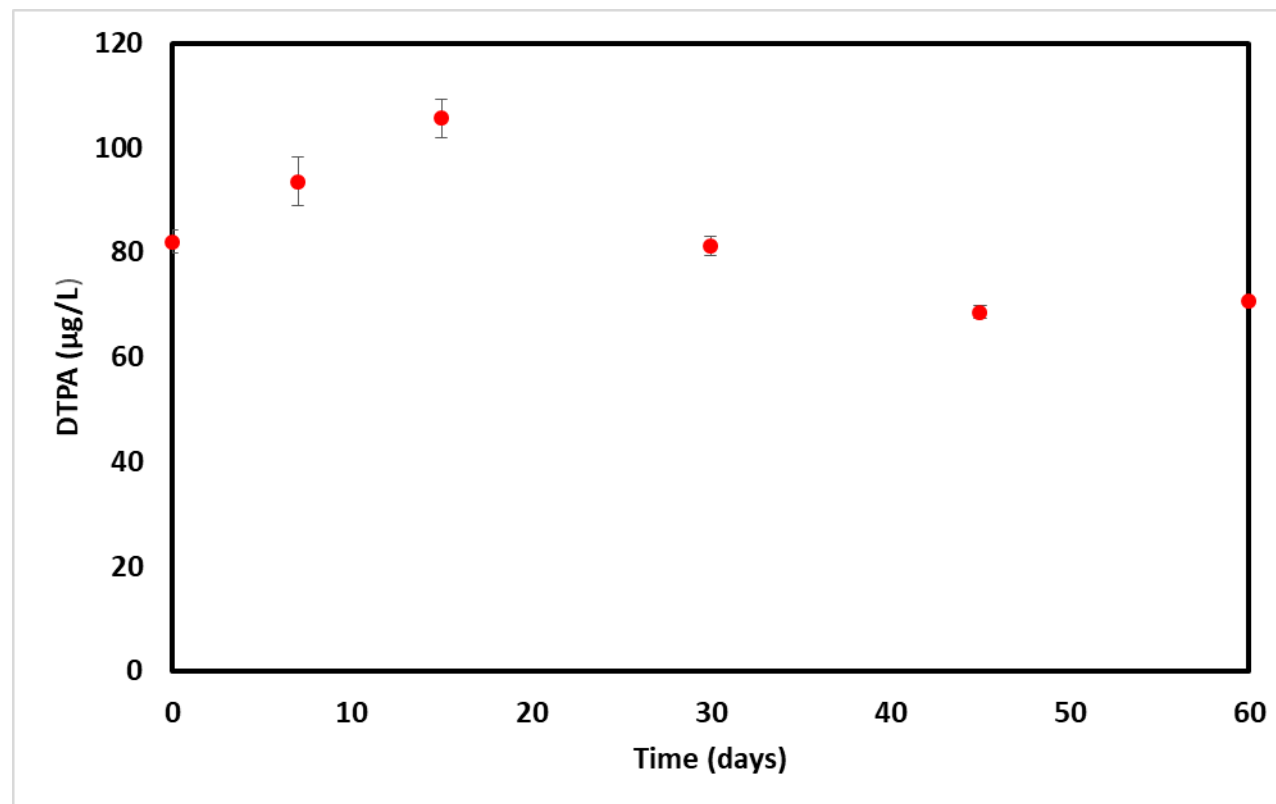
- Concentration remained stable in course of time
- Not primarily degraded in surface water
- DT50 calculated based on concentration in aqueous phase
- DT50 > 10,000 days



