

METHOD OF ANALYSIS

NMNH

1.Appearance

Take an appropriate amount of sample and spread it on white filter paper. no black spots and foreign matter can be seen to the naked eye. and the sample is Light yellow powder

2. Related Substances

➤ Procedure:

Detector: UV 340nm

Injection volume: 10 μ l

Flow rate: 1ml/min

column temperature: 25 $^{\circ}$ C

Run time: 40min

Column: Shim-pack GIST 5 μ m C18-AQ 4.6 \times 250mm. or an equivalent column

Solution A: 0.02M KH₂PO₄, Adjust the pH to 9.0 with 4 N potassium hydroxide solution (22.4 g \rightarrow 100 ml).

Solution B: acetonitrile

Diluent: Solution A

Current: see the gradient table blow.

Time	Solution A	Solution B
0	100	0
8	100	0
20	30	70
30	30	70
31	100	0
40	100	0

➤ System suitability:

Black Solution: Diluent.

Standard solution: Dissolve 25mg standard sample to a 25ml volumetric flask, and dilute of diluent to volume.

Test solution: Dissolve 25mg test sample to a 25ml volumetric flask, and dilute of

diluent to volume.

➤ Analysis:

Pipette 10 μ l of the blank solution、standsrtd solution and the test solution into the system, record the chromatogram, remove the blank solvent peak, and calculate the results by area normalization method.

3. Water

Calibration of Karl Fischer reagent: Turn on the power switch of the moisture meter. wait for the instrument to stabilize. inject 10ul of water, record the consumed Karl Fischer reagent. measure twice, and take the average value.

Sample testing: Accurately weigh an appropriate amount of sample (approximately 0.2g) and conduct testing according to the "Standard Operating Procedures for Moisture Meters".

4. pH

Weigh 50mg of the sample and add water to make a sample containing 100mg /ml. According to the general principle of "pH value determination method" in the 《USP-NF2022》. the pH value should be within the range of 7.0 to 11.0.

5. Heavy Metals

5.1 Lead

根据《USP-NF2024》通则<251>中的程序 3 ICP-MS 进行测定 Determination was made according to procedure 3 ICP-MS in USP-NF2024 General Rule <251>

5.2 Arsenic

Determination was made according to ICP-MS in USP-NF2024 General Rule <211>

5.3 Mercury

Determination was made according to procedure 5 ICP-MS in USP-NF2024 General Rule <261>

5.4 Cadmium

Determination was made according to procedure 2 ICP-MS in USP-NF2024 General Rule <233> elemental impurities—procedures

还原型 β -烟酰胺单核苷酸二钠盐检测方法

1. 外观

取适量样品铺在白色滤纸上，肉眼看不到黑点和异物。样品为淡黄色粉末。

2. 有关物质

➤ 程序

检测器：紫外 340nm

进样量：10 μ l

流速：1ml/min

柱温：25 $^{\circ}$ C

运行时间：40min

色谱柱：Shim-pack GIST 5 μ m C18-AQ 4.6 \times 250mm，或等效柱。

溶液 A：0.02M KH₂PO₄，用 4 N 氢氧化钾溶液(22.4 g \rightarrow 100 ml)调节 pH 至 9.0。

溶液 B：乙腈

稀释液：溶液 A

流路：见下列梯度表。

时间	溶液 A	溶液 B
0	100	0
8	100	0
20	30	70
30	30	70
31	100	0
40	100	0

➤ 系统适用性

黑色溶液：稀释剂。

标准溶液：将 25mg 标准样品溶解于 25ml 容量瓶中，用稀释液定容至刻度。

试验溶液：将 25mg 试验样品溶解于 25ml 容量瓶中，用稀释液定容至刻度。

➤ 分析

分别移取 10 μ l 的空白溶液、标准溶液和待测溶液入系统，记录色谱图，去除空白溶剂峰，用面积归一化法计算结果。

3. 水分

卡尔费休试剂的校准：打开湿度计的电源开关。等待仪器稳定下来。注入 10 μ l 水，记录消耗的卡尔费休试剂。测量两次，取平均值。

样品测试: 准确称量适量的样品 (约 0.2g), 并根据“水分计标准操作程序”进行测试。

4. pH

称取 50mg 样品, 加水制成 100mg/ml 样品。根据《USP-NF2022》中“pH 值测定方法”的一般原则。pH 值应在 7.0 ~ 11.0 范围内。

5. 重金属

5.1 铅

根据《USP-NF2024》通则<251>中的程序 3 ICP-MS 进行测定。

5.2 砷

根据《USP-NF2024》通则<211>中的 ICP-MS 进行测定。

5.3 汞

根据《USP-NF2024》通则<261>中的程序 5 ICP-MS 进行测定。

5.4 镉

根据《USP-NF2024》通则<233>元素杂质程序中的程序 2 ICP-MS 进行测定