Foreign Material, Unknown Objects, Ingredient Safety Considerations in Foods, Beverages, Ingredients, Supplements

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Presentation Overview

Introduction

Brand, Regulatory Considerations

Background on Analytical Techniques

Considerations with Testing Partners

Case Studies

Speakers, Contributors

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About Eurofins SF Analytical

- New Berlin, WI & Santa Ana, CA labs
- Combined over 40 Scientists, 3 PhD's
- More than $7mil of State of the Art Instruments
- Problem solving & Investigation-based analysis since the early 1970's.
- Thousands of Successful Investigations to date
- "The lab to call when you don't know who to call." ESFA_Project@eurofinsus.com

Part of the Greater Eurofins Network of ISO 17025 Laboratories.

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RAFFLE ALERT!

- Attendees today will be placed in a drawing for a $25 gift card!
- 5 Lucky winners will be selected and notified within 3 business days of our presentation.
- Thank you for attending!

Identifying Risk

- Risk is unavoidable and ever evolving.
  - Pandemic, climate change, regulatory updates, evolving consumer demands.
  - Leads to shortages of labor and supplies ➔ More Risk.
- To start, identify your hazards.
  - Upstream: Ingredients, Packaging, Process Aids
  - Change in Co-packers, Process for Manufacture
- Three main types:
  - Physical
  - Chemical
  - Biological

Why Assess Safety?

- Requirement under: Food Safety Modernization Act (FSMA).
  - 21 CFR part 111 for Dietary Supplement Ingredients
  - 21 CFR part 210/211 for OTC, Pharma, Excipients
- Increase quality, product performance, control over supply chain, confirm product label claims, enabling increased market share, improving your “bottom line”.
- Critical to bolster product and brand stewardship including your Company’s reputation. Negative Publicity can destroy a company or brand forever.
- Identify problems quickly, prevent incident from impacting production or product into marketplace
- It’s “The Right Thing to Do”.

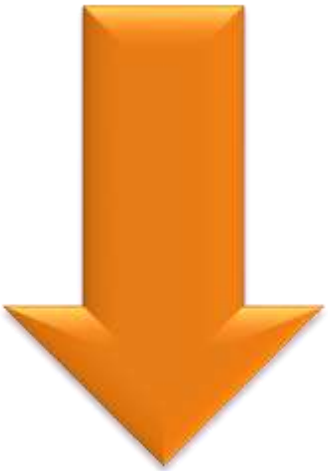
What Could Happen?

- Food Fraud and Adulteration
  - Unknown materials, undeclared allergens, colorants, drugs, and other ingredients.
- Packaging Failures – Spoilage and loss of product
- Product Rejections, Recalls, & Quarantines
- Harm to Consumers
- Negative Publicity
- Damage to brand, reputation
- Legal Action

Loss of your business and trust in your brand.

What to Do? Next Step?

- It is not about removing risk, but managing, reducing, and preventing through a robust Quality Management System and the right partners to assist with foreign material, unknown objects in ingredients, products
- What preventative tools and procedures do you have in place to assess and prevent risk to your ingredients?



Verifying your suppliers routinely is a key component to managing risk.

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Poll Question 1

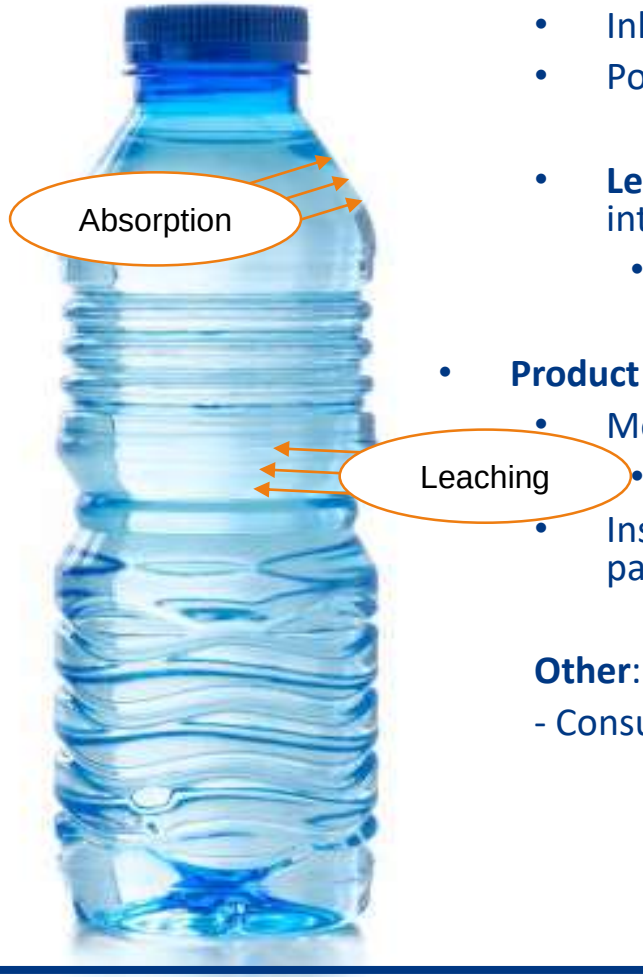
According to multiple industry sources, what was the Number One consumer complaint in food and beverages in 2021?

