AOAC SMPR® 2017.012

Standard Method Performance Requirements (SMPRs) for Quantitation of Select Nonvolatile Ginger Constituents

Intended Use: Control of Incoming Ingredients and Finished Products

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, a single-laboratory validation, or a multi-site collaborative study. SMPRs are written and adopted by AOAC stakeholder panels composed of representatives from industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC expert review panels in their evaluation of validation study data for methods being considered for *Performance Tested Methods*℠ or AOAC *Official Methods of Analysis*℠, and can be used as acceptance criteria for verification at user laboratories.

2 Applicability

The method is required to quantitate [6]-, [8]-, and [10]-gingerols and [6]-shogaol in the matrices listed in Table 1. It is desirable, but optional, for the method to quantitate [8]- and [10]-shogaols; [6]-, [8]-, and [10]-paradols; [6]- and [10]-gingerdiols; [6]-, [8]-, and [10]-gingerdiones; and zingerone.

3 Analytical Technique

Any technique that quantitates the analytes defined in the Applicability statement and satisfies the method performance requirements set forth in this SMPR.

4 Definitions

Analytes.—Refer to Table 2 for the list of analytes, their chemical attributes, and identifiers. Refer to Figure 1 for the chemical structures.

Dietary ingredient.—A vitamin; a mineral; an herb or other botanical; an amino acid; a dietary substance for use by man to supplement the diet by increasing total dietary intake; or a concentrate, metabolite, constituent, extract, or combination of any of the above dietary ingredients [Federal Food Drug and Cosmetic Act §201(ff) [U.S.C. 321 (ff)]. Dietary ingredients are conventionally presented as powders or liquids.

Dietary supplement.—A product containing a dietary ingredient intended for ingestion to supplement the diet. Dietary supplements containing dietary ingredients are commonly marketed as tablets, capsules, softgels, tinctures, or other finished dosage forms.

Limit of quantitation (LOQ).—The minimum content of analyte in a given matrix that can be reliably and precisely quantitated in agreement with the requirements set forth in this SMPR.

Repeatability.—Statistical variation in the analytical outcome arising when the maximum control over the analytical methodology is afforded. Replicate analyses are performed by the same operator within a short time period using the same instrumentation. Expressed as the repeatability standard deviation (SD_r) or % repeatability relative standard deviation (%RSD_r).

Reproducibility.—Statistical variation in the analytical outcome influenced by typical laboratory variables. Replicate analyses are conducted on different days by different operators using different sets of equipment, occasionally in different physical locations. Expressed as the reproducibility standard deviation (SD_R) or % reproducibility relative standard deviation (%RSD_R).

Recovery.—The relative percentage of the spiked analyte recovered from a given matrix following implementation of the complete analytical procedure.

Table 1. Matrices

Rhizome powder
Rhizome dry extract
Tablets or capsules containing dry extract and rhizome powder
Optional:
Rhizome soft extract
Tincture
Softgel capsules

5 Method Performance Requirements

See Table 3.

6 System Suitability Tests and/or Analytical Quality Control

Appropriate technique-specific system suitability criteria will be specified to demonstrate adequate method performance with respect to the claimed analytes.

7 Reference Material(s)

NIST SRM 3398: Ginger (*Zingiber officinale*) Rhizome
NIST SRM 3399: Ginger (*Zingiber officinale*) Extract
USP Item No. 1291504: Powdered Ginger
USP Item No. 1291446: Ginger Constituent Mixture

NIST may be able to offer additional reference materials. Contact Catherine Rimmer, Research Chemist, NIST, at email: catherine.rimmer@nist.gov or Tel: (301) 975-3651.

See Table 4 for a list of commercial sources of ginger constituents.

Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (20th Ed.), AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_f.pdf)

8 Validation Guidance

Each required analyte and each claimed optional analyte should be evaluated in all claimed matrices. For each matrix evaluated, an explicit list of analytes to which validation is applicable should be provided.

