

USP Monographs in a Small Contract Laboratory: Case Studies

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Turmeric/Curcuminoids Dietary Supplements

Supplement Facts

Serving Size - 1 Softgel
Servings Per Container - 60

	Amount per Serving	% Daily Value
Curcumin Extract (as 95% curcuminoids)	250 mg	†

† Daily Value not established.

Other Ingredients: Gelatin, Extra Virgin Olive Oil, Medium Chain Triglycerides, Glycerin, Purified Water, Yellow Beeswax, Soy Lecithin.

Suggested Use: As a dietary supplement, take 1 – 2 softgels daily. Or use as directed by your healthcare professional.

***These statements have not been evaluated by the Food and Drug Administration. This product is not intended to diagnose, treat, cure, or prevent any disease.**

Nutrition Facts

Serving Size 3 Capsules
Servings Per Container 30

Amount Per Serving

	Serving	% DV
Glucosamine Sulfate	1500mg	-
White Willow Bark Extract	250mg	-
MSM (Methylsulfonylmethane)	500mg	-
Hyaluronic Acid	4mg	-
Cayenne 40m H.U.	50mg	-
Ginger Root Extract 4:1	250mg	-
Boswellia Serrata Extract	125mg	-
Turmeric Extract 25:1	50mg	-

Proprietary Blends

Boswellia Serrata Extract

standardized to 65% boswellic acid


Turmeric Extract 25:1

standardized to 95% curcuminoids

*percent Daily Values are based on a
2,000 calorie diet.

Ingredients: Rice Flour, Gelatin,
Vegetable Magnesium Stearate And Silicon
Dioxide. CONTAINS: Crustacean Shellfish
(Shrimp, Crab).

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USP-NF

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ICON KEY

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ORIGIN OF CHANGE KEY

- ✘ Errata

Results for "curcumin extract" (6 results)

Document Results (Scroll for more results)

EXPORT TO EXCEL

Powdered Turmeric Extract
✔ *Monographs Official as of 1-May-2020*
 Powdered Turmeric **Extract**. Powdered Turmeric **Extract** is prepared from the pulverized rhizomes

Curcuminoids
✔ *Monographs Official as of 1-May-2020*
 of **curcumin**, desmethoxycurcumin, and bisdesmethoxycurcumin in the portion of Curcuminoids taken:
 Result = (r U)

Powdered Turmeric
✔ *Monographs Official as of 1-May-2017*
 of Curcuminoids.. Analysis Samples: Standard solution B and Sample solution Calculate the percentages of **curcumin**

Turmeric
✔ *Monographs Official as of 1-May-2017*
 of Curcuminoids.. Analysis Samples: Standard solution B and Sample solution Calculate the percentages of **curcumin**

Powdered Turmeric Extract
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Curcuminoids

DEFINITION

Curcuminoids is a partially purified natural complex of diaryl heptanoid derivatives isolated from Turmeric, *Curcuma longa* L. It contains NLT 95.0% of curcuminoids, calculated on the dried basis, as the sum of curcumin, desmethoxycurcumin, and bisdesmethoxycurcumin. It contains NLT 70.0% and NMT 80.0% of curcumin, NLT 15.0% and NMT 25.0% of desmethoxycurcumin, and NLT 2.5% and NMT 6.5% of bisdesmethoxycurcumin.



Sample solution under stated identification condition.

B. HPLC

Analysis: Proceed as directed in the test for *Content of Curcuminoids*.

Acceptance criteria: The retention times of the peaks for curcumin, desmethoxycurcumin, and bisdesmethoxycurcumin of the *Sample solution* correspond to those of *Standard solution A* and *Standard solution B*.

COMPOSITION

CONTENT OF CURCUMINOIDS

Mobile phase: [Tetrahydrofuran](#) and 1 mg/mL of [citric acid](#) in water (4:6)

Standard solution A: 40 µg/mL of [USP Curcuminoids RS](#) in *Mobile phase*

Standard solution B: A composite solution containing 40 µg/mL of [USP Curcumin RS](#), 10 µg/mL of [USP Desmethoxycurcumin RS](#), and 2.0 µg/mL of [USP Bisdesmethoxycurcumin RS](#) in *Mobile phase*. Use sonication if necessary. Before injection, pass through a filter of 0.45-µm pore size, and discard the initial 10 mL of the filtrate.

Sample stock solution: Transfer about 20 mg of Curcuminoids, accurately weighed, to a 50-mL volumetric flask, add 30 mL of [acetone](#), and sonicate for 30 min. Dilute with [acetone](#) to volume, mix, and centrifuge.

Sample solution: Transfer 5.0 mL of the *Sample stock solution* to a 50-mL volumetric flask. Dilute with *Mobile phase* to volume, and mix. Before injection, pass through a filter of 0.45-µm pore size, and discard the initial 10 mL of the filtrate.

Chromatographic system

(See [Chromatography \(621\) System Suitability](#).)

Mode: LC

Detector: Vis 420 nm

Column: 4.6-mm × 25-cm; 5-µm packing L1

Flow rate: 1.0 mL/min

Injection volume: 20 µL

System suitability

Samples: *Standard solution A* and *Standard solution B*

[NOTE—The relative retention times for the curcumin, desmethoxycurcumin, and bisdesmethoxycurcumin peaks are 1.0, 1.2, and 1.4, respectively.]

