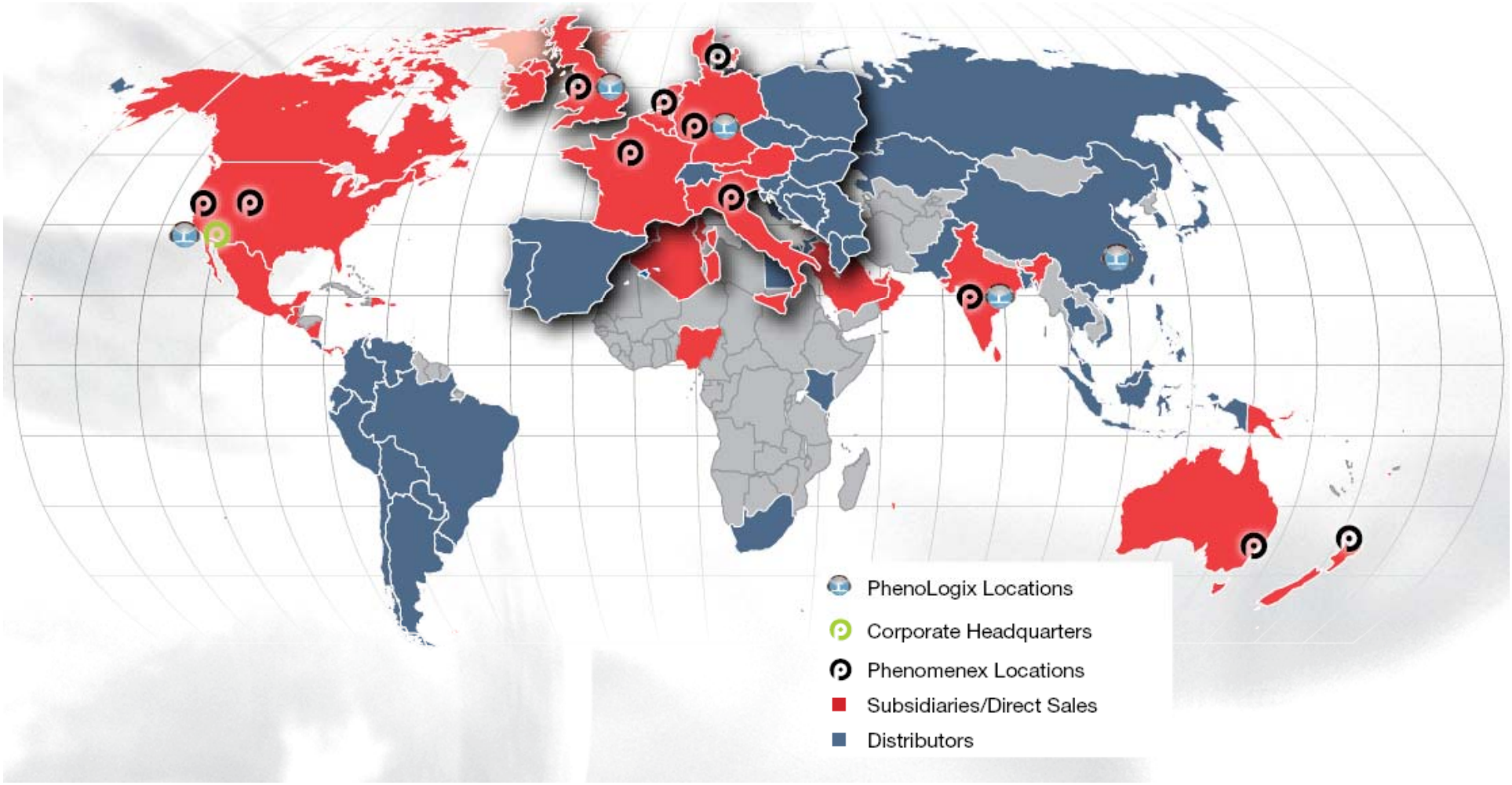
Collaboration with Other Analytical Fields: Adapting Food Safety Methodology for Environmental Monitoring Purposes

Kory Kelly
Environmental Product Manager
Phenomenex, Inc.

