

# Economic Adulteration of Citrus Juice

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Economically Motivated Adulteration: Intentional food fraud for a financial advantage

Most common adulteration practices in citrus juices:

- ◊ Addition of sugars
- ◊ Addition of pulp wash
- ◊ Undeclared dilution with water or inferior fruit juices

How much adulteration is detected by the USDA?



Diversity in adulteration techniques, environment, storage conditions, genetics, or HLB status all play a role in making the detection of adulteration a difficult task

Sophisticated adulteration techniques require sophisticated testing

Numerous methods have been developed to detect adulteration

Not a single test. Rather, multiple tests to have a complete picture



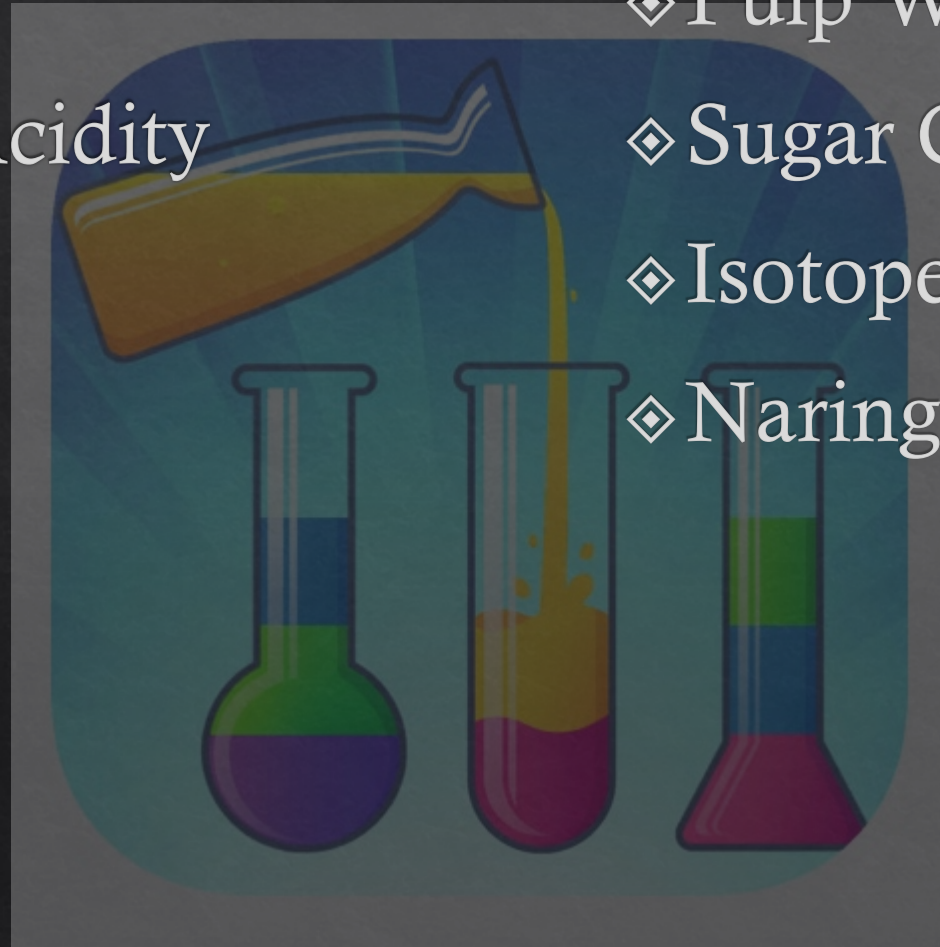
# Agricultural Marketing Service

U.S. DEPARTMENT OF AGRICULTURE

- ◆ National Science Laboratory (NSL) – Gastonia, NC
- ◆ Director: Kerry Smith, PhD
- ◆ Branch Chief: Roger Simonds
- ◆ Chemistry Section Supervisor: Demeseh Cobb
- ◆ 30 Staff at two facilities

# Juice Adulteration Analysis provided by the NSL

- ◆ Brix
- ◆ Titratable Acidity
- ◆ Formol
- ◆ Potassium
- ◆ Pulp Wash (Abs Ratio)
- ◆ Sugar Content (HPLC)
- ◆ Isotope Ratio Analysis
- ◆ Naringin (HPLC)



# BRIX

- ◆ Determines the approximate sugar content in a juice sample
- ◆ 1 degree Brix ( $^{\circ}\text{Bx}$ ) = 1g of sucrose / 100g of solution
- ◆ Must meet specification



