Determination of four polar pesticides in drinking water by IC-MS

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Keywords: Single quadrupole mass spectrometer, Dionex Integrion HPIC, Dionex IonPac AS19-4µm column, endothall, glyphosate, glufosinate, AMPA, ISQ EC, Dionex ADRS 600 suppressor

Goal

To develop a method to determine four pesticides (endothall, glyphosate, glufosinate, AMPA) in drinking water by coupling IC with single quadrupole mass spectrometry (IC-MS)

Introduction

Glyphosate is the active ingredient in the popular herbicide Roundup™ and is widely used for weed control in cultivated and uncultivated areas. There are concerns about its potential adverse effects on human health such as its potential carcinogenicity.[1] Although the bacteria in soil break down glyphosate into aminomethylphosphonic acid (AMPA), wastewater discharge and drinking water samples in the United States and Europe have tested positive for glyphosate.[2,3] It is highly water-soluble, and its monitoring in surface, underground, and potable waters is regulated by the United States Environmental Protection Agency (U.S. EPA) with a maximum contamination level (MCL) set at 700 µg/L. The determination of glyphosate and other polar compounds presents a difficult analytical challenge due to their ionic structures, poor volatility, and absence of chromophores. The U.S. EPA established Method 547 for the determination of glyphosate in drinking water by direct aqueous injections onto a high-performance liquid chromatography (HPLC) system with post-column derivatization and subsequent fluorescence detection. This method requires a post-column setup for analyte derivatization.[4]

Endothall is a widely used herbicide for both terrestrial and aquatic weeds. Exposure to endothall in excess of the MCL can cause illness. The U.S. EPA established the MCL for endothall in drinking water at 100 µg/L. The current analytical method described in U.S. EPA Method 548.1 (a GC-MS method) for the quantitation of endothall in water samples involves time-consuming sample preparation and derivatization.[5]

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Ion chromatography with mass spectrometry (IC-MS) is more suitable for endothall and glyphosate determinations because these pesticides and their metabolites are ionic. As a result, a direct analysis, i.e., no derivatization, is possible for these two U.S. EPA regulated compounds. An IC system coupled to an economical and simple-to-use single quadrupole mass spectrometer can be used to screen and confirm the presence of ionic pesticides. The easy-to-use Thermo Scientific™ ISQ™ EC Single Quadrupole Mass Spectrometer seamlessly integrates IC with mass spectrometery (MS), taking advantage of the strengths of both techniques. Anion exchange chromatography using eluent generation and suppressed conductivity detection provides chromatographic selectivity, analytes in the ionic form, and compatibility with MS. Electrospray ionization (ESI) is used to introduce the liquid IC stream (after suppression) as a fine spray into the MS source. The HESI-II probe improves the ESI interface by allowing the use of high temperatures and voltage to deliver better desolvation and enhanced sensitivity; thus, a make-up solvent is not needed.

The objective of the present work was to develop an IC-MS method that can simultaneously determine endothall, glyphosate, AMPA, and glufosinate. Using the method we developed, water samples were directly injected for analysis, and chromatographic separation was achieved in 25 min. The mass spectrometer was operated in selected ion monitoring (SIM) mode, allowing minimum sample cleanup and ensuring sensitive and selective quantification. Isotope labeled endothall-3,4,4,5,5,6-d_6 monohydrate, *N*-acetyl-d_3-glufosinate, and glyphosate (2-^{13}C, ^{15}N) were used as internal standards to ensure quantitation accuracy. Performance data for the method such as recovery, precision, sensitivity, and calibration range were also reported. Together these data show that the IC-MS can successfully determine the four targeted analytes in drinking water samples.

Experimental

Equipment

- A Thermo Scientific™ Dionex™ Integrion™ HPIC™ system (P/N 22153-60208) including: *
  - Eluent generator
  - Pump
  - Degasser
  - Conductivity detector
  - Second 6-port injection valve (P/N 22153-62027) used as a diverter valve
  - Thermo Scientific™ Dionex™ IC PEEK Viper Fitting Kit (P/N 088798)
  - Column oven temperature control
  - Detector-suppressor compartment temperature control
  - Tablet control

* This method can also be run on a Thermo Scientific™ Dionex™ ICS-5000+ system or Thermo Scientific™ Dionex™ ICS-6000 dual system using the second pump to deliver suppressor external water.

- Thermo Scientific™ Dionex™ AS-AP Autosampler (P/N 074926), with 250 µL syringe (P/N 074306), 1200 µL buffer line assembly (P/N 074998), 10 µL injection loop and 1.5 mL vial trays (P/N 074936)
- ISQ EC single quadrupole mass spectrometer (P/N ISQEC-IC) including Thermo Scientific™ HESI-II probe (P/N 70005-60155)
- Thermo Scientific™ Dionex™ AXP-MS auxiliary pump (used to deliver suppressor external water) (P/N 060684)
- Nitrogen generator with capacity for 3L/min flow at 100 psi (110 V: P/N 1R77606-1120; 230 V: P/N 1R77606-1230)

