The role of FCC in protecting the integrity of natural food colors

2022 IACM Global Color Conference

Tongtong Xu, PhD

Food Chemicals Codex at U.S. Pharmacopeia

Food Chemicals Codex | FCC

- Created by the US-FDA and the US Institute of Medicine in 1966
- Currently published by USP, a non-profit organization
- >1250 standards for additives, ingredients, and other food chemicals
- Standards are developed by expert volunteers
- A fully independent source of food ingredient standards

Ingredient standard is…

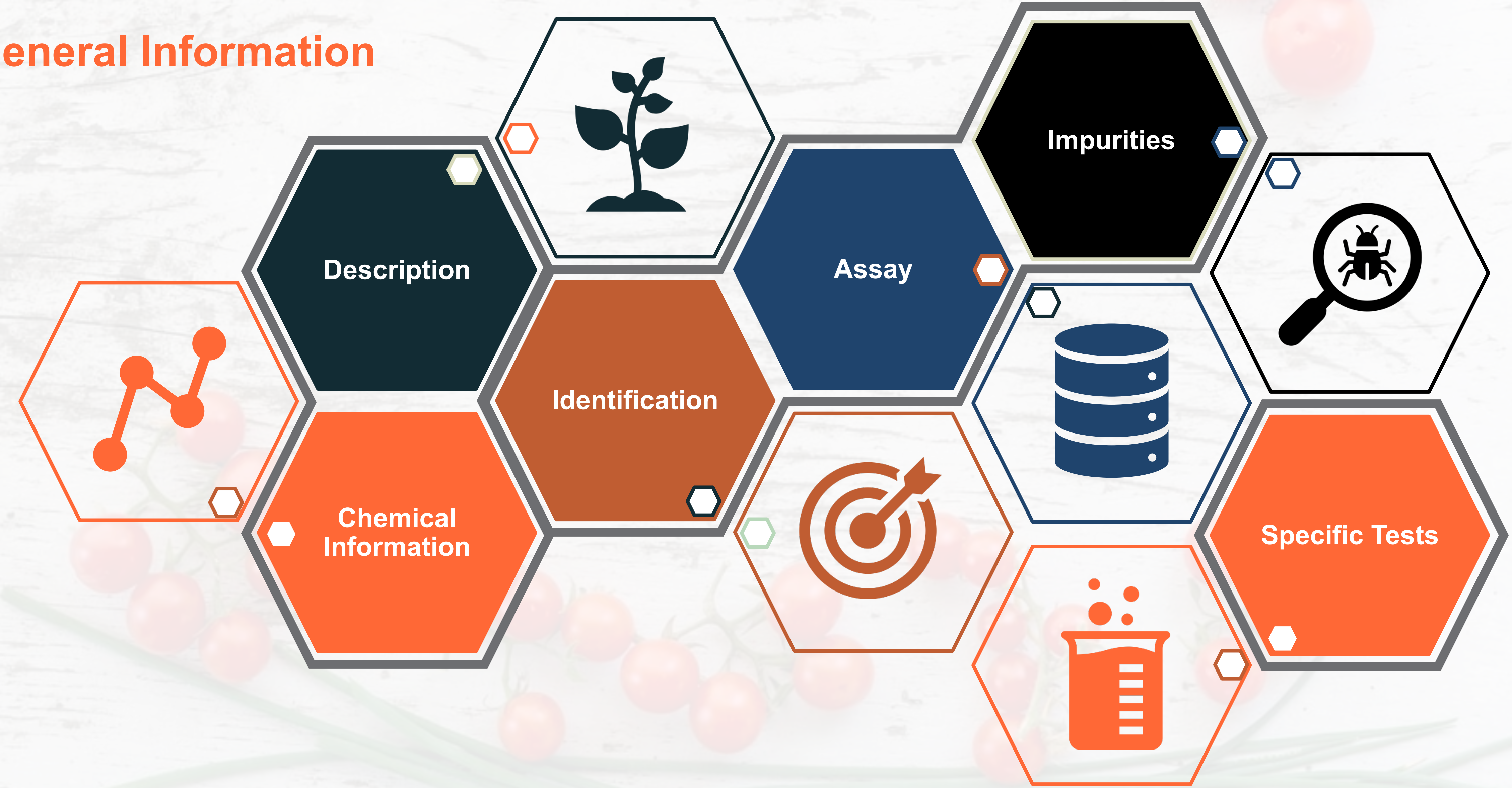
FD&C Act Sec 201 (f) The term "food" means (1) articles used for food or drink for man or other animals, (2) chewing gum, and (3) articles used for components of any such article.

21 CFR Sec. 73.450 Riboflavin "(a) Identity. (1) The color additive riboflavin is the riboflavin defined in the **food chemicals codex...***Specifications*. Riboflavin shall meet the specifications given in the ***food chemicals codex***..."

The FCC serves two key roles in this area: 1.) helping to limit the introduction of potential problems at the ingredients level, and 2.) serving as a widely acknowledged quality benchmark in the global marketplace for food ingredients. FCC standards are recognized around the world by regulatory agencies, food processors, and ingredient suppliers as the basis for defining "food grade" ingredients.

The content of an FCC standard

General Information



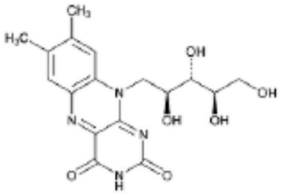
An example of an *FCC* standard – Riboflavin

Riboflavin

Riboflavin

Published in: FCC 11 2S | FCC 11 3S | FCC 12
First Published: Prior to FCC 6

Vitamin B_2



$C_{17}H_{20}N_4O_6$
Formula wt 376.37
INS: 101(i)
CAS: CAS [83-88-5]

DESCRIPTION

Riboflavin occurs as a yellow to orange-yellow, crystalline powder. When dry, it is not affected by diffused light, but when in solution, light induces deterioration. It melts at about 280° with decomposition, and its saturated solution is neutral to litmus. One g dissolves in 3000 to about 20,000 mL of water, the variations being due to differences in the internal crystalline structure. It is less soluble in alcohol than in water. It is insoluble in ether and in chloroform, but it is very soluble in dilute solutions of alkalies.

Function: Nutrient

Packaging and Storage: Store in tight, light-resistant containers.

IDENTIFICATION

- **Procedure**
  - **Sample solution:** 1 mg in 100 mL of water
  - **Acceptance criteria:** The *Sample solution* is pale green-yellow by transmitted light and has an intense yellow-green fluorescence that disappears on the addition of mineral acids or alkalies.

