

1 **AOAC SMPR 2021.XXX; Draft AOAC Standard Method Performance Requirements (SMPRs) for**
2 **Targeted Testing (TT) of Turmeric Adulterants; Version 2; May 2021**

3
4 **Intended Use**

5 AOACI SMPRs® describe the minimum recommended performance characteristics to be used
6 during the evaluation of a method. The evaluation may be a single-laboratory validation, or a
7 multi-site collaborative study.

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9 SMPRs are written and adopted by AOACI using the consensus of stakeholders representing the
10 industry, government, and academic and/or research institutions. AOACI SMPRs are used by
11 AOACI expert review panels (ERPs) in their evaluation of validation study data for method being
12 considered for *Performance Tested MethodsSM* or *AOACI Official Methods of AnalysisSM* and can
13 be used as acceptance criteria for verification at user laboratories.

14
15 **1. Applicability**

16 This document contains assessment parameters on the performance of Targeted Testing
17 methods to monitor turmeric (as a Spice or nutritional supplement) for the presence of the
18 following potential economically motivated adulterants (EMAs): Sudan 1, Metanil Yellow,
19 (sodium 3-[4-anilinophenylazo] benzenesulfonate), Acid Orange II, (sodium 4-[(2E)-2-(2-
20 oxonaphthalen-1-ylidene) hydrazinyl] benzenesulfonate, Lead Chromate, Yellow chalk
21 (soapstone) powder, *Curcuma xanthorrhoea*, *Curcuma zedoaria*, *Curcuma malabarica*, *Curcuma*
22 *aromatic*, and Cassava (*Manihot esculenta*).

23
24 **2. Analytical Technique**

25 A Targeted Testing (TT) method(s) to monitor turmeric (as a Spice or nutritional supplement) for the
26 presence of the following potential economically motivated adulterants (EMAs): Sudan 1, Metanil
27 Yellow, Acid Orange II, Lead Chromate, Yellow chalk (soapstone) powder, *Curcuma xanthorrhoea*,
28 *Curcuma zedoaria*, *Curcuma malabarica*, *Curcuma aromatic*, Cassava (*Manihot esculenta*).

29 A Targeted method to be used to monitor and enforce regulatory requirements for turmeric
30 adulterants in food.

31
32 Any quantitative method capable of detecting, identifying and quantifying the presence of an
33 adulterating ingredient in turmeric present in the food item will be considered.

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

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

39 Metanil yellow, the most frequently and widely used non-permitted food colour that include the
40 synthetic dyes such as auramine, lead chromate, rhodamine, sudan-3, sudan-4, orange 2 and

41 malachite green, are suspected to be mutagenic and carcinogenic and, therefore, present
42 potentially serious health issues to the consumer.

43

44 The allowable level of lead in turmeric powder is 2.5 ppm in Bangladesh, 10 ppm by the
45 Food and Agriculture Organization of the United Nations, and 2.5 ppm in India.

46

47 **3. Definitions**

48 **Applicability Statement** – This document contains assessment parameters on the performance
49 of Targeted Testing methods to monitor turmeric (as a Spice or nutritional supplement) for the
50 presence of the following potential economically motivated adulterants (EMAs): Sudan 1,
51 Metanil Yellow, Acid Orange II, Lead Chromate, Yellow chalk (soapstone) powder, *Curcuma*
52 *xanthorrhoea*, *Curcuma zedoaria*, *Curcuma malabarica*, *Curcuma aromatic*, Cassava (*Manihot*
53 *esculenta*).

54

55 **Authentic Turmeric** – In the US according to the FDA, the term natural flavor or natural flavouring
56 means the essential oil, oleoresin, essence or extractive, protein hydrolysate, distillate or any
57 product of roasting, heating or enzymolysis, which contains the flavouring components derived
58 from a spice, fruit or fruit juice, edible yeast, herb, bark, bud, root, leaf, or similar plant material,
59 meat, seafood, poultry, eggs, dairy products, or fermentation products thereof, whose significant
60 function is flavouring rather than nutritional.

61

62 In the EU, a natural flavouring substance shall mean a flavouring substance obtained by
63 appropriate physical, enzymatic or microbiological processes from material of vegetable, animal
64 or microbiological origin either in the raw state or after processing for human consumption by
65 one or more of the traditional food preparation processes listed in Appendix II processes which
66 include heating, smoking, curing, maturing, drying, marinating, extraction, extrusion or a
67 combination of such processes.

