Basic Food Analysis

CONTROL POINT

HANNA instruments

Welcome!

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Topics

- Basic food analysis
- Quality
- Pathogen control & food safety regulations
- HACCP Principle no. 6 – validation
- Good laboratory practices
- Instruments
- Temperature
- Question and Answer

What sector of the food industry are you in?

Basic Food Analysis

"the growth and infrastructure of the modern global food distribution system heavily relies on food analysis (beyond simple characterization) as a tool for new product development, quality control, regulatory enforcement, and problem-solving."

- R. J. Mcgorrin, "One hundred years of progress in food analysis," Journal of Agricultural and Food Chemistry, vol. 57, no. 18, pp. 8076–8088, 2009.

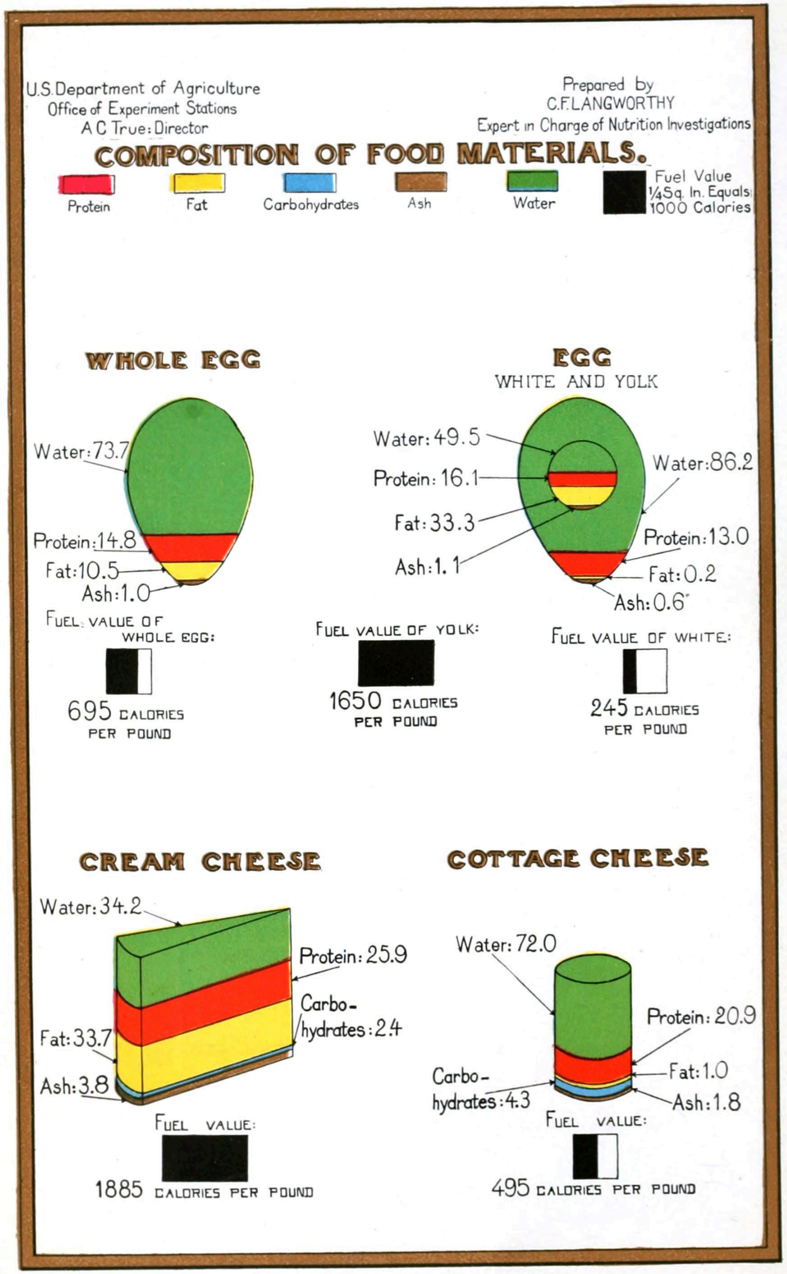
Testing

- Compliance
- Labeling
- Quality/Consistency
- Nutrition
- Safety
- Validation

Composition

- of food largely determines safety, nutrition, physicochemical properties, quality attributes, and sensory characteristic

The new household discoveries; an encyclopedia of recipes and processes (1917)



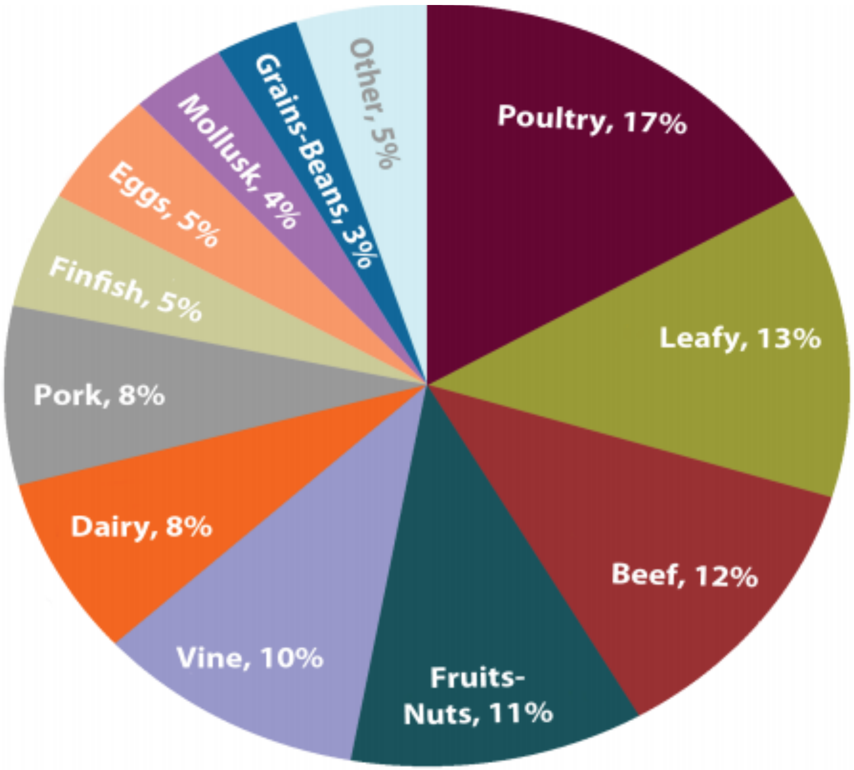
Determine Composition

- Determine the property of interest to determine the analytical procedure
  - Specific atoms
  - Specific molecules
  - Types of molecules
  - Specific substances

Food Safety

- There are 76 million cases of foodborne illness in the US
  - 325,000 hospitalizations and 5,000 deaths in 2010
  - Higher mortality among individuals with compromised immune systems

- Food safety regulations work to control FAT TOM



Microbial Spoilage

www.health.com

- Food products are constantly subjected to risk of spoilage
- Microorganisms favor food products due to their high moisture and nutrient content
  - Even food that is free from microbes can be readily spoiled if the conditions are right

Controlling Microbial Growth

Intrinsic: Relates to properties of the food

- Water activity/moisture content
- pH/Acidity
- Redox potential (ORP)
- Antimicrobials (Added or naturally occurring)