Software

Thermo Scientific™ Chromeleon™ Data System (CDS) software, version 7.2.9

Consumables

- Thermo Scientific™ Dionex™ EGC 500 KOH Cartridge (P/N 075778)
- Thermo Scientific™ Dionex™ CR-ATC 600 Continuously Regenerated Anion Trap Column (P/N 088662)
- Thermo Scientific™ Dionex™ ADRS 600 Anion Dynamically Regenerated Suppressor, 2 mm (P/N 088667)
- Dionex AS-AP Autosampler Vials 10 mL (P/N 074228)
- Fisherbrand™ Narrow-mouth Field Sample Bottles, high density polyethylene (HDPE), 125 mL, 250 mL sizes for storage of standards and samples (Fisher Scientific P/N 02-895A, B)

Reagents and standards

- Deionized (DI) water, Type I reagent grade, 18 MΩ·cm resistivity or better
- Sodium and potassium salts, A.C.S. reagent grade or better, for preparing anion standards
- Ethylenediamine, 99% (Sigma-Aldrich)
- Endothall solution, 50 µg/mL in Water (Fisher Scientific P/N US-PPS-2101)
- Glyphosate, 1000 µg/mL in DI water (Restek P/N 32426)
- Aminomethylphosphonic acid (AMPA), (Alfa Aesar P/N L09833, 250 mg)
- Glufosinate-ammonium (Chem Service P/N N-12111-100 MG)
- Glyphosate (96 chemical purity) (2-^{13}C, 99%; ^{15}N, 98+%) 100 µg/mL in H_2O (Cambridge Isotope Laboratories P/N CNLM-4666-1.2)
- Endothall-3,4,4,5,5,6-d_6 monohydrate (CDN Isotopes P/N D-7289, 10 mg)
- *N*-acetyl-d_3-glufosinate (Sigma-Aldrich P/N 05567-5MG)

Samples

Three residential drinking water samples were collected from different cities in the San Francisco Bay Area, California (DW#1-3).

Chromatographic conditions

Parameter	Setting
Columns	Thermo Scientific™ Dionex™ IonPac™ AG19-4µm guard column, 2 × 50 mm (P/N 083225) Thermo Scientific™ Dionex™ IonPac™ AS19-4µm analytical column, 2 × 250 mm (P/N 083223)
Eluent	14 mM KOH from 0 to 10 min 14–80 mM KOH from 10 to 15 min 80 mM from 15 to 22 min 14 mM from 22.1 to 25 min
Eluent source	Dionex EGC 500 KOH cartridge with Dionex CR-ATC 600
Flow rate	0.4 mL/min
Injection volume	10 µL in Push-Full injection mode
Column temperature	30 ˚C
Detection 1	Suppressed conductivity
Suppressor	Dionex ADRS 600 (2 mm) Suppressor, external water mode (flow 0.4 mL/min), 80 mA current
Detection/suppressor compartment	20 °C
Cell temperature	35 ˚C
Background conductance	<1 µS/cm
System backpressure	~3800 psi (100 psi = 689.5 kPa)
Noise	<1 nS/cm
Run time	25 min
Detection 2	Mass spectrometry
MS detector	ISQ EC single quadrupole MS
Ionization interface	Electrospray ionization (ESI), negative mode
Diverter valve switch time	0–8 min to waste, 8–25 min to MS
Sheath gas pressure	40 psi
Aux gas pressure	2 psi
Sweep gas pressure	1 psi
Source voltage	-2500 V
Vaporizer temp.	450 ˚C
Ion transfer tube temp.	350 ˚C
Chrom. filter peak width	Off
Scan mode	Table 1

Table 1. MS scan mode

Scan name	Mass list (amu)	Dwell or scan time (s)	SIM width (amu)	Ion polarity	Spectrum type	Source CID voltage (V)
AMPA	110	0.2	0.3	Negative	Centroid	10
Endothall	185	0.2	0.3	Negative	Centroid	10
Endothall IS	191.1	0.2	0.3	Negative	Centroid	10
Glufosinate	180	0.2	0.3	Negative	Centroid	10
Glufosinate IS	225.2	0.2	0.3	Negative	Centroid	10
Glyphosate	168	0.2	0.3	Negative	Centroid	10
Glyphosate IS	170.1	0.2	0.3	Negative	Centroid	10

System preparation and setup

Figure 1 shows the flow diagram of the IC-MS system. The Dionex Integrion HPIC system is plumbed as a Reagent-Free™ ion chromatography (RFIC™) system using eluent generation following the Dionex Integrion installation and operator manuals.[6] Install the suppressor in external water mode using a Dionex AXP-MS pump to provide the DI water regenerant.[7] The Dionex AXP-MS pump can be added in the instrument configuration, and thus be controlled by Chromeleon software. The ISQ-EC is installed according to the installation guide.[8]

A 6-port diverter valve is placed between the conductivity detector (CD) and the mass spectrometer. The diverter valve can be operated in two positions (Figure 2). A small piece of red PEEK™ tubing called a "jumper" is installed in the IC diverter valve connecting port 1 to port 3. In position A, eluent flows from the CD to the mass spectrometer, and the AXP delivers water to the suppressor Regen In. In position B, eluent flow is in recycle mode for the suppressor, and the AXP delivers water to the mass spectrometer. Configure the diverter valve in the instrument method script editor to divert everything to waste except the compounds of interest. Detailed instructions for configuring the IC-MS system are in Technical Note 72611.[9]

