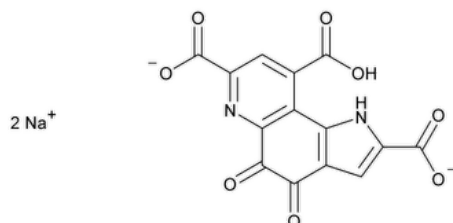


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Pyrroloquinoline Quinone Disodium



$C_{14}H_4N_2Na_2O_8$	374.17
$C_{14}H_4N_2Na_2O_8 \cdot H_2O$	392.19
$C_{14}H_4N_2Na_2O_8 \cdot 2 H_2O$	410.20
$C_{14}H_4N_2Na_2O_8 \cdot 3 H_2O$	428.22

1*H*-Pyrrolo[2,3-*f*]quinoline-2,7,9-tricarboxylic acid, 4,5-dihydro-4,5-dioxo-, sodium salt (1:2);

Sodium 9-carboxy-4,5-dioxo-4,5-dihydro-1*H*-pyrrolo[2,3-*f*]quinoline-2,7-dicarboxylate CAS RN®: 122628-50-6.

1*H*-Pyrrolo[2,3-*f*]quinoline-2,7,9-tricarboxylic acid, 4,5-dihydro-4,5-dioxo-, sodium salt (1:2) trihydrate CAS RN®: 1519046-70-8.

Change to read:

DEFINITION

Pyrroloquinoline Quinone Disodium ▲ (USP 1-Dec-2021) contains NLT 98.0% and NMT 102.0% of pyrroloquinoline quinone disodium salt

($C_{14}H_4N_2Na_2O_8$), calculated on the anhydrous basis. ▲ It may contain up to three molecules of water of hydration. ▲ (USP 1-Dec-2021)

Pyrroloquinoline quinone is also known as PQQ in the dietary supplements industry.

IDENTIFICATION

Change to read:

• **A.** The retention time of the major peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the Assay. ▲ The retention time of the sodium peak of the *Sample solution* corresponds to that of the *Standard solution* at 24 µg/mL, as obtained in the test for *Content of Sodium*. ▲ (USP 1-Dec-2021)

Add the following:

▲ **B.**

[NOTE—The requirements for *Identification B* may be met by following either *Procedure 1* or *Procedure 2*.]

Procedure 1: [SPECTROSCOPIC IDENTIFICATION TESTS \(197\)](#), [Infrared Spectroscopy](#): 197K

[NOTE—If the IR spectra of the sample and [USP Pyrroloquinoline Quinone Disodium RS](#) do not match (different hydration forms), separately dissolve about 3 mg each from the sample and from the [USP Pyrroloquinoline Quinone Disodium RS](#) in 0.6 mL of [water](#). Add 0.2 mL of [dehydrated alcohol](#), and mix well. Let the solvent evaporate from both solutions at NMT 50°. Once the solvent is evaporated, dry the sample under vacuum at room temperature.]

Procedure 2: ¹H NMR Spectrum

Diluent: [Deuterium oxide](#) containing 0.05% deuterated sodium-3-trimethylsilylpropionate (TMSP-*d*₄) NMR shift reference

Standard solution: Dissolve 2 mg of [USP Pyrroloquinoline Quinone Disodium RS](#) in 0.6 mL of *Diluent*.

Sample solution: Dissolve 2 mg of pyrroloquinoline quinone disodium in 0.6 mL of *Diluent*.

Instrumental conditions

(See [Nuclear Magnetic Resonance Spectroscopy \(761\)](#).)

Acquisition parameters

Mode: Fourier transform proton (¹H) NMR, pulsed

Frequency: NLT 300 MHz for ^1H

Pulse sequence: Single pulse

Pulse angle: 30°

Sweep width: 15 ppm (-2.0 to 13.0 ppm)

Acquired data points: NLT 32K (for 300 MHz)

Acquisition temperature: 22° – 25°

Acquisition time: NLT 2.5 s

Relaxation delay: NLT 10 s

Number of scans: Adjust to meet the requirements for *System suitability*.

Dummy scans: 2

Processing parameters

Processed spectrum data points: NLT 64K (for 300 MHz)

Window multiplication: Exponential

Line broadening factor: 1.0 Hz

System suitability

Sample: *Standard solution*

Suitability requirements

Signal-to-noise ratio (S/N): The number of transients should be adjusted until S/N for each signal of pyrroloquinoline quinone from the *Standard solution* is NLT 100:1 (see [Table 1](#)).

Analysis

Samples: *Standard solution* and *Sample solution*

Transfer the *Standard solution* and the *Sample solution* into separate NMR tubes of 5 mm in diameter. Acquire free induction decay (FID) with or without water suppression. Chemical shift for the NMR reference ($\text{TMSP-}d_4$ methyl signal) should be set to 0.00 ppm for all

Samples. Immediately acquire the ^1H NMR spectrum for the *Standard solution* and *Sample solution* using the same conditions. Phase the spectrum and correct the baseline prior to signal identification. The ^1H NMR spectrum of the *Standard solution* should display two sets of ^1H signals, corresponding to the 4,5-*ortho*-quinone and 5,5-geminal diol water adduct forms of pyrroloquinoline quinone moiety, as shown in [Table 1](#).

Table 1

Chemical shift values, δ_{H} (ppm)	Signal multiplicity	Signal assignment
7.19	singlet	C(3)H (pyrrole) ^a
7.22	singlet	C(3)H (pyrrole) ^b
8.20	singlet	C(8)H (pyridine) ^a
8.29	singlet	C(8)H (pyridine) ^b

^a Corresponds to the 4,5-*ortho*-quinone form.

^b Corresponds to the 5,5-geminal diol water adduct form.

Acceptance criteria: The chemical shift values of signals from the *Sample solution* match those obtained from the *Standard solution* and do not differ by more than ± 0.1 ppm. The ratio between the sum of integrals for the C(8)H to the sum of integrals for the C(3)H resonances is 1.0 ± 0.1 . ▲ (USP 1-Dec-2021)

ASSAY

Change to read:

• PROCEDURE

Buffer solution: Prepare a solution containing 10 mM [dibasic potassium phosphate](#) and 15 mM [tetrabutylammonium bromide](#) in [water](#).

Adjust with [phosphoric acid](#) to a pH of 7.4.

Mobile phase: [Acetonitrile](#) and *Buffer solution* (28:72)

Diluent: [Acetonitrile](#) and [water](#) (1:3)

Glycine standard solution: 0.1 mg/mL of [USP Glycine RS](#) in [water](#)