Extrinsic: Environmental controls

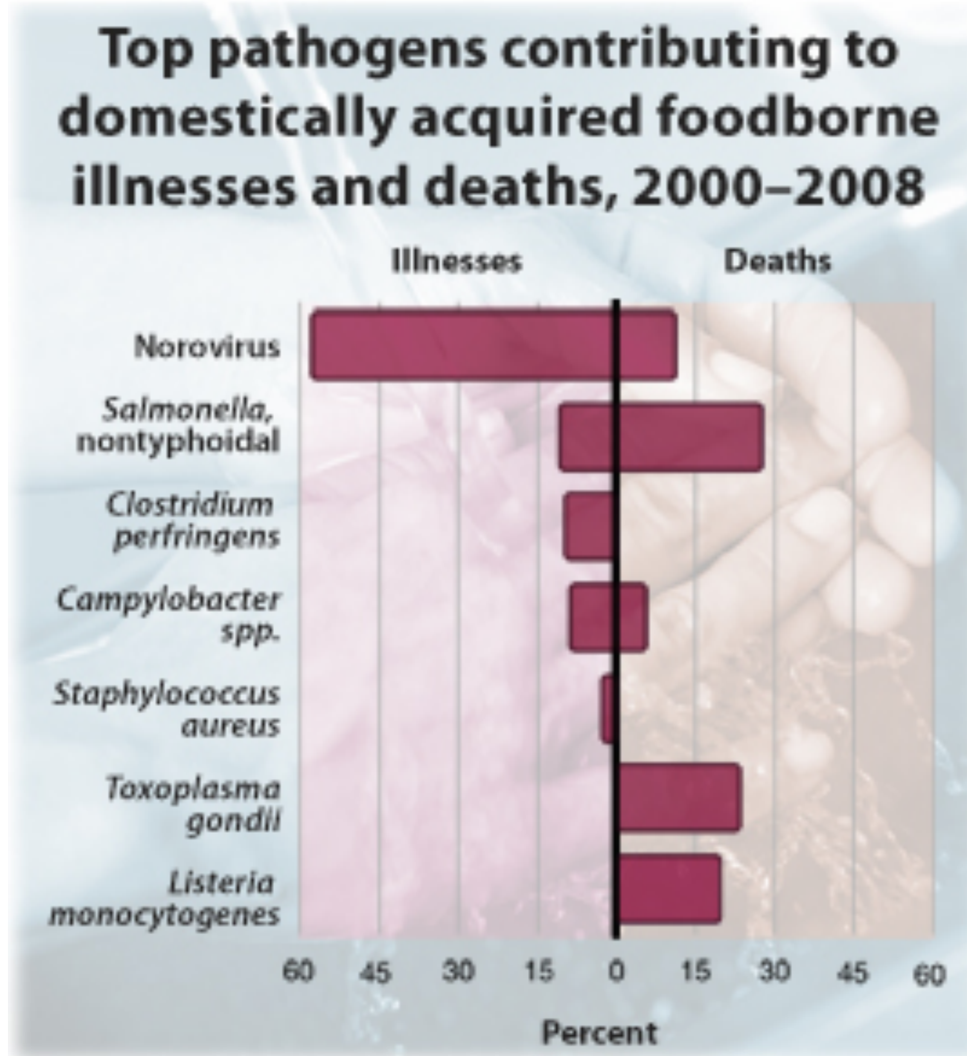
- Temperature
- Humidity
- Microbial environment

th.aectourismthai.com

FAT TOM

- (**F**): Food
- (**A**): Acidity
- (**T**): Time

- (**T**): Temperature
- (**O**): Oxygen
- (**M**): Moisture



http://www.cdc.gov/foodborneburden/resources.html

Factors Affecting the Growth and Control of Pathogenic Bacteria

Name	Temperature in ° C			Min pH	Min Aw	O2 needs
	Minimum	Optimum	Maximum			
Salmonella	7	35 - 37	45	3.8	0.94	FA*
Clostridium botulinum	3	18 - 25	45	5.0	0.96	OA*
Clostridium prefringens	12	44 - 49	50	5.5	0.93	OA*
Staphylococcus aureus	6	37	48	4.2	**0.85**	FA*
Campylobacter jejuni	30	42	45	4.9	0.98	MA*
Listeria monocytogenes	-1.5	37	45	4.4	0.92	FA*
Escherichia coli 0157:H7	7	37	46	4.4	0.95	FA*
Shigella	7	35 - 37	47	4.0	0.91	FA*
Bacillus cereus	4	30 - 37	50	4.3	0.91	FA*

Clostridium perfringens is less strictly anaerobic than Clostridium botulinum and can tolerate some exposure to oxygen.

Clostridium botulinum is pathogen associated with deadly "food poisoning".

Significant growth of ***Staphylococcus aureus*** *occurs at 15.6° C (60° C) and higher and after fermentation the sausages should be dried below this temperature. Fermentation tables are based on this fact.*

FA*: *Facultative anaerobic feels comfortable in oxygen but can live without it as well*
OA*: *Obligate anaerobic cannot grow in presence of oxygen*
MA*: *Microaerophile bacteria like very little of oxygen (5%) and low levels of CO2 (carbon dioxide-10%)*
As seen in the table Staphylococcus aureus is quite resistant to drying

http://www.meatsandsausages.com/sausage-types/fermented-sausage/safety-hurdles

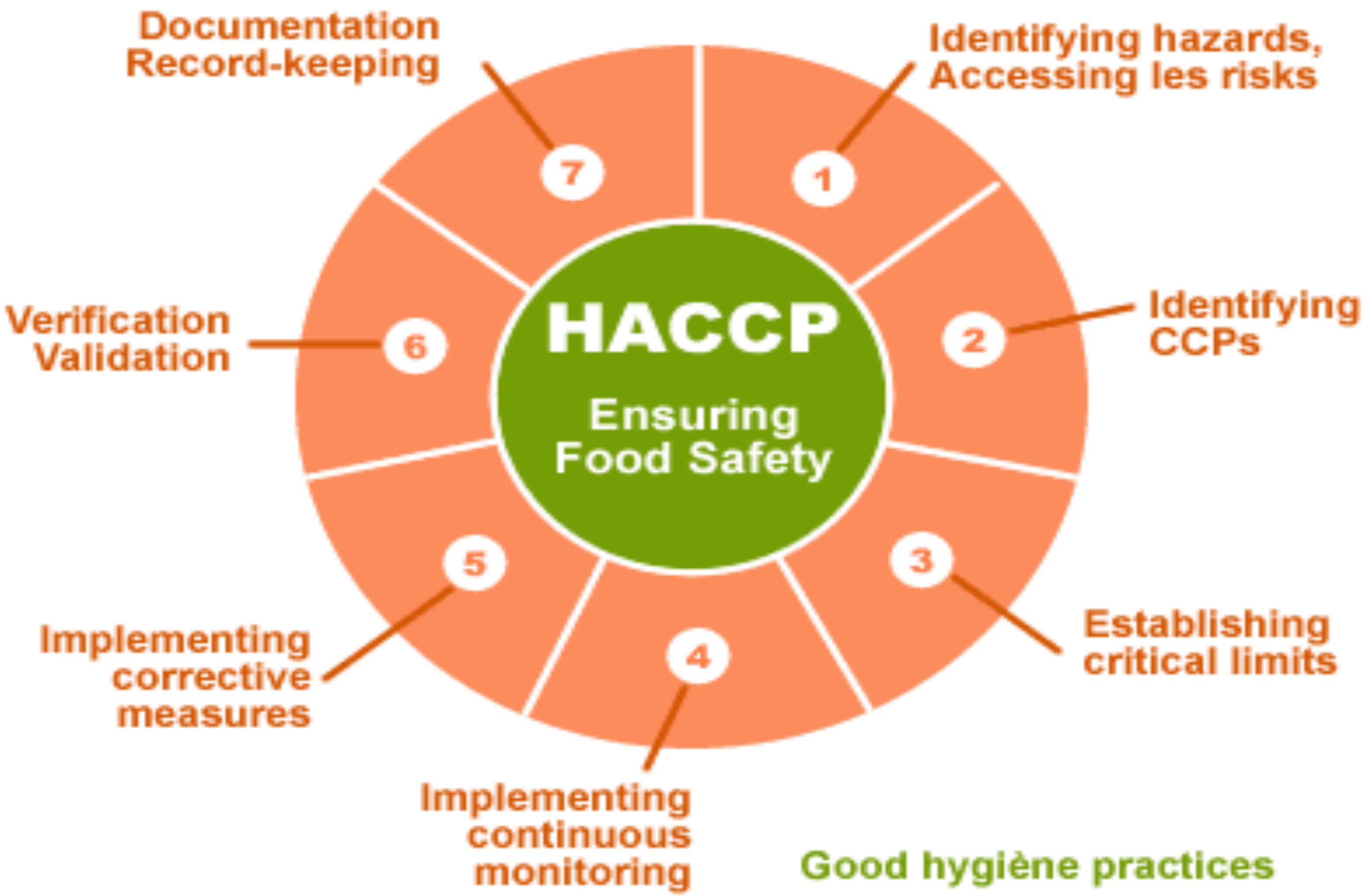
Code of Federal Regulations

Food and Drug Administration (FDA)