## Digital refractometer

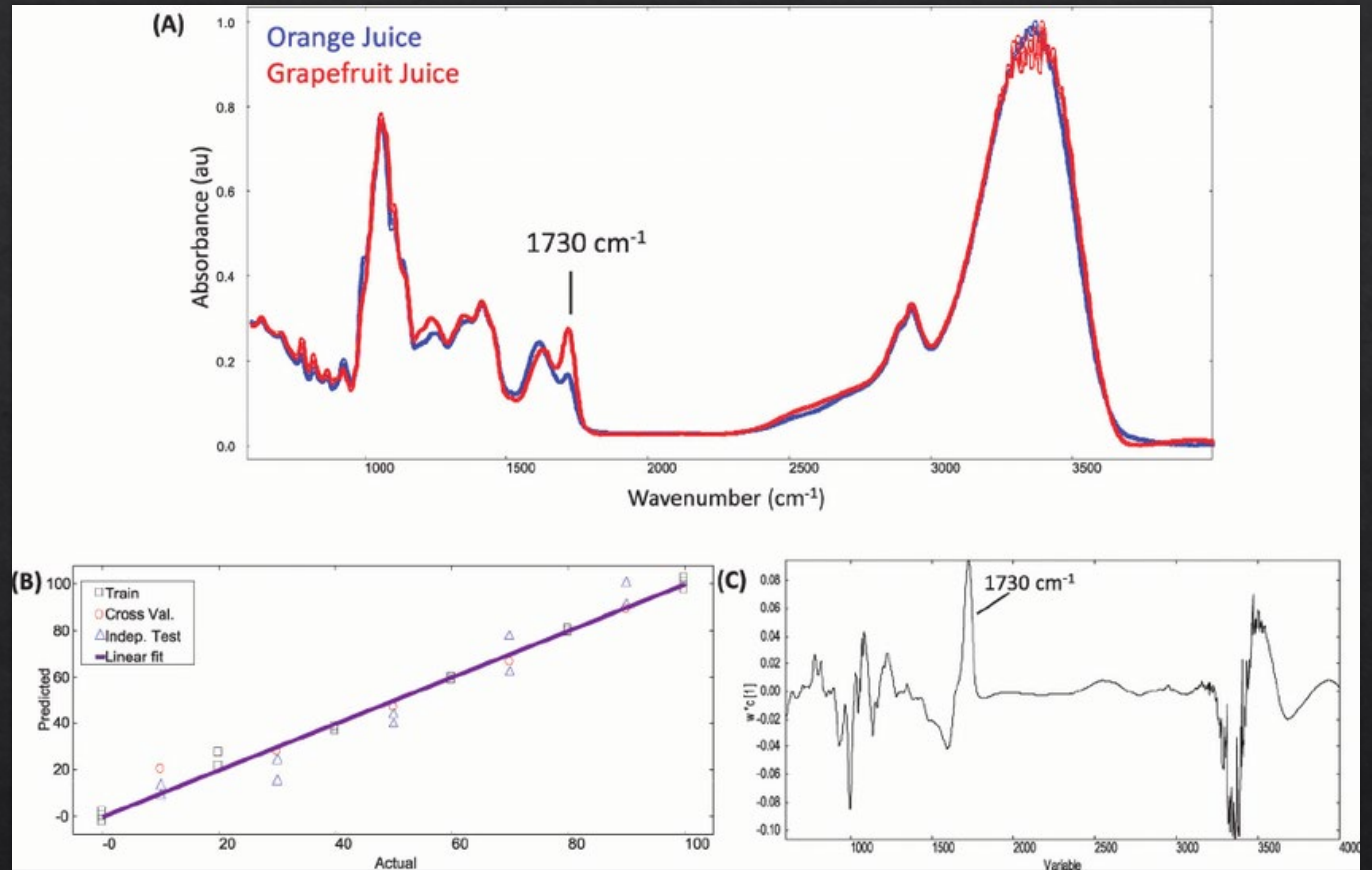
Convenient and accurate

HD display

easy to use

# Pulp Wash

- ◆ Juice Extracted by using water in a secondary step
- ◆ Determined by UV-Vis



# Titratable Acidity and Formol

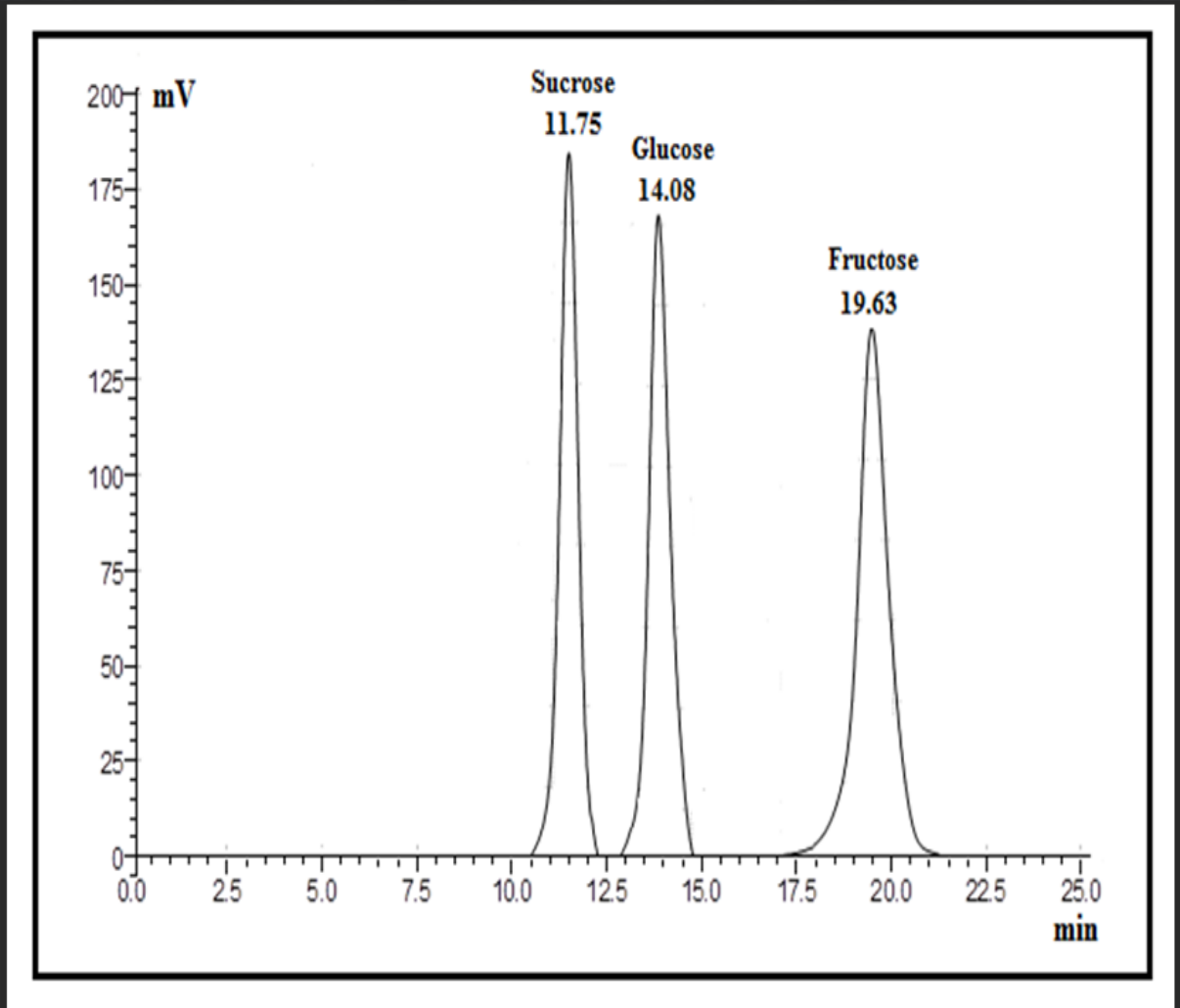


- ◇ Organic acid and amino acid determination by titration
- ◇ Excellent indicators of the authenticity of fruit juice because there naturally occurring amounts are quantifiable

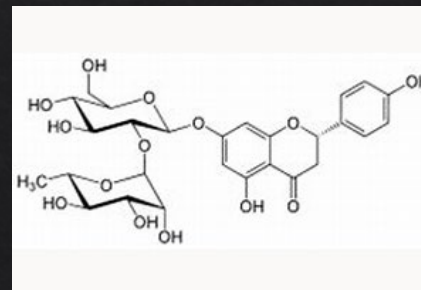


# Sugar Content by Liquid Chromatography

- ◇ Sugars are separated chromatographically
- ◇ Individual sugar values and their ratios and must meet standard
- ◇ Oligosaccharides and Polysaccharides indicative of syrup must not be present in pure citrus juice