A) Foreign Bodies
B) Unlisted Ingredients or Allergens
C) Damaged Packaging

Answer = A) Foreign bodies

- Foreign bodies form the biggest single cause of consumer complaints received by many food and drink manufacturers, retailers and enforcement authorities. The accidental inclusion of unwanted items may sometimes occur in even the best managed processes. Foreign bodies in foods are therefore quite rightly a matter of concern to all food manufacturers and retailers

Packaging Issues Can Lead to Foreign Material in Product



- **Packaging incompatibility:** packaging interacts with product
  - Acidic product or Reactive Anions degrades an improperly coated aluminum can or coated package
  - Ink can migrate thru labels, packaging into ingredient, product
  - Potential safety concern!

  - **Leaching**: unwanted molecules from the packaging material migrate into the product
    - Potential safety concern!

- **Product degradation:** degradation of profile components during storage.
  - Molecules sensitive to storage conditions
    - e.g. Light sensitive or heat sensitive
  - Insufficient preservatives or antioxidants leads to precipitation after packaging or during storage, transportation to downstream customer

Other: inadequate bottle cleaning allows “sticky particles” to remain

- Consumer backwash food, medicine into opened bottle

Sources of Contamination

Chemical

- Storage issues
  - Leaching from storage receptacle (e.g. holding tank)
  - Leaching from packaging
- Improper Cleaning
  - Insufficient rinsing procedure
  - New cleaning agent
- User error
  - Excess lubricant added to machinery during maintenance contaminates product line

Microbial

- Improper storage
  - Too long at room temperature
- Improper handling
  - Poor hygiene
- Cross-contamination
  - Exposure to raw product (e.g. meat)
- Processing failure
  - Incomplete pasteurization

Production Issues

- Equipment failure
  - Oven fails to reach proper temperature leading to spoilage or microbial growth at later date
- Process failure
  - Failure to complete defatting/pasteurization, etc.
- User error
  - Failure to recognize out of calibration equipment leads to improperly measured or processed ingredients
- Ingredient issue
  - Impure ingredient due to incomplete reaction
  - Change in quality of ingredients with new supplier

In many cases, foreign material contamination is caused by a combination of inspection, system and human errors!

Analytical Techniques

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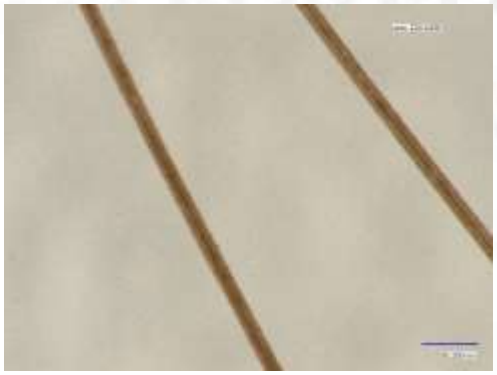
Advanced Microscopy

- Keyence Digital Microscope creates compelling digital images
- 10X to 1000X Magnification

Glucosamine Crystals



Comparison of Hair from Different Animal Sources

Ant Isolated from a Bottle

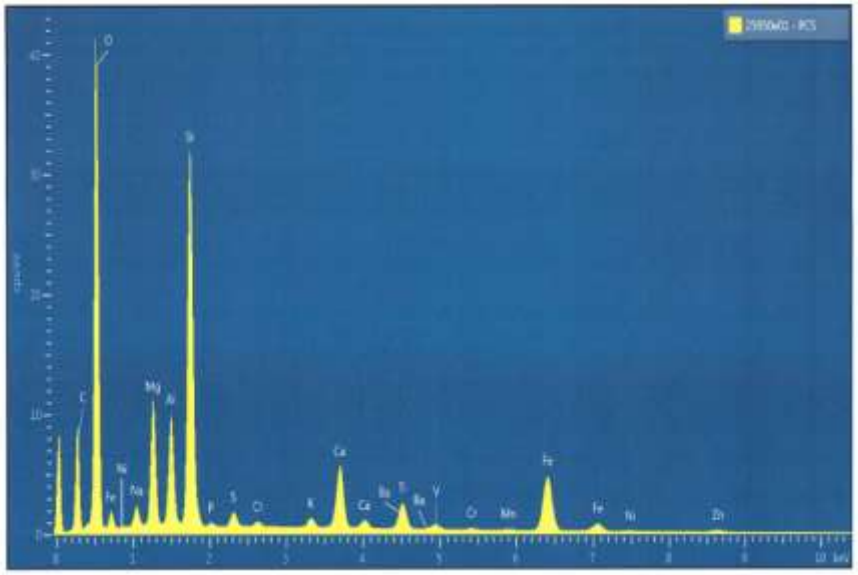
Foreign Object Found in Beverage

Glass Shard w/ 3-D imaging

SEM-EDS

Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Analysis (EDX) can be used for:

- Foreign Material Identification
- Corrosion on Metal Packaging
- Ingredient Morphology
- Failure Analysis, Pinholes in Packaging
- Elemental Images in Layers
- Multi-Layer Packaging, Film Analysis
- Semi-Quantitative Elemental Scan