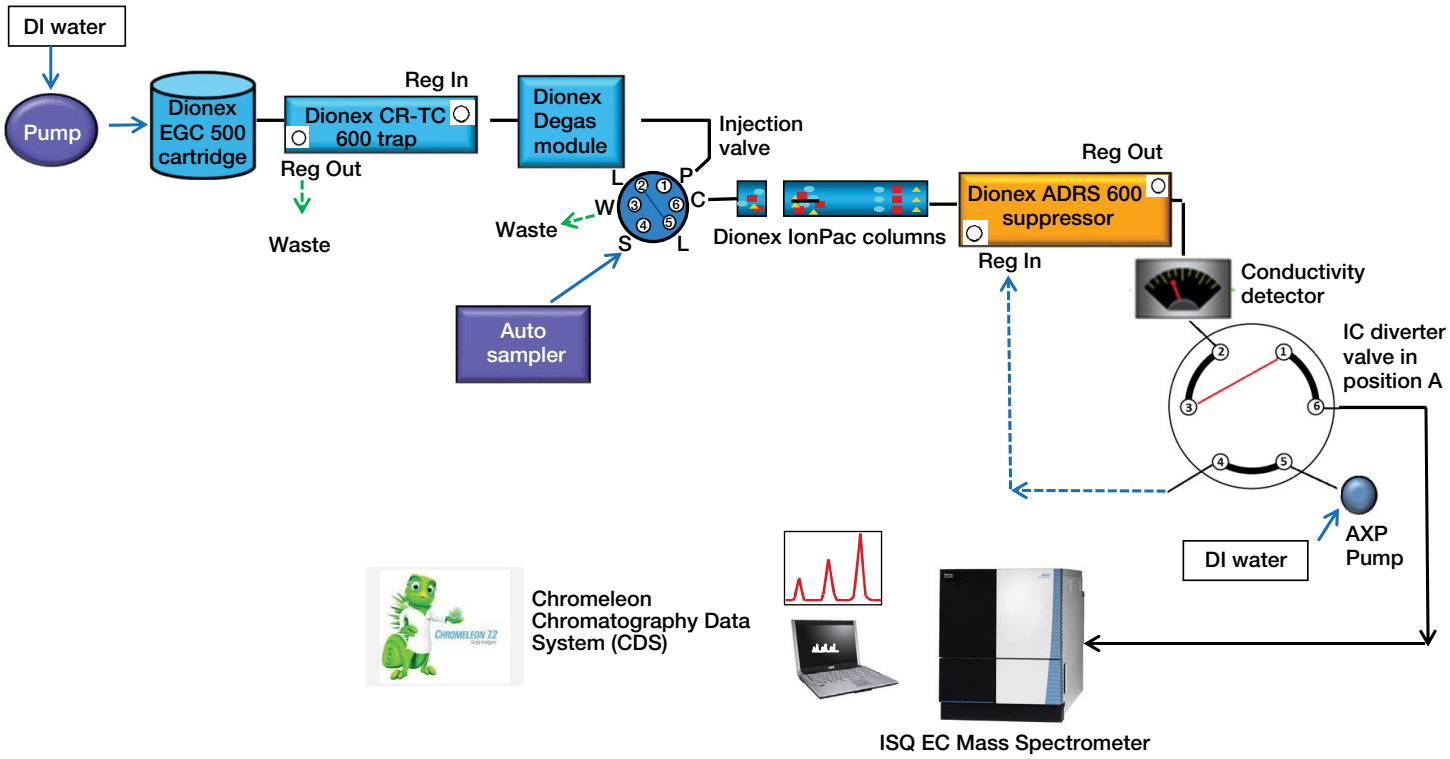

Figure 1. Flow diagram for IC-CD/MS with diverter valve in "A" position

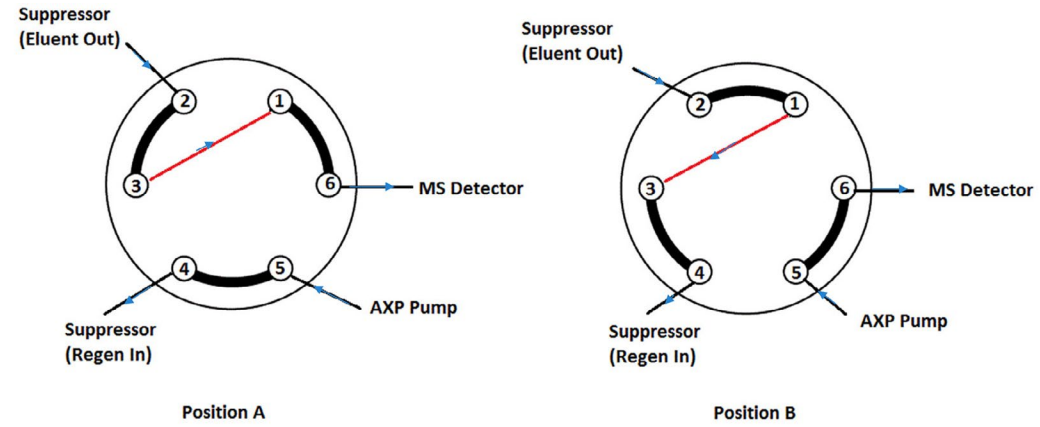

Figure 2. Diverter valve position

Precautions

1. Allow the system to equilibrate until the total conductivity is <1.5 µS/cm and then it is safe to connect the IC flow to an operating mass spectrometer. In other words, keep the divert valve in position B, with flow from the Dionex AXP-MS Auxiliary pump to the mass spectrometer until the background conductivity is below 1.5 µS/cm. This can prevent the non-volatile eluent from precipitating inside the ESI capillary.
2. The column used in this application has an inner diameter of 2 mm. Red PEEK tubing (0.005 in. i.d.) from the CD to the MS detector should be used to improve MS sensitivity. However, keep this tubing as short as possible to minimize backpressure on the suppressor. High backpressure can cause irreversible damage to the suppressor.
3. The mass spectrometer needs to be "baked out" when the system is idle for over a day or when the MS peak area reproducibility becomes poor. To bake out the mass spectrometer, set the vaporizer temperature to 500 ˚C, ion transfer temperature to 400 ˚C, sheath gas to 50 psi, aux gas to 10 psi, sweep gas to 1 psi, and then deliver DI water to the mass spectrometer at 0.1 mL/min with the Dionex AXP-MS pump. Allow the system to bake out for at least 2 h.

Preparation of solutions and reagents

Common anions stock standard solutions

Stock standard solutions (1000 mg/L) can be prepared by dissolving the appropriate amounts of the required analytes in 100 mL of DI water according to Table 2.

Stock standards for most anions are stable for at least six months at 4 ˚C. The chlorite standard is only stable for two weeks when stored protected from light at 4 ˚C. The nitrite and phosphate standards are only stable for one month when stored at 4 ˚C.

AMPA stock standard solution (1000 mg/L)

An AMPA stock standard solution can be prepared by dissolving 10 mg of AMPA in 10 mL of DI water.

Glufosinate stock standard solution (1000 mg/L)

A glufosinate stock standard solution can be prepared by dissolving 10 mg of the glufosinate-ammonium in 10 mL of DI water.

Endothall-3,4,4,5,5,6-d_6 monohydrate internal standard (ISTD) stock solution (1000 mg/L)