Suitability requirements


Chromatogram similarity: The chromatogram of *Standard solution A* is similar to the reference chromatogram provided with [USP Curcuminoids RS](#).

Resolution: NLT 2.0 between curcumin and desmethoxycurcumin peaks and desmethoxycurcumin and bisdesmethoxycurcumin peaks, *Standard solution B*

Tailing factor: NMT 1.5 for bisdesmethoxycurcumin, desmethoxycurcumin, and curcumin peaks, *Standard solution B*

Relative standard deviation: NMT 2.0% for the desmethoxycurcumin peak, in replicate injections, *Standard solution B*

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EXPORT TO EXCEL

(1094) Capsules—Dissolution Testing and Related Quality Attributes

General Chapters Official as of 1-Dec-2020
 , soft-shell capsules and hard-shell capsules are referred to as **softgels** and hardgels, respectively. **Softgels**

(1094) Capsules—Dissolution Testing and Related Quality Attributes

General Chapters Official 1-Aug-2014 to 30-Nov-2020
 to as **softgels** and hardgels, respectively. **Softgels** have a thicker shell and typically exhibit a higher degree

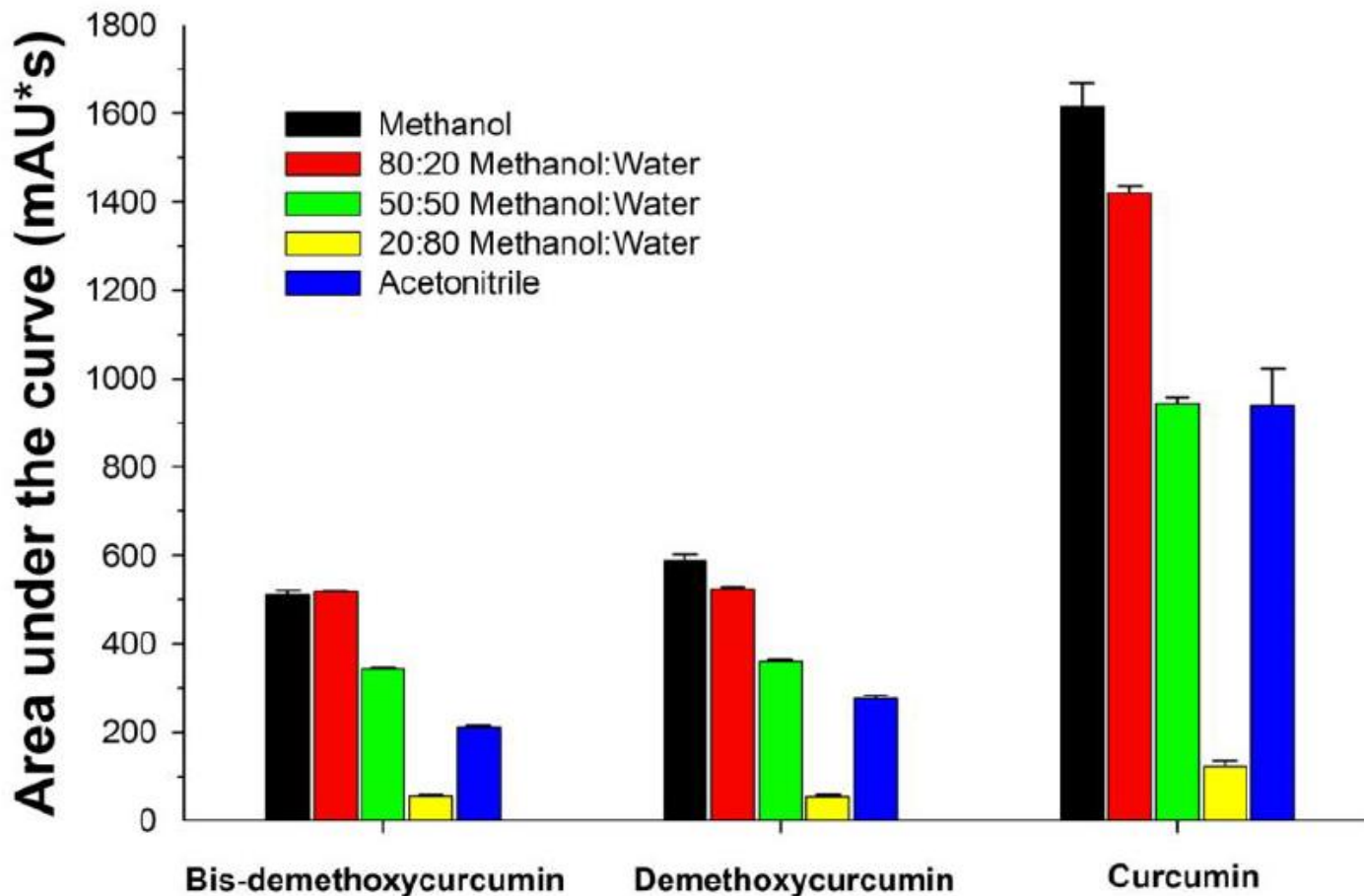
(2040) Disintegration and Dissolution of Dietary Supplements

General Chapters Official as of 1-Aug-2018
 capsules: Place 1 **softgel** capsule in each of the six tubes of the basket. Omit the use of a disk. Operate

