Establishing Standards on Colors from Natural Sources

Recommendations from an Expert Panel

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Eric A. Decker, Mario G. Ferruzzi,
Carla D. Mejia, Stephen T. Talcott

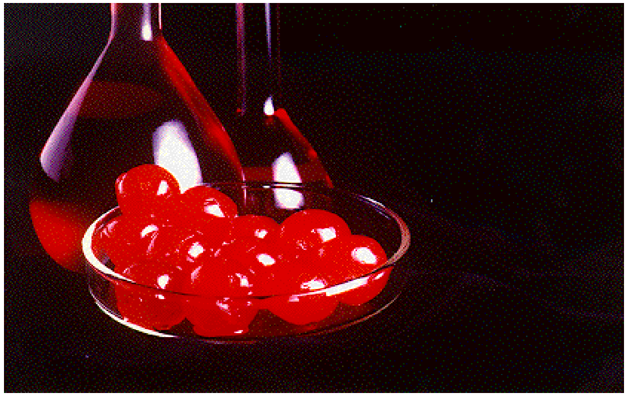
Dr. M. Mónica Giusti

- Food Engineer, UNALM (Peru)
  (Universidad Nacional Agraria La Molina)
- MS and PhD from OSU in Food Science
  - from OSU… West (Oregon State U.)
- Joined “The” OSU (Ohio State) FST in 2003

- 2 patents granted, 5 more pending
- 2010 OARDC Director’s Innovator of the Year
- 2011 TechColumbus Outstanding Woman in Technology
- 2013 Early Career Innovator of the Year, OSU.
- 2015 Plimpton Award for Outstanding Teaching

The Ohio State University

Flavonoid Research



- Potent antioxidants
- Abundant in nature
- Antibacterial, anti-allergic, antiviral and anti-inflammatory

- Isoflavones
  - Phytoestrogens
- Anthocyanins
  - Natural colorants
  - Phytonutrients
- Pro-anthocyanidins
  - Protect the urinary tract

Areas of work

- Analytical
- Horticultural
- Processing
- Bioavailability
- Health benefits

The Panel

- A panel with expertise in plant biology, food chemistry, food toxicology, food product development and manufacturing as well as food quality and regulatory affairs was convened from 2012 to 2014.

Expert Committee

Dr. James E. Simon – **Lead Author**
Rutgers University, NJ
Distinguished Professor of Plant Biology
Dept. of Plant Biology & Pathology

Dr. Eric A. Decker
University of Massachusetts Amherst
Professor and Department Head
Food Science

Dr. Carla D. Mejia
United States Pharmacopeia
Food Chemical Codex
Senior Scientific Liaison
(now with UN World Food Programme)

Dr. M. Monica Giusti
The Ohio State University
Professor of
Food Science and Technology

Dr. Mario G. Ferruzzi,
Purdue University
Professor
Food Science and Nutrition
(Now with North Carolina St. Univ., Plants for Human Health Institute)

Dr. Stephen T. Talcott
Texas A&M University
Professor of
Food Chemistry and Nutrition

Sponsored by Sensient Technologies in collaboration with the US Pharmacopeial Convention

Goals of the Committee

- Make recommendations for quality and product safety standards for the natural colors industry
- Standardization of methods used to test natural colors
- “Raising the bar” on the quality and safety of natural colors
- Development of “White Paper”

Why do we use colorants?

Research has indicated that people "see" flavors (Acree 2013, Lewis and others 2008), i.e, they form an opinion about the flavor of a food or beverage, based on its color.

- Up to 85% of consumer buying decisions are potentially influenced by color
- Color has a major impact on flavor perception and flavor acceptance.
- Effective color usage drives consumer trial and acceptance.

Some Issues Facing the Color Industry

- Southampton Study
  - Consumers and regulatory agencies questioning safety of synthetic dyes
- Supply, cost and environmental concerns on synthetic raw materials
- Climate change effecting the quality and availability of crops
- Large number of products with low quality standards commercialized as “Colors”

South Hampton Study

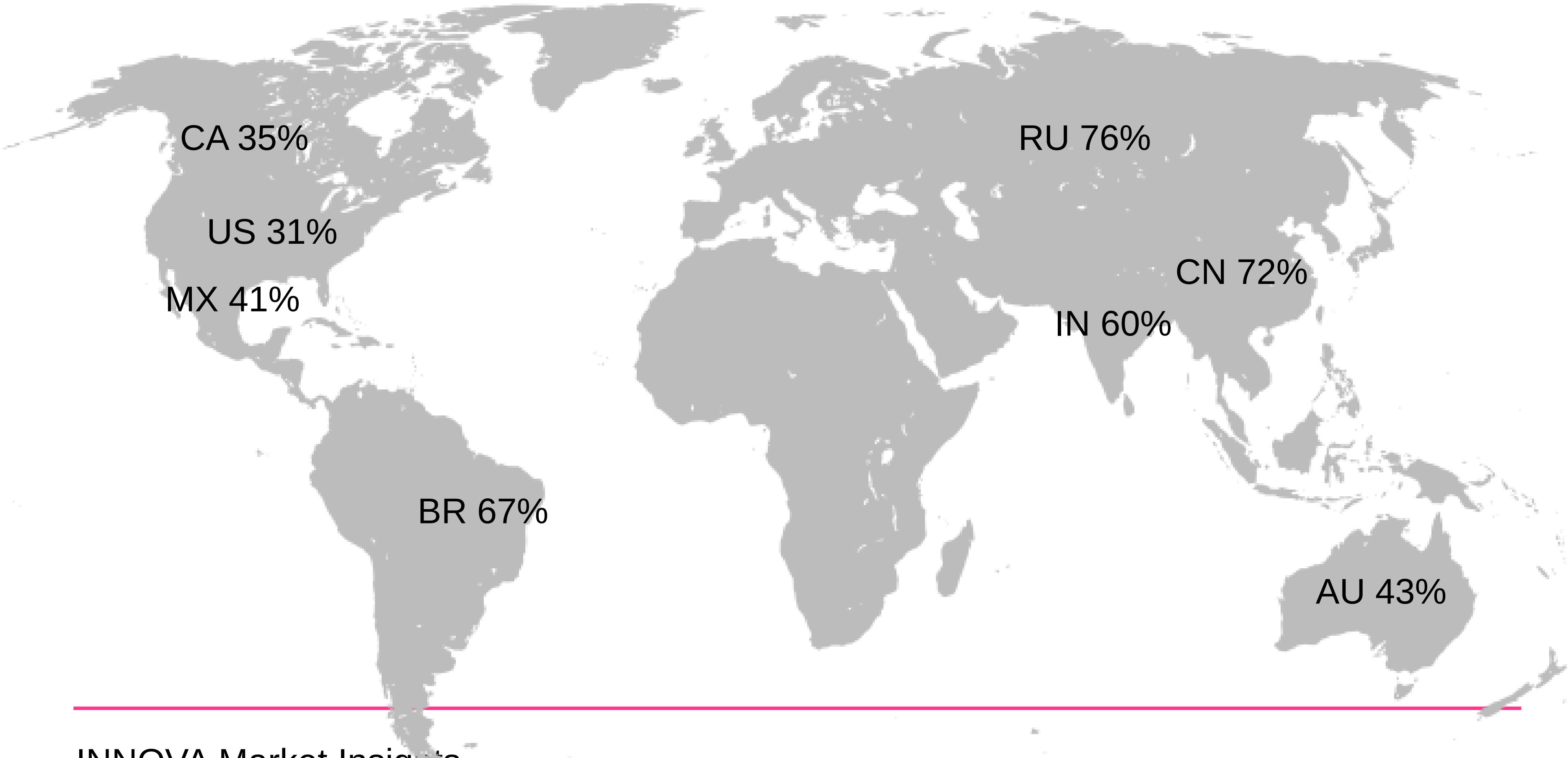
- Study conducted by the University of South Hampton in the UK
- Linked the hyperactivity in children with ADHD to certain dyes (Azo dyes)
- While the evidence was limited, the public perception and regulatory repercussions have been strong
- Lead to warning labels in the EU, Russia, GCC, and activity at watchdog groups in the US

