

Introduction

Groundwater is an important component in many industrial processes as well as irrigating our crops and recharging lakes, rivers and wetlands. Groundwater supplies drinking water for 51% of the total U.S. population and 99% of its rural population. Unfortunately, groundwater is susceptible to pollutants due to the widespread use of pesticides and fertilizers. Traditionally, syringe filtration or centrifugation have been used to remove particulates and reduce possible matrix interference prior to LC/MS analysis. However, these techniques are time consuming, adversely impact reproducibility and quantification. We investigated the potential for streamlining sample preparation method for the analysis of Prosulfuron and its metabolites in ground water using the Thomson Standard|FV compared to syringe filtration and centrifugation.

Experimental

Instrument Parameters:

HPLC: Agilent 1290 UPLC System

Analytical Column: Waters Acquity HSS T3, 2.1 x 150 mm, 1.8 μm

Column Temperature: 50 °C

Injection Volume: 20 μL

Mobile Phase A: 0.1% Formic Acid in HPLC Water

Mobile Phase B: 0.1% Formic Acid in CAN

Flow Rate: 0.6 mL/min

Gradient:

Time	Flow Rate	A(%)	B(%)	mL/min
0		0.6	99	1
0.5		0.6	99	1
2.4		0.6	40	60
4.25		0.6	35	65
4.26		0.6	0	100
5.25		0.6	0	100
5.26		0.6	99	1
5.75		0.6	99	1

Mass Spectrometer: Agilent 6490 Triple Quad

Interface: ElectroSpray Ionization (ESI)

Gas Flow: 14L/min

Temperature: 200°C

Sample Prep Procedure:

Step 1

- For recovery sample: fortify one control ground water sample at 0.1 ppb in a 10 mL volumetric flask.
- For incurred Water sample with incurred residue follow directly the steps below.

Step 2.

Prepare experimental samples in triplicate:

Exp. 1) Thomson Standard|FV, 0.2 um PTFE Filtration Vials:

a. Take 0.45 mL aliquots of the fortified control (step 1) and transfer into separate Thomson Filtration Vials.

b. Filter the samples by depressing the plunger completely.

Exp. 2) 0.2 μm PTFE 25mm syringe filter attached to a syringe:

a. Take 1.0 mL aliquots of the fortified control and field sample (step 1) and transfer into separate syringe filters.

b. Pass samples through the syringe filter and into autosampler vials.

Exp. 3) Centrifuge with spin column:

a. Centrifuge remainder of the 2 samples @ 3200 rpm for 5 minutes.

b. Transfer 1.0 mL aliquots into autosampler vials.



Step 3.

Samples ready for LCMSMS analysis.

