ASTA Adulteration and Spice Integrity Guide

Date of Last Revision: 8 November 2021

Revision No. 1

Reason for Revision: Periodic Review

1101 L Street, N.W. Suite 700. Washington, D.C. 20036.
www.astaspice.org

Contents

Introduction

What is Spice Integrity?

Spice integrity, or authenticity, is the assurance that an herb or spice is free from fraud. For a spice to be authentic, it should --

- Be of the botanical species declared, in accordance with industry agreed nomenclature, with no undeclared components; and
- Meet the global standards for extraneous vegetable matter; and
- From the country of origin declared; and
- Only be processed with any declared approved technology, officially permitted in the country of sale; and
- Be grown under the regime promoted e.g., organic, fair trade, GAP certified, etc.; and
- Where applicable, grown in the country of harvest that has been declared

It is important to note that this list is not comprehensive, and other requirements may exist for spices to meet authenticity definitions or standards.

What is Adulteration?

Adulteration is the inclusion of constituents in food whose presence is prohibited by regulation, custom and practice, or "making impure by adding inferior, alien, or less desirable materials or elements." Adulteration can also include the removal of valuable constituents.

Types of adulterants include –

- Bulking agents – These adulterants, which include starch and flour, are meant to add volume to the finished spice product so that it appears that there is more spice than there actually is.
- Substitution – Some adulterants, such as different botanical species, are added to products in place of the spice because they are cheaper alternatives.
- Concealment – These adulterants, which include artificial and natural dyes, are added to spice to conceal either poor-quality spices or other adulterants.

Testing Methods

At the time of publication, the following testing methods and technologies identified in this document are commonly used to detect adulterants in spice matrices. However, detection methods evolve over time as new technologies are developed and new adulterants are identified.

Although listed in this document, DNA-NGS testing is not known to be effective in the identification of adulteration in oregano and black pepper. Thus, the microscopic evaluation that has been defined as the ASTA Method is currently the most accurate testing methodology for the analysis of adulterants.

Cumin

Cumin has been known to be compromised due to economically motivated fraud. This section summarizes commonly known adulterants and testing methods. The impurity and testing matrix described below can be used to determine cleanliness and purity of the spice.

IMPURITY & TESTING MATRIX FOR CUMIN

Chart defines examples and testing methods used to identify potential known adulterants.

Adulterant	**Type of Adulterant**	**Testing Method**
Grass Seed - colored with charcoal dust	Concealment, Substitution	Random Amplified Polymorphic DNA
Fennel and dill seeds covered with powder, cement, or mud	Concealment, Substitution	GC, Microscopic ID, Ash
Fennel Seed - dye-coated poor-grade	Substitution	LCMS LC-UV/DAD
Synthetic cumin aldehyde – substitute used in essential oil	Substitution	SIRA SIMS Optical rotation
Almond, peanut shell	Bulking agent	Elisa for detection of allergens
Cereal, tuber flour	Bulking agent	Microscopic ID
Starch	Bulking agent	Microscopic ID Iodine-starch test
Prunus mahaleb	Bulking agent	DNA Testing – PCR LC-MS
Spent cumin	Bulking agent	Volatile oil analysis

Paprika

The adulteration of paprika is primarily made by processors (millers or blenders), as identification of adulterants is easier in whole product. Another common example of adulteration is the mislabeling of paprika, made by indication of false information about varieties, origins, and protected designations. This section summarizes commonly known adulterants in paprika and testing methods.

IMPURITY & TESTING MATRIX FOR PAPRIKA

Chart defines examples and testing methods used to identify potential known adulterants.

Adulterant	**Types of Adulterant**	**Testing Method**
Sugars and starch	Bulking agent	Microscopic ID IR spectroscopy Chromatography (sugars) Iodine-test (starch only)
Tomato skins	Bulking agent	Microscopic ID Lycopene determination
Almond shells	Bulking agent	Immunoassay – ELISA DNA testing – PCR Microscopic ID
Mineral elements (i.e., brick dust)	Bulking agent/Substitution	Ash/AIA determination
Parts of other plants	Bulking agent	Microscopic ID
Added dyes (Artificial) – Sudan group, lead oxide	Concealment	AAS (lead oxide) Chromatography (any sudan dyes)
Added dyes (Natural)—Annatto, bixin	Concealment	LC
Defatted paprika	Substitution	Solvent determination by GC Microscopic ID

Saffron

The adulteration of saffron occurs at all levels of the supply chain (farmers, brokers, processors, and packers) and is performed in both whole and ground presentations. Some adulteration in saffron can be difficult to identify even for qualified operators. One important adulteration is related to origin, as prices present significant differences depending on the country of origin of the raw material. This section summarizes commonly known adulterants in saffron and testing methods.

IMPURITY & TESTING MATRIX FOR SAFFRON

Chart defines examples and testing methods used to identify potential known adulterants.