Combustion/Ash Residue EDX Scan

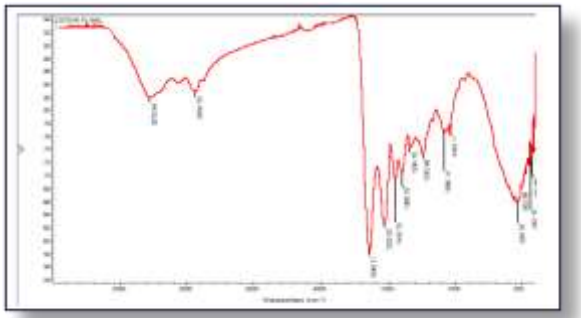
Spectroscopy

FTIR

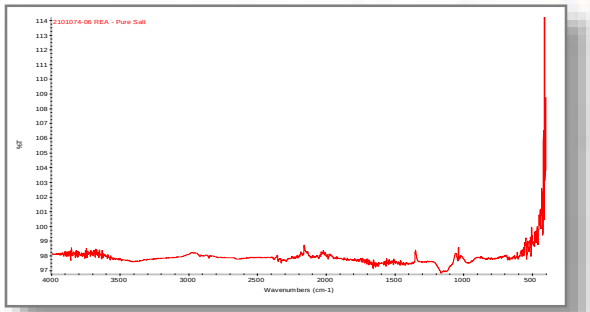
- Obtain spectra to identify organics and select inorganics by measuring absorption of IR

MicroFTIR

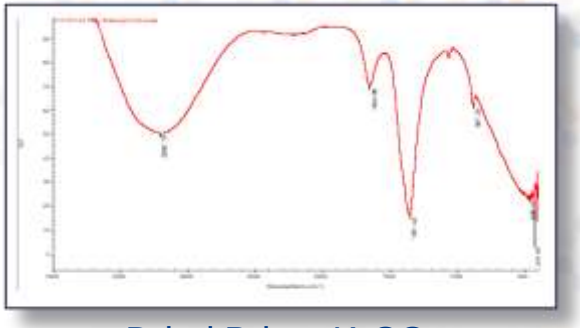
- Collect FTIR spectra of tiny particles or object/filter surfaces



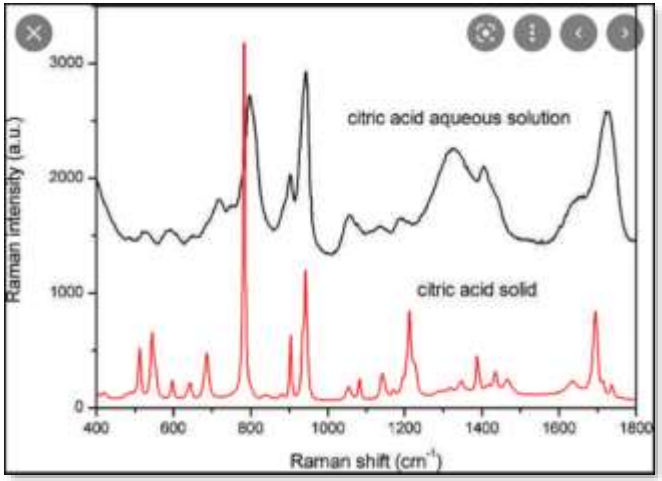
Brined Pork



Dried Brine: NaCl



Dried Brine: K_2CO_3



Raman

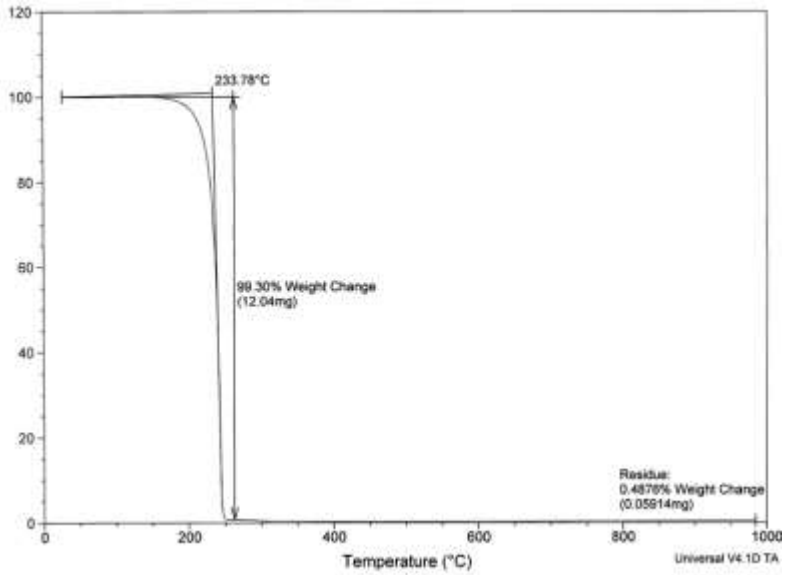
- Obtain spectra of materials by measuring light scattering
- Useful for bonds FTIR can't discern
- Mineral, gas phase inclusions
- Crystal Forms (charcoal, graphite, diamond)

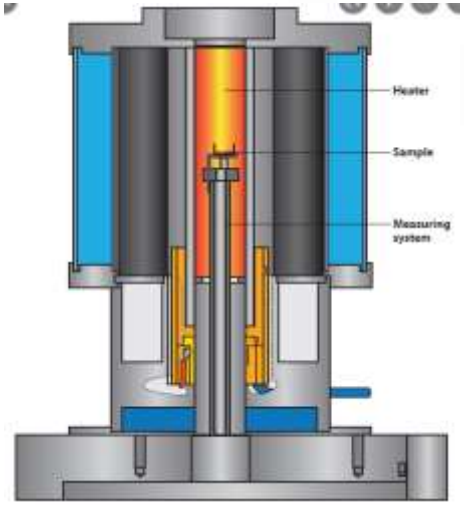
Physiochemical Analysis

Moisture

Residue on Ignition/Thermal Gravimetric Analysis (TGA)

- Measures a material's thermal stability and its volatile fraction by monitoring the weight change that occurs as a sample is heated





Inductively Coupled Plasma (ICP)