- Responsible for CFR 21

US Department of Agriculture (USDA)

- Responsible for CFR 9



Source: arcelormittal

Shelf Life Studies

Accelerated Vs. Real Time

6^{th} HACCP Principle

Validation

Validation

•process of demonstrating that the HACCP system, as designed, can adequately control identified hazards to produce a safe, unadulterated product.

- HACCP system - HACCP plan in operation, including the HACCP plan itself.
- HACCP plan - includes the hazard analysis, any supporting documentation including prerequisite programs supporting decisions in the hazard analysis, and all HACCP records.

Food Testing

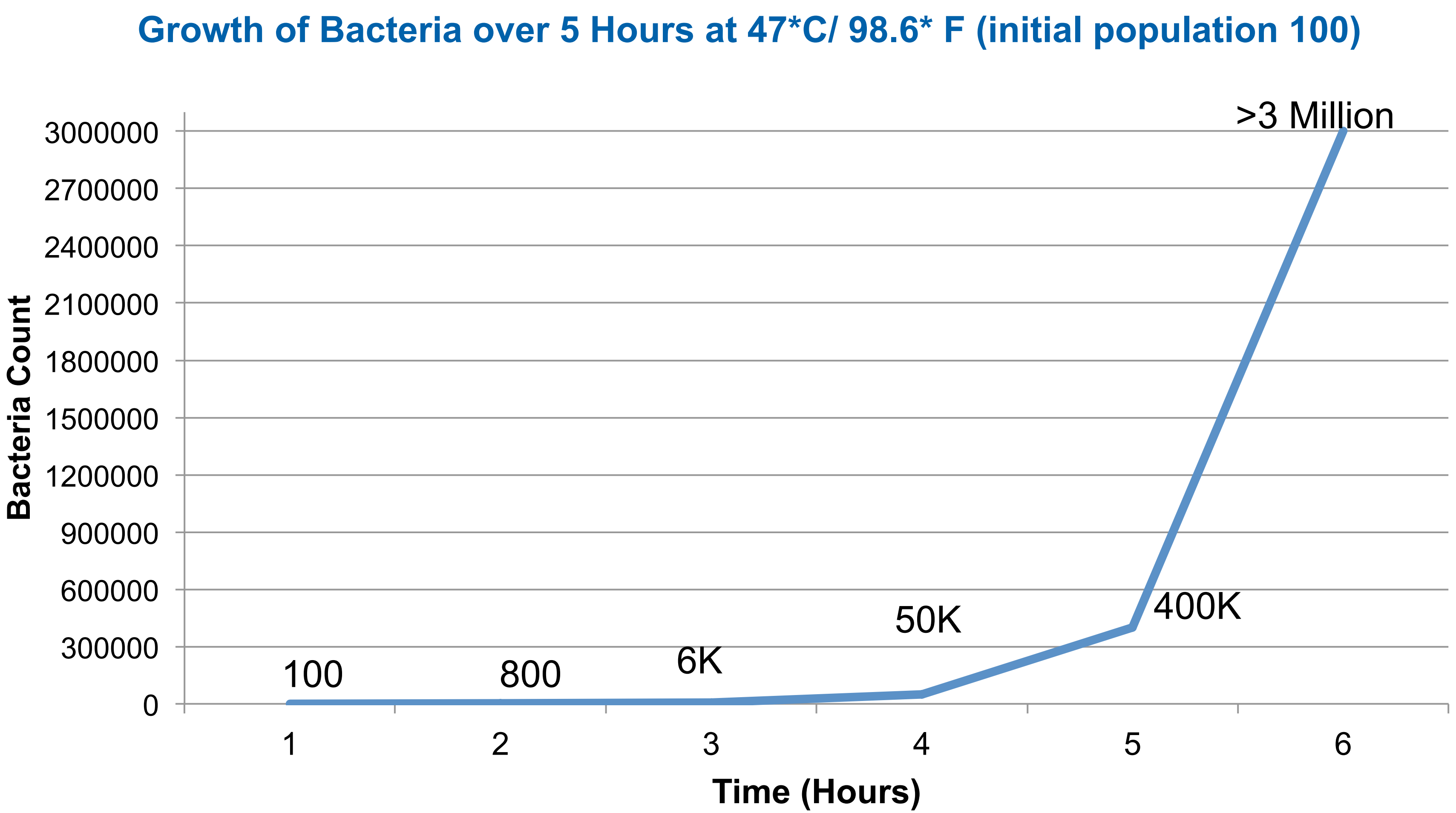
- Food Safety
- Food Labeling
- Food Quality and Consistency

Food Quality and Consistency

www.mestec.net

- Self guided initiatives
- Consistency between batches, over time
- Ensure food meets specified flavor/ texture profile

Temperature



Primary Thermometer Types

Thermocouple

- Measures the potential difference at the opposite extremes of two different metal wires

Thermistor

- Measures temperature by applying a potential difference and measuring the resistance

Pt100

- Measures a change in electrical resistance

Infrared

- Measures the heat given off

Thermometers

Infrared
HI99556
HI99551

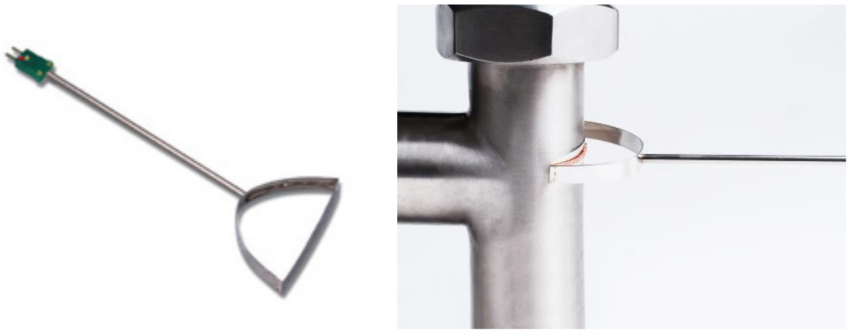
Thermocouple
HI935007N

Thermistor
HI9241
HI93501NS

Thermistor
Checktemp™ Family

Thermocouple K-Type Probe Varieties

HI766PA Roller Surface Probe



HI766PD Air and Gas Probe

HI766B1 90° Surface Probe

HI766TV1 Pipe Clamp Probe

Temperature Measurement			
	Range	**Accuracy**	**Response**
Thermistor	Limited (-50°C-220°C)	High (±0.3°C)	Medium
Thermocouple	Wide (-200°C-1371°C)	Medium (±0.5°C)	Fast
Pt100	Limited (200°C-850°C)	High (±0.2°C)	Fast
Infrared	Limited (-20°C-300°C)	Medium (±2% of Reading)	Very Fast