Synthetic Raw Materials Under Pressure

- Increasing concern around chemical industry in China: Shut downs, fines and jail time increasing for companies who violate policies
- Availability of raw materials has become more volatile
- Cost are increasing and needs for environmental controls become more wide spread
- Natural colors may become attractive alternatives from a supply security, cost and environmentally responsible standpoint.

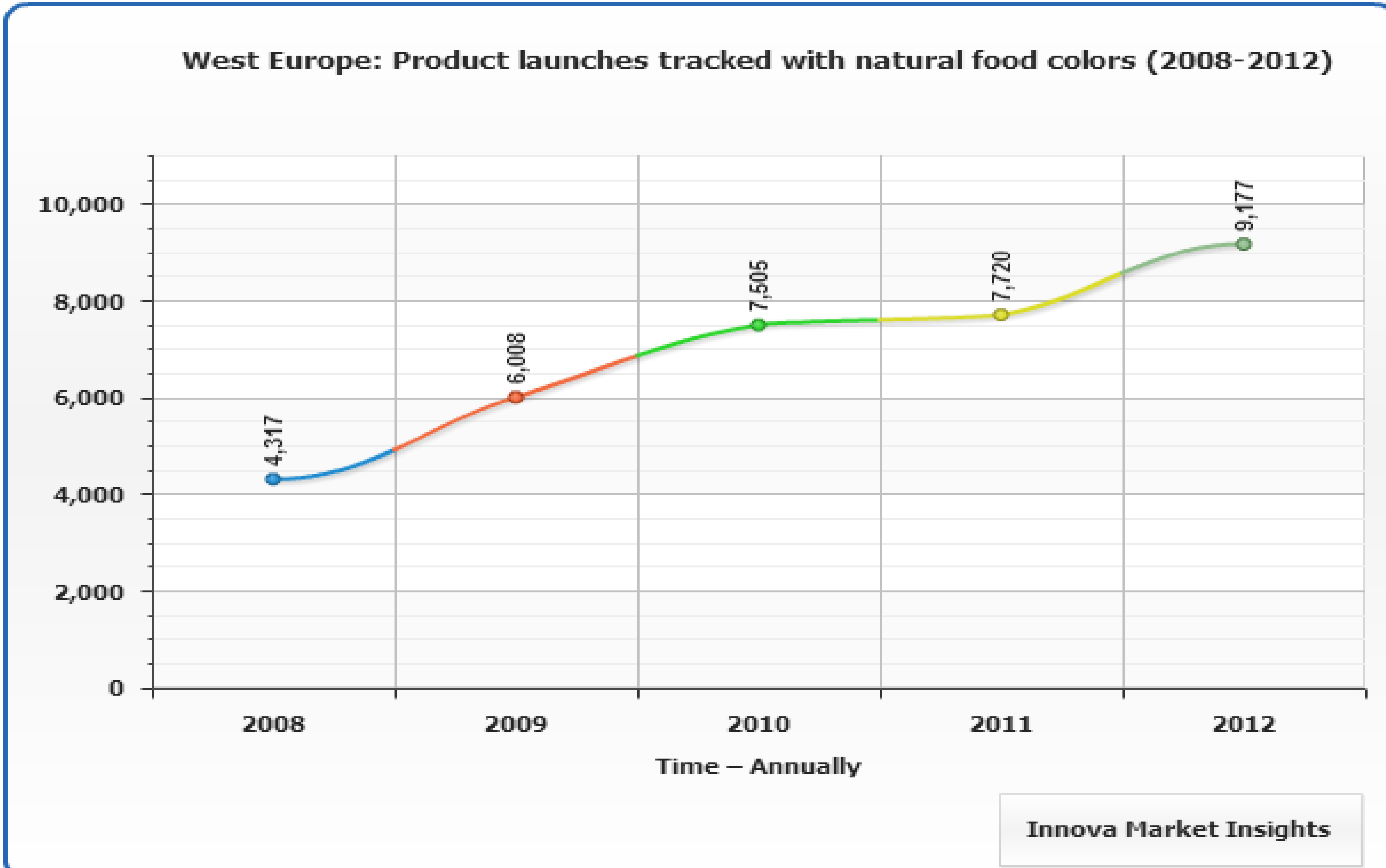
Trends Towards Color from Naturals Sources

- Proportion of consumers that report to be very/extremely concerned about food colorings:

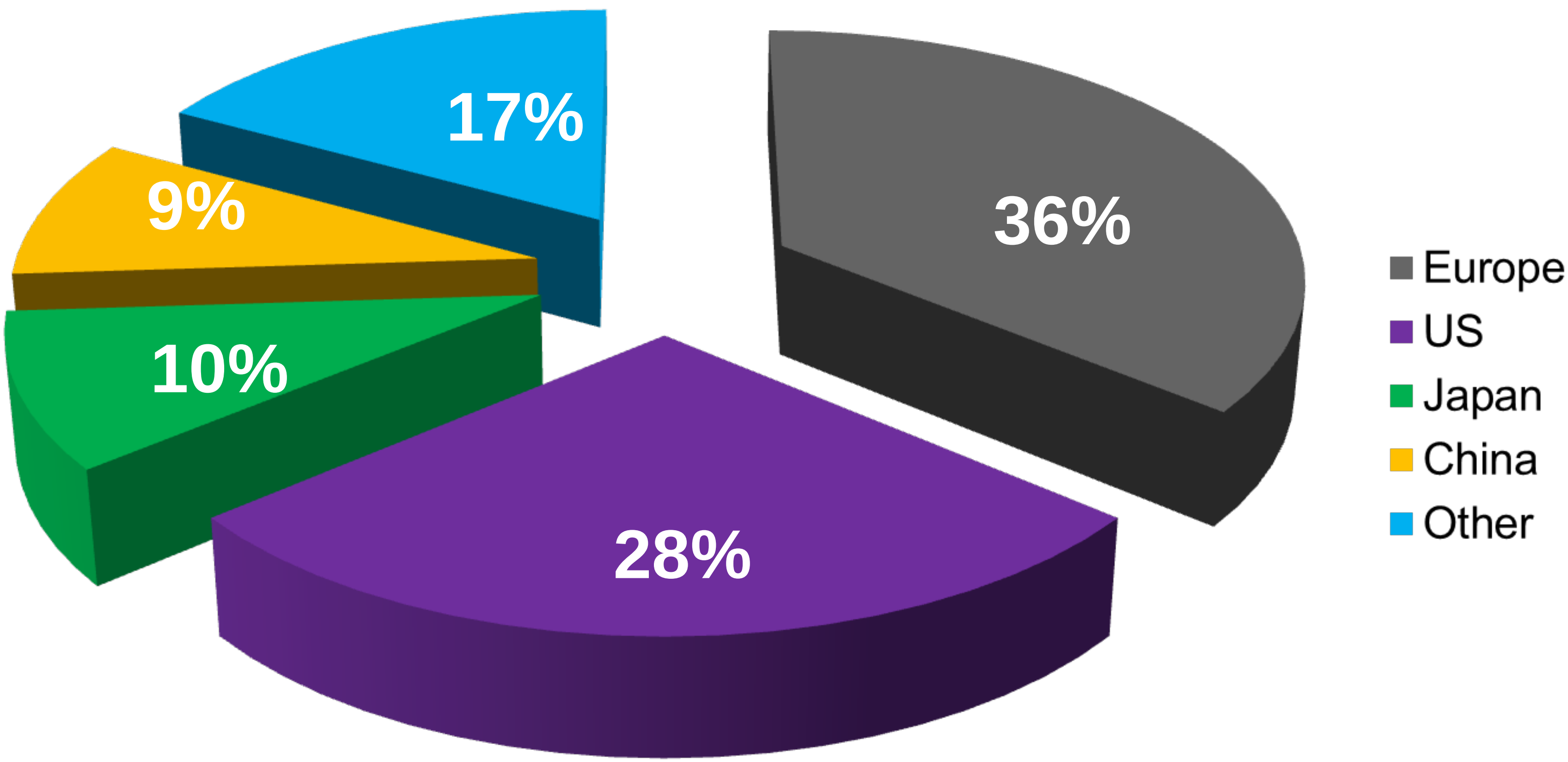


INNOVA Market Insights

Trends Towards Color from Naturals Sources



Global Food Colours Market by Country/Region, 2012



Source: Leatherhead Food Research

Increased demand for “natural” colors

- Consumers are looking for natural ingredients with the belief that they are safer, and may be even healthier.

- There is a need for reliable and consistent supply of raw materials

Climate Changes Could Increase Growing Constraints and Risks

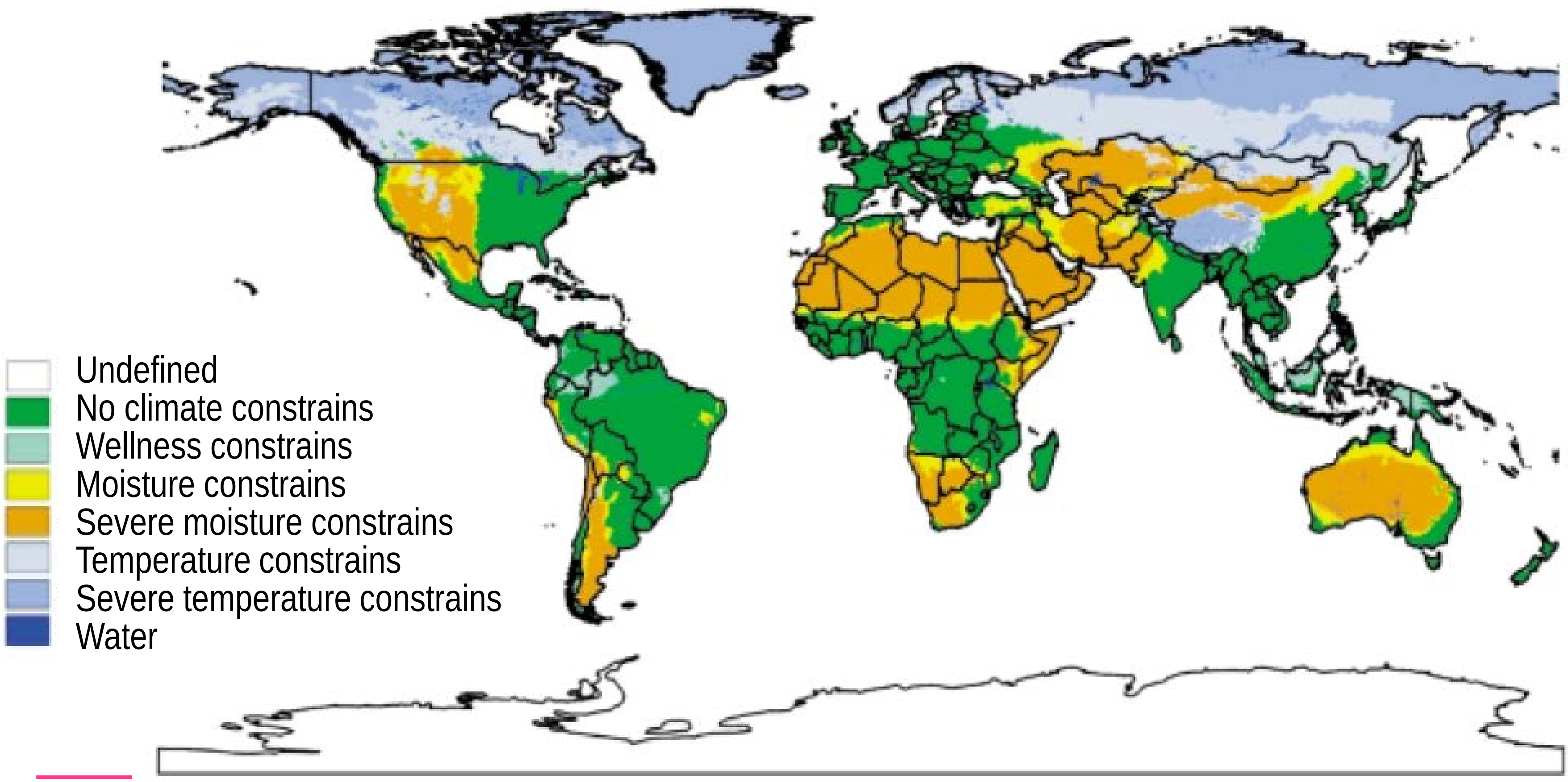


Plate E. Climate constraints.