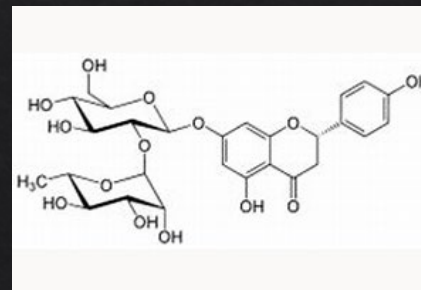
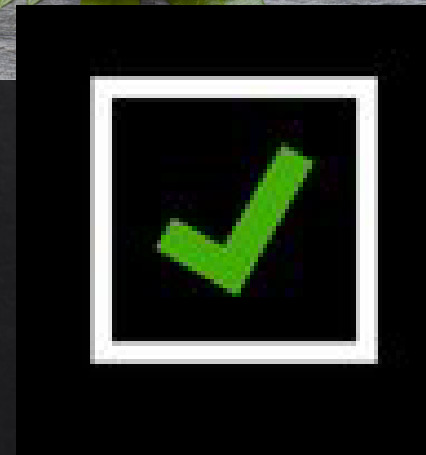
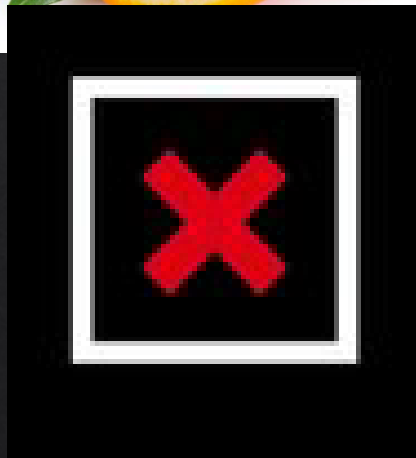
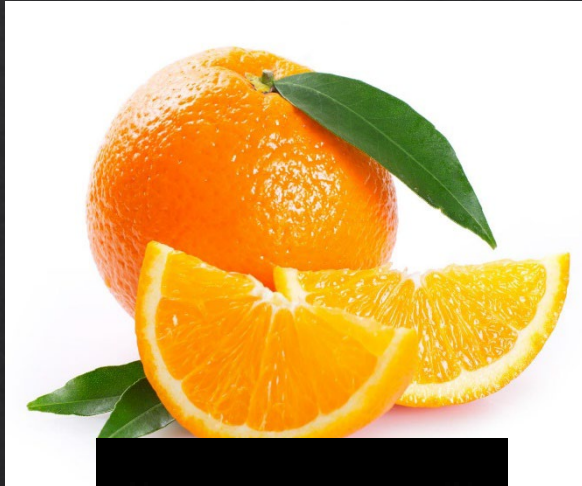