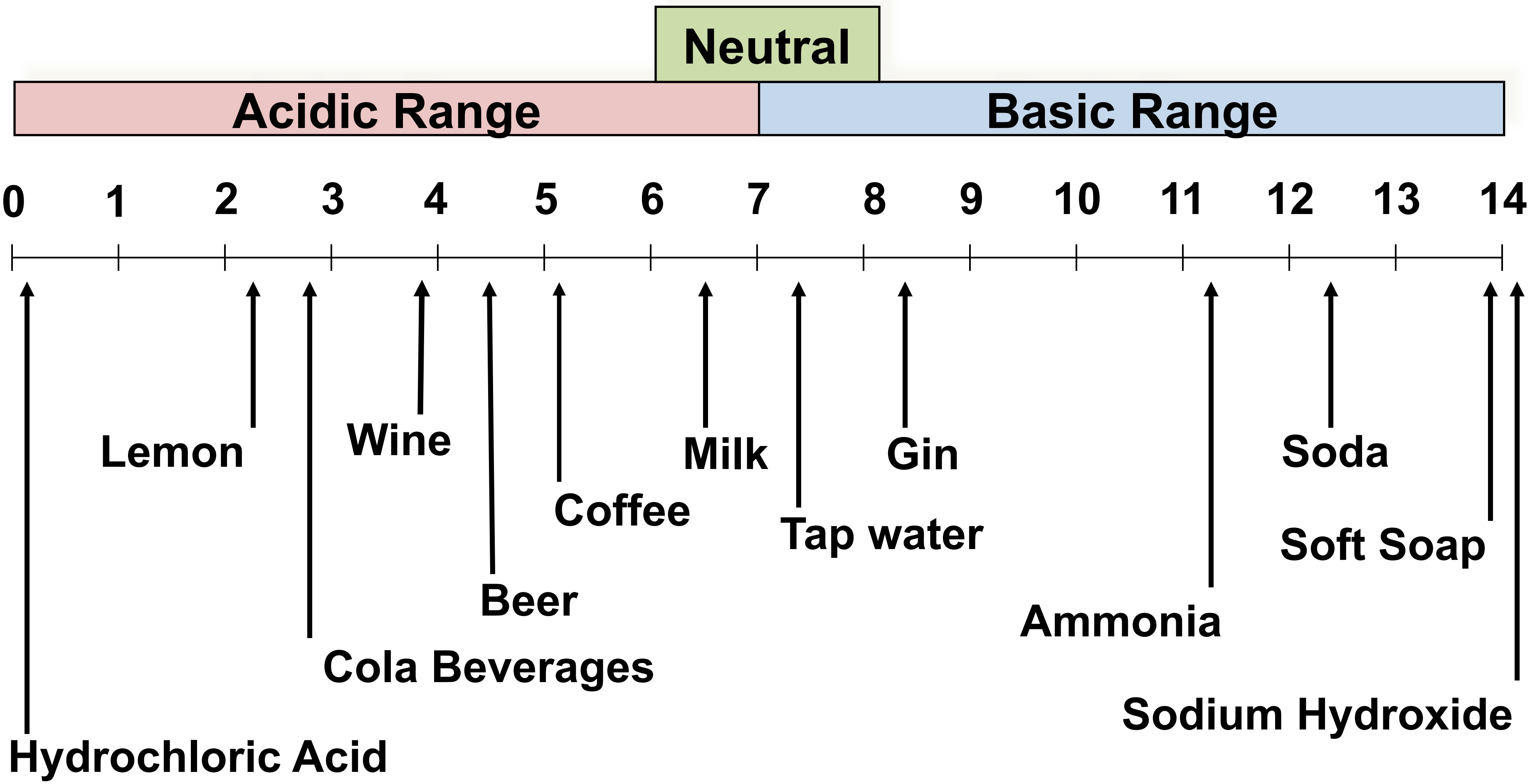
pH

$pH = -Log_{10}[H^+]$

Based on water

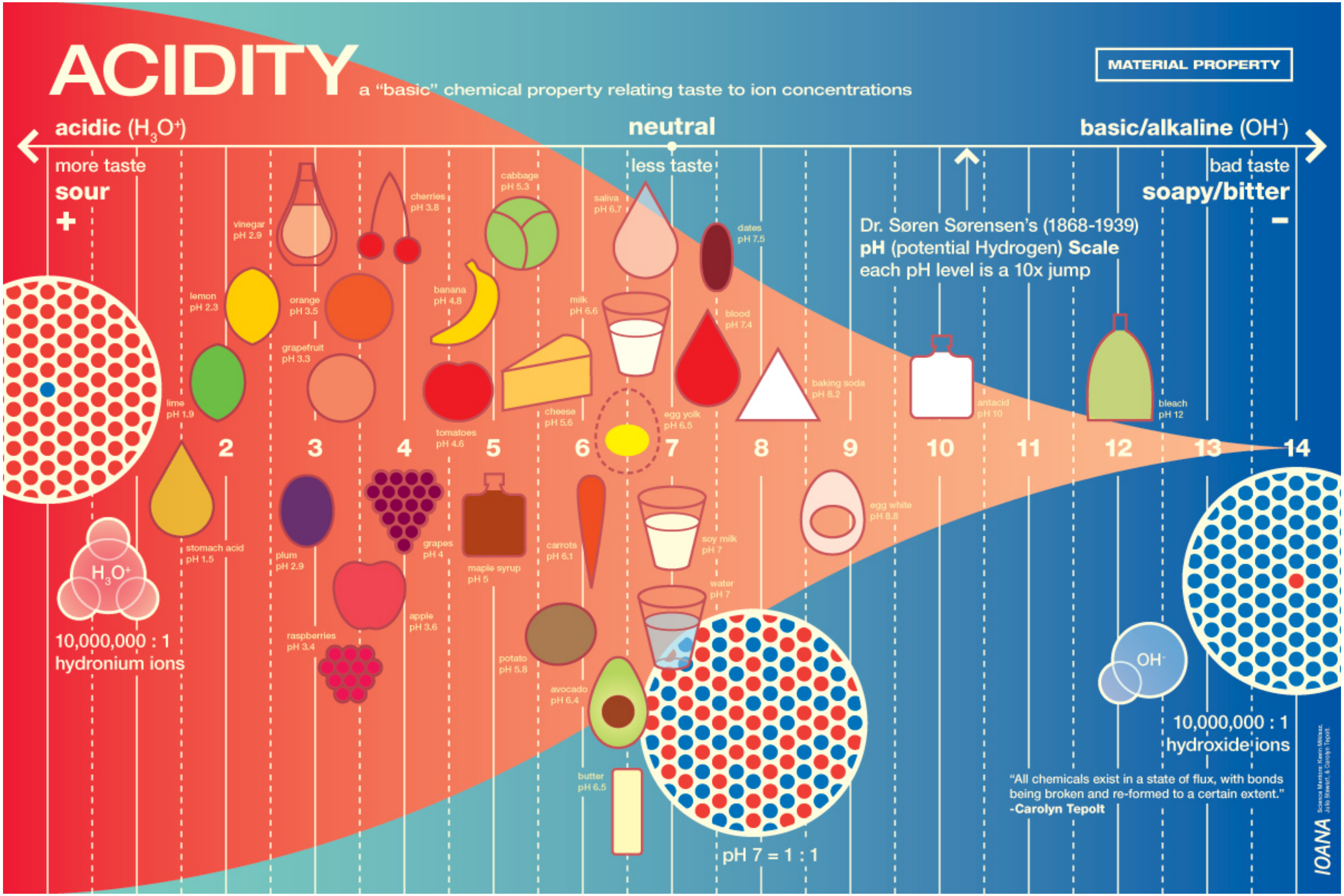
- $H_2O \rightleftharpoons H^+ + OH^-$
- In pure water, $[H^+] = [OH^-]$
- Equal (+) and (–) charges means that no voltage will be present

The pH Scale


Neutral
Acidic Range
Basic Range
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
Lemon
Wine
Milk
Gin
Soda
Coffee
Tap water
Soft Soap
Beer
Ammonia
Cola Beverages
Sodium Hydroxide
Hydrochloric Acid

Classification of Acidity in Foods

- The FDA 21 CFR defines guidelines for acidity of foods for their preservation:
  - Acid foods: Natural pH ≤4.6
  - Low-acid foods: Have a pH >4.6 and water activity >0.85 (exclusion for alcoholic beverages)
  - Acidified foods: Any low-acid foods that have had acid or acid food added such that pH ≤4.6.



