

# Analysis of Organic Acids

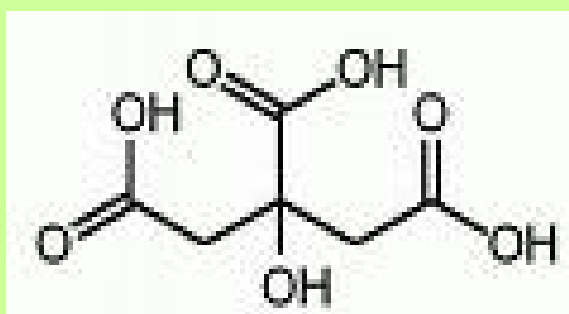


# Definition of Organic Acids

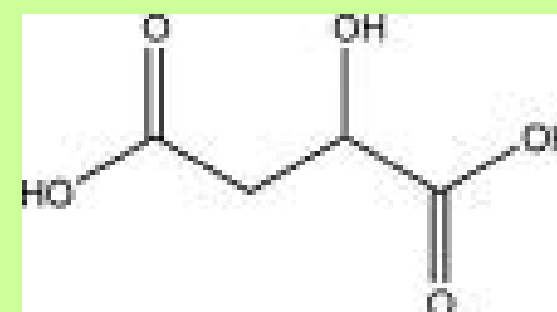
- **No definitions from Codex**
- **Broadly speaking, all organic compounds having at least one carboxylic acid functional group**

# Important OAs in Food

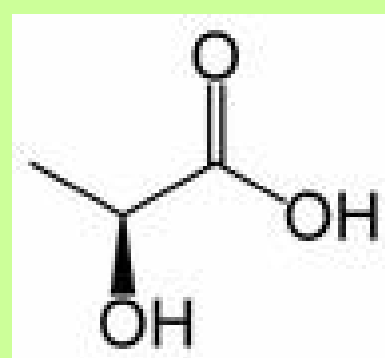
Citric acid



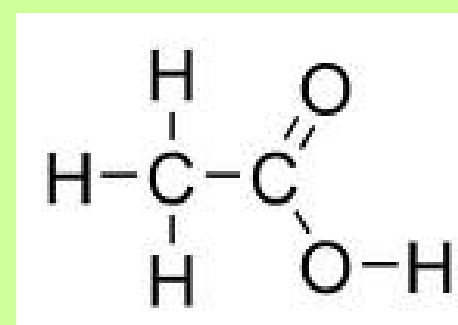
Malic acid



Lactic acid



Acetic acid



# Other OA can be found in Foods

- ◆ Formic acid
- ◆ Fumaric acid
- ◆ Maleic acid
- ◆ Malonic acid
- ◆ Oxalic acid
- ◆ Quinic acid
- ◆ Shikimic acid
- ◆ Succinic acid
- ◆ Tartaric acid

■ No exhaustive list

# Relevancy of OAs in NL

$$\begin{aligned} \text{energy (kcal in 100g)} = & \text{carbohydrate} \times 4 + \\ & \text{protein} \times 4 + \\ & \text{total fat} \times 9 + \\ & \text{ethanol} \times 7 + \\ & \text{organic acid} \times 3 \end{aligned}$$

- May contribute in energy calculation
- Not a core nutrient but voluntary labeled value must be correct

# Analytical Methods

**Q :** Can I determine the “organic acids” content by titration?

**A :** Not recommended since titration might:

- (1) over-estimate due to common additives such as benzoic acid, sorbic acid, sulphur dioxide, erythoric acid, etc. and
- (2) no suitable conversion factor for calculation