Prepare endothall internal stock standard solution by dissolving 10 mg of endothall-3,4,4,5,5,6-d_6 monohydrate in 10 mL of DI water.

N-acetyl-d_3-glufosinate internal standard (ISTD) stock solution (1000 mg/L)

Prepare glufosinate internal stock standard solution by dissolving 5 mg of *N*-acetyl-d_3-glufosinate in 5 mL of DI water.

Four pesticides working solution mixture (1 mg/L)

Prepare 1 mg/L of standard working solution mixture (endothall, glufosinate, glyphosate, AMPA) by diluting the standard stock solution with DI water. (Table 3)

Table 2. Masses of compounds used to prepare 100 mL of 1000 mg/L ion standards

Analyte	Compound	Amount (mg)
Fluoride	Sodium fluoride (NaF)	221.0
Chlorite	Sodium chlorite ($NaClO_2$), 80%	167.6
Bromate	Sodium bromate ($NaBrO_3$)	118.0
Chloride	Sodium chloride (NaCl)	164.9
Nitrite	Sodium nitrite ($NaNO_2$)	150.0
Chlorate	Sodium chlorate ($NaClO_3$)	127.5
Bromide	Sodium bromide (NaBr)	128.8
Nitrate	Sodium nitrate ($NaNO_3$)	137.1
Sulfate	Sodium sulfate (Na_2SO_4)	147.9
Phosphate	Potassium phosphate, monobasic (KH_2PO_4)	143.3
Carbonate	Sodium carbonate (Na_2CO_3)	176.6
Perchlorate	Sodium perchlorate ($NaClO_4$)	123.1

Table 3. Volume of stock used to prepare 100 mL of a four pesticides mixture (1 mg/L)

Analyte	Stock concentration (mg/L)	Stock source	Stock volume (µL)
Endothall	50	Commercially available	2000
Glyphosate	1000	Commercially available	100
Glufosinate	1000	Prepare from powder	100
AMPA	1000	Prepare from powder	100

Three internal standard working solutions (ISTD) mixture (1 mg/L)

Prepare the internal standard working solution mixture (endothall-3,4,4,5,5,6-d_6 monohydrate, *N*-acetyl-d_3-glufosinate, glyphosate (2-^{13}C, ^{15}N) by diluting the internal standard stock solutions with DI water. (Table 4)

Table 4. Volume of stock used to prepare 100 mL of three pesticides isotope internal standard mixture (1 mg/L)

Analyte	Stock concentration (mg/L)	Stock source	Stock volume (µL)
Endothall-3,4,4,5,5,6-d_6 monohydrate	1000	Prepare from powder	100
N-acetyl-d_3-glufosinate	1000	Prepare from powder	100
Glyphosate (2-^{13}C, ^{15}N)	100	Commercially available	1000

Working standard solutions

Diluted working standard solutions were prepared using the four pesticides working solution (1 mg/L). The mixed calibration standard solutions were 0.5, 1, 2, 5, 10, 25, 50, and 100 µg/L.