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Some repercussions of climate change

- In a recent article in *Nature* sited that elevated CO2 levels are reducing the quality and nutritional value of crops.
- This leads to an increased risk of adulteration, unapproved pesticides or other short cuts that are not allowed.

More Issues Facing the Color Industry

- Lack of clear quality and safety specifications in the CFR for colors exempt from certification
- Ongoing import detentions involving color
- Food colorant safety issues in the news

US Color Regulations

- Colorants are not regulated as food additives, nor are they generally on the GRAS list.
- Color Additives covered by 21CFR Parts 70–82.
  - Certified colors – synthetic colorants
  - Colors exempt from certification
    - Colors from natural sources
    - Nature identical compounds

FDA Certifies FD&C Colorants

CFR - Code of Federal Regulations

TITLE 21--FOOD AND DRUGS
CHAPTER I--FOOD AND DRUG ADMINISTRATION
DEPARTMENT OF HEALTH AND HUMAN SERVICES
SUBCHAPTER A--GENERAL

Part 74 - Listing of Color Additives Subject to Certification

U.S. Food and Drug Administration
Protecting and Promoting *Your* Health

Subpart A--Foods

- § 74.101 – FD&C Blue No. 1.
- § 74.102 – FD&C Blue No. 2.
- § 74.203 – FD&C Green No. 3.
- § 74.250 - Orange B.
- § 74.302 - Citrus Red No. 2.
- § 74.303 – FD&C Red No. 3.
- § 74.340 – FD&C Red No. 40.
- § 74.705 – FD&C Yellow No. 5.
- § 74.706 – FD&C Yellow No. 6.

7 FD&C + 2

US Color Regulations

- Certified colors
  - Every batch is tested by the FDA against a toxicological reference.
  - Nomenclature changes upon certification.
  - An FDA lot number is assigned.
  - There is an assessed fee, based on the total batch weight being tested.

So Who “Certifies” Natural Colors?

CFR - Code of Federal Regulations

TITLE 21--FOOD AND DRUGS
CHAPTER I--FOOD AND DRUG ADMINISTRATION
DEPARTMENT OF HEALTH AND HUMAN SERVICES
SUBCHAPTER A--GENERAL

Part 73 - Listing of Color Additives Exempt from Certification

Subpart A Foods

§ 73.1 - Diluents in color additive mixtures for food use exempt from certification.
§ 73.30 - Annatto extract.
§ 73.40 - Dehydrated beets (beet powder).
§ 73.85 - Caramel.
§ 73.95 - [beta]-Carotene.
§ 73.100 - Cochineal extract; carmine.
§ 73.169 - Grape color extract.
§ 73.170 - Grape skin extract
§ 73.250 - Fruit juice.
§ 73.260 - Vegetable juice.
§ 73.345 - Paprika oleoresin.
§ 73.575 - Titanium dioxide.
§ 73.585 - Tomato lycopene extract; tomato lycopene concentrate.
§ 73.600 - Turmeric.
§ 73.615 - Turmeric oleoresin.

Colors exempt from certification

- Annatto extract
- Dehydrated beets (beet powder)
- Canthaxanthin
- Caramel
- [beta]-Apo-8'-carotenal
- [beta]-Carotene
- Cochineal extract; carmine
- Sodium copper chlorophyllin
- Toast ... y ... ooked ... onseed flour
- Ferrous gluconate
- Ferrous lactate
- Grape color extract
- Grape skin extract (enocianina)
- Fruit juice
- Vegetable ju...
- Carr...
- ...aprika oleoresin
- Mica-based pearlescent pigments
- Riboflavin
- Saffron
- Sp... ...act
- ...um dioxide
- Tomato lycopene extract; tomato lycopene concentrate
- Turmeric
- Turmeric oleoresin

A total of 38 are listed, with 26 for use in human food.

Colors exempt from certification

- Annatto extract
- **Dehydrated beets (beet powder)**
- Canthaxanthin
- Caramel
- [beta]-Apo-
- [beta]-Caro
- Cochineal e carmine
- Sodium cop chlorophylli
- Toasted par defatted co cottonseed
- Ferrous gluconate
- Ferrous lactate
- Grape color extract
- Grape skin extract
- Mica-based pearlescent pigments
- Riboflavin
- Saffron

(a) *Identity.* (1) The color additive dehydrated beets is a dark red powder prepared by dehydrating sound, mature, good quality, edible beets.

(b) Specifications:

Volatile matter, not more than 4 percent.
Acid insoluble ash, not more than 0.5 percent.
Lead (as Pb), not more than 10 parts per million.
Arsenic (as As), not more than 1 part per million.
Mercury (as Hg), not more than 1 part per million.

(c) Uses and Restrictions: according to GMPs.

Colors

- Annatto extract
- Dehydrated beets (beet powder)
- Canthaxanthin
- Caramel
- [beta]-Apo-8'-caro
- [beta]-Carotene
- Cochineal extract carmine
- **Sodium copper chlorophyllin**
- Toasted partially defatted cooked cottonseed flour

(a) Identity. (1) Sodium copper chlorophyllin is a green to black powder prepared from chlorophyll by saponification and replacement of magnesium by copper. Chlorophyll is extracted from alfalfa (*Medicago sativa*) using any one or a combination of the solvents acetone, ethanol, and hexane.

(b) Specifications. GMPs and:

(1) Moisture, not more than 5.0 %.

(2) Solvent residues (acetone, ethanol, and hexane), not more than 50 ppm, singly or, in combination.

(3) Total copper, not less than 4% and not more than 6%.

(4) Free copper, not more than 200 ppm.

(5) Lead (as Pb), not more than 10 ppm.

(6) Arsenic (as As), not more than 3 ppm.

(7) Mercury (as Hg), not more than 0.5 ppm.

(8) Ratio of absorbance at 405nm to absorbance at 630 nm, not less than 3.4 and not more than 3.9.

(9) Total copper chlorophyllins, not less than 95% of the sample dried at 100 °C for 1 hr.

(c) Uses and restrictions: to color citrus-based dry beverage mixes not exceeding 0.2% in the dry mix.