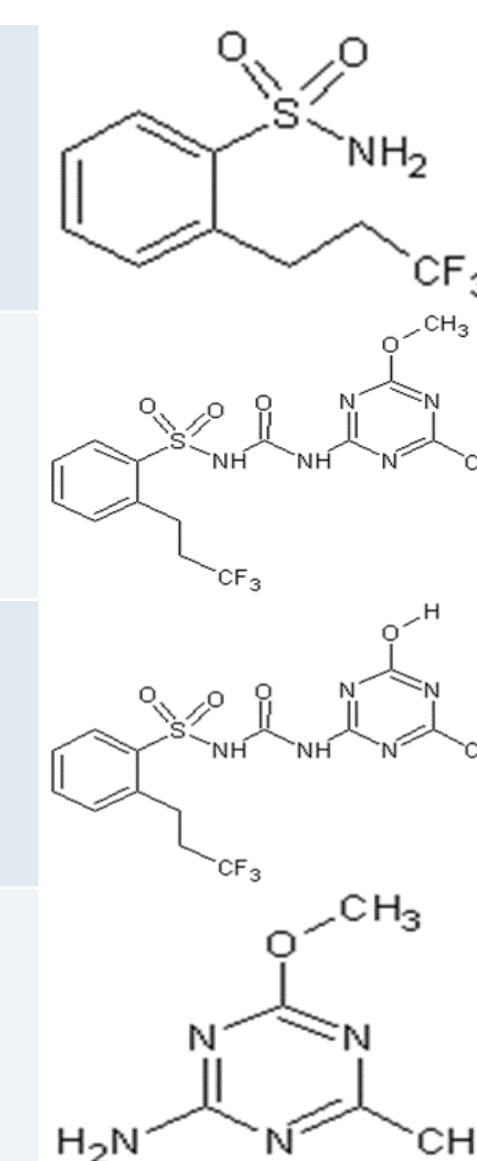
Pesticides Analyzed

Prosulfuron (CGA152005) - 1-(4-methoxy-6-methyl-biazin-2-yl)-3-[2-(3,3,3-trifluoropropyl)-phenylsulfonyl]-urea, CAS #: 94125-34-5

CGA300406 -1-(4-hydroxy-6-methyl-1,3,5-triazin-2-yl)-3-[2-(3,3,3-trifluoropropyl) phenylsulfonyl]-urea

CGA159902 -2-(3,3,3-trifluoropropyl) phenylsulfonylurea, CAS #: 94125-42-5

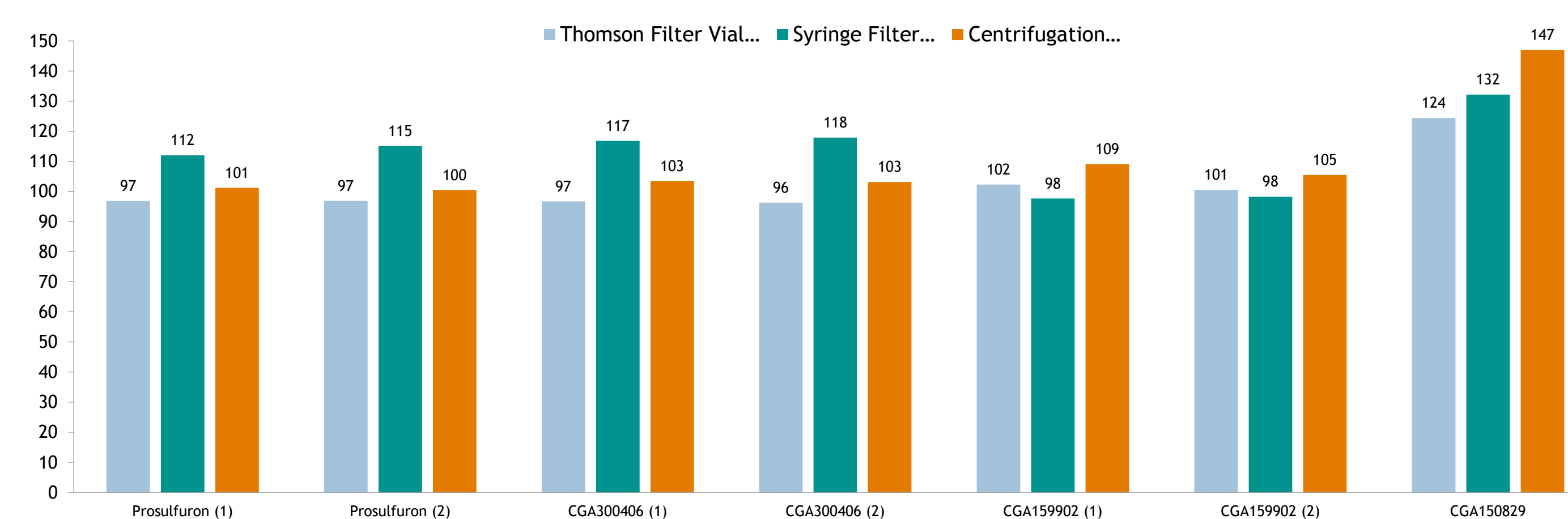
CGA150829 -2-amino-4-hydroxy-6-methyl-1,3,5-triazine, CAS #: 1668-54-8



