iCAP PRO Series ICP-OES Determination of Impurity Elements in Battery-Grade NMP

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Keywords

NMP, N-Methylpyrrolidone, iCAP PRO ICP-OES, Direct Injection

Abstract

This paper presents a rapid analytical method for detecting multiple impurity elements in *N*-Methylpyrrolidone using the Thermo Scientific™ iCAP™ PRO Series ICP-OES. The instrument's high-efficiency, variable-frequency RF generator enables direct sample introduction without dilution. By optimizing the oxygen flow using a precise mass flow controller, carbon buildup and molecular spectral interference from carbon species are minimized. Calibration curves for all measured elements exhibit excellent linearity ($r^2 > 0.999$). Detection limits reach the µg/L level, with measurement precision (RSD) < 2% and recovery rates between 85% and 110%. This method is suitable for the analysis of impurity elements in NMP.

Introduction

N-Methylpyrrolidone (NMP) is a key material used as a cathode coating solvent and conductive slurry solvent in lithium-ion batteries. Battery-grade NMP requires extremely high purity. In addition to low moisture content, the concentrations of metallic ions must be strictly controlled, as they can significantly affect battery performance and safety.

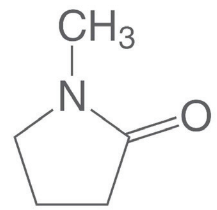

Figure 1.
N-Methylpyrrolidone (NMP)

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To ensure battery performance and reliability, impurity levels in battery-grade NMP are typically in the range of 10–20 µg/L.

In this study, the Thermo Scientific iCAP PRO Series ICP-OES was used to analyze impurity elements in battery-grade NMP. The method enables direct injection without dilution, minimizing contamination from the environment and water and thereby improving analytical accuracy and reliability.

Experimental

Instruments and Reagents

- Thermo Scientific iCAP PRO ICP-OES
- 24-element mixed standard solution (1000 mg/L, Spex)
- *N*-Methylpyrrolidone (NMP)
- Blank NMP (semiconductor-grade)
- Analytical balance (0.1 mg precision)
- 50 mL LDPE volumetric flask (Nalgene)
- Pipettes (200 µL, 1000 µL, Thermo Scientific)

Solution Preparation and Sample Pretreatment

Intermediate stock solution (20 mg/L): Transfer 1 mL of 1000 mg/L mixed standard solution into a 50 mL volumetric flask and dilute to volume with blank NMP.

Standard solutions: Transfer 0 mL, 0.05 mL, 0.125 mL, 0.25 mL, and 1.25 mL of the 20 mg/L intermediate solution into separate 50 mL flasks, dilute with blank NMP to obtain concentrations of 0, 0.02, 0.05, 0.1, and 0.5 mg/L, respectively.

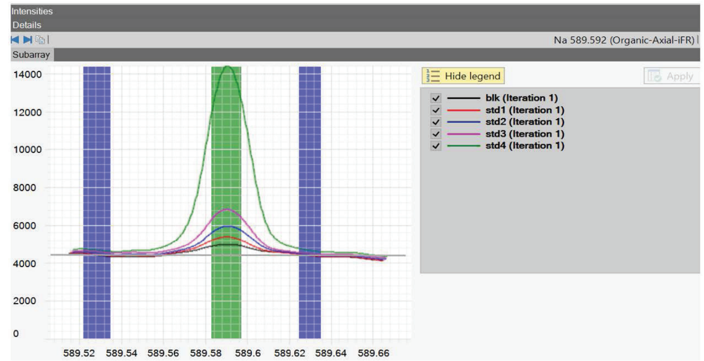
Spiked recovery samples (0.05 mg/L and 0.1 mg/L): Transfer 0.125 mL and 0.25 mL of the 20 mg/L intermediate solution into separate 50 mL volumetric flasks and dilute to volume with the sample solution. These samples were used to evaluate recovery.

Instrument Parameters and Method Optimization

Oxygen Flow Optimization

Introducing oxygen into the plasma reduces carbon deposition from organic solvents and minimizes C_2 spectral background interference near Na 589.592 nm. However, excessive oxygen can extinguish the plasma, whereas insufficient oxygen fails to remove interference effectively. An oxygen flow rate of 50 mL/min was selected, providing minimal carbon deposition while maintaining stable plasma operation.

Figure 2. Na standards at 0, 0.02, 0.05, 0.1, and 0.5 mg/L spiked in NMP at an oxygen flow rate of 50 mL/min.



Instrument Parameters

Table 1. Instrument paramaters

Model	iCAP PRO XP Duo	**Oxygen Flow**	50 mL/min
Observation Mode	Axial	**Pump Speed**	20 rpm
Nebulizer	Concentric	**RF Power**	1300 W
Torch	Quartz	**Nebulizer Gas**	0.4 L/min
Center Tube	1.0 mm alumina	**Auxiliary Gas**	1.0 L/min
Spray Chamber	Cyclonic with baffle	**Cooling Gas**	14.5 L/min

Table 2. Calibration curve correlation coefficient (R^2): The calibration standards were prepared in semiconductor-grade NMP, and measurements were performed using a matrix-matched method.