Appendix D: *Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis*, *Official Methods*

Table 2. Analytes with chemical attributes and identifiers

Compound	IUPAC name	Formula	CAS No.	UNII code	InChi key	PubChem
(5*S*)-[6]-Gingerol	**(*S*)-5-hydroxy-1-(4-hydroxy-3-methoxyphenyl)decan-3-one**	$C_{17}H_{26}O_4$	23513-14-6	925QK2Z900	NLDDIKRKFXEWBK-AWEZNQCLSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/442793
(5*R*)-[6]-Gingerol	(*R*)-5-hydroxy-1-(4-hydroxy-3-methoxyphenyl)decan-3-one	$C_{17}H_{26}O_4$	72749-01-0		NLDDIKRKFXEWBK-CQSZACIVSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/12310197
(5*S*)-[8]-Gingerol	**(*S*)-5-hydroxy-1-(4-hydroxy-3-methoxyphenyl)dodecan-3-one**	$C_{19}H_{30}O_4$	23513-08-8	LB0IJB138K	BCIWKKMTBRYQJU-INIZCTEOSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/168114
(5*R*)-[8]-Gingerol	(*R*)-5-hydroxy-1-(4-hydroxy-3-methoxyphenyl)dodecan-3-one	$C_{19}H_{30}O_4$	135272-33-2		BCIWKKMTBRYQJU-MRXNPFEDSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/11023711
(5*S*)-[10]-Gingerol	**(*S*)-5-hydroxy-1-(4-hydroxy-3-methoxyphenyl)tetradecan-3-one**	$C_{21}H_{34}O_4$	23513-15-7	ND6ZLI4J0V	AIULWNKTYPZYAN-SFHVURJKSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/168115
[6]-Shogaol	(*E*)-1-(4-hydroxy-3-methoxyphenyl)dec-4-en-3-one	$C_{17}H_{24}O_3$	555-66-8	83DNB5FIRF	OQWKEEOHDMUXEO-BQYQJAHWSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/5281794
[8]-Shogaol	(*E*)-1-(4-hydroxy-3-methoxyphenyl)dodec-4-en-3-one	$C_{19}H_{28}O_3$	36700-45-5	AV4IK2HCNT	LGZSMXJRMTYABD-MDZDMXLPSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/6442560
[10]-Shogaol	(*E*)-1-(4-hydroxy-3-methoxyphenyl)tetradec-4-en-3-one	$C_{21}H_{32}O_3$	36752-54-2	UP39BHE708	FADFGCOCHHNRHF-VAWYXSNFSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/6442612
Zingerone ([0]-Paradol)	4-(4-Hydroxy-3-methoxyphenyl)butan-2-one	$C_{11}H_{14}O_3$	122-48-5	4MMW850892	OJYLAHXKWMRDGS-UHFFFAOYSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/31211
[6]-Paradol	1-(4-Hydroxy-3-methoxyphenyl)decan-3-one	$C_{17}H_{26}O_3$	27113-22-0	BO24ID7E9U	CZNLTCTYLMYLHL-UHFFFAOYSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/94378