ACIDITY
a "basic" chemical property relating taste to ion concentrations
MATERIAL PROPERTY
acidic (H_3O^+)
neutral
basic/alkaline (OH^-)
more taste
sour
+
less taste
bad taste
soapy/bitter
−
Dr. Søren Sørensen's (1868-1939)
pH (potential Hydrogen) Scale
each pH level is a 10x jump
vinegar pH 2.9
cherries pH 3.8
cabbage pH 5.3
saliva pH 6.7
dates pH 7.5
lemon pH 2.3
orange pH 3.5
banana pH 4.8
milk pH 6.6
blood pH 7.4
grapefruit pH 3.3
baking soda pH 8.2
antacid pH 10
bleach pH 12
lime pH 1.9
tomatoes pH 4.6
cheese pH 5.6
egg yolk pH 6.5
2
3
4
5
6
7
8
9
10
11
12
13
14
stomach acid pH 1.5
plum pH 2.9
grapes pH 4
maple syrup pH 5
carrots pH 6.1
soy milk pH 7
egg white pH 8.8
water pH 7
apple pH 3.6
raspberries pH 3.4
potato pH 5.8
avocado pH 6.4
butter pH 6.5
H_3O^+
10,000,000 : 1
hydronium ions
OH^-
10,000,000 : 1
hydroxide ions
pH 7 = 1 : 1
"All chemicals exist in a state of flux, with bonds being broken and re-formed to a certain extent."
-Carolyn Tepolt
IOANA

Methods to Measure pH

- Chemical methods
  - Litmus Paper
  - Test Kit
  - Photometer

- Potentiometric
  - Meter with Electrode

Classification of Acidity in Foods

ACID LEVELS (pH)

Food	*pH*	*Classification*
Most fruits	Equal or less than 4.6	NATURALLY ACID
Most peaches		
Most apples		
Most tomatoes		
Most orange juices		
Fresh fish	Greater than 4.6	LOW-ACID
Canned green beans		
Bread		
Fresh ham		
Most protein foods		
Most vegetables and starch-based foods		
Pickled foods	Equal or less than 4.6	ACIDIFIED

Source: *Food Microbiological Control Manual*, Federal and Drug Administration, 1998.

If the finished pH of the food is ≤4.00 any method can be used. If the finished pH of the food is >4.00, the final pH reading must be determined using potentiometric methods.

Are you measuring pH in house?

Bulb Shapes

A **spherical tip** is recommended for general use in aqueous or liquid solutions and provides a wide area of contact with the solution.

A **conical tip** is recommended for semi-solid products like emulsions, cheese, meat and food, in general.

A **flat** tip is recommended for direct surface measurement on skin, leather paper, etc…

Electrode Body Types

Glass

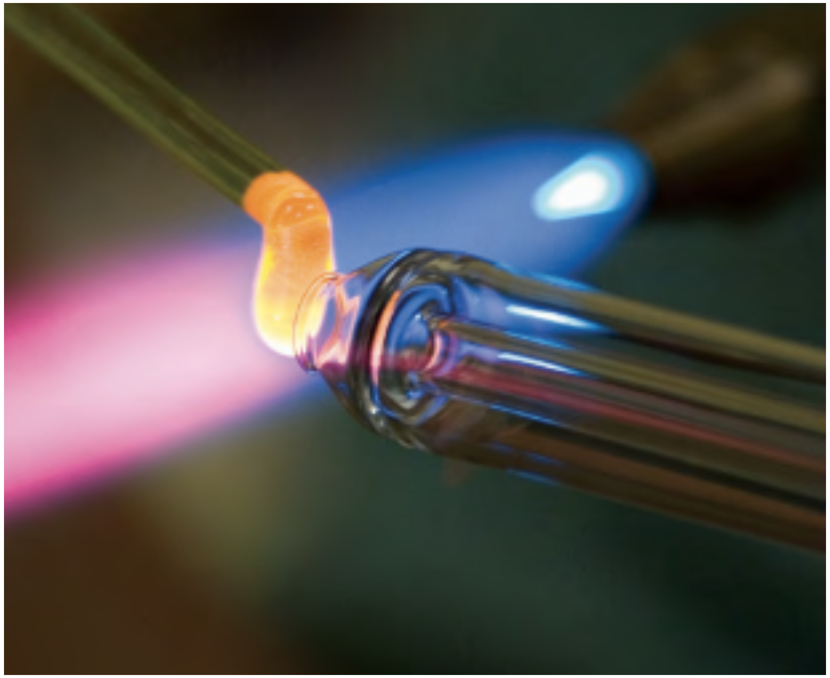
Plastic

Titanium

Stainless Steel

Hanna pH Glass Types

Identification	Range
Standard	pH = 0 – 13 0°C – 70°C
High Temperature	pH = 0 – 14 20°C – 100°C
Low Temperature	pH = 0 – 12 -10°C – 50°C
HF Glass	pH = 2 – 10 -5°C – 30°C



Types of Junctions

Single Ceramic | Triple Ceramic | PTFE | Open | Sleeve

Hanna Foodcare pH Electrodes

FC210: Creams, Yogurt, Sauces

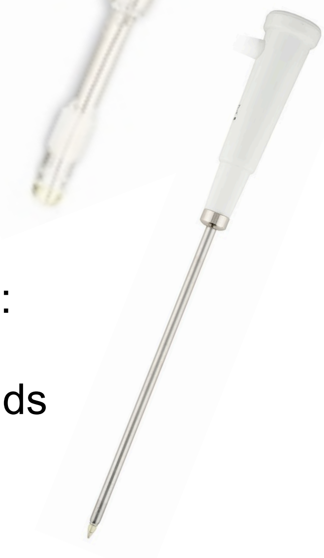
FC220: Sauces, Fruit Juice

FC230: Meat

FC202: Dairy Products, Semi-Solid Foods

HI1048: Wine Must, Juice, Viscous Samples

FC240B: Cheese, Soft Solids



Hanna Foodcare pH Electrodes

Electrode and Meter Configurations

BNC: standard electrode connection, require separate temperature probe

Benchtop (HI5221/2)