Element	Correlation coefficient (R^2)
Al 167.079	0.9997
Ca 393.366	0.9991
Cd 214.438	0.9997
Co 238.892	0.9997
Cr 267.716	0.9998
Cu 327.396	0.9994
Fe 259.940	0.9998
K 766.490	0.9998
Mg 279.553	0.9996
Na 589.592	0.9997
Ni 231.604	1.0000
Zn 213.856	0.9996

An NMP blank solution was measured 11 consecutive times. The method detection limit (MDL) under the experimental conditions was calculated as three times the standard deviation of the blank measurements and is reported in µg/L.

Table 3. MRL and test results

Element	MRL (g/L)	Test Results (g/L)
Al 167.079	3.97	ND
Ca 393.366	0.64	ND
Cd 214.438	1.01	ND
Co 238.892	1.72	ND
Cr 267.716	1.63	ND
Cu 327.396	1.56	ND
Fe 259.940	2.34	ND
K 766.490	4.13	ND
Mg 279.553	0.51	ND
Na 589.592	3.85	ND
Ni 231.604	3.57	ND
Zn 213.856	0.97	ND

**ND: Not detected*

Spike recovery experiments were performed on two NMP samples at spike levels of 0.05 and 0.1 mg/L.

The recovery results are shown below.

Table 4. Recovery study and results

Element	Study result	Standard with 0.05 mg/L	0.05 mg/L Recovery	Standard with 0.1 mg/L	0.1 mg/L Recovery
Al 167.079	ND	0.0491	98.2	0.1003	100.3
Ca 393.366	ND	0.0536	107.1	0.1049	104.9
Cd 214.438	ND	0.0514	102.9	0.0995	99.5
Co 238.892	ND	0.0513	102.6	0.0975	97.5
Cr 267.716	ND	0.0498	99.6	0.0963	96.3
Cu 327.396	ND	0.0480	96.0	0.0877	87.7
Fe 259.940	ND	0.0501	100.3	0.0973	97.3
K 766.490	ND	0.0460	92.0	0.0919	91.9
Mg 279.553	ND	0.0508	101.5	0.0983	98.3
Na 589.592	ND	0.0488	97.6	0.0917	91.7
Ni 231.604	ND	0.0540	108.0	0.1060	106.0
Zn 213.856	ND	0.0496	99.1	0.0947	94.7

**ND: Not detected*

An NMP sample spiked at 0.1 mg/L was analyzed with seven replicate injections. The relative standard deviation (RSD) was calculated to evaluate method precision.

Table 5. Repeatability study

Element	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Aver-age	RSD%
Al 167.079	0.0987	0.0970	0.0977	0.0958	0.0964	0.0983	0.0967	0.0972	1.07
Ca 393.366	0.0967	0.0959	0.0952	0.0953	0.0959	0.0962	0.0958	0.0959	0.54
Cd 214.438	0.0955	0.0960	0.0954	0.0952	0.0965	0.0962	0.0964	0.0959	0.53
Co 238.892	0.0971	0.0976	0.0973	0.0972	0.0980	0.0985	0.0981	0.0977	0.54
Cr 267.716	0.0985	0.0985	0.0976	0.0984	0.0993	0.0991	0.0992	0.0987	0.59
Cu 327.396	0.0995	0.1000	0.0991	0.0996	0.1012	0.1008	0.1003	0.1001	0.73
Fe 259.940	0.0989	0.0984	0.0973	0.0982	0.0987	0.0994	0.0995	0.0986	0.75
K 766.490	0.0979	0.0969	0.0978	0.0985	0.0988	0.0985	0.0985	0.0981	0.65
Mg 279.553	0.0975	0.0968	0.0971	0.0968	0.0978	0.0978	0.0973	0.0973	0.44
Na 589.592	0.1057	0.1055	0.1067	0.1075	0.1086	0.1065	0.1068	0.1068	1.00
Ni 231.604	0.0986	0.0970	0.0974	0.0971	0.0992	0.0984	0.0977	0.0979	0.86
Zn 213.856	0.1003	0.0990	0.1000	0.0996	0.1007	0.1004	0.1002	0.1000	0.57

Conclusion and discussion

This study established a rapid, direct-injection ICP-OES method for the simultaneous analysis of multiple elements in NMP using the Thermo Scientific iCAP PRO Series ICP-OES. Each sample injection and data acquisition were completed within one minute. The instrument's variable-frequency, high-efficiency RF generator ensures stable plasma operation even with organic samples such as NMP. Compared with digestion or dilution methods, direct injection offers higher sensitivity, lower detection limits, and superior accuracy, significantly improving analytical efficiency and meeting the stringent demands of the battery industry.