# Liquid chromatography of Naringin



Naringin

# Liquid chromatography of Naringin

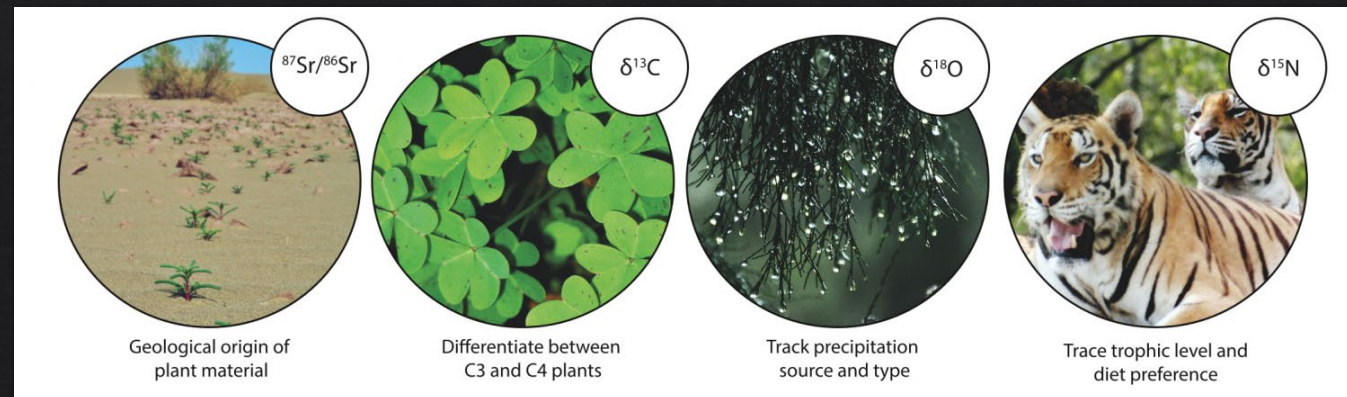


Naringin

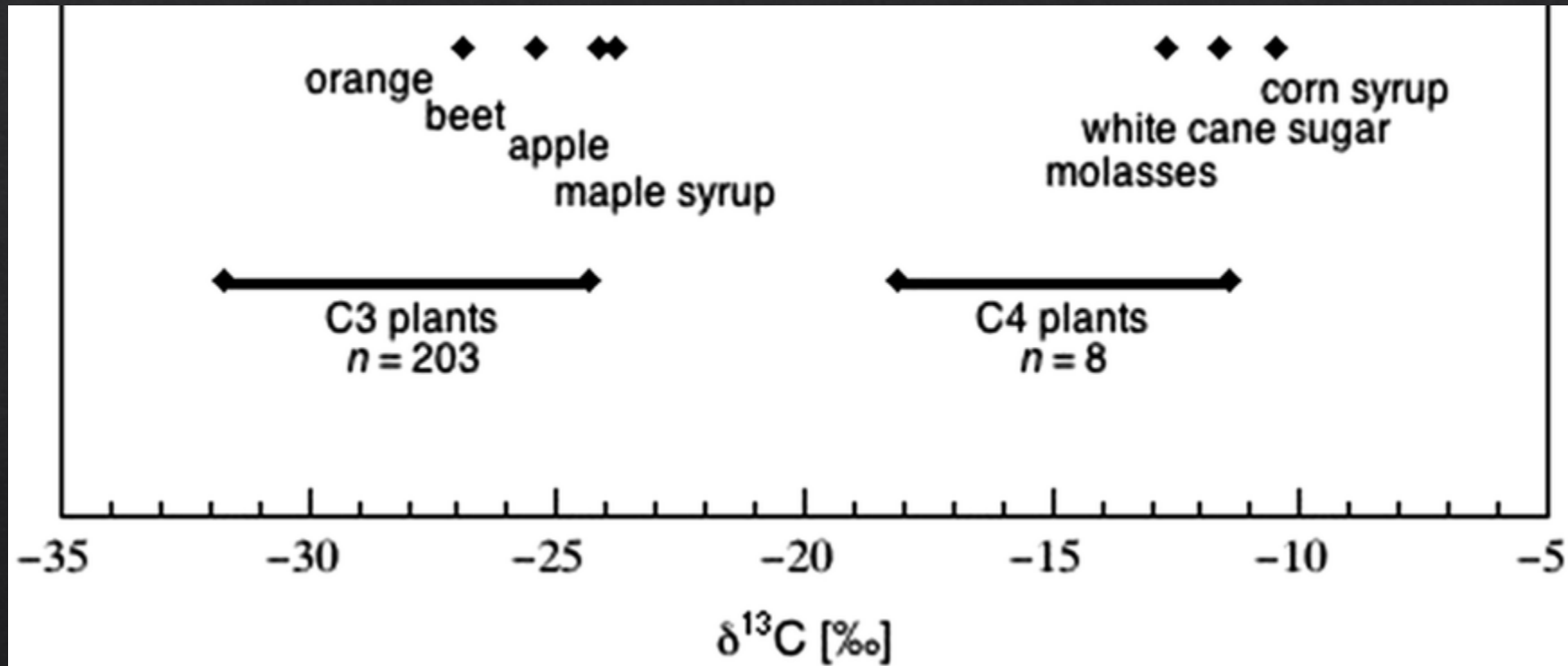
$$\delta^{13}\text{C}_{\text{Sample}} = \left\{ \frac{\left( \frac{^{13}\text{C}}{^{12}\text{C}} \right)_{\text{Sample}}}{\left( \frac{^{13}\text{C}}{^{12}\text{C}} \right)_{\text{Reference}}} - 1 \right\} * 1000$$

# Isotope Ratio Analysis

- ◇ Nature's fingerprint
- ◇ Identifies addition of sugars from C4 plants
- ◇ Identifies addition of ground water



# Stable carbon isotope ratio analysis (SCIRA)



# The future of adulteration testing for orange juice

- ◇ LC-IRMS
  - ◇ Analysis of isotope ratios of sugars or organic acids as they are separated with liquid chromatography
  - ◇ Very successful with applying this technique to detect adulteration in honey
- ◇ Current methods are targeted, but what about untargeted methods?
- ◇ Whole-picture metabolomics fingerprint testing

We already have these instruments in our lab!



# Questions?



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