

Standard Method Performance Requirements (SMPRs®) for Targeted Testing (TT) of Turmeric Adulterants

Intended Use: Surveillance and Monitoring by Trained Analysts

1 Purpose

AOAC SMPRs describe the minimum recommended performance characteristics to be used during the evaluation of a method. The evaluation may be an on-site verification, single-laboratory validation (SLV), or multi-site collaborative study. SMPRs are written and adopted by AOAC stakeholders composed of representatives from industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC method review experts, including expert review panels (ERPs), in their evaluation of validation study data for methods being considered for AOAC *Performance Tested Methods*SM, *Reviewed and Recognized*SM, or AOAC *Official Methods of Analysis*SM, and can be used as acceptance criteria for verification at user laboratories.

2 Applicability

The document contains assessment parameters on the performance of targeted testing (TT) methods to monitor turmeric (as a spice or nutritional supplement) for the presence of the following potential economically motivated adulterants (EMAs): Sudan 1, Metanil yellow (sodium 3-[4-anilinophenylazo] benzenesulfonate), Acid Orange II (sodium 4-[(2E)-2-(2-oxonaphthalen-1-ylidene)hydrazinyl] benzenesulfonate, lead chromate, Yellow chalk (soapstone) powder, *Curcuma xanthorrhoea*, *Curcuma zedoaria*, *Curcuma malabarica*, *Curcuma aromatic*, and Cassava (*Manihot esculenta*).

3 Analytical Technique

TT method(s) to monitor turmeric (as a spice or nutritional supplement) for the presence of the following potential EMAs: Sudan 1, Metanil yellow, Acid Orange II, lead chromate, Yellow chalk (soapstone) powder, *Curcuma xanthorrhoea*, *Curcuma zedoaria*, *Curcuma malabarica*, *Curcuma aromatic*, and Cassava (*Manihot esculenta*).

TT method to be used to monitor and enforce regulatory requirements for turmeric adulterants in food.

Any quantitative method capable of detecting, identifying, and quantifying the presence of an adulterating ingredient in turmeric present in the food item will be considered.

The scope of the TT method will be defined by the authentic samples and or reference standard material (if available) that were used in validating the method.

It should be noted that the maximum limit of permissible colors that may be added to any food to be consumed as specified in the Prevention of Food Adulteration Act of India (PFA) is 100 mg/kg body weight.

Metanil yellow, the most frequently and widely used nonpermitted food color that includes synthetic dyes, such as auramine, lead chromate, rhodamine, sudan-3, sudan-4, orange 2,

and malachite green, is suspected to be mutagenic and carcinogenic and, therefore, presents potentially serious health issues to the consumer.

The allowable level of lead in turmeric powder is 2.5 ppm in Bangladesh, 10 ppm by the Food and Agriculture Organization of the United Nations, and 2.5 ppm in India (1–3).

4 Definitions

Applicability statement.—The document contains assessment parameters on the performance of TT methods to monitor turmeric (as a spice or nutritional supplement) for the presence of the following potential EMAs: Sudan 1, Metanil yellow, Acid Orange II, lead chromate, Yellow chalk (soapstone) powder, *Curcuma xanthorrhoea*, *Curcuma zedoaria*, *Curcuma malabarica*, *Curcuma aromatic*, and Cassava (*Manihot esculenta*).

Authentic turmeric.—In the United States, according to the U.S. Food and Drug Administration (FDA), the term ‘natural flavor’ or ‘natural flavoring’ means the essential oil, oleoresin, essence or extractive, protein hydrolysate, distillate or any product of roasting, heating or enzymolysis, which contains the flavoring components derived from a spice, fruit or fruit juice, edible yeast, herb, bark, bud, root, leaf, or similar plant material, meat, seafood, poultry, eggs, dairy products, or fermentation products thereof, whose significant function is flavoring rather than nutritional.

In the European Union, a natural flavoring substance shall mean a flavoring substance obtained by appropriate physical, enzymatic, or microbiological processes from material of vegetable, animal, or microbiological origin either in the raw state or after processing for human consumption by one or more of the traditional food preparation processes listed in Appendix II (OF WHAT?) processes, which include heating, smoking, curing, maturing, drying, marinating, extraction, extrusion, or a combination of such processes.

They also correspond to substances that are naturally present and have been identified in nature.

The accepted Latin binomial name is *Curcuma longa* L., and the synonymous name is *Curcuma domestica*, belonging to the botanical family: Zingiberaceae. It goes by the common names: turmeric, common turmeric, Indian saffron, and yellow ginger.