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

69

70 **Economically Motivated Adulteration** – The fraudulent addition of non-authentic substances or
71 removal or replacement of authentic substances without the purchaser's knowledge for
72 economic gain of the seller.

73 **Authentic Turmeric** - The accepted Latin binomial name is *Curcuma longa* L., and the synonymous
74 name *Curcuma domestica*, belonging to the botanical family: Zingiberaceae. It goes by the
75 common names: Turmeric, common turmeric, Indian saffron, yellow ginger and the plant is native
76 to Southeast Asia, especially India. It is available in all states of India, but particularly in Tamil
77 Nadu, West Bengal, and Maharashtra. It is a tropical crop cultivated at sea level to 1,200 meters
78 above sea level and grows in light black clay loam soils and red soils under irrigated and rain-fed
79 conditions. It is also extensively cultivated in Pakistan, China, Haiti, Jamaica, Peru, Taiwan,

80 Nigeria, Bangladesh, and Thailand. Other important producers include Japan, Indonesia, Sri
81 Lanka, Burma (Myanmar), Cambodia, Malaysia, and the Philippines. It has a wide distribution as
82 a non-native species in Madagascar, Oceania. Turmeric is distinguished by the presence of the
83 orange pigment curcumin. Several other species of *Curcuma*, e.g., *C. aromatica* and *C. zedoaria*,
84 are also known to contain curcumin. Commercially, dried rhizome/root is sold either whole or in
85 powdered form. In terms of varieties, it appears there are up to 30 different varieties growing in
86 India, but only two designations are commercially significant: *Alleppey* and *Madras* turmeric,
87 both named after the places of cultivation. The *Alleppey* turmeric grows in the Thodupuzha and
88 Muvattupuzha regions of Kerala State, and this variety is predominantly imported by the United
89 States in unpolished form, where users prefer it as a spice and a food colorant. This turmeric
90 contains about 3.5-5.5% volatile oil, and 4-7% curcumin. In contrast, the *Madras*-type contains
91 only 2% of volatile oil and 2% of curcumin.

92

93 ***Non-authentic substance or adulterant*** - A food item intentionally labelled as turmeric when the
94 product developer knows that another substance or an adulterant such as those listed in the
95 applicability statement has been used to adulterate turmeric for economic gain.

96

97 ***Single Laboratory Validation*** – Demonstration by one laboratory of method performance on
98 samples described according to internationally accepted validation guidelines contained in
99 Guidance Documents such as AOAC'S Appendix D, "Guidelines for Collaborative Study
100 Procedures to Validate Characteristics of a Method of Study" the *ISO/IEC 17025:2017 Document:*
101 *"General requirements for the competence of testing and calibration laboratories"*, the *Codex*
102 *Alimentarius Committee Guidance Document CAC/GL 71- 2009 - "Guidelines for the Design and*
103 *Implementation of National Regulatory Food Safety Assurance Programme Associated with the*
104 *use of Veterinary Drugs in Food Producing Animals"* (Adopted 2009. Revision 2012, 2014); the
105 *"Harmonized ISO/IUPAC/AOAC Guidelines for Single-Laboratory Validation of Methods of*
106 *Analysis CAC/GL-49-2003 "Harmonized Guidelines For Single-Laboratory Validation Of Methods*
107 *Of Analysis"*; *"Guidelines on the use of Mass Spectrometry (MS) for Identification, Confirmation*
108 *and Quantitative analysis of Residues CAC/GL 56-2005"*; *"Establishing the Fitness for Purpose of*
109 *Mass Spectrometric methods."* and *SANTE/12682/2019. "Method Validation and Quality Control*
110 *Procedures for Pesticide Residues Analysis in Food and Feed"*- A Guidance document on analytical
111 quality control and method validation procedures for pesticide residues analysis in food and feed.

112

113 Once the method has been demonstrated to meet the minimum requirements for validation and
114 fit for purpose criteria, the method can be reviewed and considered by AOACI for classification
115 as First Action Official Method of Analysis.