- Elemental detection –
  - Nutritional Minerals (eg. Ca, Mg, K, Se)
  - Metals (including heavy metals): As, Cd, Hg, Pb
- ICP-OES (Optical Emission Spectroscopy) or MS (Mass Spectrometry)
- Mass Spec provides lower level detection

Chromatography

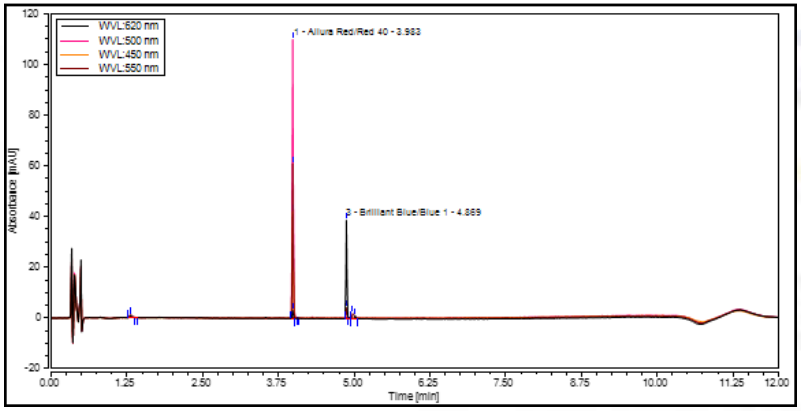
Liquid Chromatography:

- Non-volatile components
- Detectors: MS, UV, ELSD, RI
- E.g. Organic acids, dyes, sugars

Gas Chromatography:

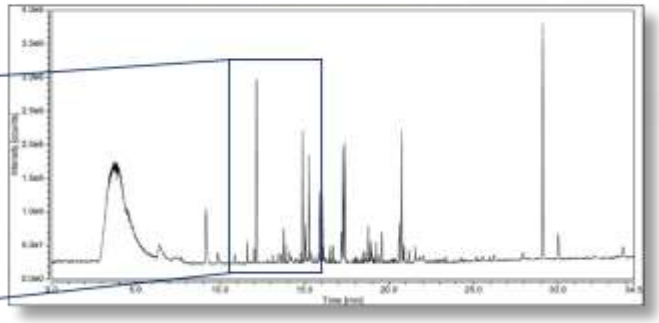
- Volatile components
- Detectors: MS, FID, TDC
- E.g. solvents

Dyes in a Purple Drink



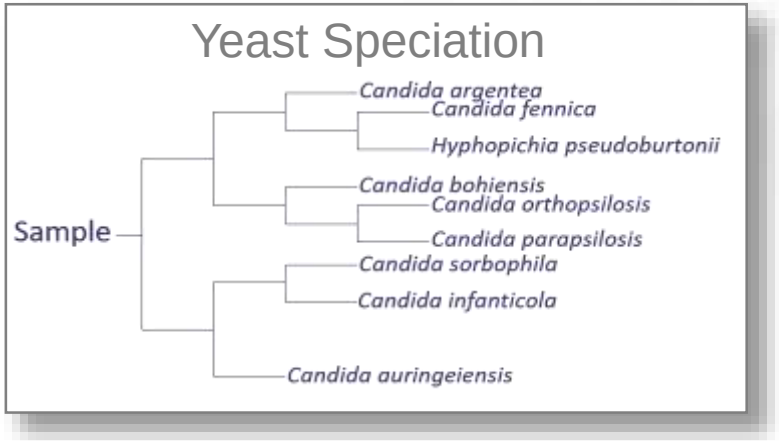
Compound	RT (min)	Odor/Aroma
Methyl-pyrazine	11.28	Nutty, roasted
2,5-dimethyl-pyrazine	12.31	Nutty, roasted
2,6-dimethyl-pyrazine	12.41	Nutty, roasted
4-propyl-pyridine	13.40	Green, fatty
2,3,5-trimethyl-pyrazine	13.74	Nutty, roasted
Acetic acid	14.27	Sour, acidic
1-(acetyloxy)-2-propanone	14.50	Fruity
Furfural	14.56	Bready, baked
Benzaldehyde	15.65	Almond, cherry