The plant is native to Southeast Asia, especially India. It is available in all states of India, but particularly in Tamil Nadu, West Bengal, and Maharashtra. It is a tropical crop cultivated at sea level to 1200 m above sea level and grows in light black clay loam soils and red soils under irrigated and rain-fed conditions. It is also extensively cultivated in Pakistan, China, Haiti, Jamaica, Peru, Taiwan, Nigeria, Bangladesh, and Thailand. Other important producers include Japan, Indonesia, Sri Lanka, Burma (Myanmar), Cambodia, Malaysia, and the Philippines. It has a wide distribution as a non-native species in Madagascar, Oceania.

Turmeric is distinguished by the presence of the orange pigment curcumin. Several other species of *Curcuma*, e.g., *C. aromatica* and *C. zedoaria*, are also known to contain curcumin. Commercially, dried rhizome/root is sold either whole or in powdered form.

In terms of varieties, it appears there are up to 30 different varieties growing in India, but only two designations are commercially significant: *Alleppey* and *Madras* turmeric, both named after the places of cultivation. The *Alleppey* turmeric grows in the Thodupuzha and Muvattupuzha regions of Kerala State, and the variety is predominantly imported by the United States in unpolished form, where users prefer it as a spice and food colorant. This turmeric contains about 3.5–5.5% volatile oil and 4–7%

Table 1. Method performance requirements for the color and noncolor adulterants in turmeric powder

Parameter	Acceptance criteria for color adulterants	Acceptance criteria for noncolor adulterants
Analytical range, %	1–30	1–30
LOQ, %	≤1	≤1
Recovery, %	80–120	80–120
Accuracy, %	±20	±20
Precision (repeatability) RSD _r	15	15
Precision (reproducibility) RSD _R	20	20
	Sudan I, Metanil yellow, acid orange II, lead chromate, Yellow chalk powder, <i>curcuma aromatica</i> , <i>curcuma malabarica</i> , <i>curcuma zedoaria</i> , <i>curcuma xanthorrhoea</i>	Noncolor adulterants include cassava, starch, and glucose

curcumin. In contrast, the *Madras*-type contains only 2% volatile oil and 2% curcumin.

Economically motivated adulteration (EMA).—Fraudulent addition of nonauthentic substances or removal or replacement of authentic substances without the purchaser’s knowledge for economic gain of the seller.

Multilaboratory validation (MLV).—Demonstration between laboratories using adulterated samples created by a third-party group and supplied blindly to the participating laboratories according to guidelines described in OMA Appendix D (4) to be considered for classification as AOAC Final Action Method and in refs. 5–8.

The predicted relative standard deviation of reproducibility (PRSD_R) is calculated from the Horwitz equation:

$$\text{PRSD}_R = 2C^{-0.15}$$

where *C* is expressed as a mass fraction.

For quantitative methods undergoing MLV, 10–12 laboratories must be recruited to provide at least eight valid data sets, two blind duplicate replicates at five concentration levels for each analyte/matrix combination to each collaborator.

$$\text{HorRat (repeatability, } r) = \text{RSD}_r / \text{PRSD}_R$$

$$\text{HorRat (reproducibility, } R) = \text{RSD}_R / \text{PRSD}_R$$

For interlaboratory studies, acceptable HorRat (*R*) of 1 with limits of acceptability of 0.5–2. For within-laboratory studies, acceptable HorRat (*r*) of 0.3–1.3.

Nonauthentic substance or adulterant.—Food item intentionally labeled as turmeric when the product developer knows that another substance or an adulterant, such as those listed in the applicability statement, has been used to adulterate turmeric for economic gain.

Single-laboratory validation (SLV).—Demonstration by one laboratory of method performance on samples described according to internationally accepted validation guidelines contained in guidance documents (4, 9–15).

Once the method has been demonstrated to meet the minimum requirements for validation and fit for purpose criteria, the method can be reviewed and considered by AOAC INTERNATIONAL for adoption and publication in the *Official Methods of Analysis of AOAC INTERNATIONAL* as First Action status.

5 Method Performance Requirements

See Table 1.

6 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blanks and appropriate check standards.

7 Reference Materials

A detailed description of the process used to obtain and evaluate authentic/reference standard materials (sources) and of the test protocol used for validating the method must be provided.

8 Validation Guidance

(a) Data demonstrating method performance is required.

(b) *Samples.*—Complete documentation for the collection and use of authentic samples must be supplied by the method authors. The scope of “authentic” samples used to validate the method must be applicable to the defined scope of the TT method. Expansion of the scope is possible with the inclusion of additional authentic samples and abbreviated validation using the protocol listed in the SMPR.

(c) *SLV studies.*—The method will be evaluated using prescribed adulterated materials as shown in Table 1. Methods approved at this level will proceed to a second level of evaluation (MLV), where blinded samples containing unknown adulterants will be sent to participating laboratories.

(d) *Statistical analysis of interlaboratory studies.*—Sample size needed to meet performance requirement on proportion.

9 Maximum Time-to-Results

None.

10 References

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