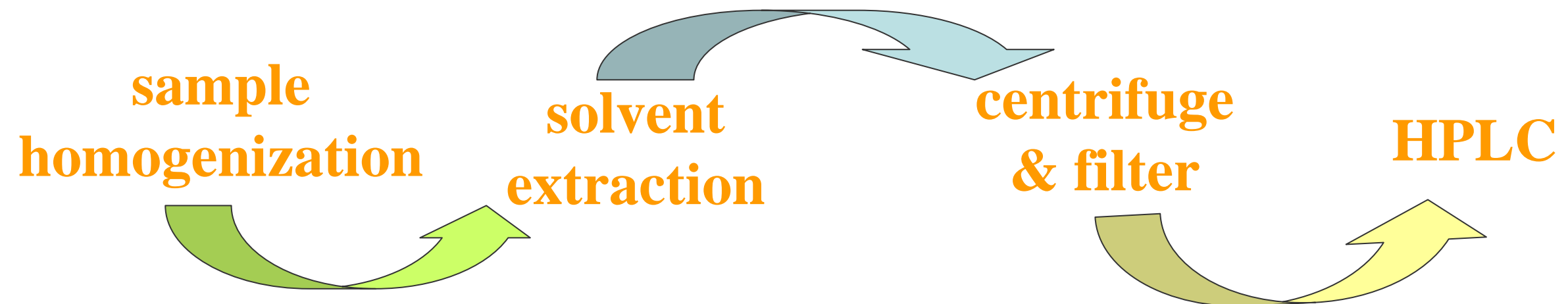
**LC methods are preferred**

# Analytical Methods

Official method	Titles
AOAC 986.13	Quinic, Malic, and Citric Acids in Cranberry Juice Cocktail and Apple Juice
GB/T 5009.157-2003	食品中有机酸的测定 Determination of Organic Acid in Foods

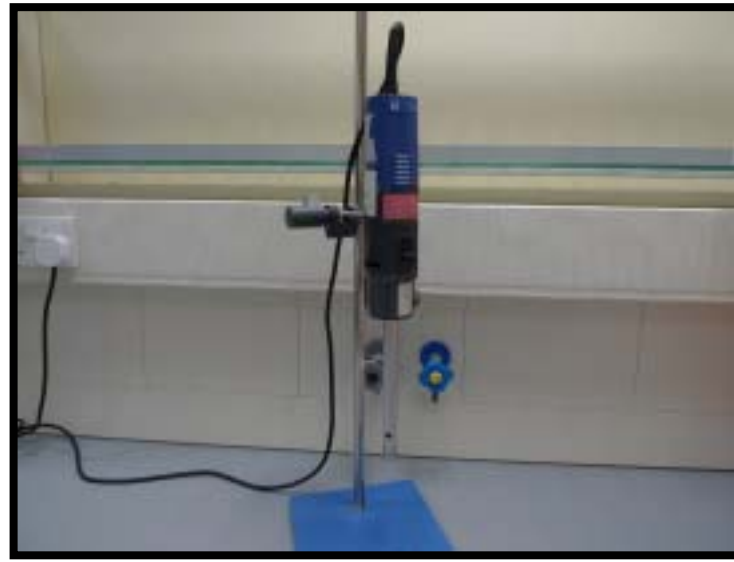
- ... and any other suitable methods for the food matrix concerned.

# FRL Method Workflow





# Equipment



**homogenizer**



**centrifuge**



**nitrogen evaporator**



**HPLC**

# Protocol for Liquid Samples

**mixing or blending**



**aliquot, add buffer, make up**



**centrifuge 3000 rpm 10 min**



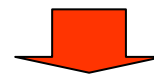
**filter supernatant, HPLC**

# Protocol for Solid Samples

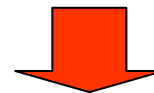
**10 g homogenized sample + 20 ml 80% EtOH**