Volatiles in Coffee



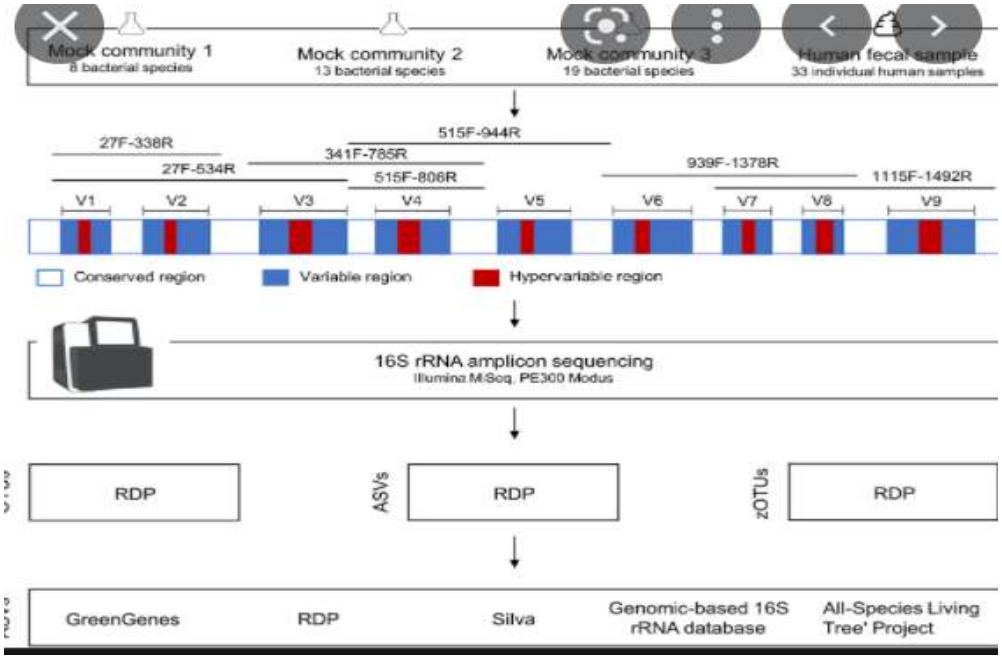
Microbial Screening & DNA Analysis

- Identify and/or quantify microbes
- Yeast, mold, bacteria such as *E. coli, Salmonella, listeria, Staph*, pathogens
- Microbial Speciation by custom-culturing followed by 16S Long Sequence Speciation in cases where Microbial ID is important to prevent reoccurance.



DNA Testing, Microbial ID

- Speciation for biological specimen in ingredients, beverages, products is often warranted when problems occur.
- Custom Culturing in 2-4 media types with 24-72 hour incubation at various conditions is employed
- Next we photograph and image dominant colony forming units using special lights, dyes.
- Dominant CFU's can often be identified by Metagenomics, 16S Long Sequence Speciation if warranted.



ELISA/ICT Assay

- Screen or quantify proteins of interest (eg. Human hemoglobin, allergens, antigens)
- Can often be performed quickly with 24-72 hours turnaround time
- Enzymatic assays for protease characterization is useful for determining why commercial thickeners fail prematurely
- Guar/xanthum gum characterization is another application of this type of testing
- ELISA kits are also useful in forensic investigations

Suspected blood residue on can (human or animal?)

Poll Question 2

Since January 2019, allegations of US Food, Beverage, and Ingredient Adulteration and related Insurance Claims have increased on average:

A) Less than 15% per year
B) 15-25% per year
C) More Than 25% per year

Answer = C – More than 25% per year

32% CAGR 2019-2022 with vegetable oil and honey adulteration claims increasing over 100% since 2020

Testing Lab Requirements

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External Testing Lab Considerations

Routine Testing Lab:

- Focus on certain sample types or tests: food, pharmaceutical, environmental
- Experienced in certain testing
- High throughput focus

Non-Routine Testing Lab:

- Specialized focus on solving obscure and complex problems
- Utilize wide array of techniques including orthogonal techniques if warranted
- Experienced problem solvers – access to credible Scientist(s)

Testing Lab Requirements

- Ethical, no conflict of interest (independent, objective)
- Client references
- Confidentiality/attorney – client privilege
- Chain of custody, securing/storing samples
- Courier Network – are they local or can they support you nationally or on global basis?
- Capabilities, quality certifications, technical credibility
- Turnaround time and sensitivity to delays. RUSH or EXPRESS Capability?
- Capacity, instrumentation, experience, industry-specific expertise
- Ask specific, targeted questions

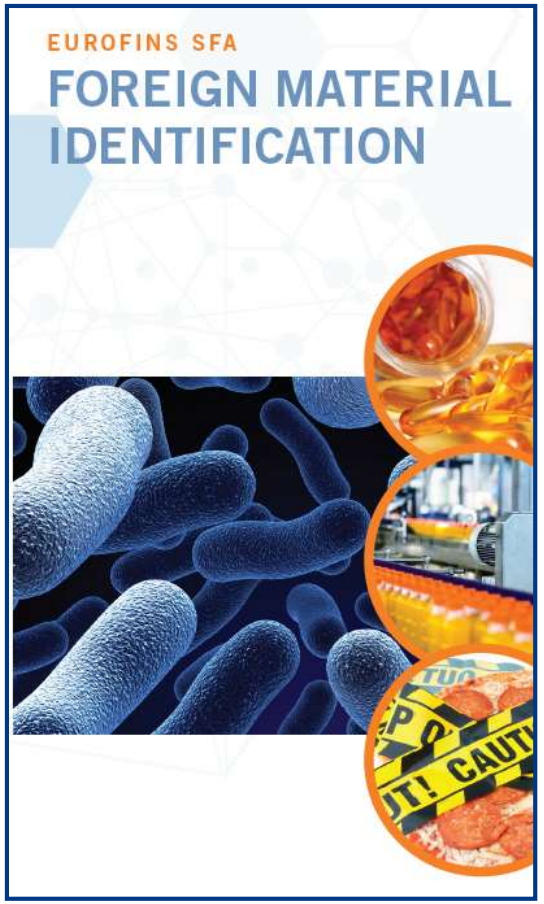
Using the Results

Deliverables:

- Data, interpretation, written reports
- Access to Scientists at each step. Are the right questions being asked?
- Determine source/cause
- Expert Services, Affidavits, Depositions, Courtroom Testimony

Case Studies

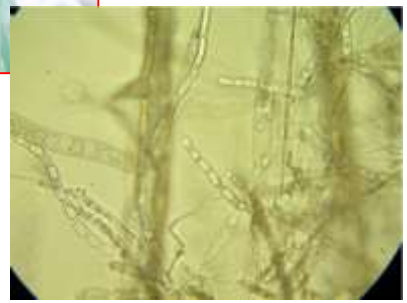
1. Gas Formation in Bottled Juice
2. Alleged Rodent Contamination of a Bun
3. Particulate Material in a Cheese Product
4. Pharmaceutical Tablet Discoloration
5. Floaters in Liquor
6. Insect Artifacts in Non Dairy Creamer
7. Combustion By Products in Dietary Supplements
8. Microbial Contamination in Dietary Supplements Process
9. Ink Migration in Jello Shots