Colors exempt from certification

- Annatto extract
- Dehydrated beets (beet powder)
- Canthaxanthin
- Caramel
- [beta]-Apo-8'-carotenal
- [beta]-Carotene
- Cochineal extract; carmin
- Sodiun chlorop
- Toaste defatted cooked cottonseed flour
- Ferrous gluconate
- Ferrous lactate
- Grape color extract
- Grape skin extract (enocianina)
- **Fruit juice.**
- Vegetable juice
- Carrot oil
- Mica-based pearlescent pigments
- Riboflavin
- Saffron
- Spirulina extract
- Titanium dioxide
- Tomato lycopene extract; tomato lycopene concentrate

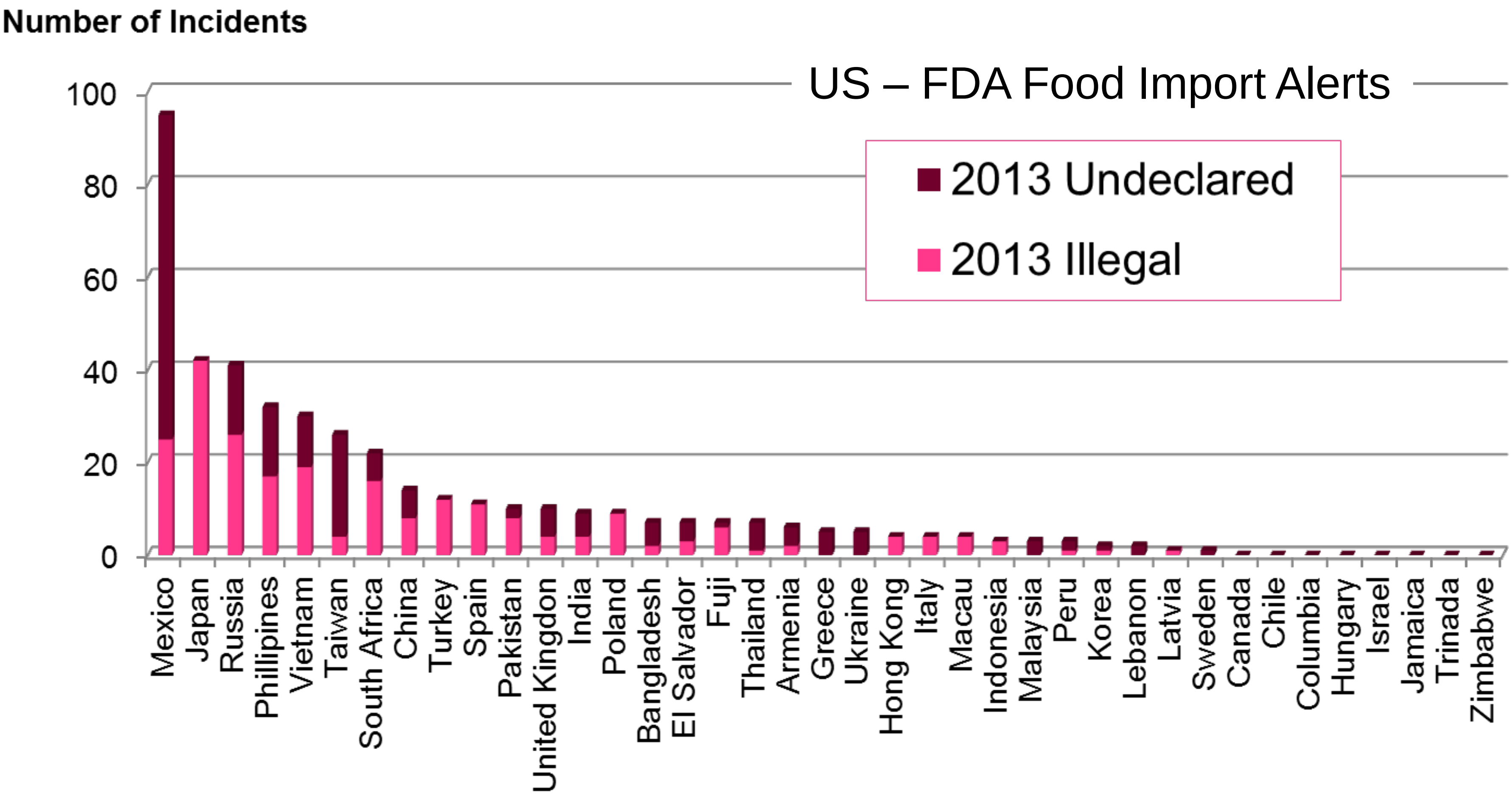
(a) *Identity.* (1) Expression of juice from fresh fruit or water infusion of the dried fruit

(b) Uses and restrictions: According to GMPs.