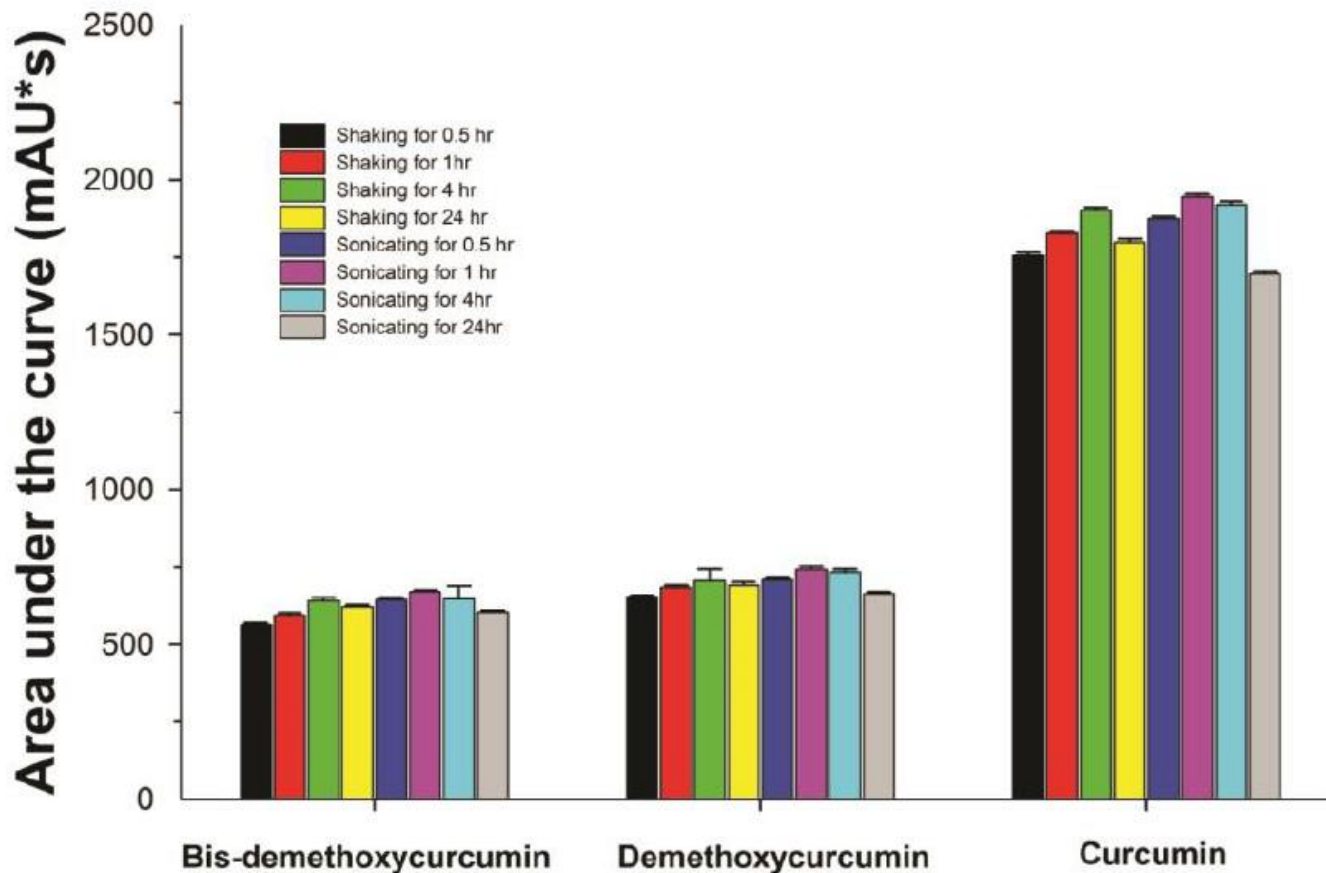
<2040> Disintegration and dissolution of dietary supplements

Delayed-release (enteric-coated) soft shell capsules: Place 1 softgel capsule in each of the six tubes of the basket. Omit the use of a disk. Operate the apparatus using simulated gastric fluid TS, maintained at $37 \pm 2^\circ$, as the immersion fluid. After 1 h of operation in simulated gastric fluid TS, lift the basket from the fluid and observe the softgels: the softgels show no evidence of disintegration or rupture that would permit the escape of the contents. Operate the apparatus with disks using simulated intestinal fluid TS, maintained at $37 \pm 2^\circ$, as the immersion fluid for NMT 60 min. Lift the basket from the fluid and observe the capsules.