ASSAY

- **Procedure**
  [Note—Conduct this assay so that the solutions are protected from direct sunlight at all stages.]
  - **Sample solution:** Transfer 50 mg of sample into a 1000-mL volumetric flask containing about 50 mL of water. Add 5 mL of 6 N acetic acid and sufficient water to make about 800 mL. Heat on a steam bath, protected from light, with frequent agitation until dissolved. Cool to about 25°, add water to volume, and mix. Dilute this solution with water, quantitatively and stepwise, to bring it within the operating sensitivity of the fluorometer used.
  - **Standard solution:** In the same manner, prepare a standard solution to contain, in each mL, a quantity of USP Riboflavin RS equivalent to that of the *Sample solution*.
  - **Analysis:** Using a fluorometer at about 530 nm, using an excitation wavelength of about 440 nm, measure the intensity of the *Standard solution's* fluorescence. Directly after the reading, add about 10 mg of sodium hydrosulfite to the *Standard solution*, stirring with a glass rod until dissolved, and immediately measure the fluorescence again. The difference between the two readings represents the intensity of the fluorescence caused by the USP Riboflavin RS. Similarly, measure the intensity of the fluorescence of the *Sample solution*, both before and after the addition of sodium hydrosulfite. Calculate the quantity of $C_{17}H_{20}N_4O_6$ in the *Sample solution* by the formula:

$$\text{Result} = C(I_U/I_S)$$

C = concentration of USP Riboflavin RS (mg/mL) in the final solution of the *Standard solution*
I_U = corrected fluorescence values observed for the *Sample solution*
I_S = corrected fluorescence values observed for the *Standard solution*

  - **Acceptance criteria:** NLT 98.0% and NMT 102.0% of $C_{17}H_{20}N_4O_6$, calculated on the dried basis

IMPURITIES

Organic Impurities

- **Lumiflavin**
  - **Alcohol-free chloroform:** Shake 20 mL of chloroform gently, but thoroughly, with 20 mL of water for 3 min, draw off the chloroform layer, and wash twice more with 20-mL portions of water. Finally, filter the chloroform through a dry filter paper, shake it well for 5 min with 5 g of powdered anhydrous sodium sulfate, allow the mixture to stand for 2 h, and decant or filter the clear chloroform.
  - **Sample preparation:** Shake 25 mg of sample with 10 mL of *Alcohol-free chloroform*, for 5 min and filter.
  - **Analysis:** Determine the absorbance of the *Sample preparation* with a suitable spectrophotometer set at 440 nm using a 1-cm cell and *Alcohol-free chloroform* as the blank.
  - **Acceptance criteria:** The absorbance of the *Sample preparation* is NMT 0.025.

SPECIFIC TESTS

- **Loss on Drying, Appendix IIC:** 105° for 2 h
  - **Acceptance criteria:** NMT 1.5%
- **Optical (Specific) Rotation, Appendix IIB**
  - **Sample solution:** 5 mg/mL in hydrochloric acid
  - **Acceptance criteria:** $[\alpha]_D^{25}$ between + 56.5° and +59.5°, calculated on the dried basis
- **Residue on Ignition (Sulfated Ash), Appendix IIC**
  - **Sample:** 1 g

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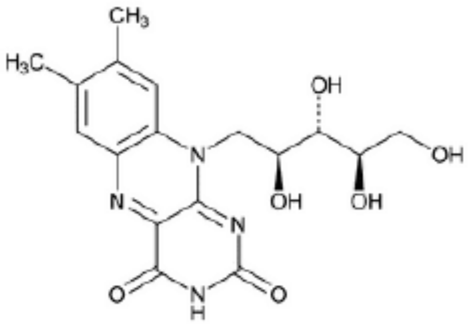

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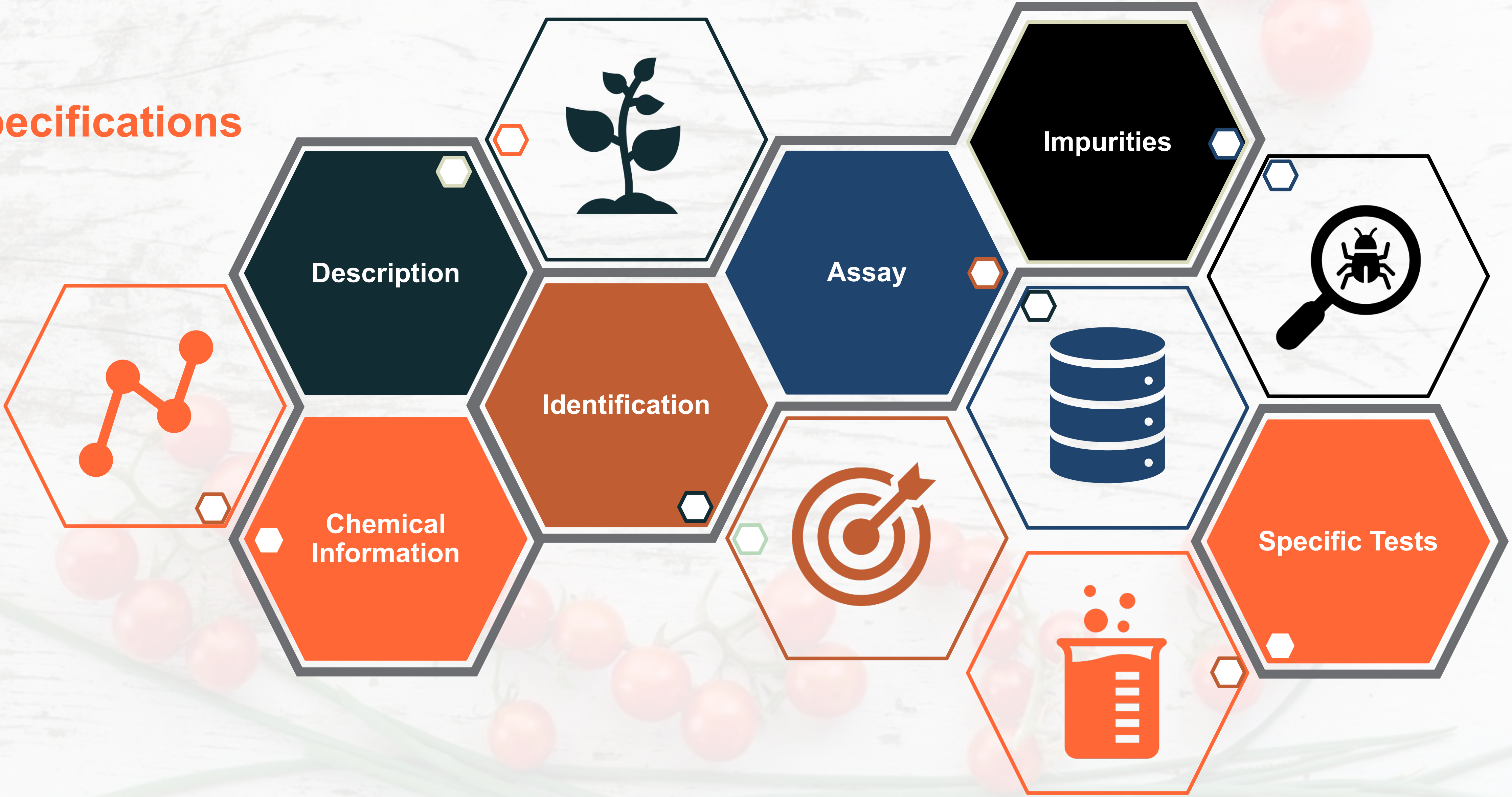
The content of an FCC standard

Specifications



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Riboflavin

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Published in: FCC 11

First Published: Prio

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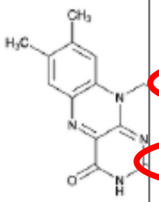