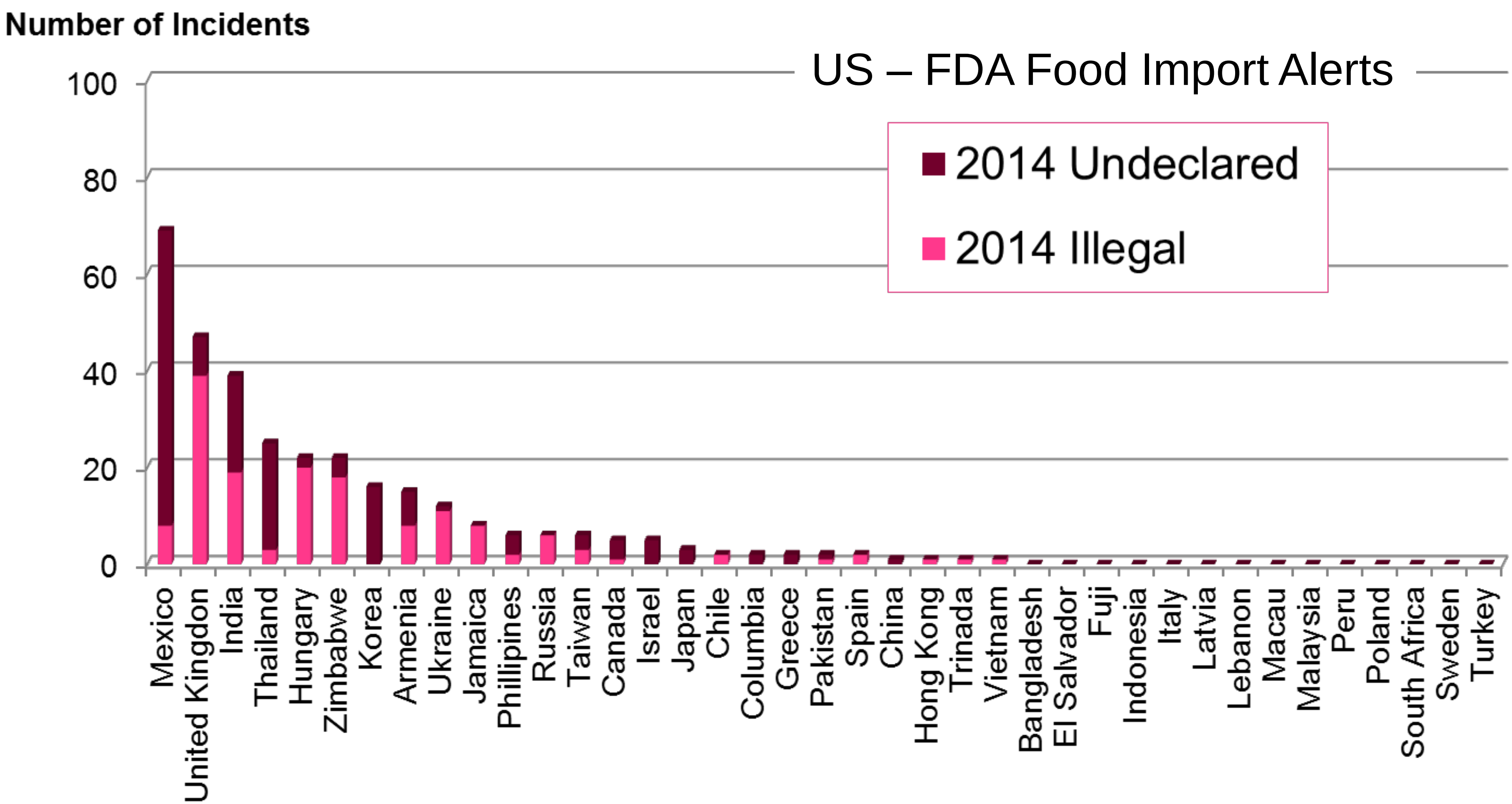
Large Disparity on CFR Specifications

	FD&C Yellow 6	Fruit Juice
	74.706	73.26
Process Specifications in the CFR	Azo reaction of Schaffer salt and sulfanilic acid	Expression of juice from fresh fruit or water infusion of the dried fruit
Total color	87% min	
Sum of volatile matter (at 135 deg. C) and chlorides and sulfates (calculated as sodium salts)	13% maximum	
Water insoluble matter	0.2% max	
Unreacted Intermediates		
Sodium salt of 4-aminobenzenesulfonic acid	0.2% max	
Sodium salt of 6-hydroxy-2-naphthalenesulfonic acid	0.3% max	
Disodium salt of 6,6'-oxybis[2-naphthalenesulfonic acid]	1% max	
Disodium salt of 4,4'-(1-triazene-1,3-diyl)bis[benzenesulfonic acid]	0.1% max	
Subdyes		
Sum of the sodium salt of 6-hydroxy-5-(phenylazo)-2-naphthalenesulfonic acid and the sodium salt of 4-[(2-hydroxy-1-naphthalenyl)azo]benzenesulfonic acid	1% max	
Sum of the trisodium salt of 3-hydroxy-4-[(4-sulfophenyl)azo]-2,7-naphthalenedisulfonic acid and other higher sulfonated subsidiaries	5% max	
Potentially carcinogenic compounds		
4-Aminoazobenzene	50 ppb	
4-Aminobiphenyl	15 ppb	
Aniline	250 ppb	
Azobenzene	200 ppb	
Benzidine	1 ppb	
1,3-Diphenyltriazene	40 ppb	
1-(Phenylazo)-2-naphthalenol	10 ppb	
Heavy Metals		
Lead (as Pb)	2 ppm	
Arsenic (as As)	3 ppm	
Mercury (as Hg)	1 ppm	
Cadmium (as Cd)	1 ppm	