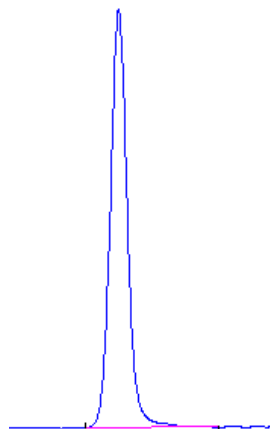
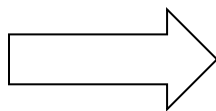
Optimization of the Extraction Method – Extraction solvent



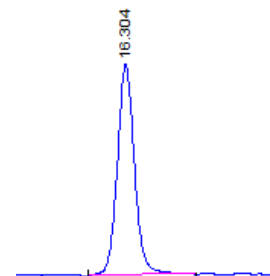
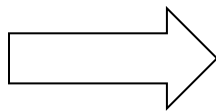
Extraction time and method



Optimization of the Extraction Method – Extraction Efficiency

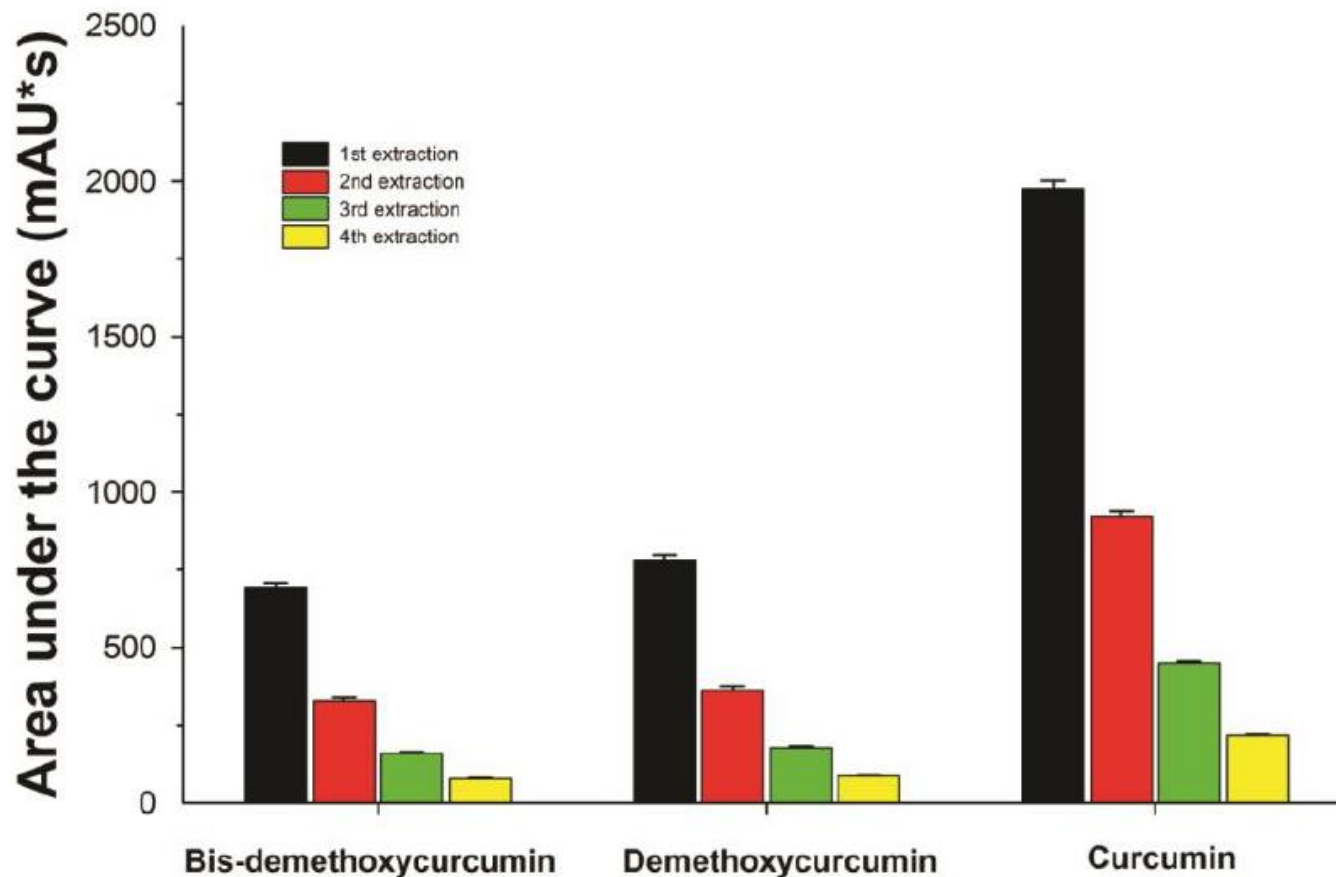


AUC: 88.1



AUC: 42.9

Optimization of the Extraction Method – Extraction Efficiency



USP method

In-house availability

Chromatographic system

(See [Chromatography \(621\), System Suitability.](#))

Mode: LC

Detector: Vis 420 nm

Column: 4.6-mm x 25-cm; 5- μ m packing L1

Flow rate: 1.0 mL/min

Injection volume: 20 μ L

Phenomenex

Kinetex Biphenyl

4.6 x 150 cm, 2.6 μ m

Method Translator - Agilent

Agilent Technologies Method Translator and Cost Savings Calculator

Basic Mode **Advanced Mode** Viscosity Table Cost Savings Calculator Agilent 1290 Infinity II LC

Original Method

System Info
Conventional LC
[Reset Advanced Mode settings](#)

Column Info
Column ID (mm) 4.6
Column length (mm) 250
Particle Size (µm) 5

Method info
Flow Rate (mL/min) 1
Injection Vol. (µL) 20
Pressure (bar) 133

Solvent Water / Methanol
Temperature (°C) 30
Max. Solvent Visc. (cP) 1.47

	Time	%B	Flow
Initial:	0.00	40	1.00
Initial Hold:			1.00
Gradient:			1.00
Hold to:			1.00
Return by:			1.00
End of Run:	60	40	1.00