**homogenize, 3000 rpm 10 min, collect supernatant**



**extract 2x with 80% EtOH, combine supernatants**



**aliquot, dry by N<sub>2</sub> (50°C), add buffer, make up**

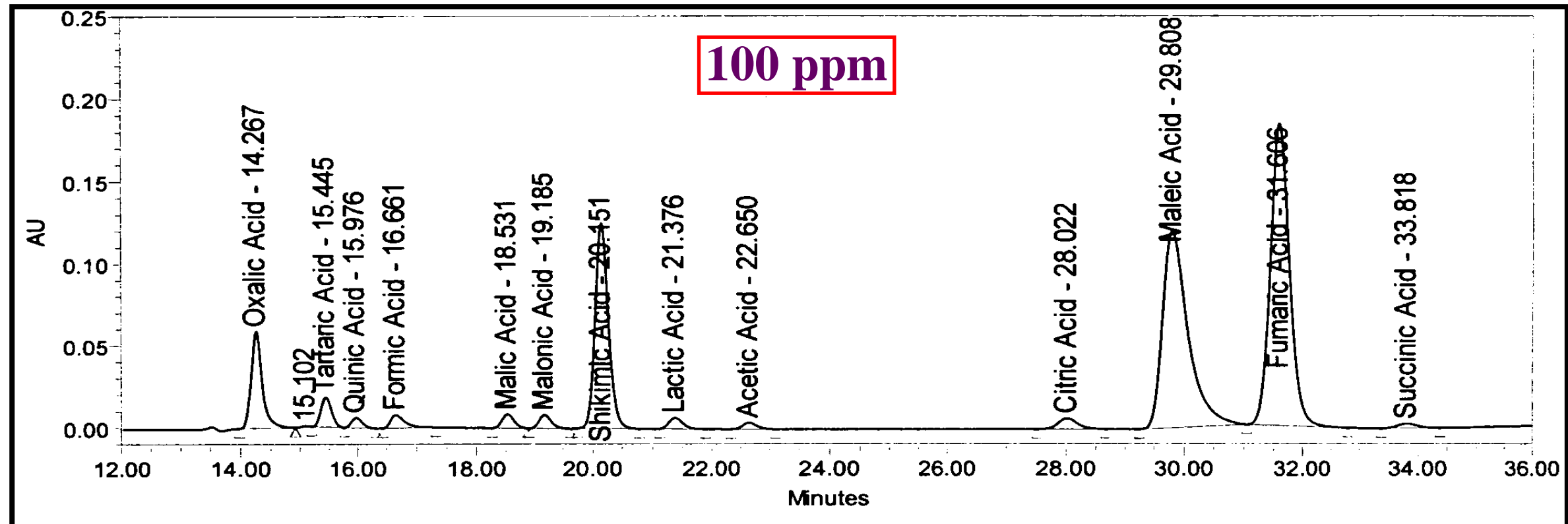


**filter supernatant, HPLC**

# HPLC Conditions

<b>Instrument:</b>	<b>HPLC-DAD/PDA</b>
<b>Column:</b>	<b>two C18 (15cm, 25cm) + one Dionex OA</b>
<b>Guard column:</b>	<b>C18 (7mm)</b>
<b>Mobile phase:</b>	<b>MeSO<sub>3</sub>H &amp; NaSO<sub>4</sub> buffer (pH 2.8)</b>
<b>Temp:</b>	<b>25 °C</b>
<b>Flow:</b>	<b>0.5 mL/min</b>
<b>Injection vol:</b>	<b>20 µL</b>
<b>UV (λ):</b>	<b>220 nm</b>
<b>Runtime:</b>	<b>40 min + 10 min post-run</b>

# Chromatogram



- All 13 OAs separated
- Frequent interferences :  
ascorbic acid and some anions