Inspection and Detention of Incoming Food Products Due to Colors is Growing



Inspection and Detention of Incoming Food Products Due to Colors is Growing



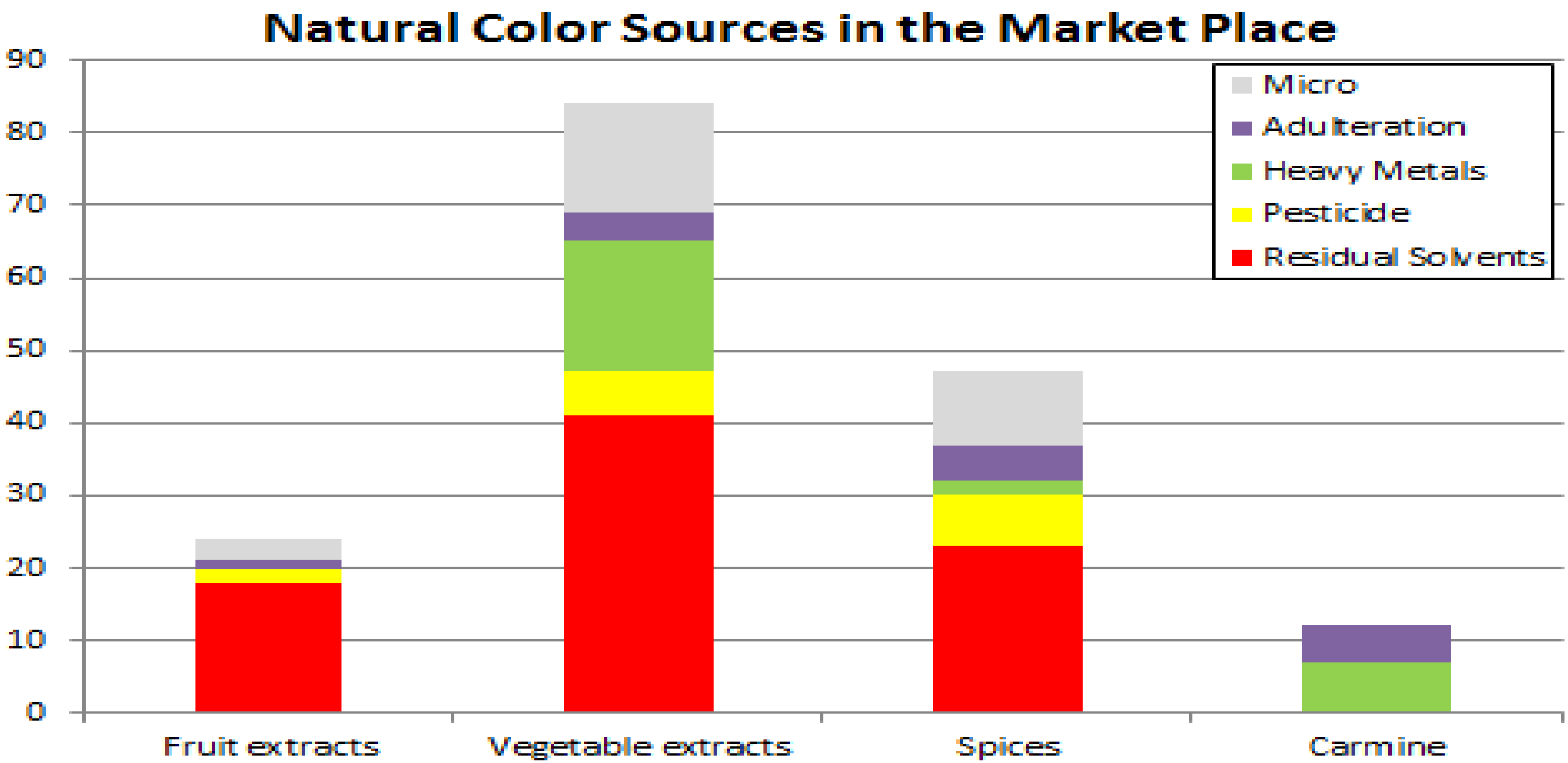
Food Safety Issues in the News

- Contamination Issues
  - Extraction solvents not approved for use in colors – varies by region
  - Residuals solvents in excess of >1,000 ppm
  - Lead and mercury contamination in Carmine
  - Pesticide residues in Paprika
  - Dioxin in Paprika
  - Bacterial and pathogen contamination

Food Safety Issues in the News

- Adulteration Issues
  - Adulterated FDA certified dyes in the market place
  - Beverage cloud adulterated with plasticizer
  - Synthetic dye adulteration of natural colors
  - Carthamus adulterated with Orange II
  - Saffron adulterated with Carthamus
  - Brick in Paprika
  - Sudan dyes in Turmeric and Paprika
  - Natural colors not approved for use in the US & EU (Huito)

Supply Expansions Efforts Uncovered Risk in Natural Raw Materials: From 650 samples imported into the US – 25% Failed QC



Some Risk Factors

Color	Tumeric	Paprika	Vegetable Juice	Cochineal
Country of Origin	India or China	India or China	India or China	Peru or Mexico
Natural/Crop/Botanical	Yes	Yes	Yes	Yes
Import Alert / Food Safety Incident / CFR	Sudan dyes & pathogens	Sudan dyes	Multiple	Synthetic dyes & pathogens
Kill Step (Supplier HACCP)?	No	No	No	No
Contains Preservative?	No	No	No	No
Contains Nutrients	Protein ~10%	Protein ~1%	Protein ~10%	Protein ~20%
Aw > 0.86	<0.86	<0.86	>0.86	<0.86
pH 4 - 10	Yes	Yes	No	Yes
Risk	High	High	High	High
Identification	Yes	Yes	Yes	Yes
Heavy Metals	Yes	Yes	Yes	Yes
Micro	Yes	Yes	Yes	Yes
Pesticides	Yes	Yes	Yes	Yes
Residual Solvents	Yes	Yes	Yes	
Adulterants	Yes	Yes	Yes	Yes

The Proposed Solution

- Development of Industry-accepted standards for colorants from natural sources.
  - Minimize risks
  - Increase consumer and industry confidence
  - Provide a self-regulatory framework

Starting from the Raw Materials

- Raw material
  - Origin of the raw material
  - Heavy metals more frequent in certain regions
  - Product specification sheets
  - Botanical authentication
    - Chemical analyses

Managing Key Risk Factors for Natural Colors

Hazard	Mitigation Strategy
Microbiological	Test all incoming raw materials for pathogens. Test all finished products for pathogens and spoilage organisms
Heavy Metals	Test for most common heavy metals (FDA, EU & Codex)
Pesticides	Test for pesticides that are not allowed (EPA, EU & Codex)
Adulteration	Test for synthetic dyes or other additives that adulterate product. Screening established for unknown adulterants.
Unauthorized Solvents	Map all supplier process and detail solvents used to ensure they meet all local regulations. Test for the presence of solvents.
Supplier Reliability	All vendors should be “certified” by the manufacturer.
Raw Material Traceability	Need full traceability for all ingredients

Microbiological Contamination

- Source of colors from natural source are food crops, therefore risk of microbial contamination exists.
  - Testing
  - Spoilage organisms
  - Indicator organisms
  - Pathogens
  - Mycotoxins

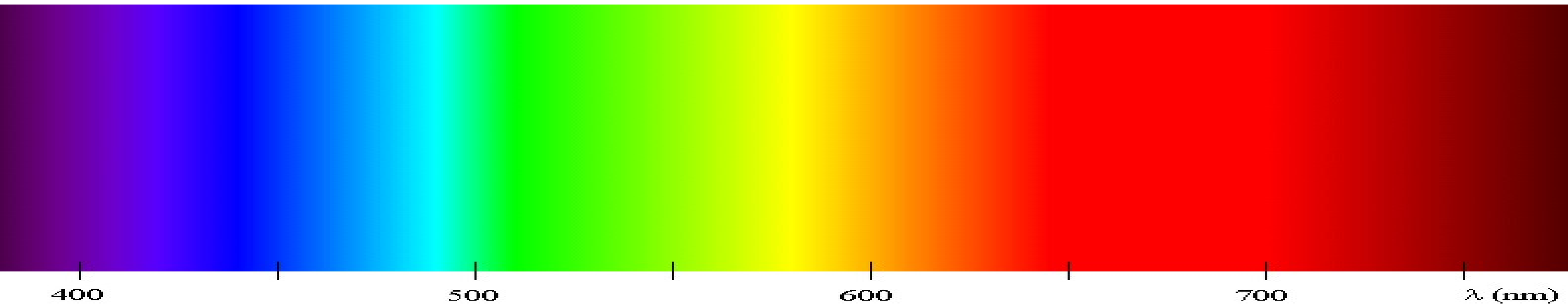
Heavy Metals

	Mercury	Arsenic	Lead	Cadmium	Total Heavy Metals
FDA 21CFR	<1 ppm	<3 ppm	<10 ppm		
HC (Canada)		<3 ppm	<10 ppm		<40 ppm
EC 231/2012	<1 ppm	<3 ppm	<2 ppm	<1 ppm	<40 ppm
JECFA	<1 ppm		<2 ppm		<40 ppm
JFSA (Japan)		<2 ppm	<2 ppm		<20 ppm
GB (China)	<1 ppm	<1 ppm	<10 ppm		<20 ppm

Pesticide Residues

- Source of colors from natural source are food crops, therefore risk of pesticide residues exists.
- Pesticide residues can be concentrated during the extraction process to much higher levels than might present in a food crops.
- Compliance to EPA, Codex and EU pesticide residue regulations.
- For colors from natural sources, a concentration factor based on coloring units, is applied similar to spice extracts.