Alerts!
Original method k* (retention factor) too high

New Method

System Info
Agilent 1290 Infinity II LC
[Reset Advanced Mode settings](#)

Column Info
Column ID (mm) 4.6
Column length (mm) 150
Particle Size (µm) 2.7

Method info
Flow Rate (mL/min) 1.00
Injection Vol. (µL) 4.0
Pressure (bar) 205
Detector Settings 0

	Time	%B	Flow
Initial:	0.00	40	1.00
Initial Hold:	0.00		1.00
Gradient:	0.00		1.00
Hold to:	0.00		1.00
Return by:	0.00		1.00
End of Run:	12.00	40	1.00

Time Saving Factor

5.0

Solvent Saving

80%

Simple Conversion
 Speed Optimized
 Resolution Optimized

GAAS Analytical Method Translator - ThermoFisher



Reset All



LC Method Transfer Calculator – an online tool to transfer methods from HPLC to UHPLC conditions

Use at your own risk. Prepopulated data are estimates only. Thermo Fisher Scientific takes no responsibility for the accuracy of data, calculations or results.

Current Column

Length (mm):

Diameter (mm):

Particle Size (μm):

Peak Details (Critical Pair)

Actual R_s (Resolution Factor):

Planned Column

Length (mm):

Diameter (mm):

Particle Size (μm):

Peak Details (Critical Pair)

Predicted R_s Change Factor: (7.4%)

Predicted R_s : Baseline resolution achieved



Method Translator - ThermoFisher

Current Method Conditions

Flow (mL/min):

Injection Volume (µL):

Max Observed Pressure:

Pressure Units: ▼

Number of samples:

Data Collection Rate (Hz):

Consider Gradient Delay Volume (GDV)

Current Gradient Table

Step	Time (min)	%A	%B	%C	%D
1	<input type="text" value="0.00"/>	<input type="text" value="60.0"/>	<input type="text" value="40.0"/>	<input type="text" value="0.0"/>	<input type="text" value="0.0"/>
2	<input type="text" value="60.00"/>	<input type="text" value="60.0"/>	<input type="text" value="40.0"/>	<input type="text" value="0.0"/>	<input type="text" value="0.0"/>

Recommended Method Conditions

Boost Factor: ▼

* Use this factor to increase the flow rate of the fast LC method. Note: if factor other than 1 is used, the resolution calculation is disabled.

Adjust Flow

Flow (mL/min):

Injection Volume (µL):

Estimated Max Observed Pressure:

Number of samples:

Data Collection Rate (Hz):

Planned Gradient Table

Step	Time (min)	%A	%B	%C	%D
1	<input type="text" value="0.00"/>	<input type="text" value="60.0"/>	<input type="text" value="40.0"/>	<input type="text" value="0.0"/>	<input type="text" value="0.0"/>
2	<input type="text" value="18.72"/>	<input type="text" value="60.0"/>	<input type="text" value="40.0"/>	<input type="text" value="0.0"/>	<input type="text" value="0.0"/>

USP Reference Standard (Curcuminoids)



LABEL TEXT

USP REFERENCE STANDARD
CURCUMINOIDS 300 mg


Labeled for qualitative use. Keep container tightly closed. Protect from light. Store in a freezer.

See certificate for any additional information.
USP, 12601 Twinbrook Pkwy, Rockville, MD, +1-301-881-0666
Cat. No. 1151866 Material mfd. in India

Jeri L. Ioth
Quality Assurance

For use with validated USP-compendial tests. Use prior to use at www.usp.org/usp.

LOT: R094N0

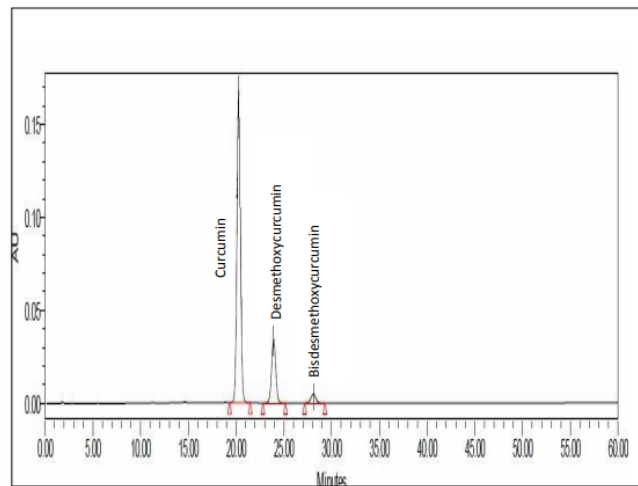




Typical Chromatogram

USP Curcuminoids RS

Catalog Number: 1151866
Lot: R094N0
Monograph: Curcuminoids
Publication: USP41/NF36
Test: Content of Curcuminoids
Sample: Standard Solution A



USP Reference Standard (Curcumin)

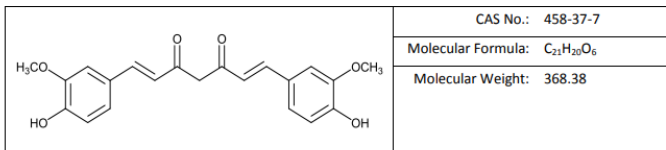


Certificate

CURCUMIN

((1E,6E)-1,7-Bis(4-hydroxy-3-methoxyphenyl)hepta-1,6-diene-3,5-dione)

USP Catalog No.: 1151855
USP Lot No.: R080W0



LABEL TEXT

USP REFERENCE STANDARD

CURCUMIN 30 mg

For quantitative applications, use a value of 1.00 mg of curcumin per mg of material on the as is basis. Keep container tightly closed. Protect from light. Store in a freezer.