Results

DTPA

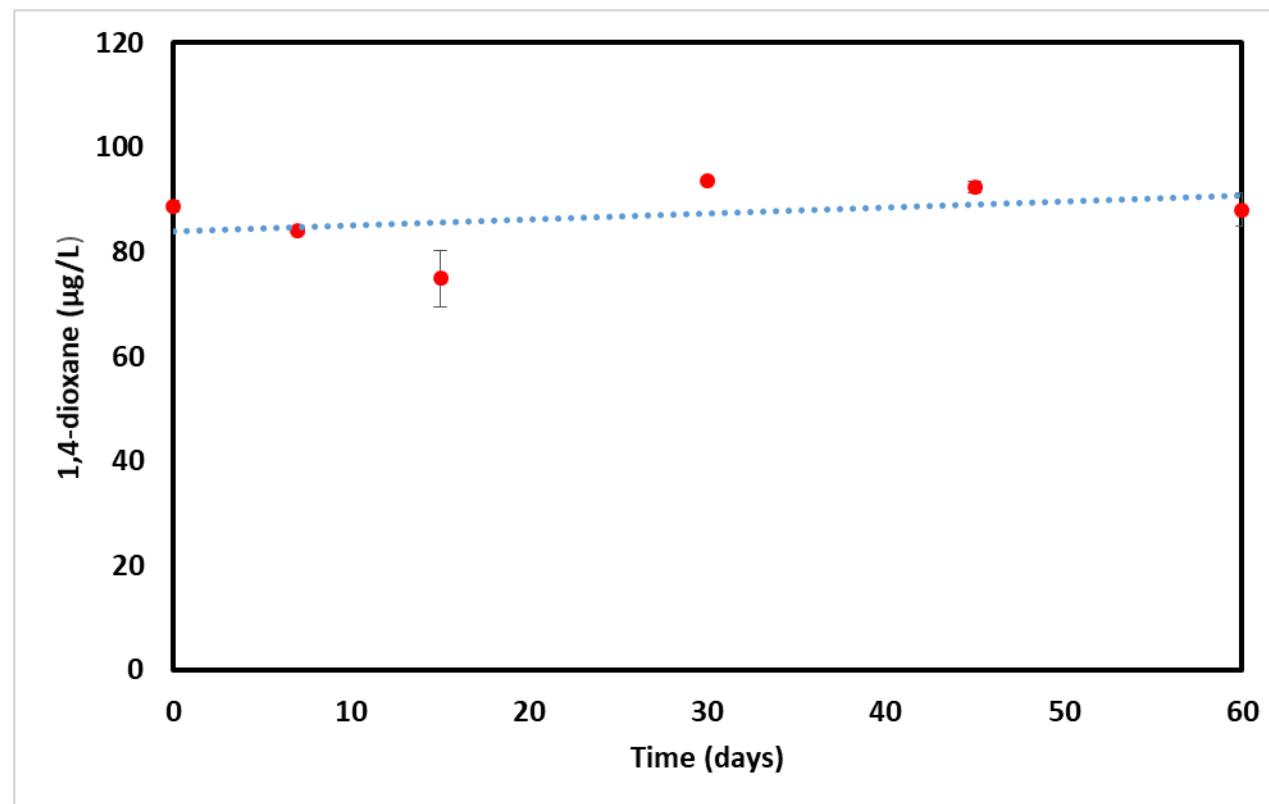
- Concentrations first seemed to increase and then decrease
- DTPA was probably primarily or ultimately degraded in surface water
- DT50 calculated on aqueous concentrations from day 5 onwards
- DT50 > 67.6 days



Results

1,4-dioxane

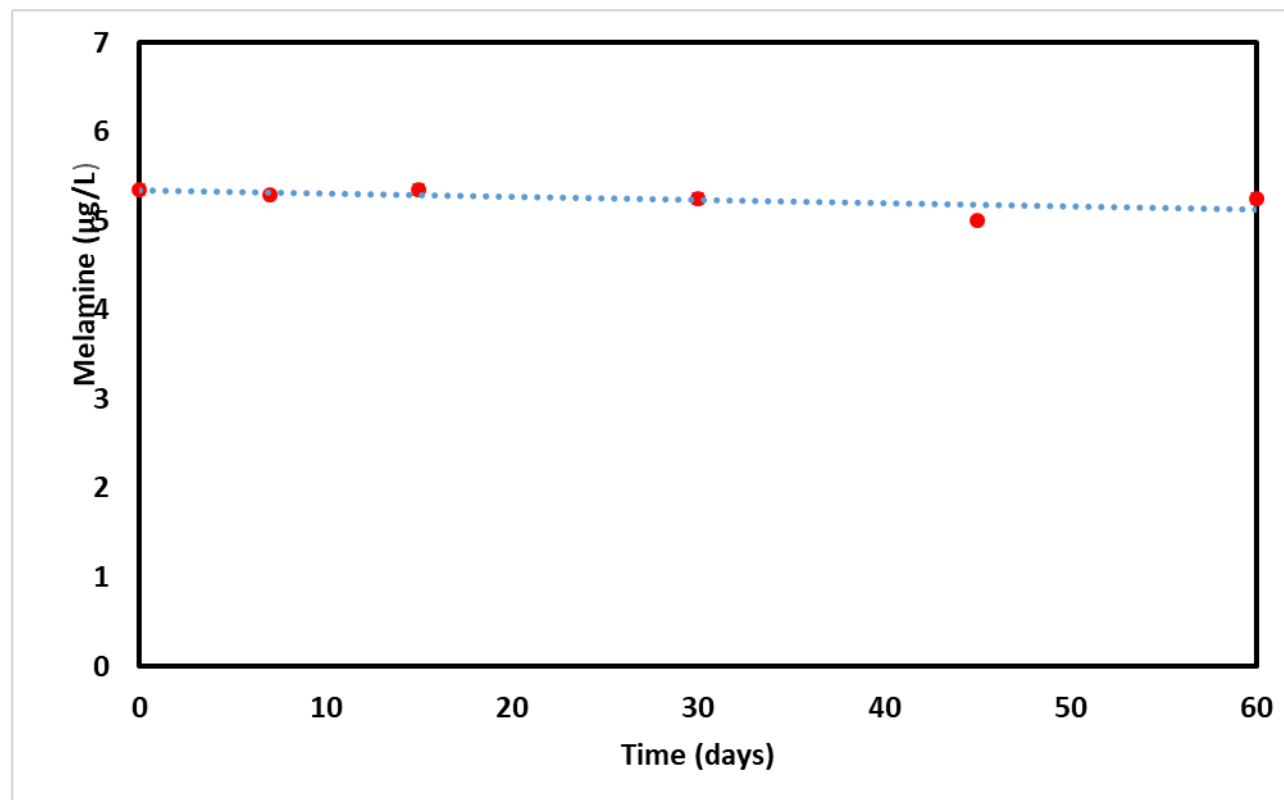
- Concentration remained stable in course of time
- Not primarily degraded in surface water
- DT50 calculated based on concentration in aqueous phase
- DT50 > 10,000 days