Adulteration – targeted approach



Purple
- Blue 2
- Rhodamine B

Blue
- Blue 1
- Patent Blue V

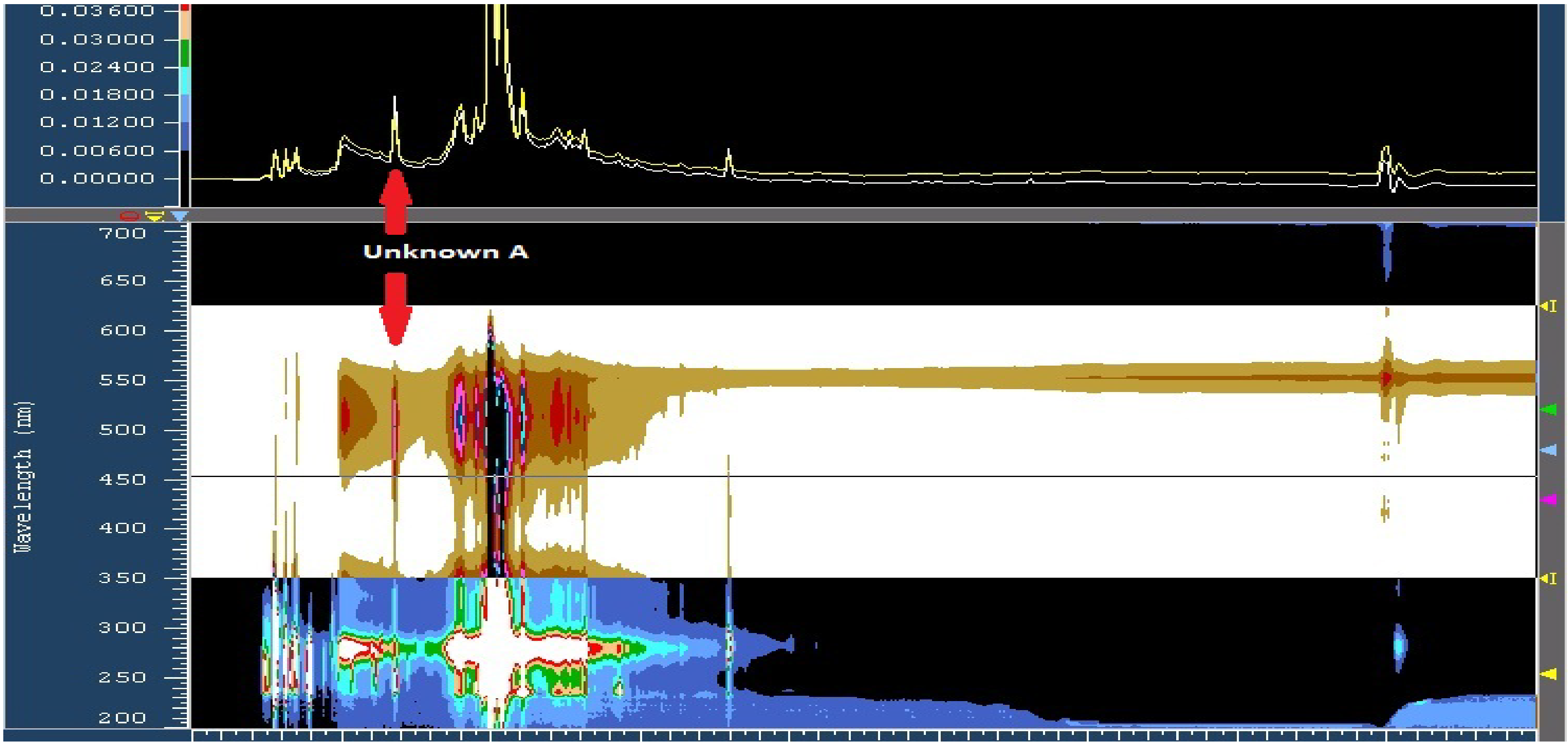
Green
- Green 3
- B1 + Y5
- Green S

Yellow
- Yellow 5
- Metamil Yellow
- Dimethyl Yellow
- Fast Garnet GBC
- Auramine
- Quinoline Yellow

Orange
- Yellow 6
- Orange II
- Sudan Orange
- Oil Orange SS
- Sudan I & II
- p-Nitroaniline
- Chrysoidine G

Red
- Red 40
- Red 3
- Para Red
- Sudan Red 7B
- Ponceau 4R
- Carmoisine
- Sudan III
- Sudan IV
- Amaranth
- Toluidine Red

Adulteration – untargeted approach



Solvent Residues (ppm)

	United States	European Union	Japan	Korea
Acetone	30	50	30	50
1, 3 –Butylene glycol	MA	--	--	--
Ethyl acetate	MA	50	--	--
Ethyl chloride	30	--	--	--
Isopropyl alcohol	50	50	50	30-50
Methyl alcohol	50	50	50	50
Methylene chloride	30	10	--	10-30
Hexane	25	50	25	25
Trichloroethylene	30	--	30	--
n-Butanol	--	50	--	--
Methyl ethyl ketone	--	50	--	--
Ethyl alcohol	--	50		--
Trichloro ethylene	--	--	30	30

Summary of Proposed Standards

		Specification
Heavy Metals	Lead (Pb) Arsenic (As) Mercury (Hg) Cadmium (Cd) Total Heavy Metals	<2 ppm <3ppm) <1ppm <1ppm <20ppm
Microbiological Properties	Total plate count Yeast Mold Coliforms E-coli Salmonella Staph aureus Listeria Aflatoxin	<1,000 cfu/g <100 cfu/g <100cfu/g <3 cfu/g Negative in 10 g Negative in 375 g Negative in 10 g Negative in 10 g <20 ppb
Pesticide Residues	Complies with EPA Complies with EU	Apply concentration factors based on plant material
Solvent Residues	Methanol Ethanol	50ppm 250 ppm

Advantages of development and publishing the proposed standards

1) Higher quality color sources available to processors, therefore raising the quality bar across the industry
2) Developing and harmonizing standards will stimulate trade
3) Ensuring a practical system relative to botanical authentication
4) Ensuring consumer product safety and consumer confidence
5) Better definition of product quality

Advantages of development and publishing the proposed standards (…continued)

6) Guidance for sourcing of natural colorants
7) Brand protection of all companies in the supply chain
8) Increased availability and reference by all in the industry
9) Stimulating industry “self-policing,” thus preventing the FDA from establishing further enforcement or restricting trade in natural colors

The White Paper

A manuscript has been submitted to the Journal of Food Science as a concise review:

Establishing Standards on Colors from Natural Sources

By: Simon, JE; Decker, EA; Ferruzzi, MG; Giusti, MM; Mejia, CD; Talcott, ST.