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• **RESIDUE ON IGNITION (SULFATED ASH),** Appendix IIC

Sample: 1 g

Ingredient standards: critical tool to protect integrity of foods

- Producing foods that are safe, nutritious, and genuine requires control of, and communication along, the whole supply chain
- Food supply chains are complex, non-linear, and subject to sudden disruption
- This creates multiple opportunities for misunderstanding, miscommunication, mishandling, fraud, and adulteration
- Some contaminants are inevitable in foods (environmental or process)
- Standards are a critical tool for mitigating these risks

FMI Food Industry Assocation Safety Code

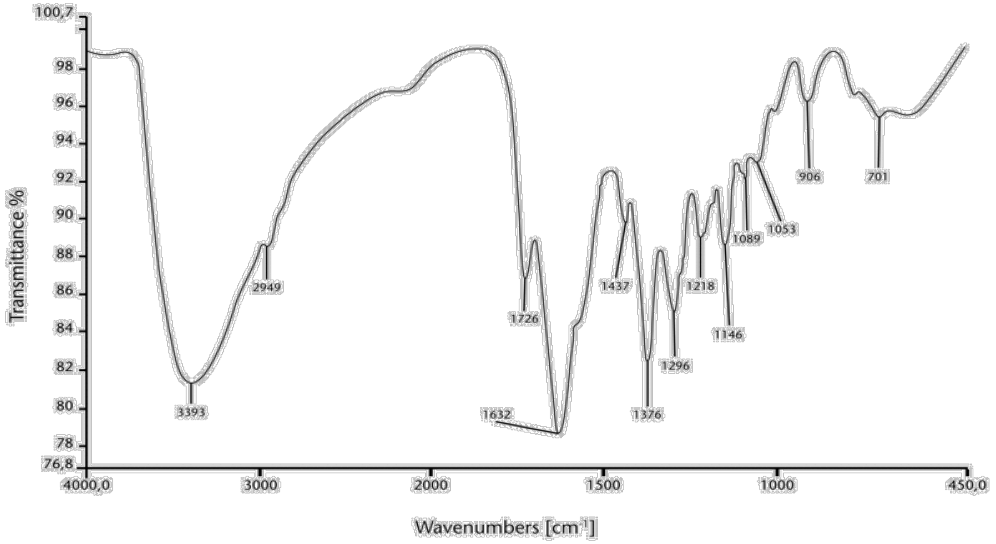
- 2.3.2.2 Specifications for all raw and packaging materials, including, but not limited to ingredients, additives, hazardous chemicals and processing aids … shall be documented and kept current
- 2.4.4.2 Material suppliers shall be selected and approved based on their ability to supply materials that meet quality specifications
- 2.4.4.3 Material suppliers shall only be accepted into the facility based on either certificates of analysis for every lot received, or inspection at receipt to ensure materials comply with specification.
- 2.4.7.1 The site shall document and implement a positive product release procedure to ensure that the food supplied complies with all agreed customer requirements including, but not limited to, product specifications

https://www.sqfi.com/wp-content/uploads/2020/11/20227FMIN_FoodManufacturing_v3-2-FINAL-w-links.pdf

Recent Work on Natural Food Colors

New Monograph: Jagua (Genipin-Glycine) Blue

- **Monograph section - Identification**
  - Visible Absorption Spectrum
    - UV-Vis spectrophotometer (maximum 590-594nm)
  - Infrared Spectra



New Monograph: Jagua (Genipin-Glycine) Blue

- **Monograph section - Assay**
  - Jagua (Genipin-Glycine) Blue Polymer

    Standard preparation

    Method: HPLC

    Detector: UV-Vis/PDA 590 nm

    Acceptance criteria: 20%–43.5% (as the blue polymer), calculated on the dried basis

  - Total Color

    FCC Appendix IIIC Color Determination

    Acceptance criteria: 25%–54.4%, calculated on the dried basis

New Monograph: Jagua (Genipin-Glycine) Blue

- **Monograph section - Impurities**
  - Arsenic NMT 1 mg/kg
  - Lead NMT 2 mg/kg
  - Cadmium NMT 1 mg/kg

  FCC Appendix IIIC ICP Method

- **Monograph section - Specific Test**
  - Genipin

  Method: HPLC

  Detector: UV-Vis 240 nm

  Acceptance criteria: NMT 0.3%, calculated on the dried basis

New Monograph: Paprika Oleoresin

- **Monograph section - Identification**
  - Color reaction
  - UV-Visible Absorption Spectrum
  - Chromatographic profile for β-carotene, capsanthin diester peak 1, and capsanthin diester peak 2 (Chromatographic system in *Assay*)
  - Approximate Relative Retention Time of the peaks for carotenoids (Chromatographic system in *Assay*)

New Monograph: Paprika Oleoresin

- **Monograph section - Assay**
  - Capsanthin and capsorubin

    Method: HPLC

    Detector: Vis 450 nm

    Column:4.6-mm × 250-mm; packing of octadecylsilane chemically bonded to 5-µm porous silica or ceramic micro-particles.Use a suitable C18 guard column.

