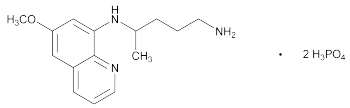
Primaquine Phosphate



$C_{15}H_{21}N_3O \cdot 2H_3PO_4$ 455.34

1,4-Pentanediamine, N^4-(6-methoxy-8-quinolinyl)-, (±)-, phosphate (1:2);
(±)-8-[(4-Amino-1-methylbutyl)amino]-6-methoxyquinoline phosphate (1:2) [63-45-6].

DEFINITION

Change to read:

Primaquine Phosphate contains •NLT 97.0% and NMT 102.0%•(RB 1-Jan-2012) of primaquine phosphate ($C_{15}H_{21}N_3O \cdot 2H_3PO_4$), calculated on the dried basis.

IDENTIFICATION

- **A. INFRARED ABSORPTION ⟨197K⟩:** Meets the requirements
- **B.** The residue obtained by ignition meets the requirements of the test for pyrophosphate, as described in *Identification Tests—General* ⟨191⟩, *Phosphate*.
- **C.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

Change to read:

- **PROCEDURE**

▲Mobile phase: Acetonitrile, tetrahydrofuran, trifluoroacetic acid, and water (9: 1: 0.1: 90)
Standard solution: 0.4 mg/mL of USP Primaquine Phosphate RS in *Mobile phase*. [NOTE—Sonicate with intermittent shaking to dissolve, if necessary.]
System suitability stock solution: 0.4 mg/mL of USP Primaquine Related Compound A RS in *Mobile phase*
System suitability solution: Transfer 1.0 mL of the *System suitability stock solution* to a 10-mL volumetric flask, and dilute with *Standard solution* to volume.
Sensitivity solution: 0.2 µg/mL of USP Primaquine Phosphate RS from the *Standard solution*
Sample solution: 0.4 mg/mL in *Mobile phase*. [NOTE—Sonicate with intermittent shaking to dissolve, if necessary.]
Chromatographic system
(See *Chromatography* ⟨621⟩, *System Suitability*.)
Mode: LC
Detector: UV 265 nm
Column: 4.6-mm × 75-mm; 3-µm packing L7
Flow rate: 1.5 mL/min
Injection volume: 10 µL
Run time: Three times the retention time of primaquine
System suitability
Samples: *Standard solution*, *System suitability solution*, and *Sensitivity solution*
Suitability requirements
Resolution: NLT 2.5 between primaquine and primaquine related compound A, *System suitability solution*
Relative standard deviation: NMT 1.0% for primaquine, *Standard solution*
Signal-to-noise ratio: NLT 10 for the primaquine peak, *Sensitivity solution*
Analysis
Samples: *Standard solution* and *Sample solution*
Calculate the percentage of primaquine phosphate ($C_{15}H_{21}N_3O \cdot 2H_3PO_4$) in the portion of Primaquine Phosphate taken:

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

r_U = peak response from the *Sample solution*
r_S = peak response from the *Standard solution*
C_S = concentration of USP Primaquine Phosphate RS in the *Standard solution* (mg/mL)
C_U = concentration of Primaquine Phosphate in the *Sample solution* (mg/mL)

Acceptance criteria: •97.0%–102.0%•(RB 1-Jan-2012) on the dried basis▲*USP35*

IMPURITIES

Add the following:

▲• ORGANIC IMPURITIES
Mobile phase, Standard solution, System suitability solution, Sensitivity solution, Sample solution, Chromatographic system, and **System suitability:** Proceed as directed in the *Assay*.
Analysis
Sample: *Sample solution*
Calculate the percentage of each impurity in the portion of Primaquine Phosphate taken:

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

r_U = peak response of each impurity from the *Sample solution*
r_S = peak response of primaquine phosphate from the *Sample solution*

Acceptance criteria: See *Table 1*. Disregard any impurity less than 0.05%.

Table 1

Name	Relative Retention Time	Acceptance Criteria, NMT (%)
Specified unidentified impurity	0.24	0.20
Specified unidentified impurity	0.29	0.60
Primaquine related compound A[a]	0.80	2.0
Primaquine	1.0	—
Specified unidentified impurity	1.8	•0.50•(RB 1-Jan-2012)
Any other individual impurities	—	0.20
Total impurities	—	3.0

[a] 8-[(4-Aminopentyl)amino]-6-methoxyquinoline.

▲*USP35*

SPECIFIC TESTS

- **LOSS ON DRYING ⟨731⟩**
Analysis: Dry a sample at 105° for 2 h.
Acceptance criteria: NMT 1.0%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed, light-resistant containers.

$C_{15}H_{21}N_3O$ 259.35▲*USP35*

Change to read:

- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Primaquine Phosphate RS
  ▲USP Primaquine Related Compound A RS
  8-[(4-Aminopentyl)amino]-6-methoxyquinoline.