USP, 12601 Twinbrook Pkwy, Rockville, MD, +1-301-881-0666
Cat. No. 1151855 Material mfd. in India

LOT:R080W0



Juri L. Ioth

Quality Assurance

Demethoxy-curcumin and bis-demethoxy-curcumin reference standards are also available.



Case Study #2 – Quantitation of Curcuminoids and Gingerols

Nutrition Facts

Serving Size 3 Capsules
Servings Per Container 30

Amount Per Serving

	Serving	% DV
Glucosamine Sulfate	1500mg	-
White Willow Bark Extract	250mg	-
MSM (Methylsulfonylmethane)	500mg	-
Hyaluronic Acid	4mg	-
Cayenne 40m H.U.	50mg	-
Ginger Root Extract 4:1	250mg	-
Boswellia Serrata Extract	125mg	-
Turmeric Extract 25:1	50mg	-

Proprietary Blends

Boswellia Serrata Extract

standardized to 65% boswellic acid

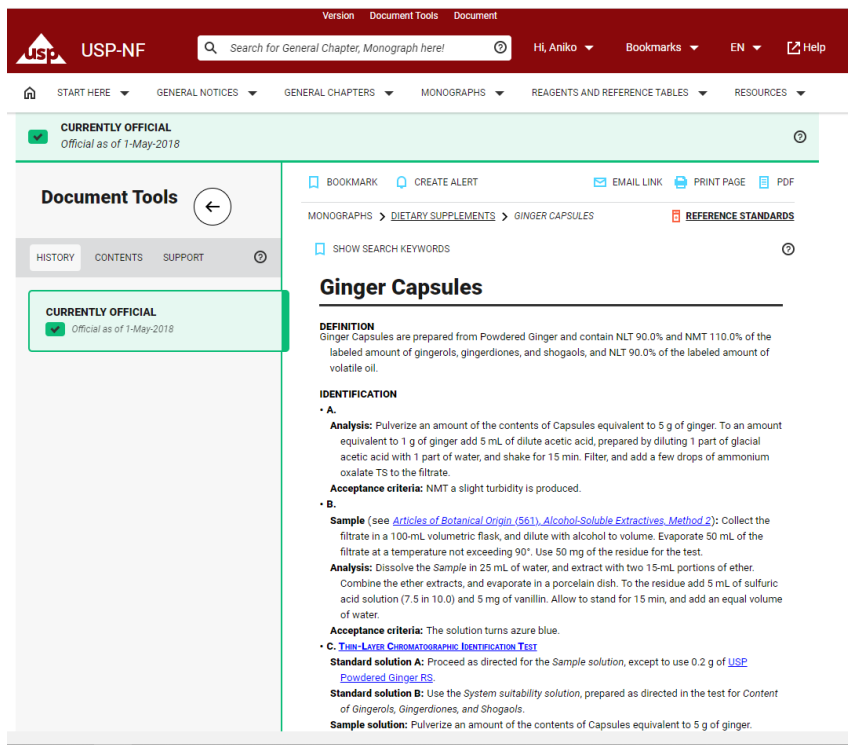
Turmeric Extract 25:1

standardized to 95% curcuminids

*percent Daily Values are based on a
2,000 calorie diet.

Ingredients: Rice Flour, Gelatin,
Vegetable Magnesium Stearate And Silicon
Dioxide. **CONTAINS:** Crustacean Shellfish
(Shrimp, Crab).

USP Monograph Ginger Capsules



USP-NF Search for General Chapter, Monograph here!

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Ginger Capsules

DEFINITION
Ginger Capsules are prepared from Powdered Ginger and contain NLT 90.0% and NMT 110.0% of the labeled amount of gingerols, gingerdiones, and shogaols, and NLT 90.0% of the labeled amount of volatile oil.

IDENTIFICATION

- A.**

Analysis: Pulverize an amount of the contents of Capsules equivalent to 5 g of ginger. To an amount equivalent to 1 g of ginger add 5 mL of dilute acetic acid, prepared by diluting 1 part of glacial acetic acid with 1 part of water, and shake for 15 min. Filter, and add a few drops of ammonium oxalate TS to the filtrate.

Acceptance criteria: NMT a slight turbidity is produced.
- B.**

Sample (see [Articles of Botanical Origin \(661\)](#), [Alcohol-Soluble Extractives, Method 2](#)): Collect the filtrate in a 100-mL volumetric flask, and dilute with alcohol to volume. Evaporate 50 mL of the filtrate at a temperature not exceeding 90°. Use 50 mg of the residue for the test.

Analysis: Dissolve the Sample in 25 mL of water, and extract with two 15-mL portions of ether. Combine the ether extracts, and evaporate in a porcelain dish. To the residue add 5 mL of sulfuric acid solution (7.5 in 10.0) and 5 mg of vanillin. Allow to stand for 15 min, and add an equal volume of water.