    Acceptance criteria: NLT 30%

- **Monograph section - Specific Tests**
  - Color Value

    Acceptance criteria: NLT 500 units, as specified on the label

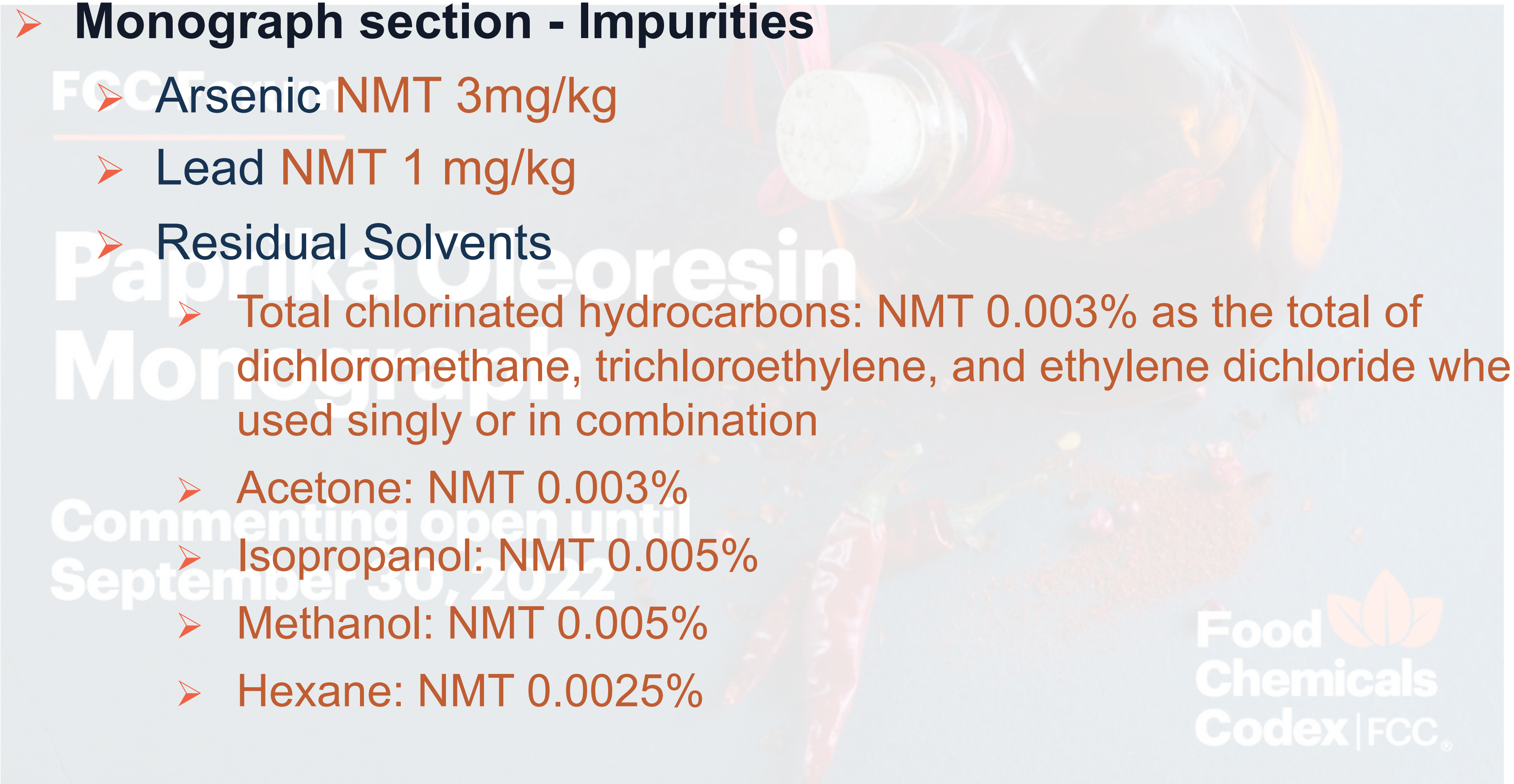
  - Total Capsaicinoids Content

    Acceptance criteria:

    Color applications: NMT 200 mg/kg

    Flavor applications: NMT 0.5%

New Monograph: Paprika Oleoresin



- **Monograph section - Impurities**
  - Arsenic NMT 3mg/kg
  - Lead NMT 1 mg/kg
  - Residual Solvents
    - Total chlorinated hydrocarbons: NMT 0.003% as the total of dichloromethane, trichloroethylene, and ethylene dichloride when used singly or in combination
    - Acetone: NMT 0.003%
    - Isopropanol: NMT 0.005%
    - Methanol: NMT 0.005%
    - Hexane: NMT 0.0025%

FCC Appendix XIII: Residual Solvents in Food Colors

Chromatographic parameters

Mode: Headspace GC

Detector: FID

Column: Fused silica, 30-m × 0.25-mm (id) with a 1.4-µm coating of 6% cyanopropylphenyl–94% dimethylpolysiloxane stationary phase

Gas flow
Air: 400 mL/min
Hydrogen: 40 mL/min
Nitrogen column flow: 1.5 mL/min
Makeup flow: 25 mL/min (nitrogen)
Injection type: Split injection, split ratio of 1:1

Solvent	LOD (µg/g)	LOQ (µg/g)
Methanol	3	10
Ethanol	3	10
Acetone	0.6	2
Isopropanol	3	10
Methylene chloride	3	10
Hexane	0.16	0.63
Ethyl methyl ketone	1.2	4
Ethyl acetate	1.2	4
Ethylene dichloride	4.5	15
Trichloroethylene	4.5	15

FCC Appendix XIII: Residual Solvents in Food Colors

- Effective as of Dec 2021
- Method developed in USP internal lab
- Fully validated in the food color matrices:
  - Annatto extracts
  - Tagetes extract
  - Sodium Copper Chlorophyllins
  - Paprika Oleoresins

Monograph Modernization

The Residual Solvent test method is modernized to *FCC Appendix XIII* method
Effective as of Dec 2021

Tagetes extract

Acceptance criteria: NMT 0.005% acetone, hexane, ethanol, ethyl methyl ketone, isopropanol, and methanol, individually or in combination

Annatto extracts

Acceptance criteria:
Acetone: NMT 0.003%
Ethylene dichloride: NMT 0.003%
Hexanes: NMT 0.0025%
Isopropyl alcohol, ethanol, and ethyl acetate: NMT 0.005%, individually or in combination
Methyl alcohol: NMT 0.005%
Trichloroethylene and Dichloromethane: NMT 0.003%, individually or in combination

Future FCC Work on Natural Food Colors

Expected June 2023 FCC Forum

Two Potential New Monographs:

- Chlorophyllins, Copper Complexes sodium and Potassium Salts
- Cochineal Extract

One Potential Monograph Modernization:

- Lutein

Free access to *FCC Forum*:
https://www.foodchemicalscodex.org/fcc-forum

Food Chemicals Codex | FCC

Future FCC Work on Natural Food Colors

Four Potential New Natural Food Colors

- Black Carrot Extract
- Purple Sweet Potato Color
- Red Beet
- Red Cabbage Color

USP® Food Chemicals Codex | FCC

Thank you

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IACM Global Color Conference
November 16-17, 2022

The Role of FCC in Protecting the Integrity of Natural Food Colors

Eric Schwartz Ph.D.,
Food Chemicals Codex at U.S. Pharmacopeia

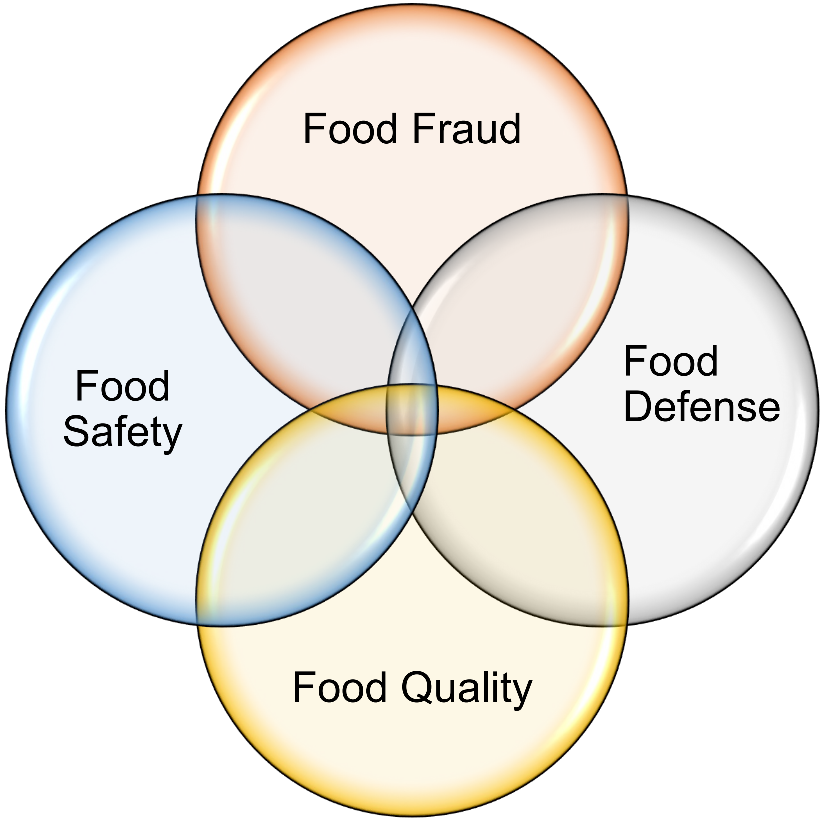
Food Chemicals Codex Scope

- Monographs (>1250)
- Appendices
  - General tests and assays (>150)
  - Guidelines
    - Developing and Validating Non-Targeted Testing
    - Olive Oil Guidance, Methods and Applicable Resources
    - Food Fraud Mitigation