[8]-Paradol	1-(4-Hydroxy-3-methoxyphenyl)dodecan-3-one	$C_{19}H_{30}O_3$	27113-23-1		TYQRTQZWHUXDLG-UHFFFAOYSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/213821
[10]-Paradol	1-(4-Hydroxy-3-methoxyphenyl)tetradecan-3-one	$C_{21}H_{34}O_3$	36700-48-8		XNBUKRQGYHYOOP-UHFFFAOYSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/51352076
[6]-Gingerdione	1-(4-Hydroxy-3-methoxyphenyl)decane-3,5-dione	$C_{17}H_{24}O_4$	61871-71-4	L2L6JCL6YY	KMNVXQHNIWUUSE-UHFFFAOYSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/162952
[8]-Gingerdione	1-(4-Hydroxy-3-methoxyphenyl)dodecane-3,5-dione	$C_{19}H_{28}O_4$	77334-06-6	70E1Y63Q2L	QDSRAFNZQKMHPZ-UHFFFAOYSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/14440537
[10]-Gingerdione	1-(4-Hydroxy-3-methoxyphenyl)tetradecane-3,5-dione	$C_{21}H_{32}O_4$	79067-90-6		QPSYZJDGMPQMSV-UHFFFAOYSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/14440539
(3*R*,5*S*)-[6]-Gingerdiol	**(+)-(3*R*,5*S*)-1-(4-hydroxy-3-methoxyphenyl)decane-3,5-diol**	$C_{17}H_{28}O_4$	154905-69-8	4C9F8U79BX	QYXKQNMJTHPKBP-LSDHHAIUSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/11369949
(3*S*,5*R*)-[6]-Gingerdiol	(-)-(3*S*,5*R*)-1-(4-hydroxy-3-methoxyphenyl)decane-3,5-diol	$C_{17}H_{28}O_4$	53318-09-5			
(3*S*,5*S*)-[6]-Gingerdiol	(3*S*,5*S*)-1-(4-hydroxy-3-methoxyphenyl)decane-3,5-diol	$C_{17}H_{28}O_4$	143615-76-3		QYXKQNMJTHPKBP-GJZGRUSLSA-N	https://pubchem.ncbi.nlm.nih.gov/compound/15839040
(3*R*,5*S*)-[8]-Gingerdiol	**(3*R*,5*S*)-1-(4-hydroxy-3-methoxyphenyl)dodecane-3,5-diol**	$C_{19}H_{32}O_4$	53254-76-5		RLBBNYBPCMIQMG-DLBZAZTESA-N	https://pubchem.ncbi.nlm.nih.gov/compound/101941698
(3*R*,5*S*)-[10]-Gingerdiol	**(3*R*,5*S*)-1-(4-hydroxy-3-methoxyphenyl)tetradecane-3,5-diol**	$C_{21}H_{36}O_4$	53254-77-6		LGSIUDXMEDKEPY-RBUKOAKNSA-N	
(3*S*,5*R*)-[10]-Gingerdiol	(3*S*,5*R*)-1-(4-hydroxy-3-methoxyphenyl)tetradecane-3,5-diol	$C_{21}H_{36}O_4$	1339934-29-0		LGSIUDXMEDKEPY-QINVSXPYNA-N	
(3*S*,5*S*)-[10]-Gingerdiol	(3*S*,5*S*)-1-(4-hydroxy-3-methoxyphenyl)tetradecane3,5-diol	$C_{21}H_{36}O_4$	1438241-35-0		LGSIUDXMEDKEPY-OALUTQOASA-N	https://pubchem.ncbi.nlm.nih.gov/compound/101572265