Acceptance criteria: The solution turns azure blue.
- C. Thin-Layer Chromatographic Identification Test**

Standard solution A: Proceed as directed for the Sample solution, except to use 0.2 g of [USP Powdered Ginger RS](#).

Standard solution B: Use the System suitability solution, prepared as directed in the test for Content of Gingerols, Gingerdiones, and Shogaols.

Sample solution: Pulverize an amount of the contents of Capsules equivalent to 5 g of ginger.

STRENGTH

• CONTENT OF GINGEROLS, GINGERDIONES, AND SHOGAOLS

Solution A: Acetonitrile, dilute phosphoric acid (1 in 1000), and methanol (55:44:1)

Solution B: Acetonitrile

Mobile phase: Use *Solution A* for NLT seven times the retention time of capsaicin.

Column washing: After each chromatographic run, wash the column, using [Table 1](#).

Table 1

Time (min)	Solution A (%)	Solution B (%)
0	100	0
2	0	100
12	0	100
14	100	0
29	100	0

Standard solution: 0.1 mg/mL of [USP Capsaicin RS](#) in methanol

System suitability solution: Reconstitute the content of 1 vial of [USP Ginger Constituent Mixture RS](#) in 1 mL of the *Standard solution*.

Sample solution: Mix and finely powder the contents of NLT 20 Capsules, and transfer an amount equivalent to 2.0 g of powdered ginger to a glass-stoppered conical flask. Add 50 mL of alcohol, insert a stopper into the flask, and macerate for 24 h, shaking frequently during the first 8 h, and then allowing to stand for 16 h. Filter, and use the filtrate.

Chromatographic system

(See [Chromatography \(621\)](#), [System Suitability](#).)

Mode: LC

Detector: UV 282 nm

Column: 4.6-mm × 25-cm; packing L1

Flow rate: 1 mL/min

Injection size: 25 µL

System suitability

Samples: *Standard solution* and *System suitability solution*

[Note—The relative retention times for 6-gingerol, capsaicin, and 6-shogaol are about 0.8, 1.0, and 1.9, respectively, *System suitability solution*.]

Suitability requirements

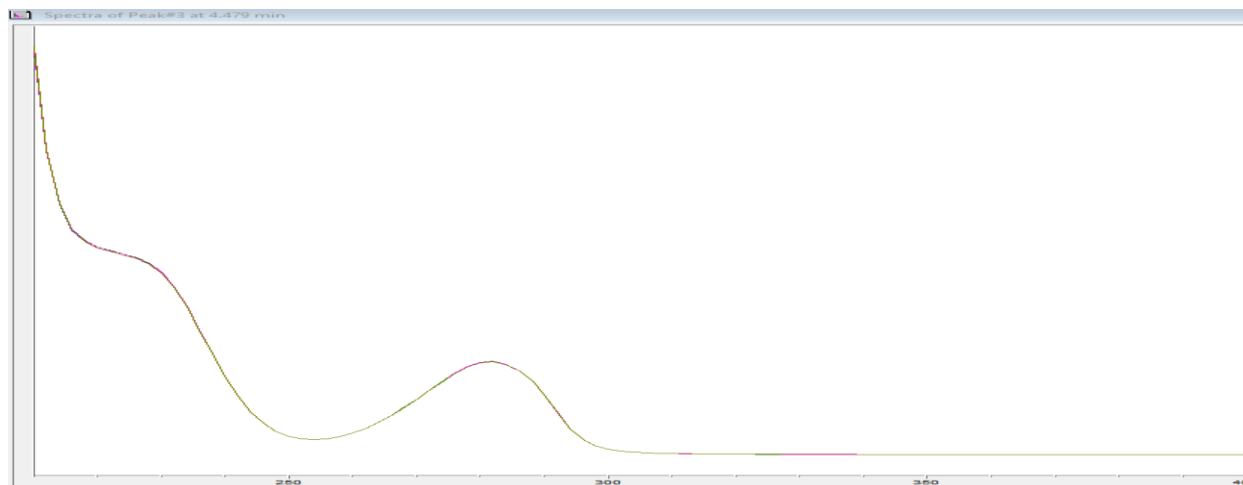
Resolution: NLT 3.0 between the 6-gingerol and capsaicin peaks and NLT 10.0 between the capsaicin and 6-shogaol peaks, *System suitability solution*

Tailing factors: NMT 2.0 for the 6-gingerol, capsaicin, and 6-shogaol peaks, *System suitability solution*

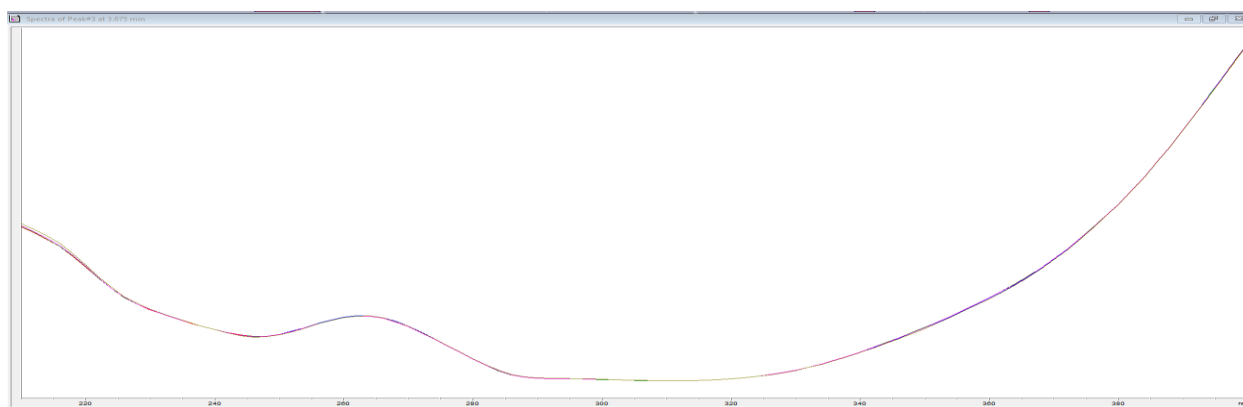
Relative standard deviation: NMT 2.5% for the capsaicin peak for replicate injections, *Standard*