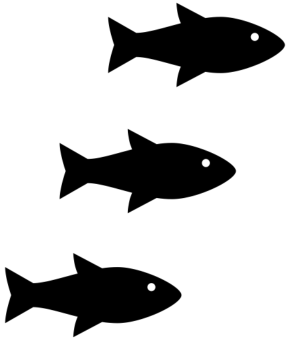
Food Chemicals Codex | FCC

Protecting public health and the integrity of the food supply worldwide

Food Integrity is a Major Public Health Challenge



Elements of food integrity



Source: GSFI

Importance of Standards in Maintaining Ingredient Integrity

- Food supply chains are complex, non-linear, and subject to sudden disruption which can affect food integrity
- While traceability of an ingredient may be known, without proper oversight and controls, the potential for fraud exists
- Standards are integral to the reduction of an ingredient's vulnerability to food fraud and protection of ingredient integrity.
  - especially when the methods and specifications in the standard well characterize the ingredient and contain appropriate validated methods for adulteration detection.

Importance of Quality and Safety for Natural Colors

- With the rising demand for natural ingredients, quality and safety are extremely important topics and opens the door for both unintentional and intentional economic adulteration
- Because up to 85% of consumer buying decisions are potentially influenced by color, appropriate application of color additives and their safety is critical
- Globally, natural colors are used more often in food and beverage launches compared to artificial colors or whole foods that add color

Simon, J.E., Decker, E.A., Ferruzzi, M.G., Giusti, M.M., Mejia, C.D., Goldschmidt, M. and Talcott, S.T. (2017), Establishing Standards on Colors from Natural Sources. Journal of Food Science, 82: 2539-2553.

US Color Regulations

- Under current U.S. Food and Drug Administration regulations, colors fall into 2 categories (covered by 21CFR Parts 70-82)
  - Certified colors - those subject to an FDA certification process
  - Exempt from certification - often referred to as “natural” colors by consumers because they are sourced from plants, minerals, and animals
- "Natural" colors do not undergo a certification process by FDA to assure their quality and safety before they may be marketed
- Legal definitions and labeling requirements for certified dyes and lakes are better defined than those for noncertified, or natural colors

Specification Differences in CFR for Colors

	FD&C Yellow 6	Fruit Juice
	74.706	73.250
Process	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	

Identified Chemical Contaminants in Natural Colors

- **Environmental, Process and other Contaminants**
  - Extraction solvents not approved for use in colors –varies by region
  - Residual solvents in excess of >1,000 ppm
  - Lead and mercury contamination in Carmine
  - Pesticide residues in Paprika
  - Dioxin in Paprika
  - Bacterial and pathogen contamination

- **Contaminants used for Economically Motivated Adulteration**
  - Orange II in safflower
  - Finely ground red bricks in paprika
  - Synthetic dyes in saffron
  - Sudan dues in turmeric and paprika

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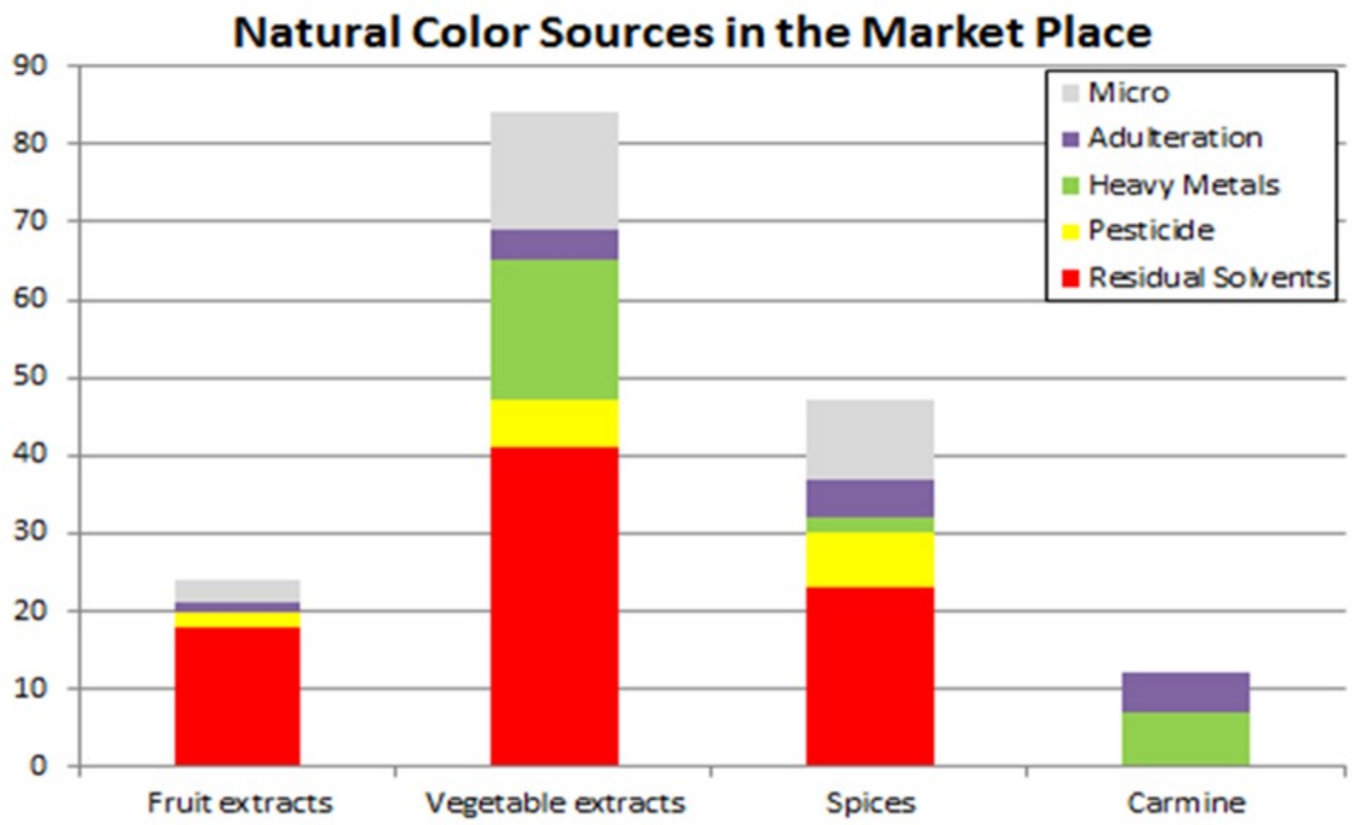
Identified Chemical Contaminants in Natural Colors