# Calculation of Available Carbohydrate when OAs is of concern

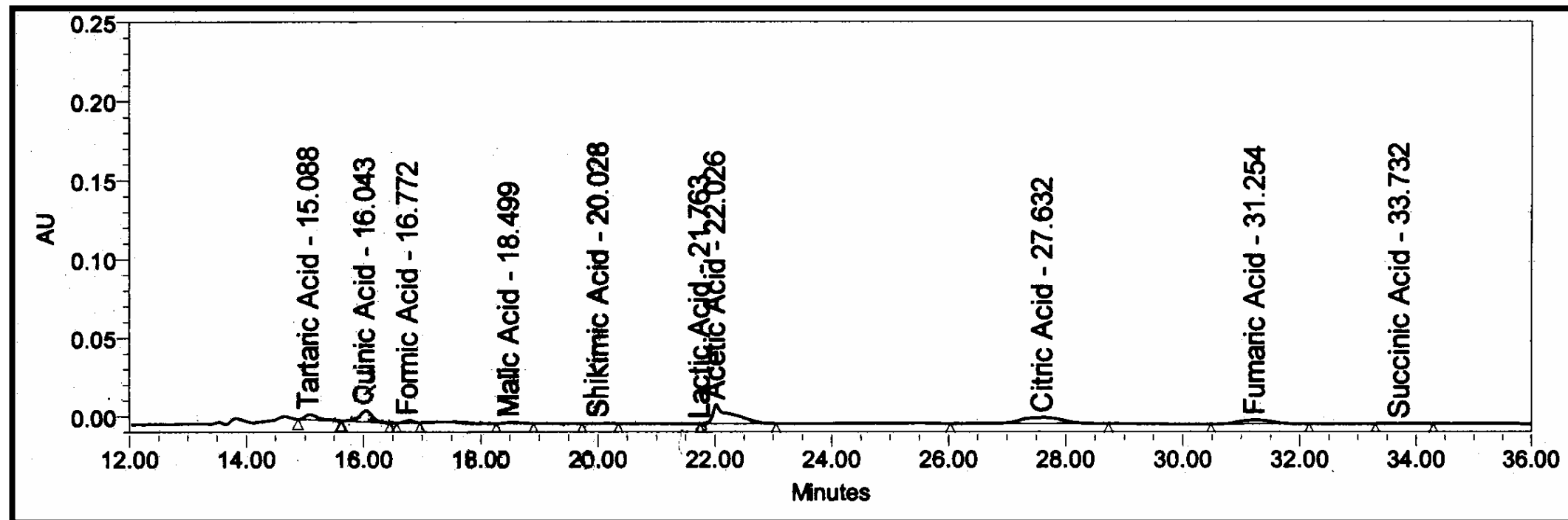
**Carbohydrate = 100 g – ( water + ash + DF  
(available) + protein + fat + ethanol +  
organic acid )**

**Q : How would energy content be affected?**

**A : (1) Due to different conversion factors for  
carbohydrates & OA (i.e. 4 vs 3)**

**(2) The extent depends on cases**

# Example 1 – Thousand Island Sauce



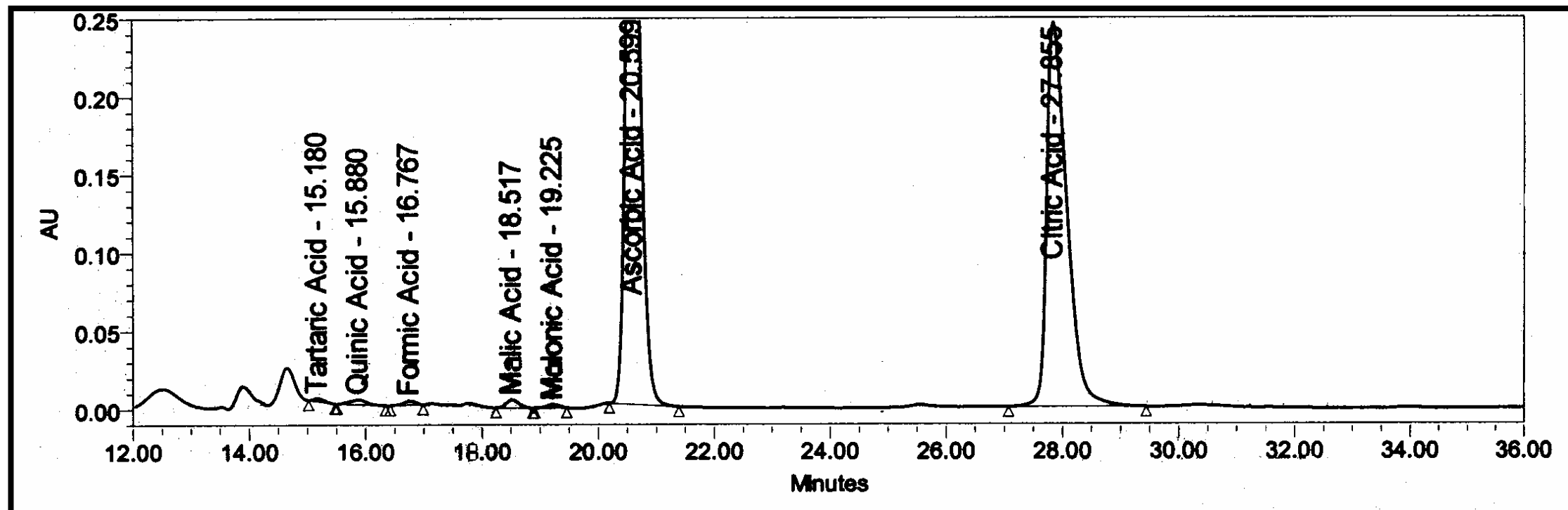
# Example 1 – Thousand Island Sauce

Nutrient (g/100 ml)	original	revised
Protein	1.1	1.1
Fat	45.5	45.5
Carbohydrate	12.3	11.3
Organic acid	Not determined	1.0
Ethanol	0.0	0.0
Energy (kcal/100ml)	<b>463</b>	<b>462</b>