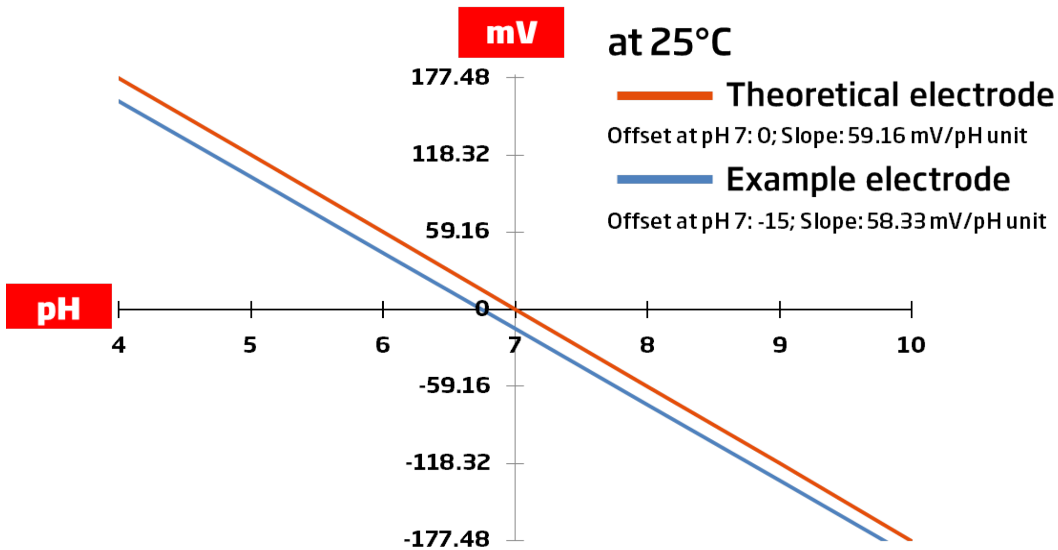
DIN: proprietary electrode connection, built in temperature sensor

Hybrid (edge®, HALO®)

Bluetooth: proprietary wireless electrode connection, built in temperature sensor

Portable (HI99163, HI98190)

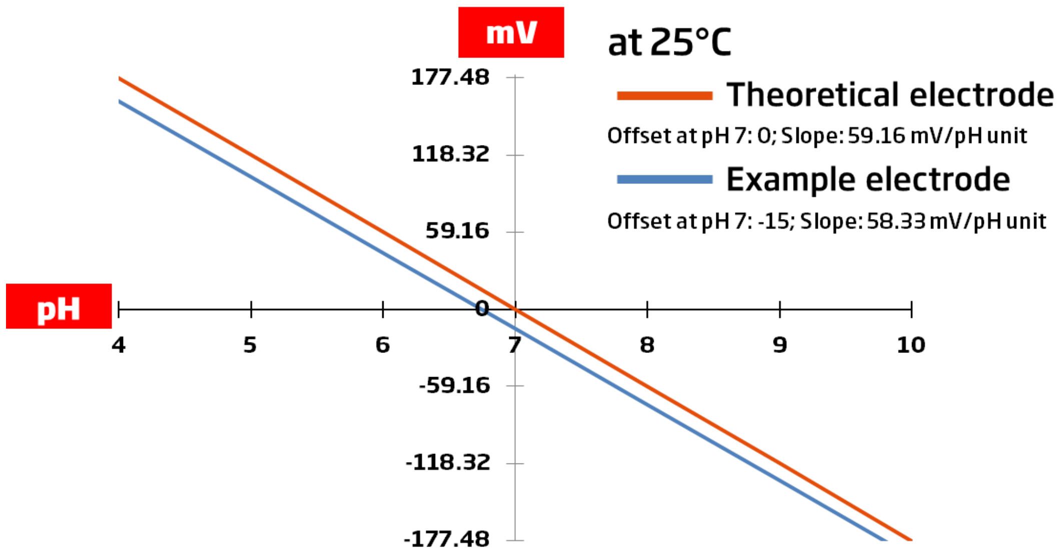
Why Calibrate?



pH electrodes differ from ideal behavior due to:

- Electrode condition and cleaning
- Electrode aging
- Manufacturing process

Why Calibrate?



Calibration tries to **compensate** for all the above effects without knowing which effect is causing the deviation from ideal behavior.

Measuring pH in Foods

- Calibration: bracket expected pH range
  - Always calibrate at pH 7.0 and at least one other point
  - Calibration points should be > 1 pH unit apart

Lemon:
pH 2.0

1.68, (4.01), 7.01

Milk:
pH 6.5

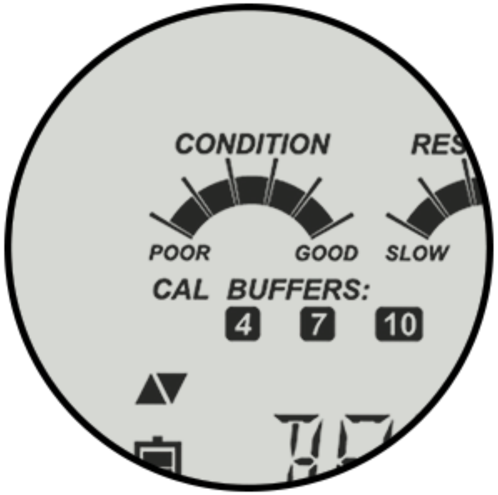
4.01, 7.01

Alka-Seltzer:
pH 12.00

7.01, (10.01), 12.45

CAL Check™

- Hanna-exclusive feature found in bench-tops and portables.
- Provides indicators to potential problems during the calibration process.



Store Electrode Properly

HI700300L: pH/ORP Electrode Storage Solution

If storage solution is not available electrode can be stored in pH 4.01 or pH 7.01 buffer.

Do not store electrodes in alkaline solutions long term.

NEVER store electrodes in deionized water.

Clean Periodically

HI8061L: General Purpose Cleaning Solution in FDA bottle

HI 7073: Cleaning Solution for Proteins

HI 70641L: Cleaning and Disinfection Solution for Dairy Products

HI70604L Cleaning Solution for Milk Deposits

HI70635L Cleaning Solution for Wine Deposits

HI8077L: Cleaning Solution for Oil and Fats in FDA bottle

HI70631L Grease and Fats Alkaline Cleaning Solution

HI70630L Grease and Fats Acid Cleaning Solution

Acidity and pH

- Acidity is the amount of acid in a sample, or the amount of base required to neutralize a sample (titration).
- pH is the strength of an acid, or the negative log of the activity of the hydrogen ion
- In foods, both the strength and quantity of the acid in solution is important, so both parameters are commonly monitored

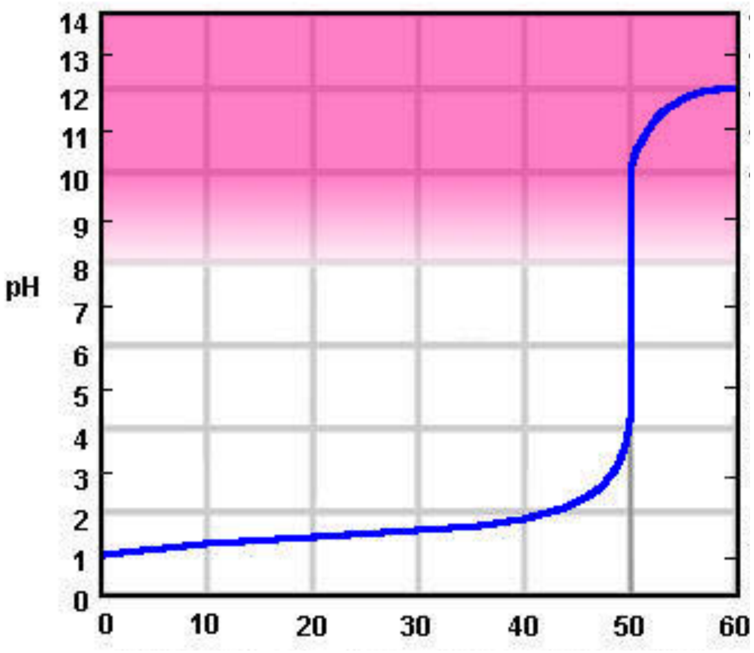
What is titration?