- Research was conducted by Sensient over a 4-y period
- Evaluated 650 samples against limits set by Sensient
- Results of the tests indicated 25% of the samples failed to meet specifications resulting in rejection from the supply chain

Prevalence of failures of natural colors in the marketplace from fruit, vegetable, spice, and carmine



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The Need for Industry Guidance on Natural Colors Highlighted in Review Article

- Review article published in *Journal of Food Science*
  - Simon JE, Decker EA, Ferruzzi MG, Giusti MM, Mejia CD, Goldschmidt M, Talcott ST. Establishing Standards on Colors from Natural Sources. J Food Sci. 2017 Nov;82(11):2539-2553.
- Article resulted from a group of individuals with expertise in plant biology, food chemistry, food toxicology, food product development and manufacturing as well as food quality and regulatory affairs
- Objectives of Review Article
  - Make recommendations for quality and product safety standards for the natural colors industry
  - Promote standardization of methods used to test natural colors
  - "Raise the bar" on the quality and safety of natural colors

Concise Reviews & Hypotheses in Food Science

Feature Article

Establishing Standards on Colors from Natural Sources

James E. Simon, Eric A. Decker, Mario G. Ferruzzi, M. Monica Giusti, Carla D. Mejia, Mark Goldschmidt, and Stephen T. Talcott

Abstract: Color additives are applied to many food, drug, and cosmetic products. With up to 85% of consumer buying decisions potentially influenced by color, appropriate application of color additives and their safety is critical. Color additives are defined by the U.S. Federal Food, Drug, and Cosmetic Act (FD&C Act) as any dye, pigment, or substance that can impart color to a food, drug, or cosmetic or to the human body. Under current U.S. Food and Drug Administration (FDA) regulations, colors fall into 2 categories as those subject to an FDA certification process and those that are exempt from certification often referred to as "natural" colors by consumers because they are sourced from plants, minerals, and animals. Certified colors have been used for decades in food and beverage products, but consumer interest in natural colors is leading market applications. However, the popularity of natural colors has also opened a door for both unintentional and intentional economic adulteration. Whereas FDA certifications for synthetic dyes and lakes involve strict quality control, natural colors are not evaluated by the FDA and often lack clear definitions and industry accepted quality and safety specifications. A significant risk of adulteration of natural colors exists, ranging from simple misbranding or misuse of the term "natural" on a product label to potentially serious cases of physical, chemical, and/or microbial contamination from raw material sources, improper processing methods, or intentional postproduction adulteration. Consistent industry-wide safety standards are needed to address the manufacturing, processing, application, and international trade of colors from natural sources to ensure quality and safety throughout the supply chain.

Keywords: adulteration, food additives, food safety, natural colors, synthetic colors

Introduction

Current regulations for colors exempt from certification, also referred to as "natural" colors, noncertified colors, or exempt colors because they are not subject to certification testing by the FDA prior to use, lack consistent definitions and universally accepted quality control and product safety specifications that typically comprise a harmonized regulatory framework (Burrows 2009; Scotter 2011; Oplatowska-Stachowiak and Elliott 2017). Additionally, there is a lack of agreement on standard testing methods that would help to insure the safety, quality, and purity of these color additives. A survey conducted by Innova Database reported that 31% of American consumers surveyed were very or extremely concerned about food colorings (Innova Database 2013). This survey highlights the growing importance of natural colors and the opportunities for the natural colorings industry to address both safety and consumer concerns.

Even a single product safety incident involving natural color additive could adversely impact consumer confidence and alter the marketplace. History has shown that problems with adulterants or contaminants in foods, perceived safety and side-effects of synthetic colors, and even the revelation that cochineal red was an insect-based color have resulted in public demand for clarity that has changed FDA rulings on ingredient labels (Burrows 2009). To ascertain a need for consistent standards for the manufacture and application of natural colors, scientists from the fields of plant biology, food chemistry, food toxicology, food product development and manufacturing, and food quality and regulatory affairs organized to address quality attributes and potential safety hazards. This paper arose from those multidisciplinary discussions, with a focus on understanding potential hazards through the perspective of manufactures, importers, and end-users of natural colors. This work proposes standards for testing natural colors to ensure their quality and safety. The objective of this review is to outline current issues involving natural color additives in the marketplace and propose solutions that lead to recommendations for establishing industry standards for colors from natural sources. Our intent for this work is to initiate discussion among members of the academic, regulatory, and industrial communities who are responsible for realistic, practical approaches to define standards for natural colors. This work was initiated by Sensient Technologies Corp. (Sensient; Wisc., U.S.A.) in collaboration with the U.S. Pharmacopeial Convention (USP), but the content of this work does not

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Certification: An Expert Committee Report

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FCC's Efforts to Provide Stakeholder Guidelines on Natural Colors

- Currently a new guideline document (new FCC appendix) is in development based on information from the review article and will entail recommendations, links, and references for the safety and quality of natural colors on
  - Heavy metals
  - Microbiology
  - Residual solvents
  - Pesticides
- Incorporation of links to relevant FCC appendices and USP-NF chapters
  - Links to natural color monographs
  - Links to relevant CFR text
  - Appendix XIII: Adulteration and contaminants in food ingredients
  - Food fraud mitigation guidance
  - USP <563> Identification of articles of botanical origin

Critical Factors and Mitigation Strategies for Natural Colors

Risk	Mitigation strategy	USP Reference
Microbiological	Test all incoming raw materials for wholesomeness and spoilage. Test all finished products for pathogens, spoilage organisms, and mycotoxins.	APPENDIX XV: Microbial Food Cultures Including Probiotics
Heavy metals	Test most common heavy metals based upon FDA,[a] EU,[b] and JECFA[c] guidelines.	APPENDIX III: Chemical Tests and Determinations – Elemental Impurities
Pesticides	Test for presence of pesticides that are not allowed (EPD[d] and EU)	APPENDIX XIII: Adulterants and Contaminants in Food Ingredients – Pesticide Residues

[a]U.S. Food and Drug Administration [b]European Union [c]Joint FAO/WHO Expert Committee of Food Additives [d]Environmental Product Declaration [e]American Spice Trade Assn., Inc.