Laboratory synthetic sample matrix (LSSM)

Additional anions listed in Table 2 were used to prepare a LSSM containing 250 mg/L chloride, 20 mg/L nitrate, 150 mg/L carbonate, and 250 mg/L sulfate.

Preservation solution

Dilute 2.8 mL of ethylenediamine (EDA) to 25 mL with DI water according to section 7.4 in U.S. EPA Method 300.1[10] to prepare a 100 mg/mL solution. Preserve the standards or samples by adding 50 µL of the EDA preservation solution per 100 mL of sample.

Sample preparation

Drinking water samples are treated with the EDA preservation solution and kept in HDPE bottles.

Standard and sample with ISTD

Add 10 µL of ISTD (1 mg/L) to each 1 mL of calibration standard or sample.

Results and discussion

Separation

The Dionex IonPac AS19-4µm hydroxide-selective anion-exchange column is a high capacity and high-resolution column, which are critical factors for the determination of pesticides at the low µg/L concentrations in samples containing high concentrations of common anions such as chloride, nitrate, and sulfate.[11] Figure 3 shows a separation of common anions and four pesticides within 25 min using the Dionex IonPac AS19-4µm column. The top chromatogram displays the CD profile of all anions.

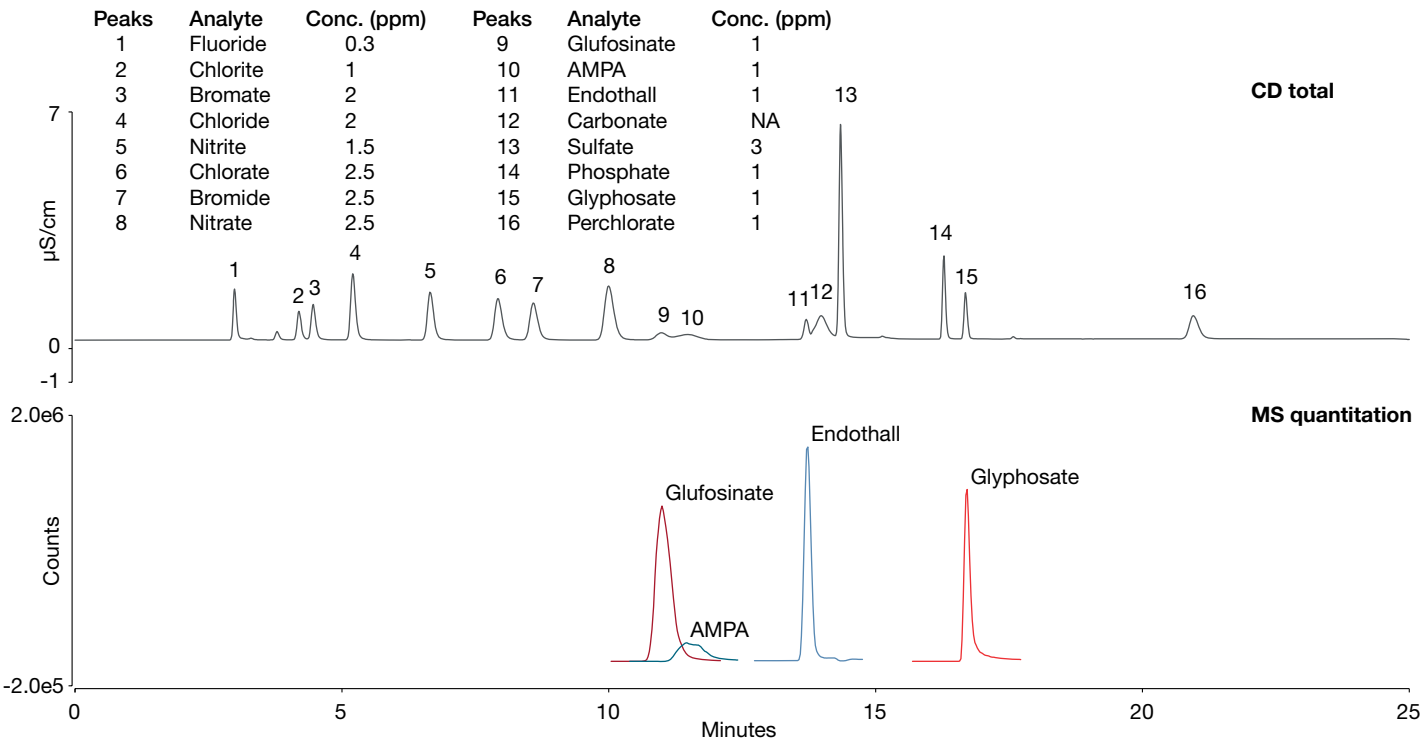

Figure 3. Separation of common anions and four pesticides

The bottom chromatogram displays the MS channel of the four analytes of interest, glufosinate, AMPA, endothall, and glyphosate. As Figure 3 shows, glufosinate, AMPA, endothall, and glyphosate were resolved from the common inorganic anions.