Results

Melamine

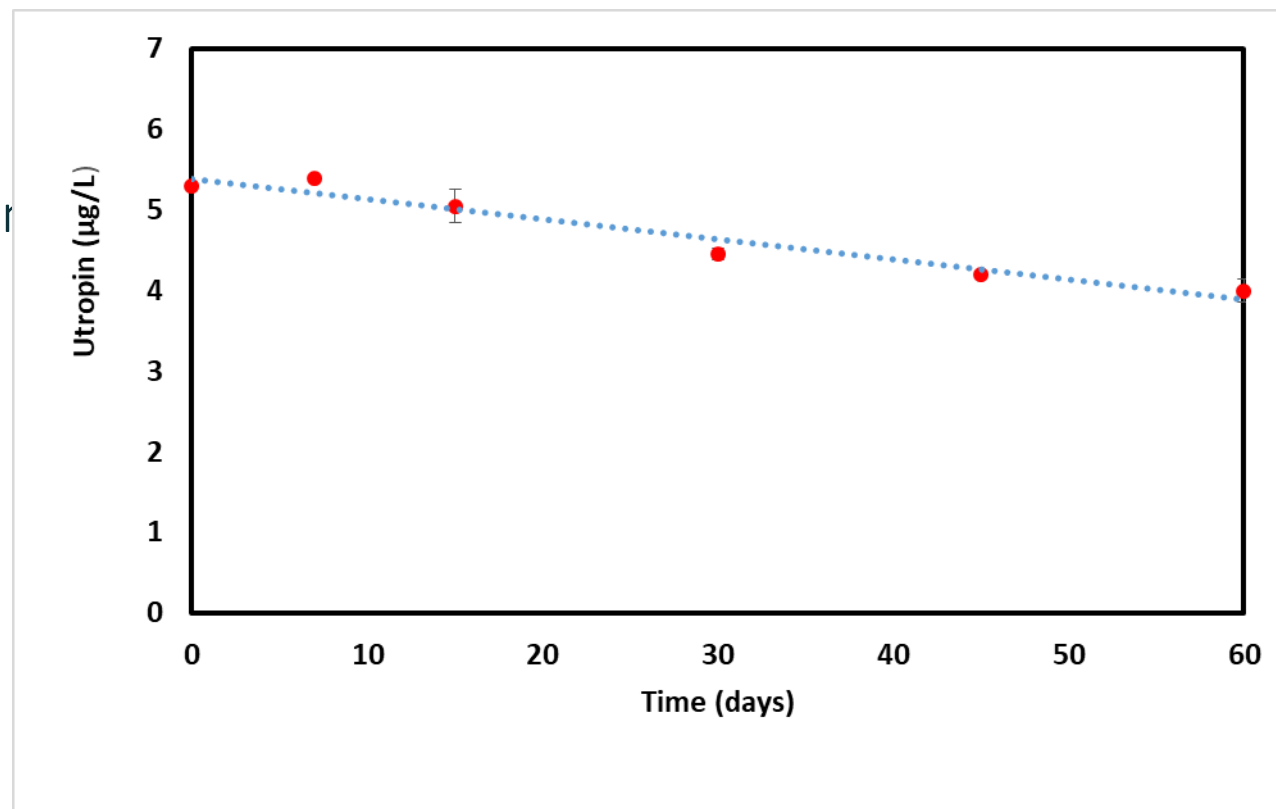
- Concentration remained stable in course of time
- Not primarily degraded in surface water
- DT50 calculated based on concentration in aqueous phase
- DT50 > 10,000 days



Results

Urotropin

- Concentrations slightly decreased in course of time
- Probably primarily or ultimately degraded in surface water
- DT50 calculated based on aqueous concentrations
- DT50 > 128 days





Conclusions

No degradation:

- Gabapentin
- 1H-benzotriazole
- Diglyme
- 1,4-dioxane
- Melamine

Slow degradation:

- DTPA
- Urotropin

Very slow or negligible abiotic and biotic degradation in surface water

All compounds $DT_{50} > 60$ days → very persistent in surface water



Conclusions

Present method is suitable for determination persistence assessment of OMP which are deemed to have low biodegradation potential

N.B. If there is a certain indication for biodegradation, transformation products should also be analytically determined.

Questions

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Congress information

- Title
- Location
- Date
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