Method 523: 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)

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Method 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

1) **Sample Preparation**
    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 Na_2SO_4 collecting it in a 40-mL centrifuge tube. Pre-rinse the Na_2SO_4 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 ºC
    h. Warm the 40-mL tubes to 35 °C in a water bath under a stream of N_2 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 ºC

Complete details at Office of Water (MLK 140) EPA Document No. 815-R-11-002 February 2011
http://www.epa.gov/safewater/

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