Wavelength Selection

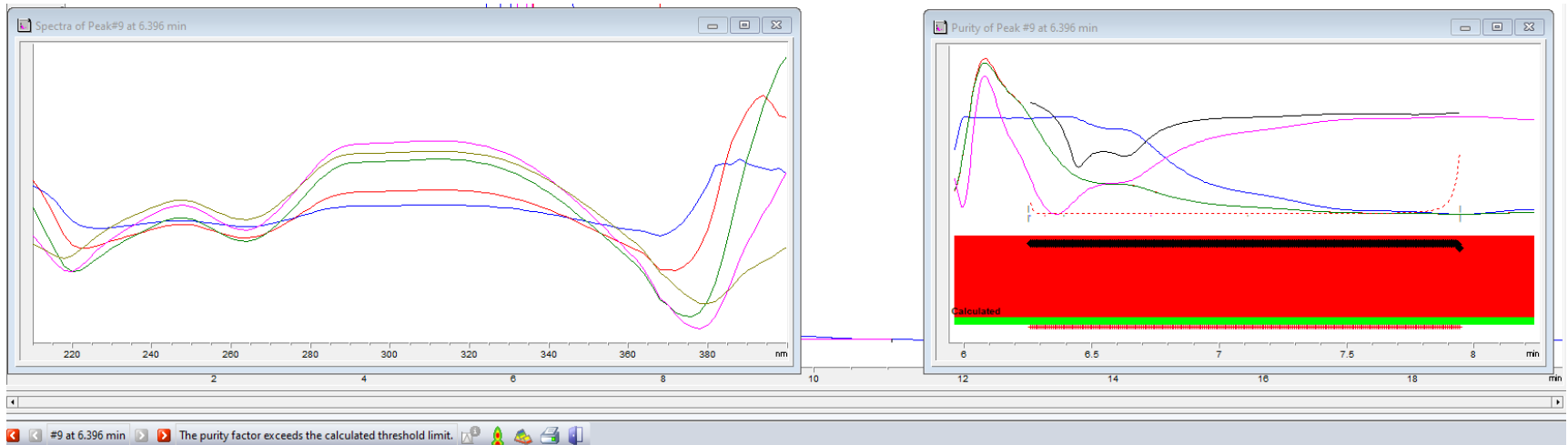
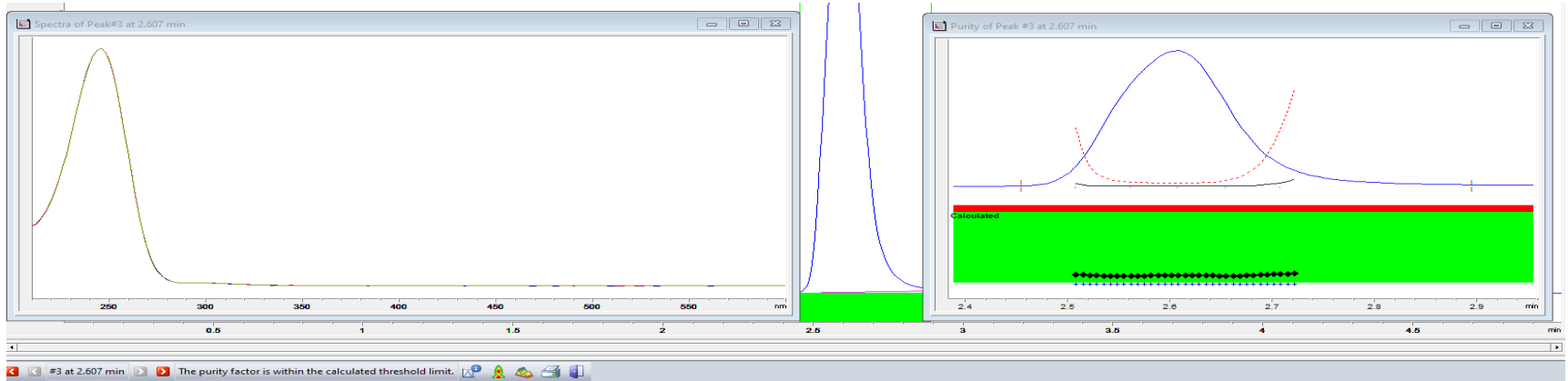
6-Gingerol



Curcumin



Peak Purity/Resolution



QUESTIONS?

Thank you!

Anikó Sólyom, Ph.D.

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520-975-0411

www.gaasanalytical.com