Residue analysis: How to sample and why the results are variable

Wilma du Plooy

Postharvest Researcher

CRI Postharvest Workshop 2026

Back to some basics

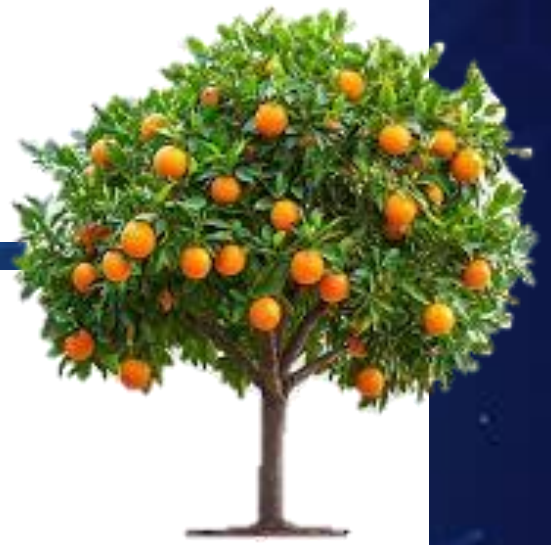
- Take care to consider the postharvest context
  - Fruit is still alive after picking
  - Orchards are not homogenous
    - Variables include soil, wind, incline, aspect (→ temperature)
  - Tree canopies are individual
    - Unequal growth, sunshine and shade
  - It's never one size fits all when fruit develop
  - **Fruit are not tennis balls**

citrus research international

First hurdle: non-uniform residue distribution

- Environment plays a definitive role in rind development
  - Thickness
  - Amount of intracuticular oil
  - Amount of wax deposited on outermost layer
- Rind characteristics are what determine uptake of chemicals
  - Interactions include hydrophobicity, lipophilicity, amphiphilic
- Postharvest chemicals are mostly lipophilic
  - Connect the dots: intracuticular oil… wax… chemicals
- Fruit maturity in a single tree is variable

Sampling for a fair test



- What is a fair test?
  - (Ideally) we should have only one variable at a time, e.g.
    - Use the same type of plant
    - Use comparable plant parts
    - Plants grown in same environmental conditions
  - Fruit is bringing the ends closer together
    - Same orchard, grown under same environmental and horticultural conditions
    - Same cultivar
    - Same packhouse treatments
- HOWEVER: Fruit have not all developed alike, regardless of above

Practical implications -1

- Rind oil contents differ
  - Content affects pesticide absorption and retention
  - Lipophilic pesticides dissolve in oils, affecting residue levels
  - Variable oil content = inconsistent residue uptake
- Rind wax thickness differ
  - Acts as a barrier to penetration
  - Thicker wax may trap more surface residues
  - Affects extraction efficiency during analysis

Practical implications -2

- Cutin thickness
  - Has a similar barrier-effect to wax - it influences how deeply pesticides penetrate and impacts what extraction methods can recover
- Colour and size
  - May be indicators of maturity stage
  - Correlates with other characteristics

Impossible to do a fair test in a packhouse?

- Look at a batch of fruit coming into the packhouse:
  - Single cultivar per batch
  - May be from a single orchard
  - Always come from 100’s of trees
  - Individuals have different developmental features:
    - Rind characteristics
    - Size
    - Maturation

Impossible to do a fair test in a packhouse?

- Look at a batch of fruit coming into the packhouse:
  - Single cultivar per batch
  - May be from a single orchard
  - Always come from 100’s of trees
  - Individuals have different developmental features:
    - Rind characteristics
    - Size
    - Maturation

OK… to summarise to this point

- Within the same batch, individual fruits have different chemical constitutions due to its position on tree, sun exposure, soil variations, and natural biological differences
- Chemical degradation occurs differently across samples:
  - Time between harvest and analysis
  - Storage conditions
  - Time between sampling and sample preparation
- Which part of the fruit is tested, how it's ground or processed, and extraction procedures can yield different concentrations from identical starting material.

