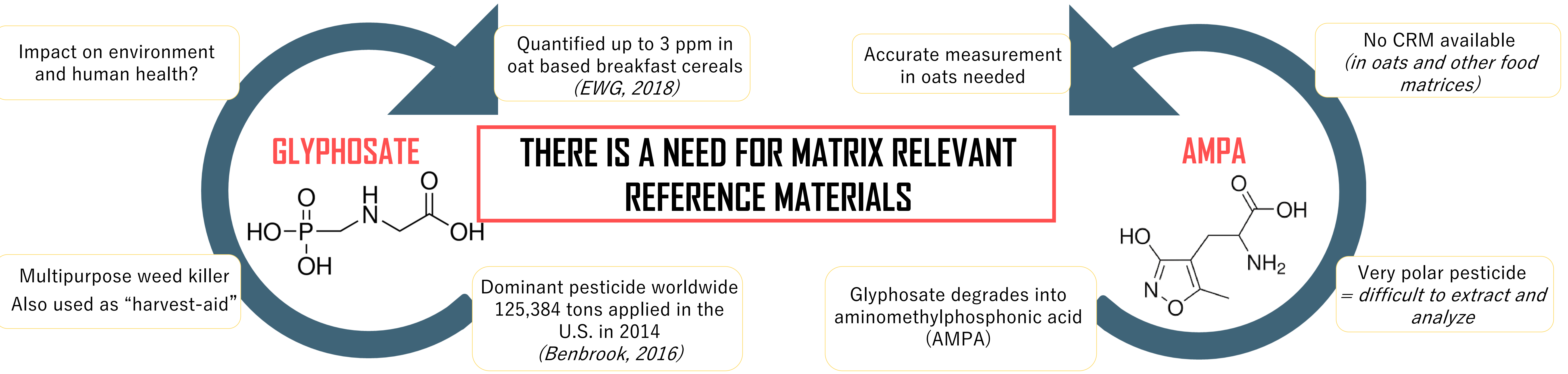


SCREENING GLYPHOSATE AND AMPA IN OAT CEREALS FOR THE SELECTION OF CANDIDATE REFERENCE MATERIALS

Justine M. Cruz, Jacolin A. Murray and Katrice A. Lippa
 National Institute of Standards and Technology, Chemical Sciences Division, Gaithersburg, MD 20899, USA
justine.cruz@nist.gov jacolin.murray@nist.gov katrice.lippa@nist.gov

WHY IS IT IMPORTANT TO DEVELOP GLYPHOSATE REFERENCE MATERIALS?



