

Determination of Triazine Pesticides and their Degradates in Drinking Water by Gas Chromatography/Mass Spectrometry (GC/MS) Version 1.0



UCT Part Numbers

EC5232506
250 mg GCB, 6 mL cartridge

Summary:

This is a gas chromatography/mass spectrometry (GC/MS) method for the determination of triazine pesticides and their degradation products in finished drinking waters. Samples are pH adjusted, dechlorinated with ammonium acetate and protected from microbial degradation with 2-chloroacetamide during collection. Analytes are extracted from a **250 mL sample** using 250 mg carbon cartridges.

The following compounds can be determined using this method:

Analyte	CASRN
Atrazine	1912-24-9
Atrazine-desethyl	6190-65-4
Atrazine-desethyl-desisopropyl	3397-62-4
Atrazine-desisopropyl	1007-28-9
Cyanazine	21725-46-2
Propazine	139-40-2
Simazine	122-34-9
Terbuthylazine-desethyl	30125-63-4
Terbuthylazine	5915-41-3
Prometon	1610-18-0
Prometryn	7287-19-6
Ametryn	834-12-8



Procedure:

1. Sample Pretreatment

- a) Allow samples to reach room temperature prior to extraction.
- b) Add an aliquot of the Surrogate Primary Dilution Standards (PDS) to each sample.
- c) Fortify Laboratory Fortified Blanks, Laboratory Fortified Sample Matrices, or LFSM Duplicates, with an appropriate volume of analyte PDS and the atrazine-desethyl-desisopropyl stock standard.
- d) Cap and invert each sample several times to mix.
- e) Proceed with sample extraction using SPE carbon cartridges.

2. Cartridge Cleaning & Conditioning

- a) Set up extraction cartridges on the SPE vacuum manifold.
- b) Using low vacuum (approximately 1 to 2 inches Hg), rinse each cartridge with two 6 mL aliquots of DCM drawing completely through.
- c) Rinse each cartridge with a 6 mL aliquot of MeOH.
- d) Draw MeOH to the top of the cartridge frit.

Note: Do not let the cartridge dry after addition of MeOH.

- e) Add a 6 mL aliquot of reagent water (RW) to the cartridge.
- f) Draw RW to the top of the cartridge frit.

3. Sample Extraction

- a) Add an additional 4 mL of RW to each cartridge.
- b) Attach sample transfer lines to the cartridges. The additional volume prevents the SPE cartridge bed from going dry while the dead volume in the transfer lines is being filled.
- c) Extract 250 mL of sample at a cartridge flow rate of 10 mL/minute.
- d) Dry the cartridges under high vacuum for 10 seconds.
- e) Release vacuum, then add a 0.25 mL aliquot of MeOH to each cartridge.
- f) Draw the MeOH to waste, then dry cartridge under full vacuum for 10 minutes.

4. Sample Elution

- a) Place 15 mL conical tubes into the manifold for collection.
- b) Add 2 mL of EtOAc to the cartridge and elute dropwise.
- c) Add 2 x 6 mL aliquots of 9:1 DCM/MeOH to cartridge.
- d) Allow the cartridge beds to briefly soak in solvent, then draw the solvent through the cartridges.
- e) Dry the eluate by passing it through approximately 3 grams of anhydrous Na₂SO₄ collecting it in a 40 mL centrifuge tube. Pre-rinse the Na₂SO₄ with a 1 mL aliquot of 3:1 DCM/EtOAc.
- f) Rinse with 1 mL aliquot of 3:1 DCM/EtOAc collecting it in the centrifuge tube.
- g) The dried extracts may be stored overnight in the 40 mL tubes at -10 °C.
- h) Warm the 40 mL tubes to 35 °C in a water bath under a stream of N₂ and evaporate solvent to less than 1 mL but no less than 0.5 mL.
- i) Transfer the concentrated eluate to 1 mL volumetric tubes.
- j) Rinse the conical tube with a small volume of EtOAc, and transfer the rinse to the volumetric.
- k) Add IS solution and adjust to volume.
- l) Transfer the extracts to autosampler vials for analysis or store in a freezer ≤ -10 °C.



References:

[1] Complete details at Office of Water (MLK 140) EPA Document No. 815-R-11-002 February 2011

<http://www.epa.gov/safewater/>

DCN-905280-112

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