A delay time of 0.17 min is applied to the MS channel to match the retention time in the CD channel. The delay time is the time required for the analyte to travel from one detector to another when they are in series. Here, the analyte goes through the CD cell before going into the mass spectrometer. The delay time can be set in the Chromeleon software in: Processing Method-Advanced Settings-Delay Time.

Limits of detection (LOD) and method detection limits (MDL), lowest concentration minimum report level (LCMRL), DL (detection limit)

Several approaches for determining the detection limit are possible. The LOD method is based on the signal-to-noise ratio (S/N). Determination of the S/N is performed by comparing measured signal from a low concentration standard and blank samples and establishing the minimum concentration at which the analyte can be reliably detected. A S/N = 3 is used for estimating the limit of detection (LOD)[12]. In this study, the baseline noise was first determined by measuring the peak-to-peak noise in a representative 1-min segment of the baseline where no peaks elute, but close to the peak of interest. The signal was determined from the average height of three injections of standard (0.5 µg/L).

The MDL method is based on the standard deviation of the response. MDLs were determined by performing seven replicate injections of standards at a concentration three to five times the estimated instrument detection limits (endothall 1 µg/L, AMPA 1 µg/L, glufosinate 0.5 µg/L, glyphosate 0.5 µg/L).

Calculate the MDL as follows: MDL = (t) × (S), where t = Student's value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom (t = 3.14 for seven injections), S = standard deviation of the replicate analysis. The estimates of MDL and LOD for glufosinate, AMPA, endothall, and glyphosate are summarized in Table 5.

The lowest concentration minimum report level (LCMRL) was also determined. The U.S. EPA provides a statistical approach for determining a single-laboratory LCMRL using linear regression and prediction intervals.[13] The LCMRL is the lowest true concentration at which the future recovery is predicted to have a 99% confidence between 50 and 150% recovery. In this study, four pesticide mixtures at concentrations of 0.2, 0.3, 0.4, 0.5, 1, and 2 µg/L were prepared. Four concentration levels for each analyte (Table 5) and five replicate injections were analyzed. The data from these injections were inserted into the system program provided on the U.S. EPA website, which produced an LCMRL and DL as shown in Table 5.[13]

Figure 4 shows the chromatographic profile of a 1 µg/L calibration standard with MS detection. The four anions of interest are detected with good sensitivity.

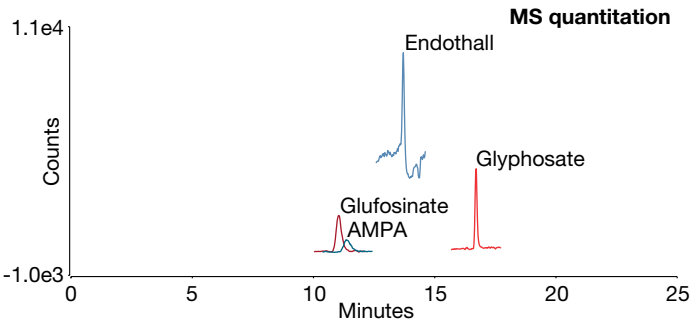

Figure 4. Four pesticides standard (1 ppb) in DI water

Table 5. Method detection limits (MDL) and limits of detection (LOD), DL, and LCMRL

	LOD (µg/L)	MDL (µg/L)	DL (µg/L)	LCMRL (µg/L)	Standards used to determine LCMRL (µg/L)
Endothall	0.33	0.33	0.36	0.40	0.4, 0.5, 1, 2
Glyphosate	0.08	0.10	0.11	0.34	0.2, 0.3, 0.4, 0.5
AMPA	0.31	0.32	0.43	0.75	0.4, 0.5, 1, 2
Glufosinate	0.16	0.13	0.13	0.31	0.3, 0.4, 0.5, 1

Calibration

Calibration standard mixtures (glufosinate, AMPA, endothall, and glyphosate) in the range of 0.5 to 100 µg/L were prepared in DI water, and then EDA was added as a preservative. The three ISTD mixture was spiked into each calibration standard at 10 µg/L. The internal standard method provides a means to account for losses in ionization efficiencies due to components of the matrix that may compete for ion formation in the source. The use of isotopically labeled internal standards ensures that both compound identification and compound quantification are of the highest degree of precision and accuracy possible. Table 6 summarizes the calibration results. Calibration curves were generated using internal standard calibration (Figure 5). The coefficient of determination is greater than 0.999 for all components.

Sample analysis

Three residential drinking waters were collected from three different cities in San Francisco Bay Area, California. Drinking water was analyzed directly without filtration. The four pesticides were not detected in all three drinking water samples. Figure 6 shows the MS chromatographic profiles of drinking water #1 spiked with the 10 µg/L four pesticides mixture. Figure 7 shows the MS chromatographic profiles of drinking water #1 spiked with the 1 µg/L four pesticides mixture.