Adulterant	**Type of Adulterant**	**Testing Method**
Starch and sugars	Bulking agent	Microscopic ID IR spectroscopy Chromatography (sugars) Iodine-test (starch only)
Added dyes (Artificial, annatto)	Concealment	LCMS/MS or other chromatography based detection
Other vegetal species and extracts (Gardenia, Safflower, Paprika, Turmeric, Beetroot, Marigold)	Substitution	Spectroscopy DNA based methods
Floral waste – parts of the flower of *Crocus sativus* other than pistils (styles, statements and pollen grains, ovary rests, petals and other rests, annatto)	Substitution	Microscopic ID

Black Pepper

The adulteration of black pepper predominantly occurs at processing or milling sites, and not at farms. Another common adulteration is the mislabeling of black pepper, made by an indication of false information about varieties, origins, and protected designations. This section summarizes commonly known adulterants in black pepper and testing methods.

IMPURITY & TESTING MATRIX FOR BLACK PEPPER

Chart defines examples and testing methods used to identify potential known adulterants.

Types of Adulterant	**Adulterant**	**Testing Method**
Buckwheat	Bulking agent	Spectroscopy ELISA
Coffee husks	Bulking agent	Microscopic ID Chromatography (caffeine)
"Spent" pepper (de-oiled pepper)	Bulking agent	Volatile oil analysis, fat, moisture
Papaya seeds	Substitution & Concealment	GC and glucose release – determination of benzyl glucosinolate (glucotropaeolin)
Silt cotton tree seeds	Substitution & Concealment	Microscopic ID
Dyed gravel	Substitution & Concealment	Microscopic ID Ash/AIA Ash/Spectroscopy and Chemometric modelling/NIR FTIR
Long pepper (*Piper longum*)	Substitution & Concealment	Organoleptic/NIR/GC fingerprint of volatile oils NGS
Mineral oil	Substitution & Concealment	LC-MS

Turmeric

This section summarizes commonly known adulterants in turmeric and testing methods.

IMPURITY & TESTING MATRIX FOR TURMERIC

Chart defines examples and testing methods used to identify potential known adulterants.

Types of Adulterant	**Adulterant**	**Testing Method**
Starches	Bulking agent	Microscopic ID Spectroscopy Iodine-starch test
Saw dust	Bulking agent	Microscopic ID
Chalk powder	Bulking agent	Spectroscopy Microscopic ID
Rice paddy husk	Bulking agent	Microscopic ID
Rice flour	Bulking agent	Multispectral analysis Iodine-starch test
Corn flour	Bulking agent	Iodine-starch test
Talcum powder	Bulking agent	Ash AAS ICP-OES
Mineral elements	Bulking agent/Substitution	Ash/AIA determination
“Spent” (de-oiled turmeric)	Bulking agent	Determination of defatted turmeric, total fat
Non-permitted colorants (i.e., lead chromate)	Concealment	GC/MS/MS (ASTA 28.0 or 29.0)
Soap stone - yellow	Substitution	Physical and colorimetric tests
Other Curcuma species (*zedoaria/malabarica*)	Substitution	DNA Testing – PCR Microscopic ID Thin-layer chromatography

Oregano

This section summarizes commonly known adulterants in oregano and testing methods.

IMPURITY & TESTING MATRIX FOR OREGANO

Chart defines examples and testing methods used to identify potential known adulterants.

Types of Adulterant	**Adulterant**	**Testing Method**
Olive leaves	Substitution	Microscopic analysis GC NIR FT-IR
Sumac leaves	Substitution	
Myrtle leaves	Substitution	
Hazelnut leaves	Substitution	
Cistus	Substitution	
Pellets of oregano stems	Substitution	
Spent oregano	Substitution	
Other green leaves	Bulking agent	

Additional Information

ASTA has produced a guide on Economically Motivated Adulteration to provide companies with the tools to effectively identify circumstances where adulteration may occur and then take steps to prevent it. The guide can be found at: https://www.astaspice.org/food-safety-technical-guidance/best-practices-and-guidance/identification-prevention-adulteration-guidance-document/

Appendix 1: Analytical Methodology Glossary

Acronym	Definition
AAS	Atomic Absorption Spectrometry
AIA	Acid Insoluble Ash
FTIR	Fourier Transform Infra-Red
GC	Gas Chromatography
GC-MS/MS	Gas Chromatography with Tandem Mass Spectrometry
IR	Infra-Red
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
LC-MS	Liquid Chromatography with Mass Spectrometry
LC-MS/MS	Liquid Chromatography with Tandem Mass Spectrometry
LC-UV	Liquid Chromatography with Ultraviolet detector
LC-DAD	Liquid Chromatography with Diode array detector
NIR	Near Infra-Red
PCR	Polymerase Chain Reaction
SIRA	Stable Isotope Ratio Analysis
SIMS	Secondary Ion Mass Spectrometry