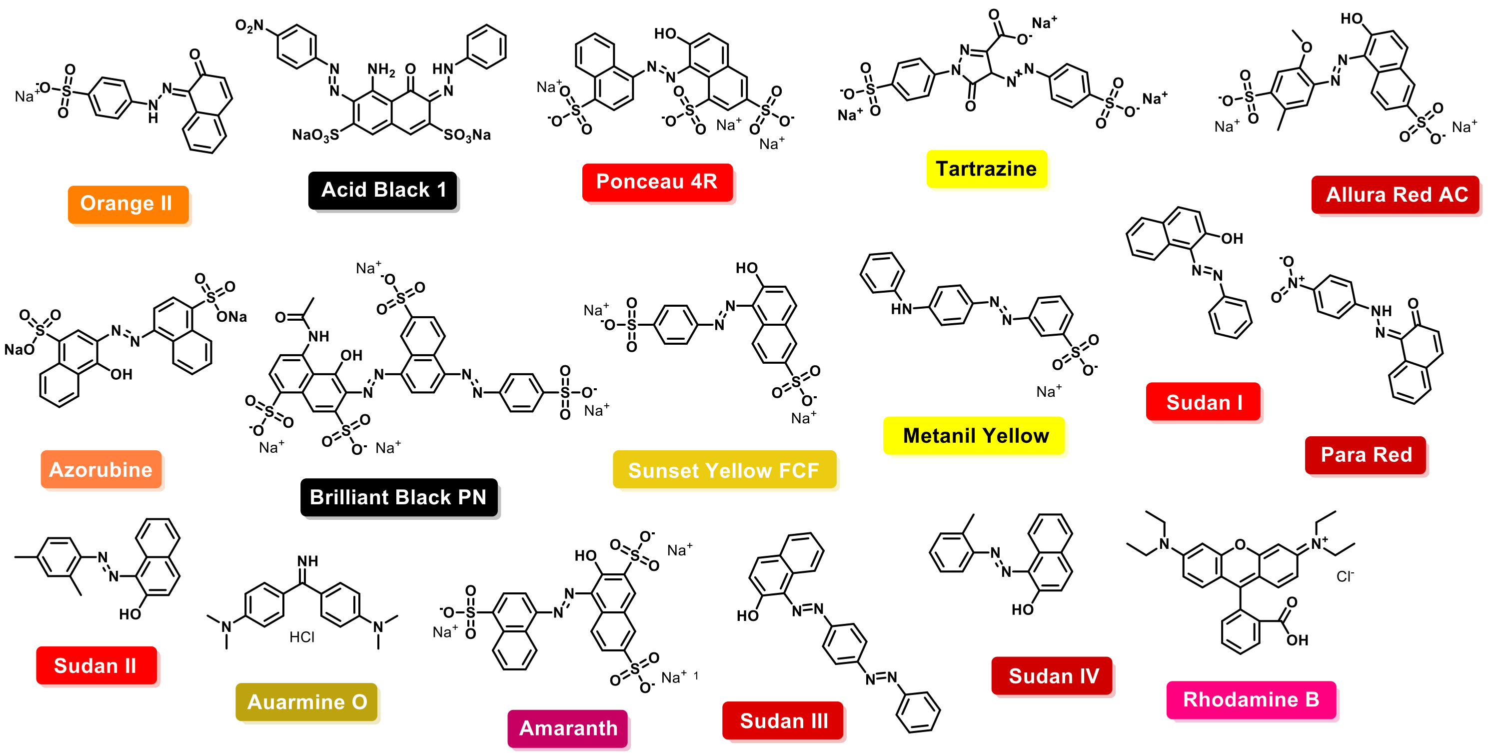
Critical Factors and Mitigation Strategies for Natural Colors

Risk	Mitigation strategy	USP Reference
Intentional Adulteration	Test for synthetic dyes or known additives that physically or chemically adulterate. Screen for unknown adulterants.	APPENDIX XIII: Adulterants and Contaminants in Food Ingredients – Added Colors in Spices APPENDIX XVII: Food Fraud Mitigation Guidance
Extraneous/foreign materials	Botanically identify source materials. Screen for extraneous materials including soil, insects, and physical hazards. Use ASTA[e] and FDA guidelines .	USP <561> Articles of Botanical Origin
Solvents and solvent residues	Evaluate supplier process for approved and unapproved solvents. Test finished products for unapproved solvents and solvent residue.	APPENDIX XIII: Adulterants and Contaminants in Food Ingredients – Residual Solvents in Food Colors

[a]U.S. Food and Drug Administration [b]European Union [c]Joint FAO/WHO Expert Committee of Food Additives [d]Environmental Product Declaration [e]American Spice Trade Assn., Inc.

FCC Appendix XIII: Adulterants and Contaminants in Food Ingredients

- Effective text published in FCC Appendix XIII
  - Non-targeted tests for the detection of undeclared colors in paprika, chili powder, turmeric, curry, and sumac
    - Thin-layer chromatography screening method
    - Three different validated procedures for detection of a total of 17 colors



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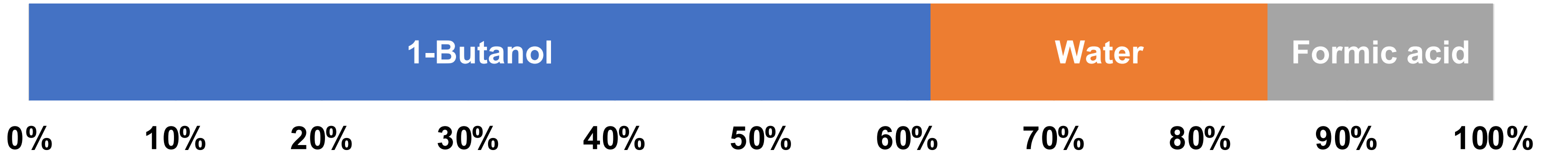
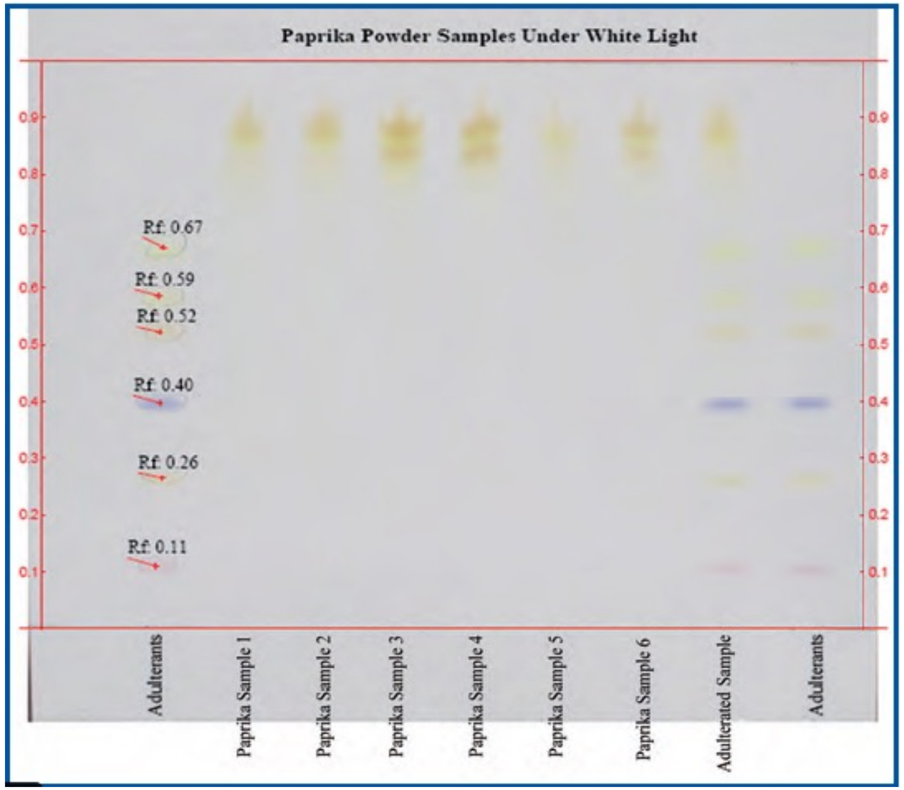
FCC Appendix XIII: Adulterants and Contaminants in Food Ingredients