Analyte	• Substance being analyzed, unknown concentration
Titrant	• Substance being added to analyte, known concentration
Equivalence Point	• Point during titration at which the analyte and titrant are present in equal parts; theoretical end of titration
Endpoint	• Actual point at which the titration is terminated, determined by physical or sensing indicator

Acidity Determination

- All acidity titrations require a base as a titrant
  - NaOH most common
- Titrations are carried out to neutralization and are indicated by a fixed pH endpoint or an equivalence point
- Titration determines the amount of titratable acidity, but does not differentiate acids

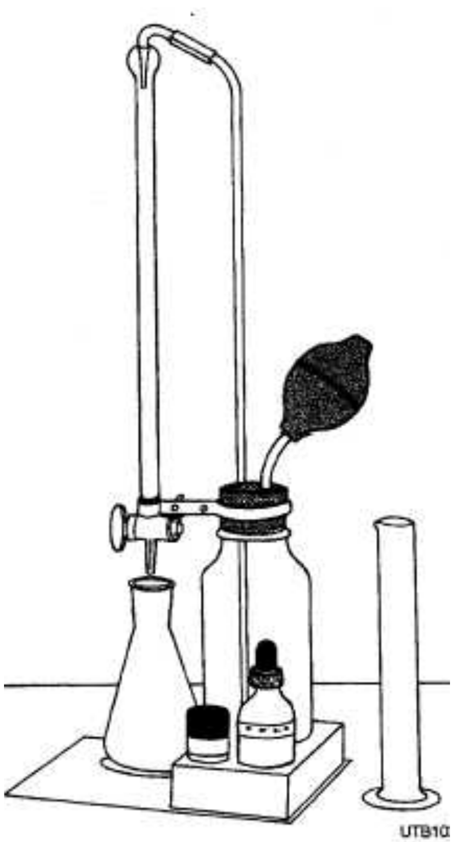
Acidity Titration



- Manual titration to color indicator
- Manual titration to fixed endpoint
- Manual titration to fixed endpoint or equivalence point

Do you perform titrations in house?

Manual vs Automatic Titration

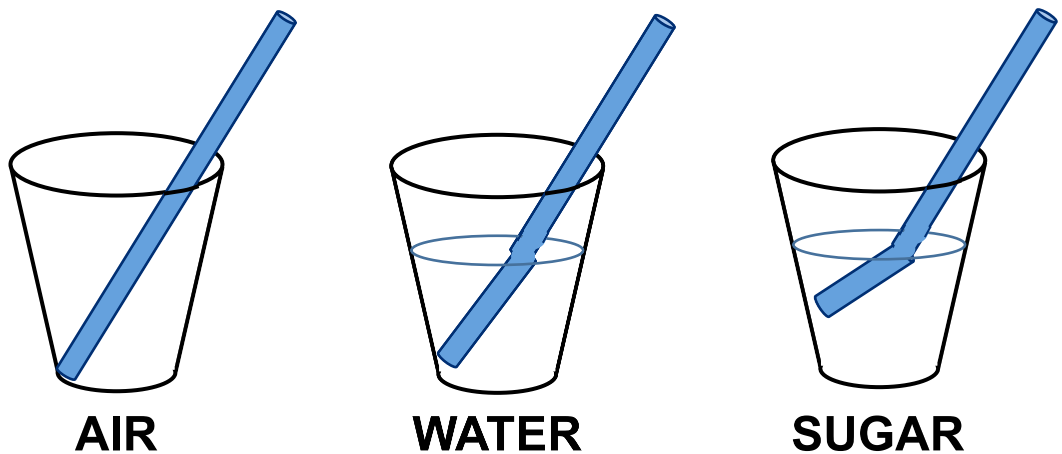


Sugar

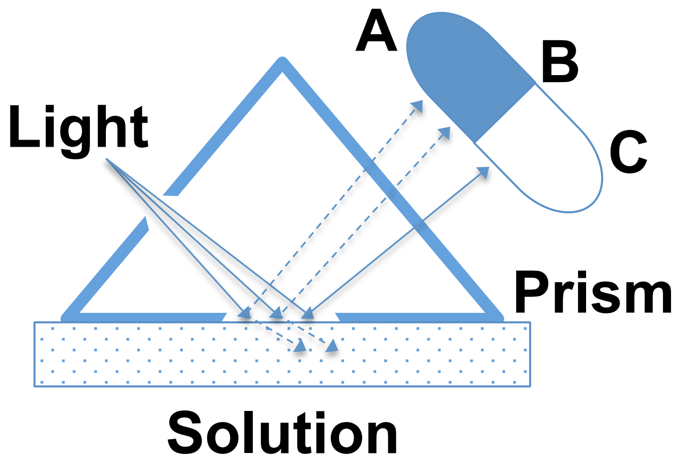
- Refractometry
- Nonspecific
  - Measure total refraction
  - Reports results in units of choice
  - Quantitative in binary solutions
  - Qualitative in complex solutions

Refractometry

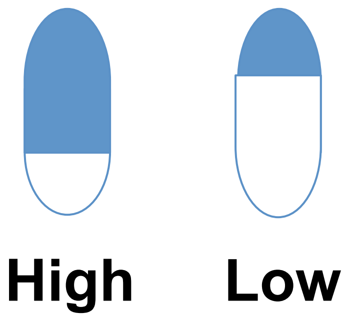
Three glasses with a straw in each	Air
	Water
	Sugar Water

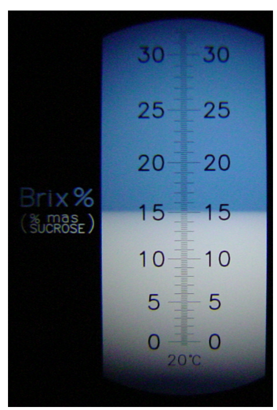


Refractometry

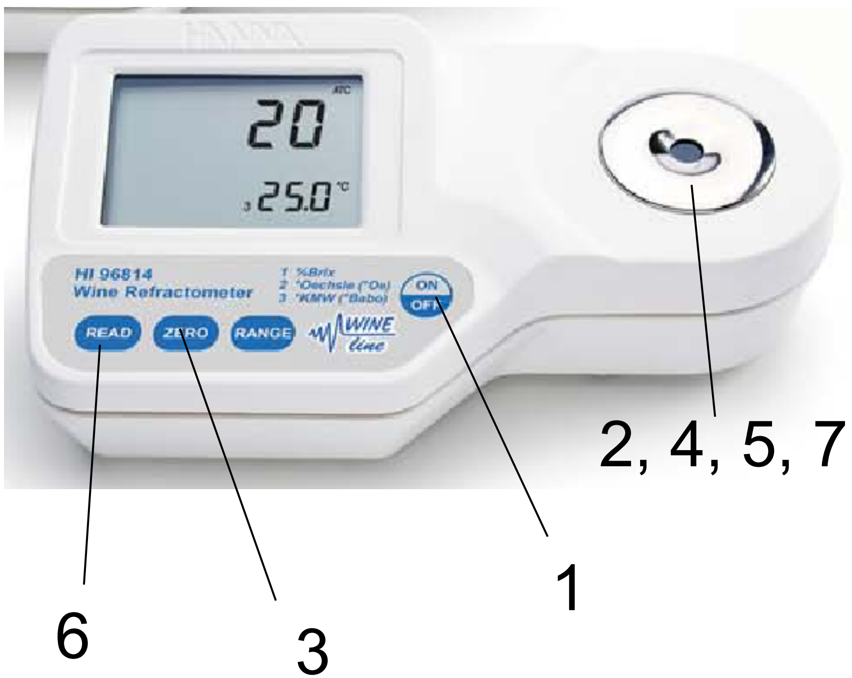


A – Total Refraction
B – Transition Point
C – Total Reflection





HI 968XX Measurement



1. Turn on
2. Add deionized or distilled water to sample stage
3. ZERO instrument
4. Wipe off prism
5. Add sample
6. Take reading
7. Wipe off prism for next sample