Note: Naturally prevalent stereoisomers are shown in bold: (5*S*) configuration for gingerols, (3*R*,5*S*) configuration for gingerdiols.

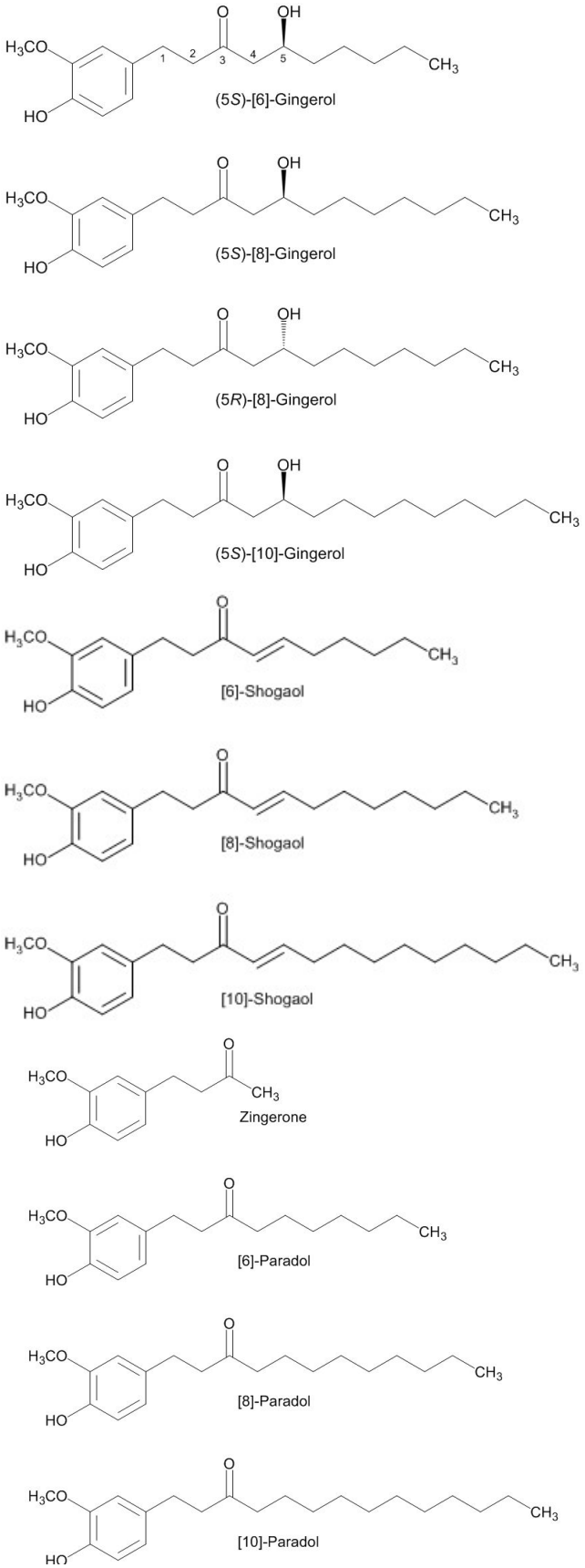

Figure 1. Chemical structures of gingerols, shogaols, paradols, zingerone, gingerdiones, and gingerdiols.

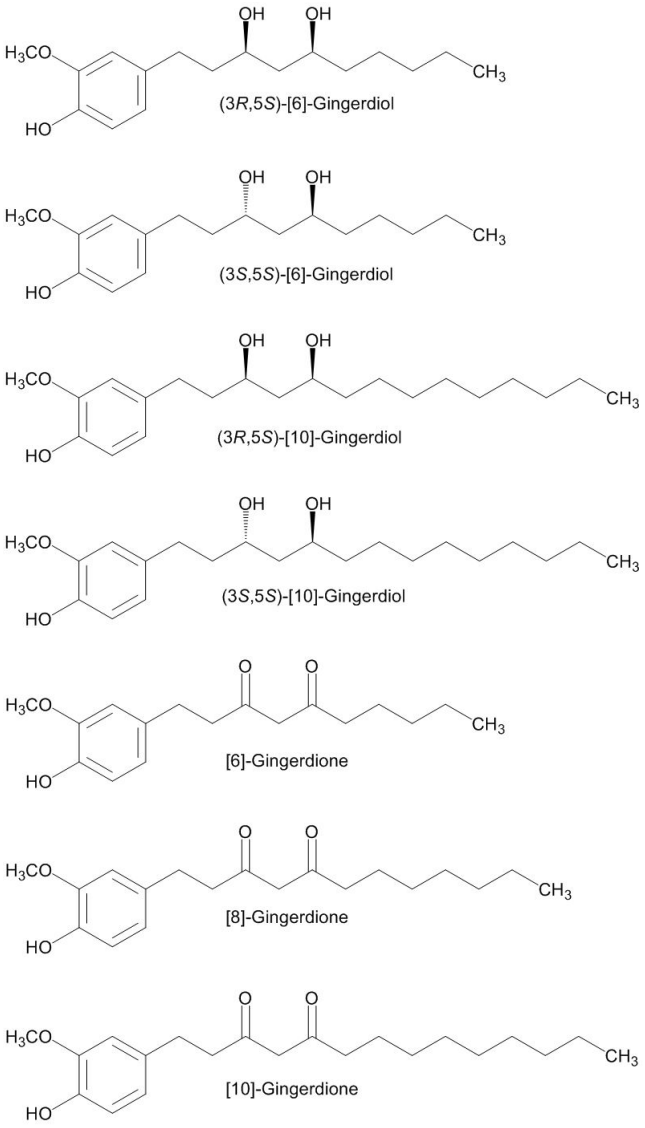

Figure 1. (*continued*)

Table 3. Method performance requirements

Parameter	Requirement, %
Analytical range	0.05–50
Limit of quantitation (LOQ)	0.05
Recovery	90–107
RSD_r	≤5
RSD_R	≤8

