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An improved method for measuring metaldehyde in surface water using liquid chromatography tandem mass spectrometry

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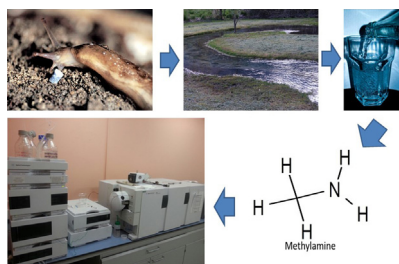
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GRAPHICAL ABSTRACT



ABSTRACT

The molluscicide metaldehyde (2,4,6,8-tetramethyl-1,3,5,7-tetraoxocanemetacetaldehyde) is an emerging pollutant. It is frequently detected in surface waters, often above the European Community Drinking Water Directive limit of 0.1 µg/L for a single pesticide. Gas chromatography mass spectrometry (GC–MS) can be used to determine metaldehyde in environmental waters, but this method requires time consuming extraction techniques prior to instrumental analysis. Use of liquid chromatography–tandem mass spectrometry (LC–MS/MS) can overcome this problem. We describe a novel LC–MS/MS method, using a methylamine mobile phase additive, coupled with on-line sample enrichment that allows for the rapid and sensitive measurement of metaldehyde in surface water. Only the methylamine adduct of metaldehyde was formed with other unwanted alkali metal adducts and dimers being suppressed. As considerably less collision energy is required to fragment the

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methylamine adduct, a five-fold improvement in method sensitivity, compared to a previous method using an ammonium acetate buffer mobile phase was achieved. This new approach offers:

- A validated method that meets regulatory requirements for the determination of metaldehyde in surface water.
- Improved reliability of quantification over existing LC–MS/MS methods by using stable precursor ions for multiple reaction monitoring.
- Low limits of quantification for tap water (4 ng/L) and river water (20 ng/L) using only 800 μ L of sample; recoveries > 97%.

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ARTICLE INFO

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Keywords: Metaldehyde, Surface water, Liquid chromatography–tandem mass spectrometry, On-line enrichment, Methylamine, Molluscicide

Method details

Reagents and standards

Acetonitrile and methanol of LC–MS grade purity were from VWR International Ltd. (Lutterworth, UK). Deuterated metaldehyde- d_{16} (>99 atom% deuterium) was from QMX Laboratories Ltd. (Thaxted, UK). Metaldehyde (99%) and methylamine (2 M) were from Sigma-Aldrich Ltd. (Gillingham, UK). Ultrapure water (18 M Ω · cm) was used throughout and was produced from an Elga Purelab Prima water system (High Wycombe, UK).

Glassware is cleaned using a 10% Decon-90 solution (Decon Laboratories Ltd., Hove, UK), then rinsed with tap water, ultrapure water and finally methanol. Metaldehyde stock solution is prepared by dissolving 25 mg metaldehyde powder in 25 mL methanol to give a concentration of 1 g/L. The solution is kept in the dark at room temperature. Subsequent dilutions in methanol are undertaken to produce a final concentration of 50 μ g/L, this solution is used to produce the aqueous calibration standards.

An internal standard stock solution (deuterated metaldehyde- d_{16}) is prepared by dissolving 10 mg of the powder in 10 mL methanol to give a concentration of 1 g/L. Subsequent dilutions in methanol are

Table 1
Mass spectrometer source conditions.

Gas Temp ($^{\circ}$ C)	250
Gas Flow (L/min)	5
Nebuliser pressure (psi)	60
Sheath gas heater ($^{\circ}$ C)	300
Sheath gas flow (L/min)	11
Capillary voltage (V)	3000
Nozzle voltage (V)	1000

Table 2
Solvent elution timetable.

Time (min)	Solvent B (%)
0	30
3	67.5
3.5	100
4.5	100
5.0	30

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