Table 6. Calibrations

	Range (µg/L)	Calibration type	Internal standard (ISTD)	ISTD concentration µg/L	Coefficient of determination (r^2)	Source CID voltage (V)
Endothall	0.5–100	Internal, quadratic	Endothall-3,4,4,5,5,6-d_6 monohydrate	10	1.000	10
Glyphosate	0.5–100	Internal, quadratic	Glyphosate (2-^{13}C, ^{15}N)	10	1.000	10
AMPA	0.5–100	Internal, quadratic	*N*-acetyl-d_3-glufosinate	10	1.000	10
Glufosinate	0.5–100	Internal, quadratic	*N*-acetyl-d_3-glufosinate	10	0.9994	10

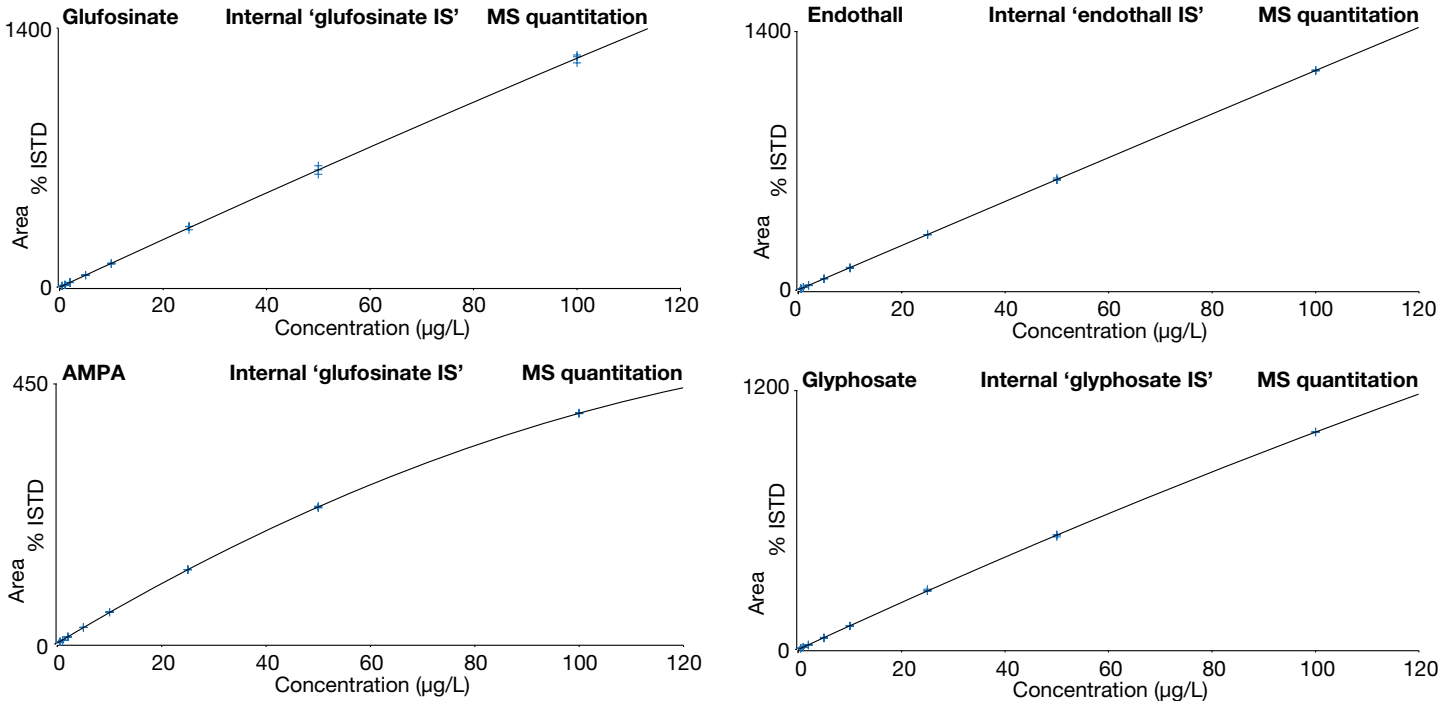

Figure 5. Calibration

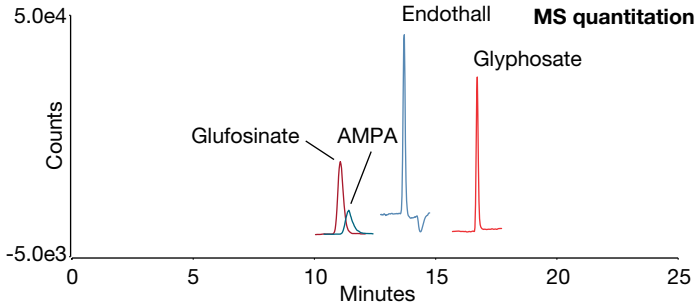

Figure 6. Sample 1 spiked with 10 ppb each of four pesticides

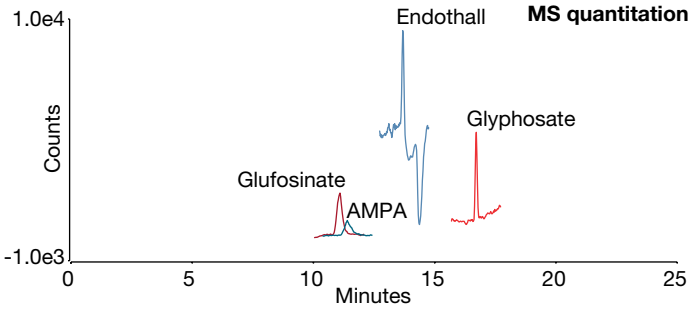

Figure 7. Sample 1 spiked with 1 ppb each of four pesticides

Matrix effects

To examine the influence of a high concentration anion matrix on the measurements, a laboratory synthetic sample matrix (LSSM) was prepared. The LSSM is a solution of common anions prepared at high concentration (250 mg/L chloride, 20 mg/L nitrate, 150 mg/L carbonate, 250 mg/L sulfate) relative to their typical occurrence in drinking water. A four pesticides mixture with a concentration of 10 µg/L was prepared using this LSSM. Figure 8 shows the comparison of chromatograms of four pesticides spiked in DI water, Drinking water #1, and LSSM. For glufosinate and AMPA, almost no matrix dependency was observed. However, endothall and glyphosate were strongly affected by the high ionic strength of the LSSM, as sulfate, one of the major anionic components, elutes in the vicinity of the pesticide apparently causing ion suppression effects in the MS. Table 7 shows the retention time and peak area of four pesticides in three matrices. Retention time shifts 0–0.43 min in LSSM matrix and peak area decreases 4.55–44.6% in LSSM. (Table 7). Therefore, it is important to use an internal standard to account for losses in ionization efficiencies due to components of the matrix that may compete for ion formation in the source. It should be noted that the LSSM was selected to simulate very high anion concentrations in drinking water. Most drinking water samples have lower concentrations of the major anions.

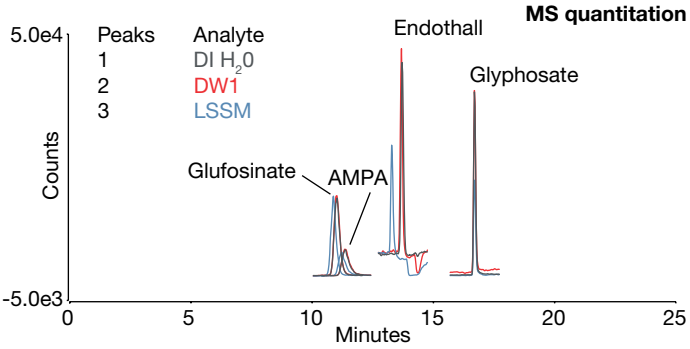

Figure 8. Pesticides (10 µg/L) in DI water vs. DW1 vs LSSM

Table 7. Matrix effect

	Retention time				MS peak area			
Component	Glufosinate	AMPA	Endothall	Glyphosate	Glufosinate	AMPA	Endothall	Glyphosate
DI H20	11.02	11.37	13.71	16.69	4380	1959	4838	3850