Hanna Refractometers

Model	Parameter	Range	Resolution	Accuracy
HI 96801	Sucrose (% Brix)	0 – 85 % Brix	0.1	+/- 0.2
HI 96802	Fructose	0 – 85 % mass	0.1	+/- 0.2
HI 96803	Glucose	0 – 85 % mass	0.1	+/- 0.2
HI 96804	Invert Sugar	0 – 85 % mass	0.1	+/- 0.2
HI 96821	Food Salinity	0 –28 g/100 g 0 – 34 g/100 mL 1.000 – 1.216 SG 0 – 26 °Baume	0.1 0.1 0.001 0.1	+/- 0.2 +/- 0.2 +/- 0.002 +/- 0.2
HI96800	Refractive Index Brix	1.3300-1.50580 nD 1.3330-1.5040nD_{20} 0.0-85.0% Brix	0.0001nD 0.0001nD_{20} 0.1% Brix	+/- 0.0005nD +/- 0.0005nD_{20} +/- 0.2% Brix

REMEMBER! Refractometers cannot differentiate between sugars, sugar/salt, etc.
Meters differ by internal algorithms only (calculation, ATC compensation)

Salt

- With regards to food, “sodium” and “salt” are not necessarily synonymous.
- Although most of the sodium in the food we eat is derived from NaCl, there are other potential sources of sodium including:
  - Baking soda
  - Other preservatives
  - Monosodium Glutamate

Salt in Food

Sample Preparation

- Most methods of analysis require liquid samples
- Solid foods required additional sample preparation

Source: Medical Expo

- **Slurries:** Salt is extracted from finely ground sample into water
- **Digestions:** Chemical treatment to facilitate extraction of salts

Salt in Food

Method Selection

- In methods of analysis, there are often multiple methods to measure the same parameter
- Method selection depends on:
  - Accuracy requirements
  - Sample matrix/sample prep
  - Ease of use
  - Type of industry and budgetary concerns
  - Prevalence of interferences

Salt in Food

Method Selection

- Simple Methods
  - Ideal for binary solutions or qualitative analysis
    - Conductivity
    - Refractometry

- Analytical Methods
  - Standard method compliance
  - Specific ion analysis
    - Titration
    - Ion Selective Electrode

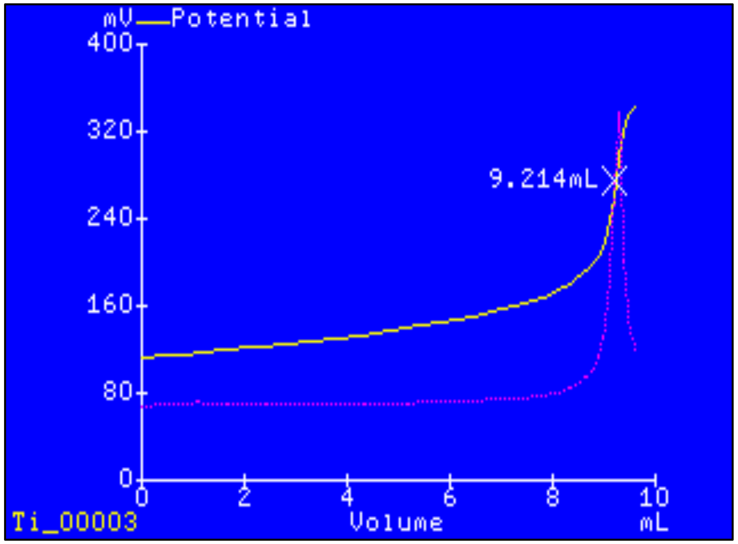
www.today.caricom.org

Titrimetric Analysis

- Direct measurement of chloride; NaCl inferred
  - $AgNO_{3\,(aq)} + Cl^{-}_{(aq)} \rightarrow AgCl_{(s)} + NO_{3}^{-}{}_{(aq)}$
- Most common method of analysis in standard methods
  - AOAC, IDF, ISO
- Can be performed manually to color indicator, or automatically to equivalence point

Titrimetric Analysis

- Direct measurement of chloride; NaCl inferred
  - $AgNO_{3\,(aq)} + Cl^-_{(aq)} \rightarrow AgCl_{(s)} + NO_{3\,(aq)}^-$
- Most common method of analysis in standard methods
  - AOAC, IDF, ISO
- Can be performed manually to color indicator, or automatically to equivalence point



Salt in Food

Method Selection

Method	Refractometry	Manual Titrimetric	Potentiometric
Apparatus	Manual or Digital/	Manual burette	Potentiometric titrator, chloride or silver-sulfide ISE
Principle	Sodium content is inferred from refraction	$AgNO_3$ reacts with Cl^-salts, color indicator detects EP	$AgNO_3$ reacts with Cl^- salts, endpoint occurs when Cl^- is no longer in solution
Cost*	$200	$300	$3000->$6000
Advantages	Simple, rapid	Inexpensive	Reference method, most accurate, automated
Disadvantages	Subject to interferences (salts, sugar, fats, acids)	Subjective, less accurate than titration	Capital Investment
Applications	Brines, semi-quantitative analyses	Finished product, brines	Finished product, brines

Moisture in Food

- Water is used in several stages of food production
  - **Processing and preparation**
  - Disinfection of equipment, etc.

- Water is a significant component of many foods
  - Added directly as an ingredient
  - Present as part of raw materials

Source: MEET-BIS.org

Free Water vs. Bound Water

- Water may be **free** and/or **bound**
  - Based on the chemical makeup of the food product

- The analytical method used to determine water is based on:
  - Sample composition
  - Customer preferred result

Source: latenitelabs.com

Analytical Methods

Loss on Drying

- Theory: measure the mass of a sample before and after drying
- Common method of analysis
- Can be performed via manual or automated analysis

Analytical Methods

Water Activity

- Water can be bound with salt, sugar, pectin, etc…
- A lower water activity means less water available for microorganisms

Analytical Methods

Water Activity

Range	Microorganisms	Examples
0.20-0.30	No growth	Milk powder, dried vegetables
0.30-0.40	No growth	Crackers, cookies, cereal, pet food
0.40-0.50	No growth	Flour, gum, dry beans
0.50-0.60	No growth	Pasta, spices, rice, wheat
0.60-0.65	Yeast, mold	Dried fruits, caramels, honey
0.65-0.75	Yeast, mold	Sugar cane, molasses, nuts
0.75-0.80	Halophilic bacteria	Marmalade, marzipan, beef jerky
0.80-0.87	Yeast, mold	Fruit concentrates, jams, jellies
0.87-0.91	Yeast, bacteria	Salami, dry cheeses, margarine
0.91-0.95	Salmonella, botulinum, yeast, molds	Cured meat, some cheeses, bread
0.95-1.00	E. Coli, pseudomonas, shigella, yeast	Canned fruits, vegetables, milk, fish

Analytical Methods

Karl Fischer Titration

- Titration to determine free and/or bound water
  - Titrant: iodine
  - Analyte: water
- HI903 Volumetric Karl Fisher Titrator
  - Higher concentrations of water
- HI904 Coulometric Karl Fischer Titrator
  - Lower concentrations of water

Determination of Moisture

Comparison of Methods

	Loss on Drying	Water Activity	Karl Fischer
Measures	All volatiles	Free water	Total moisture
Time for analysis	Several hours	Several minutes	Several minutes
Advantages	-Inexpensive	-Fast -Small instrument	-Reports water content
Disadvantages	-Time consuming -Measures volatiles -Requires several instruments	-Only measures free water	-Needs solvents -Large instrument

Conclusions

Bringing testing in house

- Faster turn around time on testing results
- Less $$$ per test
- Test more frequently
- Improve compliance
- Increase food quality and consistency

Questions & Answer Session

Thank you!

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