**Difference = 0.2 %**



# Example 2 – Grapefruit Juice

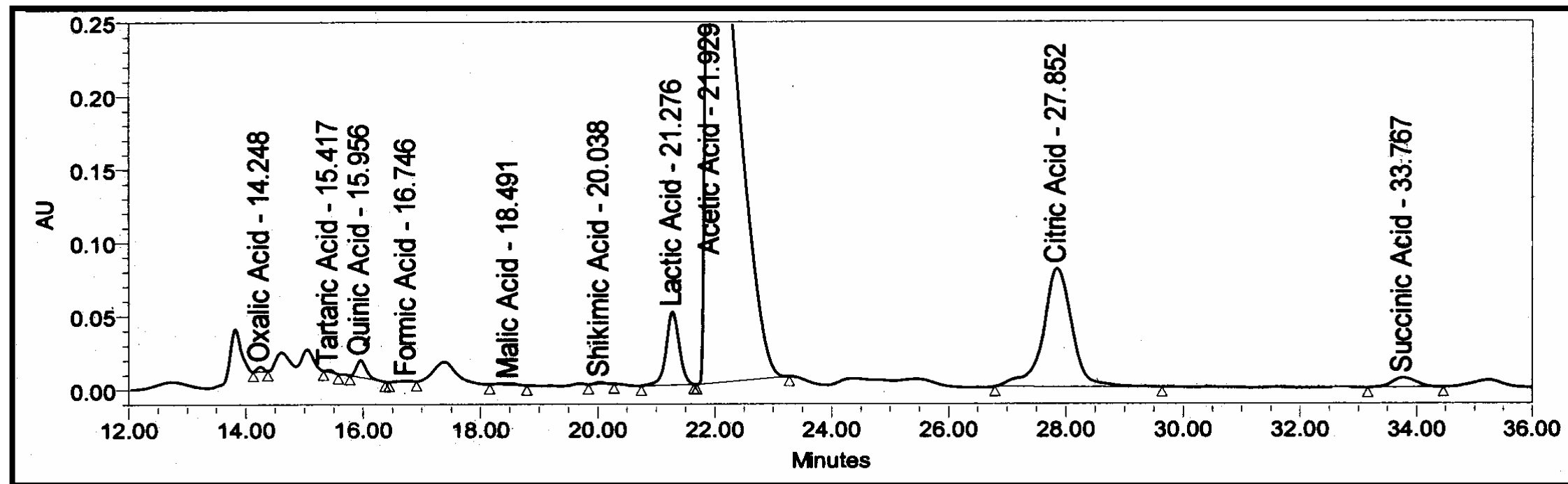


## Example 2 – Grapefruit Juice

Nutrient (g/100 ml)	original	revised
Protein	0.0	0.0
Fat	0.0	0.0
Carbohydrate	13.5	12.8
Organic acid	Not determined	0.7
Ethanol	0.0	0.0
Energy (kcal/100ml)	54	53.3

**Difference = 1.3 %**

# Example 3 – Chinese Red Vinegar



## Example 3 – Chinese Red Vinegar

Nutrient (g/100 ml)	original	revised
Protein	0.0	0.0
Fat	0.0	0.0
Carbohydrate	10.0	5.0
Organic acid	Not determined	5.0
Ethanol	0.0	0.0
Energy (kcal/100ml)	40	35

**Difference = 12.5 %**

# Points to Note

- **HPLC method is preferred for OA analysis**
- **Carbohydrate content should be recalculated after OA analysis**
- **OA analysis is only important for samples of low energy content or with high level of OA**

# THANK YOU