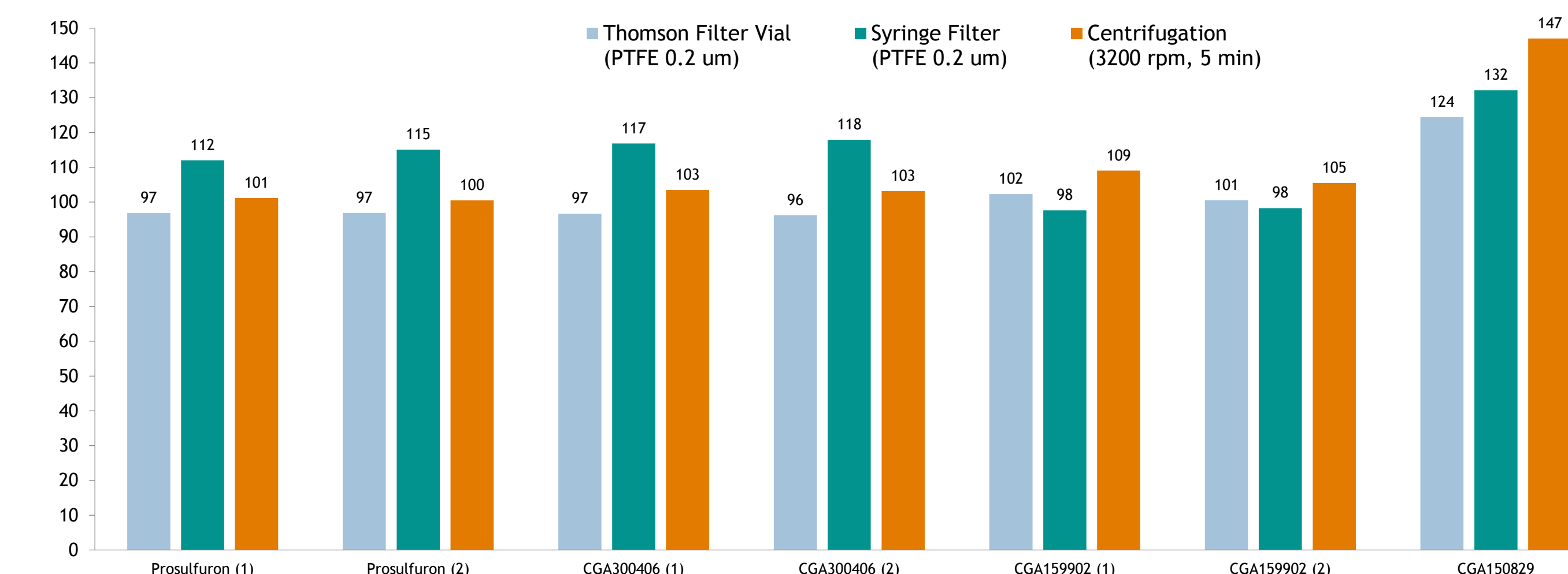
Results



Comparison of Recoveries for Ground Water Fortified with Prosulfuron and Metabolites at 0.1 ppb



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The clean-up method for ground water for the analysis of Prosulfuron and three of its metabolites was evaluated using Thomson Standard|FV, 0.2um PTFE; Syringe and 25mm filter, 0.2um PTFE and spin column, 0.2um PTFE. Recoveries were calculated for both spiked and incurred samples using a calibration curve, 0.002ng/mL - 10ng/mL. Ground water samples were run in triplicate at 0.1ppb and 4.5ppb.

- Thomson Standard|FV yielded excellent linearity and replicates with a Standard Deviation of 0.1.
- Syringe and 25mm filter yielded good linearity and replicates with a Standard Deviation of 23.
- Centrifugation with a spin column yielded excellent linearity and replicates with a Standard Deviation of 2.0.

Conclusion

- Thomson Standard Filter Vials showed slight improvement over centrifugation and significant improvement over syringe filters in mean concentration and Standard Deviation.
- Using the Thomson Standard Filter Vials for sample prep of ground water is simple, sensitive, easy to use for pesticide analysis in ground water.
- Thomson Standard Filter Vials provides a more convenient and simpler approach reducing the number of steps, time, and cost per sample.