DW1	11.02	11.35	13.68	16.69	4589	2007	4722	3853
LSSM	10.88	11.23	13.28	16.69	4181	1753	2738	2132

Method accuracy

Method accuracy was evaluated through recovery studies using drinking water samples. Table 8 shows recoveries of four pesticides spiked in water samples. The recoveries for four pesticides in the three samples were in the range of 94–105%.

Precision

The precision of the method was determined by triplicate injections of the 10 µg/L calibration standard on three separate days. As shown in Table 9, the calculated peak area precision varied from 0.47 to 2.43% with retention time precision <0.24% for all target anions. The high precision of this method is consistent with results typically obtained with an RFIC system.

Table 8. Recoveries of pesticides spiked in drinking water samples and LSSM

Sample	Spike level (µg/L)	Glufosinate	AMPA	Endothall	Glyphosate
Drinking water #1	1	104	97.7	102	105
	5	102	103	95.6	100
	10	99.0	102	99.4	100
Drinking water #2	1	100	97.2	100	104
	5	97.7	100	96.1	100
	10	98.4	102	97.1	101
Drinking water #3	1	98.7	97.8	100	98.4
	5	95.6	96.0	96.8	97.9
	10	93.6	93.6	94.0	99.1
LSSM	1	96.6	96.8	97.3	103
	5	94.2	96.1	97.8	100
	10	91.3	95.6	97.2	93.7

Table 9. Retention time and peak area precisions

Component	Retention time (RSD)	MS relative peak area to ITSD (RSD)
Glufosinate	0.12	1.98
AMPA	0.24	0.48
Endothall	0.10	2.43
Glyphosate	0.08	0.47

Conclusion

This study describes the simultaneous direct determination of endothall, glyphosate, AMPA, and glufosinate in drinking water by IC-MS. The four pesticides can be determined sensitively and accurately using a Dionex IonPac AS19-4µm column and an ISQ EC single quadrupole mass spectrometer. The Reagent-Free ion chromatography system provides excellent reproducibility, thereby yielding greater quantification accuracy and consistently reliable results.

The developed method has many benefits in comparison with currently approved U.S. EPA methods for endothall and glyphosate in drinking waters. First, one method can replace two methods. Second, due to direct injection there is no long and laborious sample preparation. The method is sensitive, fast, and avoids errors that may result from sample preparation. Third, this method also determines two major glyphosate degradation products. Adopting this method gives routine laboratories the potential to reduce cost, provide more reliable results, and increase sample throughput.

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