116

117 ***Multi-laboratory Validation*** – Demonstration between laboratories using adulterated samples
118 created by a third-party group and supplied blindly to the participating laboratories according to

119 guidelines described in the AOAC *Appendix D*, "Guidelines for Collaborative Study Procedures to
 120 Validate Characteristics of a Method of Study" be considered for classification as AOAC Final
 121 Action Method; "*Protocol for the design, conduct and interpretation of method performance*
 122 *studies*". Pure and Applied Chemistry, Horwitz, W. 1995. 67:331-343; "*Guidelines for the*
 123 *Assessment of the Competence of Testing Laboratories Involved in the Import and Export Control*
 124 *of Food*" - CAC/GL 27-1997; "*Harmonized IUPAC Guidelines for the use of Recovery Information in*
 125 *Analytical Measurement*" - CAC/GL 37-2001; and "*Harmonised Guidelines for the Use of Recovery*
 126 *Information in Analytical Measurement*"

127

128 The Predicted (PRSD_R) of REPRODUCIBILITY is calculated from the Horwitz equation

129 $PRSD_R = 2C^{-0.15}$ Where C is expressed as a mass fraction

130

131 For Quantitative methods undergoing MLV 10 –12 laboratories must be recruited to provide at
 132 least 8 valid data sets; two blind duplicate replicates at five concentration levels for each
 133 analyte/matrix combination to each collaborator.

134

135 HorRat (Repeatability, r) = RSD_r/PRSD_R

136 HorRat (Reproducibility, R) = RSD_R/PRSD_R

137

138 For Inter-laboratory studies: acceptable HorRat (R) of 1 with limits of acceptability of 0.5 to 2;

139 For Within-Laboratory studies: acceptable HorRat (r) of 0.3 – 1.3

140

141 4. Method Performance Requirements

142

143 **Table 1: Method Performance Requirements for the Colour and non-colour adulterants in**
 144 **Turmeric Powder**

Analytical Parameter	Acceptance Criteria for color adulterants	Acceptance Criteria for non-color 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 and Yellow Chalk powder, <i>curcuma aromatica</i> , <i>curcuma malabarica</i> , <i>curcuma zedoaria</i> , <i>curcuma xanthorrhoea</i>	Non-colour adulterants include cassava, starch, glucose

145

146 **5. System Suitability Tests and/or Analytical Quality Control**

147 Suitable methods will include blanks, and appropriate check standards.

148

149 **6. Reference Materials**

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

153

154 **7. Validation Guidance**

155 a. Data demonstrating method performance is required.

156

157 b. Samples: Complete documentation for the collection and use of authentic samples must
158 be supplied by the method authors. The scope of “authentic” samples used to validate
159 the method must be applicable to the defined scope of the TT method. Expansion of the
160 scope is possible with the inclusion of additional authentic samples and abbreviated
161 validation using the protocol listed in this SMPR.

162

163 c. For single lab validation studies, the method will be evaluated using prescribed
164 adulterated materials as shown in Table 1 above. Methods approved at this level will
165 proceed to a second level of evaluation (multi-laboratory) where blinded samples
166 containing unknown adulterants will be sent to participating laboratories.

167

168 d. Statistical analysis of interlaboratory studies. Sample size needed to meet performance
169 requirement on proportion.

170

171 **8. Maximum Time-to-Results**

172 None.

173

174 **References:**

175 *CAC/GL 27-1997 - "Guidelines for the Assessment of the Competence of Testing Laboratories*
176 *Involved in the Import and Export Control of Food"*

177 http://www.fao.org/input/download/standards/355/CXG_027e.pdf

178

179 *CAC/GL 37-2001 - "Harmonized IUPAC Guidelines for the use of Recovery Information in*
180 *Analytical Measurement"*

181 http://www.fao.org/input/download/standards/376/CXG_037e.pdf

182

183 *CAC/GL-49-2003 - "Harmonized ISO/IUPAC/AOAC Guidelines for Single-Laboratory Validation*
184 *of Methods of Analysis*

185 <http://www.fao.org/fao-who-codexalimentarius/codex-texts/guidelines/en/>
186 CAC/GL 56-2005 – CXG 56 "Guidelines on the use of Mass Spectrometry (MS) for Identification,
187 Confirmation and Quantitative analysis of Residues"
188 <http://www.fao.org/fao-who-codexalimentarius/codex-texts/guidelines/en/>
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190 Codex Alimentarius Committee Guidance Document CAC/GL 71- 2009 - "Guidelines for the
191 Design and Implementation of National Regulatory Food Safety Assurance Programme
192 Associated with the use of Veterinary Drugs in Food Producing Animals" (Adopted 2009.
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196 ISO/IEC 17025:2017 Guideline Document: "General requirements for the competence of
197 testing and calibration laboratories", the Codex Alimentarius Committee Guidance Document
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215 the American Society for Mass Spectrometry 14: 528-541.
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222 337-348.
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225 mandatory certification marks scheme. Turmeric powder BDS 991:
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