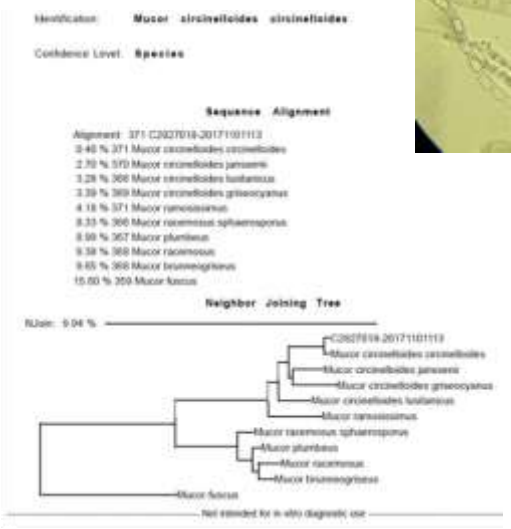


Case Study 1

Gas Formation in Bottled Juice

- Problem
  - Hot-packed juice product
  - Cloudiness a few weeks later
  - Gas formation
- Approach
  - Yeast/mold culturing, testing
  - Microscopy
  - Gene sequence-based ID
- Solution
  - Fungus contamination
  - Packaging process modified





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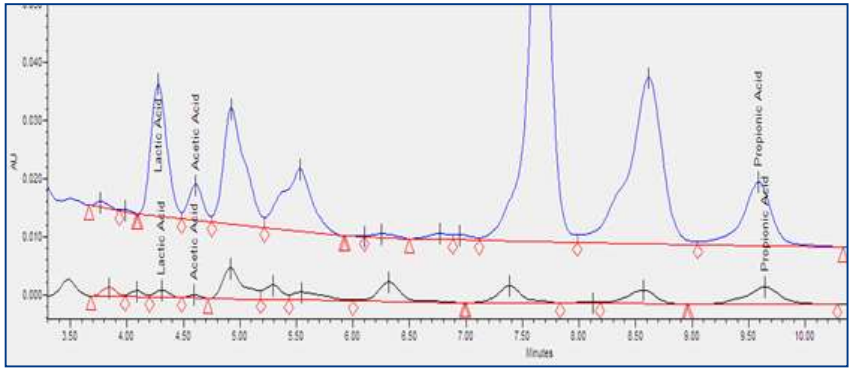
Case Study 2

Alleged Rodent Contamination of a Bun

- Problem
  - Consumer claimed rodent contamination
  - Large legal claim filed against our client

- Approach
  - Microscopy and chemical analysis (routine, HPLC)
  - Protein, starch, organic acid, preservatives
  - Comparison to reference bun

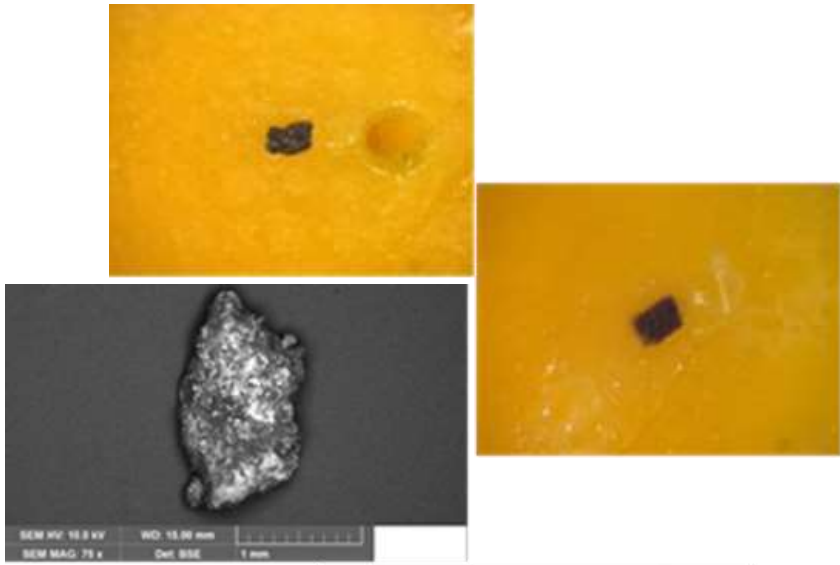


- Solution
  - Organic acid/preservative content differed
  - Suspect bun not manufactured by client
  - Favorable outcome for client

Case Study 3

Particulate Material in a Cheese Product

- Problem
  - Small pieces of dark material
  - Found prior to packaging
  - Suspected corroded metal or carbon
- Approach
  - Non-destructive testing needed
  - SEM-EDXA
- Solution
  - Metallic element content very low
  - Carbon, oxygen content high
  - Morphology examined
  - Confirmed to be filter media



Element	Weight %	Atomic %	Net Int.
C K	68.8	77.9	16699.48
O K	21.38	18.18	3777.08
Fe L	2.11	0.51	208.57
Na K	0.86	0.51	299.63
Mg K	0.43	0.24	205.7
Al K	1.11	0.56	510.49
Si K	0.91	0.44	399.87
P K	1.15	0.51	386.14
S K	0.29	0.12	91.61
Cl K	0.67	0.26	174.55
K K	0.34	0.12	62.65
Ca K	1.94	0.66	266.73