A Global Company

Contacts, Communications, and Cooperation Around The World



World-Wide Experience

- Strong experience in Europe, Asia, Africa, South America, Australia\NZ, North America
- Ongoing discussions with analytical laboratories throughout the world
  - Some are strictly following the USEPA Methods
  - Some have adapted USEPA Methods
  - Some have diverged completely

Europe

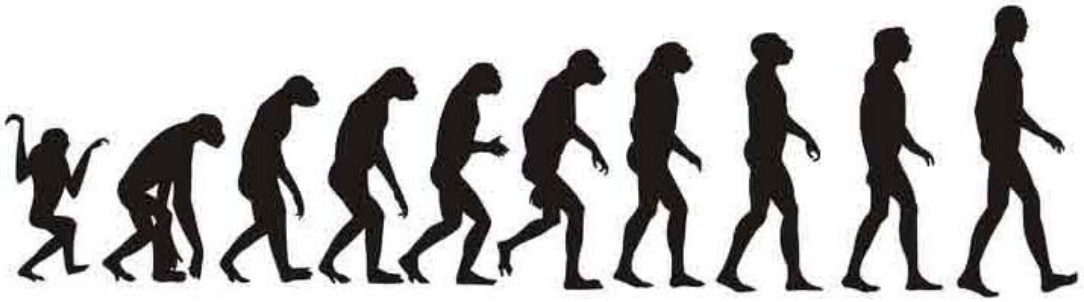
- Regulations are structured quite differently
  - Compliance monitoring is ISO-based
  - Have specific compound lists
  - Have mandated detection limits
  - Some even specify individual matrices
- No other conditions are specified!
    - Don’t Specify Detectors
    - Don’t Even Specify A Technique

- Allows labs the freedom to change and improve approaches and then validate via proficiency test

European Discussions

All Environmental Methods are based on EPA methods

- Semi-Volatiles still done by 8270, but with a slightly different compound list
- Volatiles also still done by 8260, but with slightly different list
- However, pesticides have significantly evolved, having used EPA for the original inspiration……..



The USEPA Legacy

- USEPA Methods have lead the world for many years and have been very successful, particularly for pesticides
- The chlorinated list in particular has been monitored throughout the world for decades and the detection of these compounds is now very rare
- However, this success has created some ironies
  - Most Labs (including US Labs) have not had a positive hit for some pesticides in years
  - Some EU labs are using USEPA target compounds as Surrogates and Internal Standards
  - Unfortunately, other significant peaks were present in chromatograms, but remained unidentified

Pesticide Compounds

- List is constantly growing
  - Most farmers are following regulations and using approved pesticides
  - Some farmers are using new pesticides that may be common outside of the EU, but not regulated by the EU
  - They can then use the pesticides without fear of losing crops
    - Due to pests
    - Due to confiscation when exceeding regulated levels
  - Labs are forced to evolve with the evolving list of analytes



European Pesticide Trends

- Followed USEPA methods as written until 10-15 years ago
- New regulations continued to add more and more analytes
- Gradual migration from individual detectors (ECD & NPD) to GC/MS Screening
  - Original methods used ECD and NPD for detection with MS verification
  - More methods are now only GC/MS using Internal Standards for quantitation
  - Some labs even using LC/MS/MS

Limitations of USEPA Methods

Regulations have not kept pace with additional analytes

- Some methods are from 1995 or earlier (8151, 507, 508, 525)
- 8081B, 8151, and 8318 are from 2007 but not without significantly increased compound lists
    - 8081B has 19 ‘possible’ additional analytes, but not ‘extensively validated’
    - Most labs are still analyzing the original 20 chlorinated pesticides
- Of the 20 pesticides most commonly used in the US, some are not yet in regulated methods

Limitations of Methods (cont.)

Methods have not kept up with advances in instrumentation

- Still using ECD and NPD
  - Very sensitive, very selective, and inexpensive (sort of…)
  - Not qualitative for additional compounds

Is it Time to Update the Science?

- Current US Methods have approximately160 total pesticide analytes
  - 500 + 600 + 8000 series
- However, with exception of 525.2, all methods use ECD and NPD for sensitivity but cannot identify unknown analytes

Elements of a Solution

- Demand for a solution
- Technology
  - Procedure
  - Instrumentation
- Skills (labor)
- Time
- Physical means
  - Instrumentation
  - Funding
  - Lab space
- Verification / Validation / Implementation

Reviewing Past Success

- Many success stories
  - Some just discussed
- Current collaborations
  - Experience and knowledge from universities
  - Supplies, technical help, and funding from private industry
  - Criteria, guidance and experience from EPA

Where does this leave pesticides?