The reality you experience

Same batch, different results

- You send samples from the same harvest batch to your regular laboratory
- Results come back with unexplained differences

Example: Pesticide residue results

Sample A: 0.15 mg/kg

Sample B: 0.22 mg/kg

Sample C: 0.08 mg/kg

All from Block 7, Row 12-15
Harvested same morning

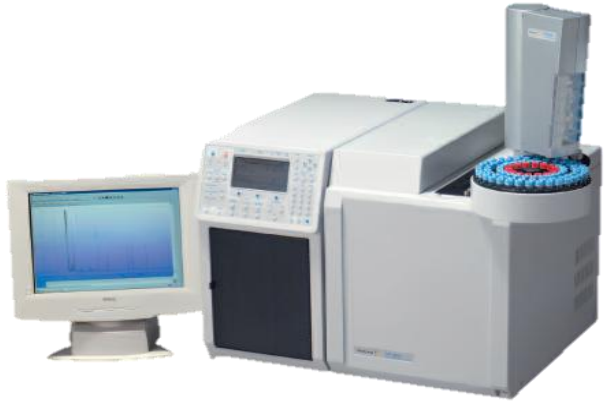
This variation is often normal and expected, not a laboratory error
Results can vary even with excellent laboratory practices

Technical and analytical factors

- Instrument precision limits
  - Even high-end analytical equipment has inherent measurement uncertainty typically ±5-15% for trace chemicals
- Method Variability
  - Different analytical methods (LC-MS vs GC-MS) or protocol variations can produce systematically different results
- Laboratory environmental variability
  - Temperature, humidity, and seasonal changes affect instrument performance and chemical stability during analysis.

What is an acceptable variation range?

- Industry standards typically accept
  - ± 20% variation for pesticide residues
  - ± 15% variation for heavy metals
  - ± 25% variation for some organic compounds



- These ranges reflect the **combined uncertainty** from sampling, preparation, and analysis - **not laboratory errors**
- Variation within these ranges indicates proper analytical practices

What this means for you

Best practices

Sample More Strategically

Take composite samples from multiple locations and fruits. Average results from 3-5 samples for better representation.

Understand Your Limits

Know the maximum residue limits (MRLs) for your markets

Consistent Protocols

Use the same sampling method and laboratory consistently for trend comparisons over time

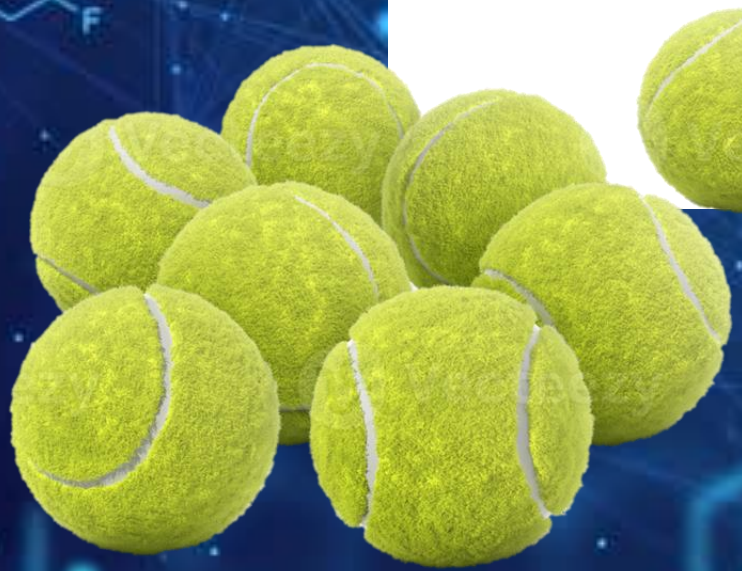
When to Investigate

Large systematic differences
Results consistently 50%+ different from expected patterns or historical data

Compliance concerns
Any result approaching or exceeding regulatory limits requires additional confirmation testing

- Remember: Variation is normal.
- Your lab partner should explain their quality controls
- Work with your laboratory to understand their specific methods and quality assurance processes

THANK YOU!



citrus research international