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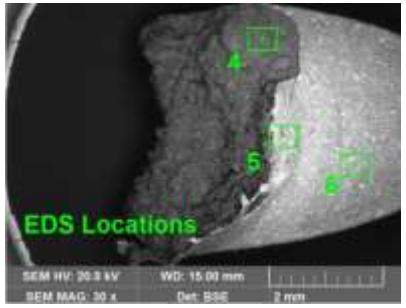
Case Study 4

Pharmaceutical Tablet Discoloration

- Problem
  - Client observed tablet discoloration
  - Contamination, formulation problem?
- Approach
  - Isolate discolored/normal spots
  - Spatial variation
  - Reference comparison
  - FTIR – organic compounds
  - SEM-EDXA – elemental analysis
- Solution
  - FTIR - inconclusive
  - EDXA – variation in Ti (TiO_2)
  - Possible light degradation

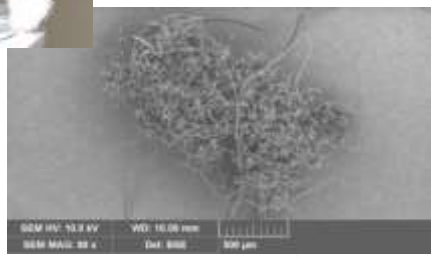


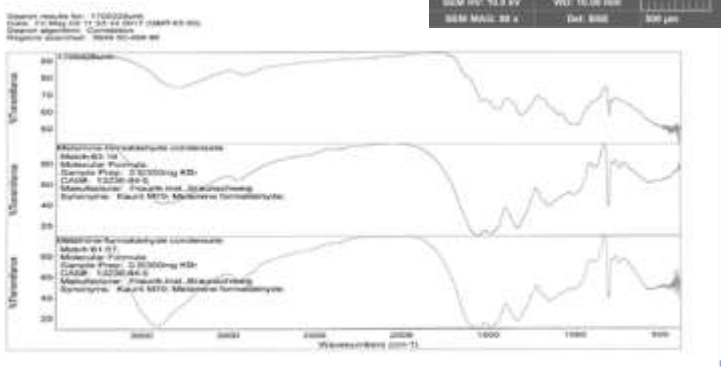
Analyte	Spot 4	Spot 5	Spot 6	Control
Titanium	0.26%	12.87%	16.43%	15.46%

Case Study 5

Floaters in Liquor

- Problem
  - Floating material observed in bottled liquor
  - Appeared after a water treatment change
- Approach
  - Filtered/isolated foreign material
  - Appeared to be fibrous
  - SEM – Morphology
  - FTIR – Organic Material
- Solution
  - SEM – unique fiber structure
  - FTIR- biological composition
  - Solution: Better filtration to
  - eliminate biofilm





Case Study 6

Insects, Artifacts in Bulk Non-Dairy Creamer

- Problem
  - Insect Infestation in Truckloads of supersacks or raw materials from overseas used to fill non-dairy, powered creamer containers at co-packer. Over 80Klbs of product quarantined, possible Insurance Claim. Were insects from overseas or local via co-packer in USA?
- Approach
  - Inspected, Sampled, Sieved powder from population of unopened, work in process supersacks, Inspected co-packer, review pest control records, spoke to operators, also compared with insects found in packaged product during final QC inspection
- Solution
  - Optical Microscopy of Insects, Pupae, artifacts found in sample population and comparisons
  - Insect ID by Entomologist determined local insect, NOT from Europe.
  - Favorable Outcome for client

Case Study 7

Combustion By Products in Dietary Supplement Factory

Problem: **Suspected Fire in dietary supplements factory could have resulted in hazardous combustion by-products being dispersed throughout facility including raw material, work in process and packaging, finished product areas impacting operations and product quality, purity, safety.**

Field Sampling: **Sterile 70% IPA prep (swab) samples where collected and placed in sterile vials according to scientifically defensible sampling plan outlined by ESFA scientists according Visual Area Estimation as defined in EPA 600/R-93/116 across 10 cm x 10 cm area (about 16 square inches) wearing sterile gloves**

Analysis Performed: **Level 2,3 Combustion By-Product Analysis for Soot, Char, Ash using a variety of optical, electron microscopy methods (SEM/TEM) were used including Confirmatory identification of aciniform soot by Transmission Electron Microscopy (TEM) per ASTM D6602-13 allowing fast return to operations by client after several rounds of area sampling/testing**

Sample Identification:	200-2022-04220047				
Sample Description:	Sample 11 – Scoops				
Combustion-by-Products:	(%)	**Fibrous Particulate:**			(%)
Char	<1	Asbestos:		(Total)	ND
Ash	ND	MMVF's:		Fibrous Glass/RCF's	ND
Black Carbon /Soot	ND			Mineral Wool	ND
		Cellulosic:		Processed/Paper Pulp	15
Common Minerals/Construction Dust:	(%)			Natural/Wood	5
Quartz	5			Starch	15
Calcite/Dolomite	2	Synthetic:		(Total)	15
Gypsum/Anhydrite	2	Hair:		Human	ND
Clays/Feldspars	1			Animal	ND
Biological:	(%)		**Additional Particulate:**		(%)
Mold	<1			Paint	2
Pollen	1			Rubber Dust	ND
Diatoms	ND				
Insect Fragments	ND				
Dust Mites	ND				
Skin Fragments	30	**Unidentified:**		Opaque Particles	5 (A)

LOQ: 1% (by Visual Area Estimation Method)