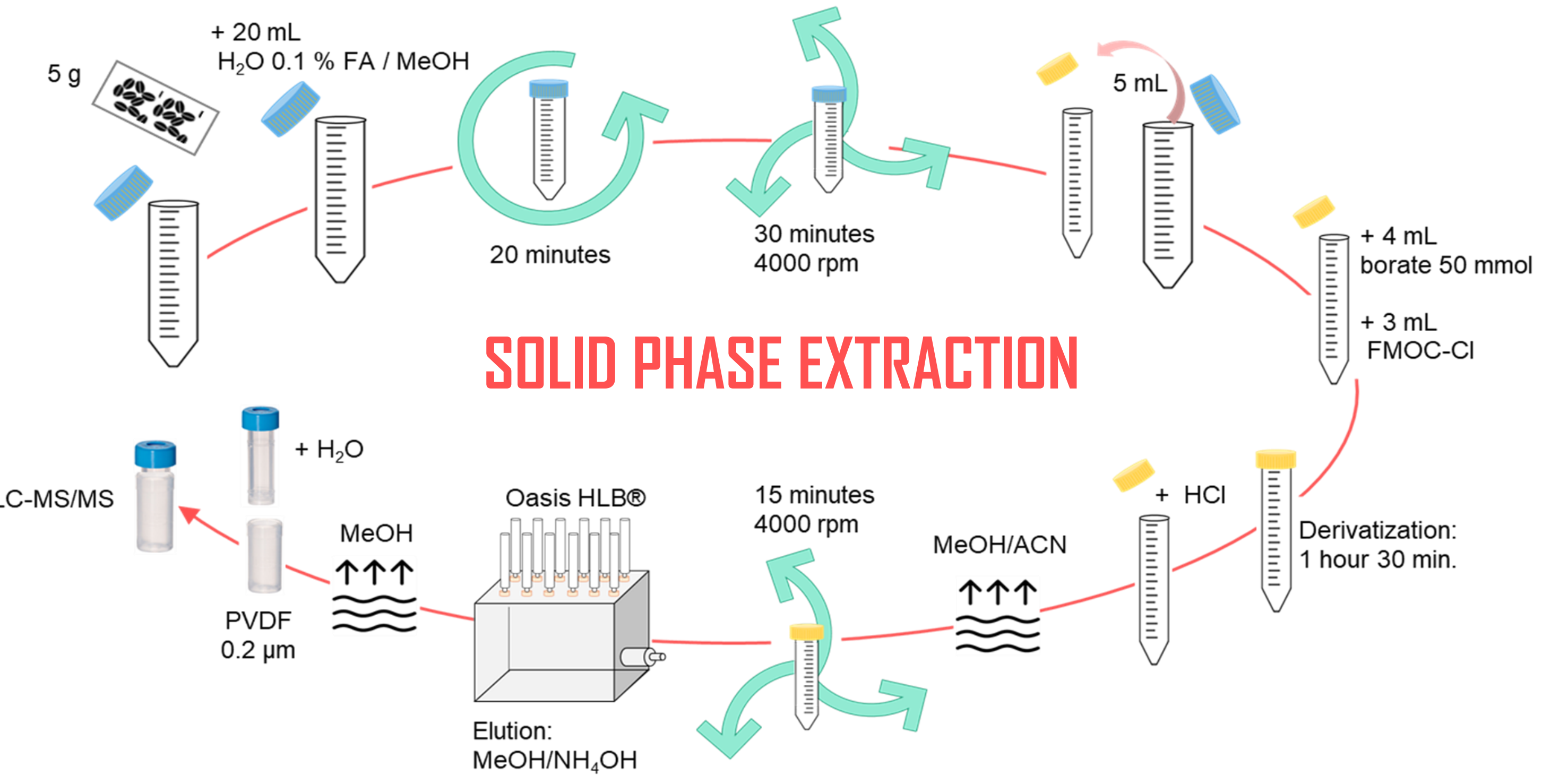
OBJECTIVES OF THE STUDY:

- Optimize and validate a robust method for the extraction and analysis of glyphosate (and AMPA)
- Apply the method for the screening of commercially available oat-based breakfast cereals to look for candidate reference materials

HOW ARE GLYPHOSATE AND AMPA ANALYZED AND MEASURED?

LC-MS/MS analysis:

- Optimized for FMOCl (9-fluorenylmethyl-chloroformate) derivatized compounds
- Agilent 1290 Infinity/Agilent 6490A
- Column: ACQUITY UPLC BEH C18 1.7 μm (Waters)
- Phases: H₂O 5 mmol ammonium acetate pH=9 / Acetonitrile