Table 4. Commercial sources of ginger constituents

	Gingerols			Shogaols			Paradols			
	[6]-	[8]-	[10]-	[6]-	[8]-	[10]-	[6]-	[8]-	[10]-	Zingerone
Chengdu Biopurify	a	b	c	d	e	f				g
Chromadex	h	i	j	k	l	m				
Dalton Research	n	o	p	q			r	s	t	
Extrasynthese	u	v		w						
Phytolab	x	y	z	aa	bb	cc				
Sigma-Aldrich	dd	ee	ff	gg		hh				ii
Tokiwa	jj	kk	ll	mm						

a http://www.phytopurify.com/6Gingerol-p-282.html

b http://www.phytopurify.com/8Gingerol-p-4051.html

c http://www.phytopurify.com/10Gingerol-p-4027.html

d http://www.phytopurify.com/6Shogaol-p-4048.html

e http://www.phytopurify.com/8Shogaol-p-5749.html

f http://www.phytopurify.com/10Shogaol-p-5750.html

g http://www.phytopurify.com/Zingerone-p-4784.html

h https://www.chromadex.com/product-detail?ItemDetailNo=ASB-00007164-010

i https://www.chromadex.com/product-detail?ItemDetailNo=ASB-00007163-010

j https://www.chromadex.com/product-detail?ItemDetailNo=ASB-00007162-025

k https://www.chromadex.com/product-detail?ItemDetailNo=ASB-00019211-025

l https://www.chromadex.com/product-detail?ItemDetailNo=ASB-00019212-025

m https://www.chromadex.com/product-detail?ItemDetailNo=ASB-00019214-025

n https://www.daltonresearchmolecules.com/merchant/product/23513-14-6a

o https://www.daltonresearchmolecules.com/merchant/product/23513-08-8

p https://www.daltonresearchmolecules.com/merchant/product/23513-15-7

q https://www.daltonresearchmolecules.com/merchant/product/555-66-8

r https://www.daltonresearchmolecules.com/merchant/product/27113-22-0

s https://www.daltonresearchmolecules.com/merchant/product/27113-23-1

t https://www.daltonresearchmolecules.com/merchant/product/36700-48-8

u http://www.extrasynthese.com/6-gingerol.html

v http://www.extrasynthese.com/8-gingerol.html

w http://www.extrasynthese.com/shogaol.html

x http://phyproof.phytolab.de/6-gingerol.html

y http://phyproof.phytolab.de/8-gingerol.html

z http://phyproof.phytolab.de/10-gingerol.html

aa http://phyproof.phytolab.de/6-shogaol.html

bb http://phyproof.phytolab.de/8-shogaol.html

cc http://phyproof.phytolab.de/10-shogaol.html

dd http://www.sigmaaldrich.com/catalog/product/sial/50866

ee http://www.sigmaaldrich.com/catalog/product/sial/01514

ff http://www.sigmaaldrich.com/catalog/product/sial/42630

gg http://www.sigmaaldrich.com/catalog/product/sial/39303

hh http://www.sigmaaldrich.com/catalog/product/sial/91287

Table 4. (*continued*)

	Gingerols			Shogaols			Paradols			
	[6]-	[8]-	[10]-	[6]-	[8]-	[10]-	[6]-	[8]-	[10]-	Zingerone

[ii] https://www.chromadex.com/product-detail?ItemDetailNo=ASB-00026600-250

[jj] http://www.tokiwaph.co.jp/en/product/2012/06/6gingerol.html

[kk] http://www.tokiwaph.co.jp/en/product/2012/06/8gingerol.html

[ll] http://www.tokiwaph.co.jp/en/product/2012/06/10gingerol.html

[mm] http://www.tokiwaph.co.jp/en/product/2012/06/6shogaol.html

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Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (20th Ed.), AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_f.pdf)

Appendix K: *Guidelines for Dietary Supplements and Botanicals*, *Official Methods of Analysis of AOAC INTERNATIONAL* (20th Ed.), AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_k.pdf)

9 Maximum Time-to-Result

None.

Approved by the AOAC Stakeholder Panel on Dietary Supplements (SPDS). Final Version Date: April 3, 2017. Effective Date: April 3, 2017.