- **Adsorbent:** 0.2-mm layer of silica
- **Developing solvent system:**



Procedure 3 Retention Factors (Rf)

Color	R_F Value
Amaranth (Acid red 27)	0.08
Yellow 6 (Food yellow 3)	0.24
Acid black 1	0.38
Orange II (Acid orange 7)	0.52
Metanil yellow (Acid yellow 36)	0.58
Auramine O	0.67

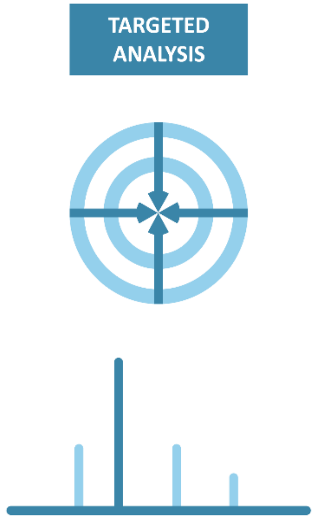


FCC Appendix XIII: Adulterants and Contaminants in Food Ingredients

- Current work
  - Targeted test for the detection of undeclared colors in paprika, chili powder, turmeric, curry, and sumac
    - LC-MS/MS validated screening method for the detection of 25 undeclared colors
    - Involves matrix matched calibration curves for quantitation

Compound Name	RT (min.)	Parent (m/z)	Cone (V)	Daughter 1 (m/z)	Collision Energy 1 (V)	Daughter 2 (m/z)	Collision Energy 2 (V)	Recovery (%)
Basic Red 9	4.9	289	60	151	50	195	30	75.6
Safranin O	6.3	315	60	210	55	237	45	75.5
Auramin O	6.9	268	60	147	30	252	35	88.9
Basic Yellow 13	7.2	307	60	170	35	292	25	90.9
Basic Yellow 28	7.4	322	60	136	25	160	15	89.9
Alizarin	7.8	241	60	157	25	185	22	93.1
Malachite Green	8.2	329	60	208	40	313	35	91.2
Quinoline Yellow	8.9	274	60	105	30	228	35	94
Disperse Orange 11	9.0	238	60	165	35	223	25	79.8
Rhodamine 6G	9.0	443	60	341	50	415	35	84.2
Rhodamine B	9.1	443	80	355	60	399	42	92.4
Chrysodine G	9.2	213	60	77	30	121	15	92.5
Sudan orange G	9.2	215	34	93	25	122	20	99.2
Crystal Violet	9.2	373	60	340	50	356	40	86.6
1-Methyl Amino anthraquinone	9.8	238	60	167	35	223	25	91.6
Brilliant Green	9.8	385	60	297	55	341	40	85.5
Butter Yellow	10.7	226	42	77	30	121	25	92.5
Para Red	11.0	294	34	128	34	156	18	81
Sudan Red G	11.5	279	25	80	55	108	35	92.6
Sudan I	11.6	249	30	128	28	156	20	78.2
Citrus Red II	11.6	309	20	138	40	152	10	81.6
Sudan II	12.9	277	30	121	20	156	20	91.8
Sudan III	13.5	353	50	156	25	197	19	93.5
Sudan Red 7B	14.2	380	34	169	30	183	20	99.4
Sudan IV	14.4	381	50	106	44	224	22	99.4

Colors, retention times, MRM transitions, and recovery of 25 colors in chili at 10 ppm



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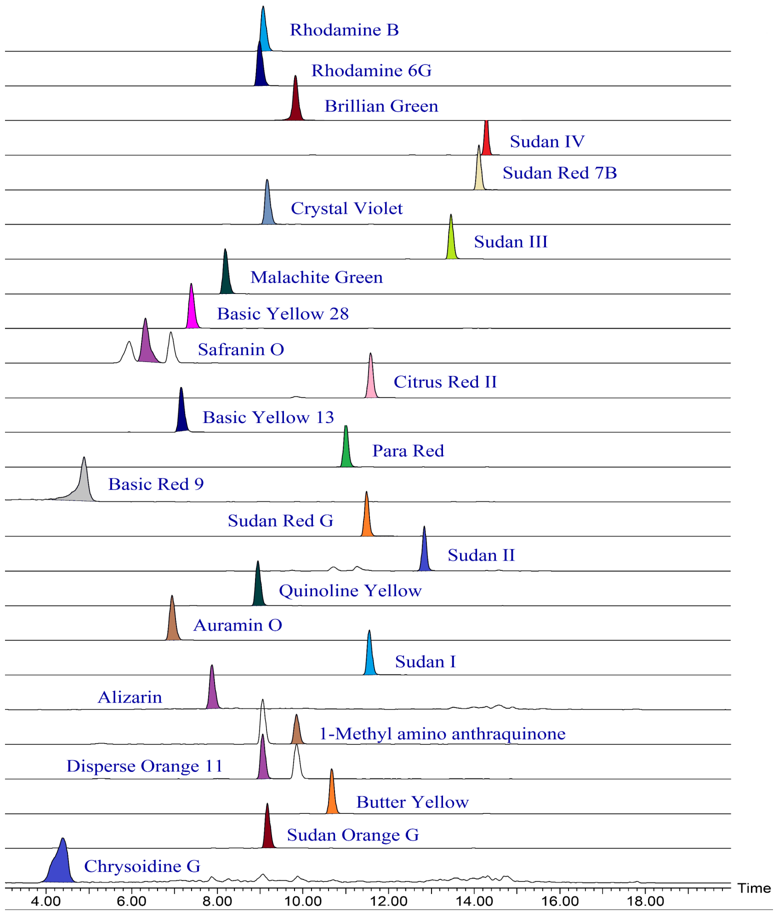
FCC Appendix XIII: Adulterants and Contaminants in Food Ingredients

Data from commercial chili powder (rejected samples) in ppm

Color	Source-1	Source-2	Source-3	Source-4	Source-5	Source-6	Source-7
Butter Yellow	ND	3.41	6.91	ND	ND	ND	ND
Sudan I	1672.67	1095.17	1209.78	1455.98	ND	ND	100.11
Sudan II	ND	ND	ND	ND	14.73	ND	ND
Para Red	ND	ND	ND	0.48	ND	ND	ND
Malachite Green	ND	ND	ND	ND	ND	ND	0.47
Sudan III	1.74	11.94	12.09	21.62	ND	ND	1.41
Sudan Red 7B	0.5	ND	ND	ND	ND	ND	0.24
Sudan IV	212.74	101.87	90.54	114.73	0.74	10.68	ND

Stacked spectra of 25 colors spiked in chili powder



Summary

- Maintaining food and ingredient integrity is a major challenge evidenced by numerous food safety incidents throughout history
- A persistent problem with quality relative to contamination exists in colors from natural sources
- Consistent industry-wide safety standards are needed to address the manufacturing, processing, application, and international trade of colors from natural sources
- FCC is developing a new appendix based on the article published in the Journal of Food Science
  - which will include guidelines and best practices to help ensure quality and safety throughout the supply chain for natural colors
- FCC has tools in place to protect the integrity of natural colors - ingredient monographs and appendix tests

Thank You

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Stay Connected

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