Mission of the EPA

The mission of EPA is to protect human health and the environment.

EPA's purpose is to ensure that:

- **all Americans are protected from significant risks to human health and the environment where they live, learn and work;**
- **national efforts to reduce environmental risk are based on the best available scientific information;**
- federal laws protecting human health and the environment are enforced fairly and effectively;
- environmental protection is an integral consideration in U.S. policies concerning natural resources, human health, economic growth, energy, transportation, agriculture, industry, and international trade, and these factors are similarly considered in establishing environmental policy;
- all parts of society -- communities, individuals, businesses, and state, local and tribal governments -- have access to accurate information sufficient to effectively participate in managing human health and environmental risks;
- environmental protection contributes to making our communities and ecosystems diverse, sustainable and economically productive; and
- the United States plays a leadership role in working with other nations to protect the global environment.

Learning from the Food Industry

Similarities

- Testing for multiple classes of pesticides to ensure public health
- Similar analytes at similar concentration levels

Differences

- More complicated matrix issues
- List of analytes is expanded to account for pesticides used abroad that may be on imported food products.
- Higher exposure to fungicides
- Lower exposure to herbicides

Many Diverse Sources

- Japan – Has a ‘Positives List’ that includes over 700 compounds, of which many are pesticides
- Europe – Already running Environmental Pesticide Screens, but also running Multi-Residue Screens on food.
  - 60% of methods by GC/MS; 40% by LC/MS/MS
- Asia – Both Environmental and Food Residue Pesticide methods
- Brazil and Latin America – Currently developing extensive programs for Environmental and food exports testing

- USFDA – Both screening and quantifying pesticides
  - Domestic samples from across state lines as well as international

FDA Pesticide Methods

- Methods are designed to test for US regulated pesticides – and many others!
- Originally followed USEPA Methods
- Switched to GC/MS methods and increased the list to 360 pesticides
  - Most samples are negative
  - Scan for qualitative identification
  - Use a ‘Targeted’ method to quantify

Further Evolution of FDA Methods

- This approach worked well, but FDA wanted lower detection
- Therefore, methods are migrating from GC/MS to QQQ
  - Provides better quantitation than ECD and NPD, but also provides verification
- LC/MS/MS is playing a much larger role in pesticide analysis
  - Hundreds of compounds accessible by LC/MS/MS

Some FDA Observations

- FDA expects very low levels because of application cycles
  - Possibly have already overcome sensitivity problems
- Organophosphorus pesticides are not common any more
- Fewer Carbamate pesticides are seen
- Still seeing positives for Chlorinated pesticides
- Constantly identifying new pesticides
- Current list of analytes is over 650!

- Fungicides are common (although applied mostly post-harvest)
  - Boscalid is the most commonly detected compound
  - Still seeing carbendazim often
  - Seeing pyrimethanil, fludioxonil, cyprodinil, thiabendazole

Some FDA Observations (cont.)

- QQQ offers unforeseen advantages and savings
  - Easier sample preparation (no fractionation)
  - Decreased sample preparation cost (first quad acts as filter)
  - Lower detection levels than GC/MS, NPD and ECD
  - Provides conclusive answers, particularly with complicated samples / chromatograms

Project Needs

- Demand for a solution
- Technology
  - Procedure
  - Instrumentation
- Skills (labor)
- Time
- Means
  - Instrumentation
  - Time
  - Funding
- Verification / Validation / Implementation

The USEPA Pesticide Challenge

- USEPA has already developed Multi-Pesticide Methods
- The Agency is currently working on improvements
- People may already be reaching outside of the EPA
- However, a widespread, systematic collaboration with other disciplines and world areas would be highly beneficial

- Remember that nobody has to work alone

External Collaborations Offer

- Exposure to new approaches
- Experience with their methods
- Experience with their analytes
- In the case of FDA: access to their extensive libraries for GC/MS, GC/MS/MS, and LC/MS/MS
- Additional resources!
  - Time
  - Instruments
  - Monetary
- A starting point…

Conclusion

- I am not proposing that we discard what we have taken so long to build
- Not proposing we adopt GC/MS/MS (at least, not yet)

- Scientists and regulators in the global food industry share our interests in creating a more healthy world
- The food industry has developed many advanced methods for monitoring pesticides and other environmental pollutants
- I am proposing collaboration – on a global scale – to attack our common environmental analytical challenges

Questions?