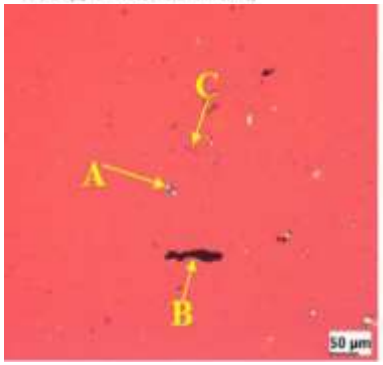


Figure 6. PLM image of particles in sample "200-2022-04220053"
A: Starch
B: Rust/Metal Dust
C: Skin Fragment

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Case Study 8

Dietary Supplement Factory Microbial Contamination, ID

Problem: **Microbial Contamination from spore forming ingredients, poor cleaning, decontamination practices, municipal water use in facility including raw material, work in process and packaging, finished product areas impacting operations and product quality, purity, safety including non-conforming product.**

Field Sampling: 28 Bulk raw material, finish product samples as well as area swab samples (4" x 4" areas of plant, process equipment) were collected according to sampling plan and testing/acceptance protocol drafted by ESFA scientists.

Analysis Performed: Custom Culturing, Incubation followed by photography of dominant colony forming units for harvesting was performed by ESFA GMP Microbiology Lab in Horsham, PA followed by metagenomics 16S long sequencing, speciation to ID source of contamination as bacillus strains enabling improved cleaning, disinfection of process areas, equipment, municipal water system and 10X reduction in out of spec product afterwards.

HiCulture Swab used with 3M phosphate buffer as collection solvent

Eurofins Sample ID	Sample Description	Sample Type	TAPC Result	Clean/ Dirty?*
996-2022-02240038	Gowning Hand Sink Rm 114	Water	920 cfu/mL	Dirty
996-2022-02240039	Weigh Stainless Table Rm 105	Swab	10 cfu/swab	Clean
996-2022-02240040	Weigh Scale (1 Floor) Rm 105	Swab	< 10 cfu/swab	Clean
996-2022-02240041	Weigh Scale (1 Bench Top) Rm 105	Swab	1,480 cfu/swab	Dirty
996-2022-02240042	Weigh Water Supply Sink Rm 105	Water	9,300 cfu/mL	Dirty
996-2022-02240043	Weigh Water Supply Sink Rm 104	Water	1,130 cfu/mL	Dirty
996-2022-02240044	Mixer 1 Rm 103	Swab	< 10 cfu/swab	Clean
996-2022-02240045	Mixer 2 Rm 106	Swab	< 10 cfu/swab	Clean
996-2022-02240046	Mix 1 Drain Rm 103	Swab	< 10 cfu/swab	Clean

Eurofins Sample ID (TAPC)	Sample Description	Sample Type	TAMC Result	Eurofins Sample ID (Speciation)	Identification
996-2022-02240038	Gowning Hand Sink Rm 114	Water	920 cfu/mL	996-2022-03030077	*Mycobacterium mucogenicum / phocaicum*
996-2022-02240042	Weigh Water Supply Sink Rm 105	Water	9,300 cfu/mL	996-2022-03030078	*Acidovorax temperans*
996-2022-02240049	Mix 2 HVAC Return Grill Rm 102	Swab	30 cfu/swab	996-2022-03030079	*Weizmannia coagulans*
996-2022-02240056	Wash HVAC Return Grill Rm 102	Swab	270 cfu/swab	996-2022-03030080	*Bacillus velezensis*
996-2022-02240057	Wash HVAC Exhaust Grill Rm 102	Swab	100 cfu/swab	996-2022-03030081	*Bacillus velezensis*

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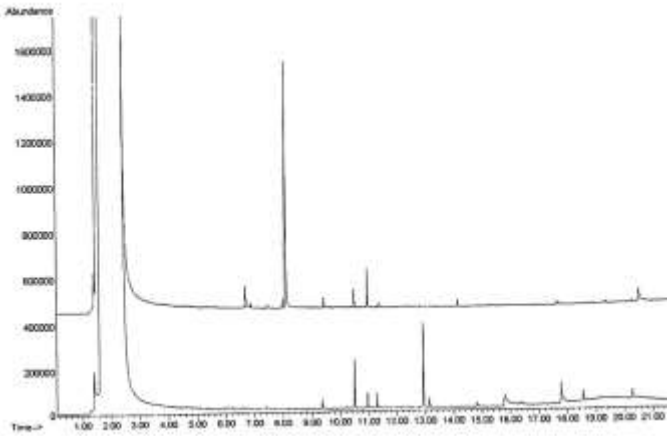
Case Study 9

Ink Migration into Jello Shots

- Problem
  - Bitter Taste in Jello Shots, customer complaints
  - Appeared after move to new co-packer and automated packaging line to support rapid expansion in jello shot demand.



- Approach
  - Compared good, suspect jello shots
  - Extractions, FTIR, GCMS, LCMS screens
  - Compared Results
- Solution
  - Caprolactum found to be contaminant
  - Discussions, questions with co-packer determined slip sheet on foil lids was eliminated to save cost.
  - Caprolactum in Ink was migrating to underside of jello shot closures when lids were stacked feeding into automated packaging line
  - Slip sheet added, different ink/cure process used on top of closure label to eliminate migration

Summary & Conclusions

- A wide portfolio of techniques exist for isolation, identification of foreign materials, unknown objects or analyte migration via product packaging
- Proactive Inspection, Testing, Risk Mitigation measures can reduce risk and help improve company brand, reputation with customers
- There are many factors to consider if engaging a 3rd party lab for this kind of investigation.

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Questions?

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