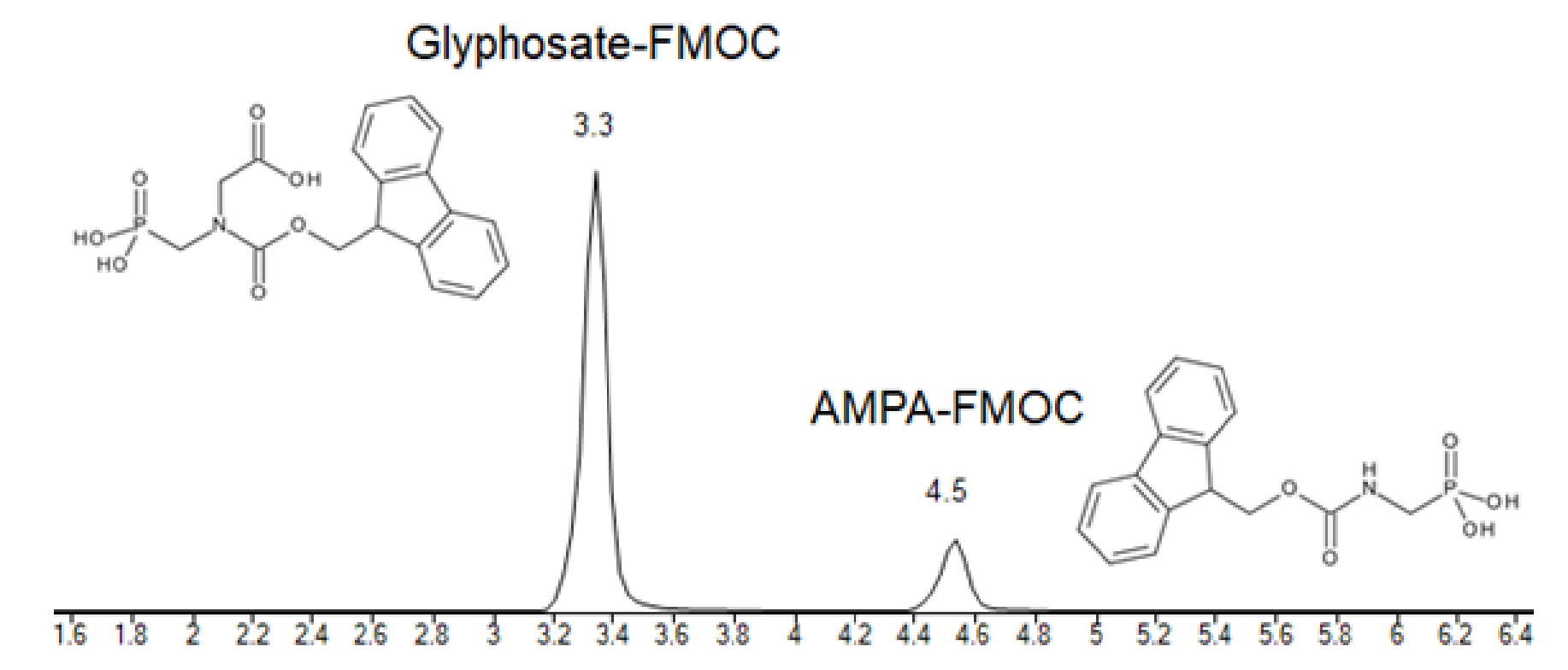


Quantification:

Isotopically labelled internal standards were added to samples at the beginning of the extraction

Glyphosate-1,2-¹³C,¹⁵N

AMPA-¹³C,¹⁵N



METHOD PERFORMANCES:

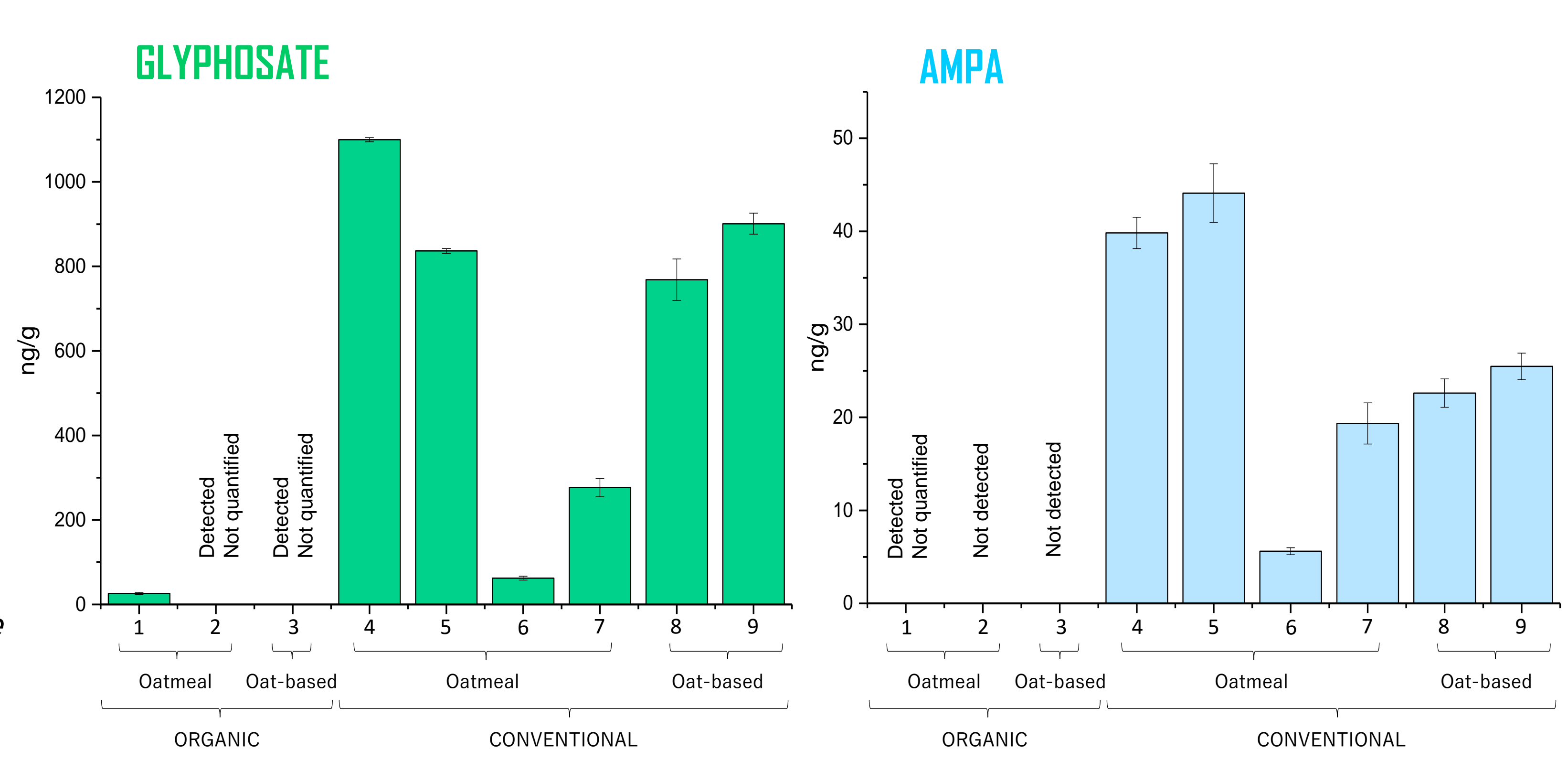
- Validation (linearity/recoveries) for glyphosate at 5 - 1000 ng/g, for AMPA at 10 - 100 ng/g
- Limit Of Quantification = 5 ng/g for both glyphosate and AMPA

WHAT LEVELS WERE FOUND IN THE COMMERCIALLY AVAILABLE BREAKFAST CEREALS?

Mass fractions (ng/g) measured in the oatmeal and oat-based breakfast cereals (n=3 replicates)

9 commercial breakfast cereals were tested:
 oatmeal/oat-based cereals slightly to highly processed conventional/organic agriculture

Samples were ground into a fine powder before extraction



Glyphosate:

- Detected in all 9 samples
- Smaller mass fractions (under 26 ng/g) were measured in samples from organic agriculture
- Mass fractions in samples from conventional agriculture ranged from 62 to 1100 ng/g

AMPA:

- Not detected in 2 samples (from organic agriculture)
- Lower mass fractions than glyphosate
- Mass fractions were under 44 ng/g

WHAT DOES THE LEGISLATION SAY?

Mass fractions were < U.S EPA tolerance = 30 ppm (30,000 ng/g) (for glyphosate in oats)

WHAT SHOULD BE DONE NEXT TO DEVELOP THE REFERENCE MATERIAL?

- Investigate glyphosate and AMPA stability in oat materials: *How should the material be prepared? How should it be stored?*
- Define the optimal mass fraction: *What do the stakeholders need?*
- Interlaboratory study coming soon!

REFERENCES

Benbrook, C. M. (2016). Trends in glyphosate herbicide use in the United States and globally. *Environmental Sciences Europe*, 28(1), 3. <https://doi.org/10.1186/s12302-016-0070-0>

EWG. (2018, October 24). Roundup for Breakfast, Part 2: In New Tests, Weed Killer Found in All Kids' Cereals Sampled. Retrieved from <https://www.ewg.org/release/roundup-breakfast-part-2-new-tests-weed-killer-found-all-kids-cereals-sampled>

DISCLAIMER

Certain commercial equipment, instruments, software, or materials are identified in this presentation in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose