Introduction

Glyphosate is the active ingredient in the popular herbicide Roundup® and is used throughout the world. Glyphosate is a broad-spectrum systemic herbicide. It is an organophosphorus compound, specifically a phosphonate. Recently, its safe use has come into question. This has heightened the concern is the affinity of these compounds to not only extraction from food but in its analysis. Of concern is the affinity of these compounds to make system-to-system reproducibility difficult. This shows the performance of the extraction of glyphosate, its major metabolite and seven other metabolites and polar pesticides.

Polar Pesticides Analyzed

Glyphosate m/z 168/463/79  Aminomethylphosphonic acid (AMPA) m/z 110 → 463/79

Glufosinate m/z 180 → 134/63  Fosetyl (A+ salt) m/z 109 → 79/63

N-acetylglufosinate m/z 222 → 136/59  2-hydroxyethyphosphonic acid (HEPA) m/z 125 → 95/79

Ethephon m/z 145 → 107  3-methylphosphinocarboxylic acid (MPPA) m/z 151 → 91/33/63

malic hydrazide m/z 111 → 82/42

Experimental

Sample Preparation

At least 2 g of sample is weighed to a 15 mL VWR plastic centrifuge tube. The sample is then extracted following the QuPPE method where high water content samples (<80%) are extracted with an equal amount of acidified methanol and lower water content samples have added water to equal the amount of acidified methanol.1

Results and Discussion

Chromatographic Separation of standards in water and in QuPPE extraction solvent

Figure 2 shows the separation and sensitivity obtained with a standard in water. There is excellent separation of AMPA and fosetyl. However, the same separation is altered by dissolving the standard in the extraction solvent. Figure 3 shows AMPA and fosetyl much closer together while MPPA and ethephon actually switch in elution.

Conclusions

QuPPE with direct analysis of polar pesticides provides good recovery and detection for high water content foods

- Recovery (not shown) for strawberry, orange, bell pepper, spinach, and peach is greater than 80%. Corn is about 50%.
- All PEEK provides repeatable results even though sample and standard solvent impact chromatography. QuPPE extraction provides good reproducibility among different foods
- LOQs for high water content foods is below 10 ppb

References