Valine

Type of Posting	Revision Bulletin (POSTPONEMENT)
Posting Date	29–April–2016; notice corrected 01–Jun-2016[1]
Official Date	01–May–2016
Expert Committee	Non-Botanical Dietary Supplements
Reason for Revision	Compliance

In accordance with the Rules and Procedures of the 2015-2020 Council of Experts, the Non-Botanical Dietary Supplements Expert Committee has revised the Valine monograph. The purpose for the revision is to postpone the revision to the *Related Compounds* section of this monograph recently published in *USP 39–NF 34*, because of comments received regarding the performance of the analytical procedure and potential impurity limit compliance issues.

The Valine Revision Bulletin supersedes the revision of the Valine monograph published in *USP 39–NF 34,* which is scheduled to become official May 1, 2016. The Revision Bulletin will be incorporated in the *USP 40–NF 35*.

Should you have any questions, please contact Huy Dinh, Senior Scientific Liaison (301–816–8594 or htd@usp.org.)

[1] The first sentence of the Revision Bulletin Notice was corrected on June 1, 2016 to state, “In accordance with the Rules and Procedures of the 2015-2020 Council of Experts.” The original version of the notice incorrectly referenced the 2010-2015 Rules and Procedures.

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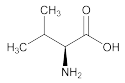

$C_5H_{11}NO_2$ 117.15
L-Valine [72-18-4].

DEFINITION
Valine contains NLT 98.5% and NMT 101.5% of L-valine ($C_5H_{11}NO_2$), calculated on the dried basis.

IDENTIFICATION
- **A. INFRARED ABSORPTION ⟨197K⟩**

ASSAY
- **PROCEDURE**

Sample: 110 mg of Valine
Blank: Mix 3 mL of formic acid and 50 mL of glacial acetic acid.
Titrimetric system
(See *Titrimetry* ⟨541⟩.)
Mode: Direct titration
Titrant: 0.1 N perchloric acid VS
Endpoint detection: Potentiometric
Analysis: Dissolve the *Sample* in 3 mL of formic acid and 50 mL of glacial acetic acid. Titrate with *Titrant*. Perform the blank determination.
Calculate the percentage of valine ($C_5H_{11}NO_2$) in the portion of Valine taken:

$$\text{Result} = \{[(V_S - V_B) \times N \times F]/W\} \times 100$$

V_S = *Titrant* volume consumed by the *Sample* (mL)
V_B = *Titrant* volume consumed by the *Blank* (mL)
N = actual normality of the *Titrant* (mEq/mL)
F = equivalency factor, 117.2 mg/mEq
W = *Sample* weight (mg)

Acceptance criteria: 98.5%–101.5% on the dried basis

IMPURITIES
- **RESIDUE ON IGNITION ⟨281⟩:** NMT 0.1%
- **CHLORIDE AND SULFATE ⟨221⟩,** *Chloride*

Standard solution: 0.50 mL of 0.020 N hydrochloric acid
Sample: 0.73 g of Valine
Acceptance criteria: NMT 0.05%

- **CHLORIDE AND SULFATE ⟨221⟩,** *Sulfate*

Standard solution: 0.10 mL of 0.020 N sulfuric acid
Sample: 0.33 g of Valine
Acceptance criteria: NMT 0.03%

- **IRON ⟨241⟩:** NMT 30 ppm

Delete the following:

•• **HEAVY METALS,** *Method I* ⟨231⟩: NMT 15 ppm
• (Official 1-Jan-2018)

Change to read:

- **RELATED COMPOUNDS**

▲Buffer: 0.05 M monobasic potassium phosphate
Mobile phase: Acetonitrile and *Buffer* (65:35)
Diluent: Acetonitrile and *Buffer* (50:50)
System suitability solution: A mixture of 5 mg/mL of USP L-Valine RS, 0.025 mg/mL of USP L-Leucine RS, 0.025 mg/mL of USP L-Isoleucine RS, 0.02 mg/mL of USP L-Alanine RS, and 0.01 mg/mL of USP L-Phenylalanine RS in *Diluent*
Phenylalanine standard solution: 0.01 mg/mL of USP L-Phenylalanine RS in *Diluent*
Leucine standard solution: 0.025 mg/mL of USP L-Leucine RS in *Diluent*
Isoleucine standard solution: 0.025 mg/mL of USP L-Isoleucine RS in *Diluent*
Alanine standard solution: 0.02 mg/mL of USP L-Alanine RS in *Diluent*
Sample solution: 5 mg/mL of Valine in *Diluent*
Chromatographic system
(See *Chromatography* ⟨621⟩, *System Suitability*.)
Mode: LC
Detector: UV 210 nm
Column: 4.6-mm × 25-cm; 5-µm packing L8
Column temperature: 25°
Flow rate: 1 mL/min
Injection volume: 50 µL
System suitability
Sample: *System suitability solution*
[NOTE—See *Table 1* for relative retention times.]
Suitability requirements
Resolution: NLT 1.5 between leucine and isoleucine
Relative standard deviation: NMT 10.0% each for phenylalanine, leucine, isoleucine, and alanine
Analysis
Samples: Standard solutions and *Sample solution*
Calculate the percentage of each amino acid impurity in the portion of Valine taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of phenylalanine, leucine, isoleucine, or alanine from the *Sample solution*
r_S = peak response of phenylalanine, leucine, isoleucine, or alanine from the corresponding Standard solution
C_S = concentration of USP L-Phenylalanine RS, USP L-Leucine RS, USP L-Isoleucine RS, or USP L-Alanine RS in the corresponding Standard solution (mg/mL)
C_U = concentration of Valine in the *Sample solution* (mg/mL)

Calculate the percentage of any other amino acid or unspecified impurity in the portion of Valine taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak response of any other amino acid or unspecified impurity from the *Sample solution*
r_T = sum of all the peak responses from the *Sample solution*

Acceptance criteria: See *Table 1*.

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Phenylalanine	0.65	0.2
Leucine	0.75	0.5
Isoleucine	0.83	0.5
Valine	1.00	—
Alanine	1.45	0.4
Any other amino acid	—	0.5

Table 1 *(Continued)*

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Any unspecified impurity	—	0.05
Sum of other amino acids	—	1.0

•(Postponed until 1-March-2017)• (RB 1-May-2016)

▲*USP39*

SPECIFIC TESTS

- **Optical Rotation** ⟨**781S**⟩**,** *Procedures, Specific Rotation*
  Sample solution: 80 mg/mL in 6 N hydrochloric acid
  Acceptance criteria: +26.6° to +28.8°
- **pH** ⟨**791**⟩
  Sample solution: 50 mg/mL
  Acceptance criteria: 5.5–7.0
- **Loss on Drying** ⟨**731**⟩
  Analysis: Dry at 105° for 3 h.
  Acceptance criteria: NMT 0.3%

ADDITIONAL REQUIREMENTS

- **Packaging and Storage:** Preserve in well-closed containers.

Change to read:

- **USP Reference Standards** ⟨**11**⟩
  ▲USP L-Alanine RS
  USP L-Isoleucine RS
  USP L-Leucine RS
  •(Postponed until 1-March-2017)• (RB 1-May-2016)▲*USP39*
  USP L-Phenylalanine RS
  USP L-Valine RS