

Determination of the two relevant impurities *iso*-temephos and temephos-oxon in formulations containing temephos by UHPLC

Vanessa LECOCQ, Régis DE BRUYNE and Marie BAES

Walloon Agricultural Research Centre (CRA-W), Agriculture and Natural Environment Department, Plant Protection Products and Biocides Physico-chemistry and Residues Unit, Rue du Bordia 11, B-5030 Gembloux, Belgium (v.lecocq@cra.wallonie.be)

Objectives

Temephos is an organophosphorus active substance used as larvicide to treat waters infested by disease-carrying insects such as mosquitoes, midges and black flies.

The WHO Pesticide Evaluation Scheme has evaluated and recommended emulsifiable concentrate (EC) and granules (GR) formulations for vector control in public health.

WHO specifications, based on data provided by two manufacturers, have been published for quality control of products containing temephos. The parameters to be checked include the determination of two relevant impurities content: "*iso*-temephos" and "temephos-oxon".

A HPLC method is proposed in the specifications for this analysis but it has been shown not strictly applicable for products from other manufacturers. Its possible optimization is the use of a recent chromatographic technique like UHPLC equipped with a DAD.



Anopheles albimanus, vector of malaria
Photo: <http://phil.cdc.gov/>

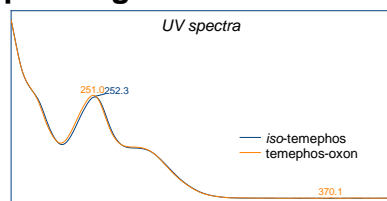
Relevant impurities

Formulations usually contain 500 g/L of temephos for EC formulations and 1% w/w of temephos for GR formulations.

	<i>iso</i> -Temephos	Temephos-oxon
Its content in formulations shall not exceed	1.4% of the temephos content (0.014% in a 1 GR formulation)	0.3% of the temephos content (0.003% in a 1 GR formulation)

Analytical challenges are: for 500 EC formulation, many adjuvants to separate, differing from one manufacturer to the others; for 1 GR formulation, low contents to measure.

Operating conditions

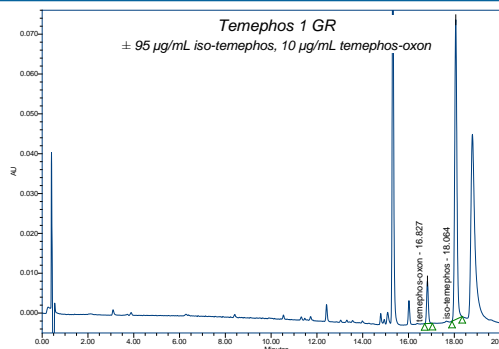
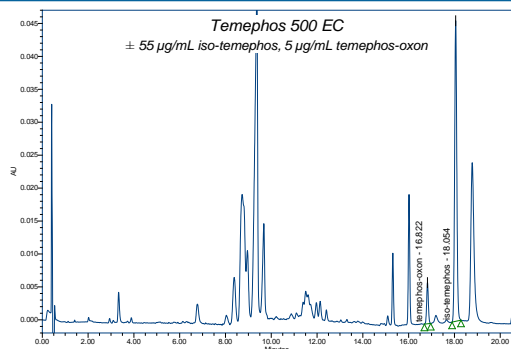


UHPLC column: Waters Acquity CSH™ Fluoro-Phenyl, 1.7 μm, 100 x 2.1 mm i.d.
Mobile phase: gradient elution
Flow rate: 0.7 mL / minute
Column temperature: 45°C
Detector wavelength: 245 nm
Injection volume: 2 μL

No change in preparation of solutions
3 determinations, 2 injections

Time [minutes]	Water + 0.2% H ₃ PO ₄ [%]	Acetonitrile [%]
0	95	5
1.5	95	5
8	80	20
15	65	35
20	65	35
21	5	95
25	5	95
26	95	5

Results



	<i>iso</i> -Temephos	Temephos-oxon
Linearity checked on 5 points between	10 - 310 μg/mL (r = 1.0000)	3 - 70 μg/mL (r = 1.0000)
7 samples analysed: > 6 of 500 EC from 4 manufacturers	detected in all the samples, measured between 0.6 - 6.3% of the temephos content	not detected in 2 samples, measured in others between 0.07 - 0.26% of the temephos content
> 1 of 1 GR	measured at 0.018% in formulation	measured at 0.002% in formulation

Conclusion

This optimization of chromatographic parameters to determine impurities leads to specificity, non-analyte interference and repeatability.