APPLICATION NOTE

Analysis of NMP in batteries by GC-FID

AN190 v1; SCION Instruments

Introduction

N-methyl-2-pyrrolidone (NMP) is a widely used polar aprotic solvent in the manufacturing of lithium-ion batteries. It serves primarily as the solvent for polyvinylidene fluoride (PVDF) binders during electrode slurry preparation. Due to its excellent solvency, chemical stability and high boiling point, NMP enables uniform dispersion of active materials and consistent electrode coating. However, residual NMP can remain trapped within electrode pores after drying, potentially affecting electrochemical performance and posing health and safety concerns due to its classification as a reproductive toxicant under current regulatory frameworks.[1]

As regulatory limits on NMP exposure become increasingly stringent, particularly under EU REACH and U.S. EPA (Toxic Substances Control Act) TSCA regulations, accurate quantification of residual NMP in battery electrodes has become an important aspect of both quality control and regulatory compliance.[2] Determination of NMP at low concentration levels is critical for process optimization, verification of solvent removal efficiency, and assessment of occupational exposure risk during electrode manufacturing.

This application can be performed on either the SCION Instruments 8300 GC or 8500 GC (Figure 3) platform with an Flame Ionization Detector (FID) and the SCION 8400PRO Autosampler. A SCION-WAXMS column was used to obtain the best separation.

Helium has long been the standard carrier gas for GC applications however, due to recent global shortages many companies are keen to find alternatives[3]. The application described in this note has been performed using an alternative carrier gas, nitrogen. This can be produced with generators using renewable energy sources and so is a cleaner, greener option as well as avoiding the current issues over helium supply. The GC uses nitrogen as a carrier and make-up gas for the FID.

Figure 1 SCION Instruments 8300 & 8500-GC equipped with 8400 PRO Autosampler.

Experimental

For this application an NMP standard, dimethylformamide (DMF), dimethyl sulfoxide (DMSO) and ethanol (HPLC grade), copper electrode plates, 50 mL centrifuge tubes and 0.45 µm PTFE syringe filters were purchased.

A stock NMP standard was prepared at a concentration of 100,000 mg/L in ethanol.

Table 1 shows the developed method parameters used for this application.

Table 1 Instrumentation operating conditions for GC

GC Part	Settings
S/SL Injector	250°C Split 20:1
Injection Volume	1.0 µL
Column	SCION-WAXMS 30m x 0.25mm x 0.25µm
Carrier Gas	Nitrogen 2 mL/min
Oven Program	60°C (hold 2.0 min), 20°C/min to 250°C (hold 3.50 min)
Detector	FID 250°C Make-up (N_2): 28 mL/min Combustion (H_2): 30 mL/min Combustion (Air): 300 mL/min
Run Time	15.0 min
Software	CompassCDS

Sample preparation

Linearity samples were prepared in ethanol at 7 levels for the high level curve: 0.5, 1, 5, 10, 20, 50 and 100 mg/L and at 5 levels for the low level curve: 0.5, 1, 5, 10 and 20 mg/L.

The system precision was performed on 4 data points, 0.5, 1, 10 and 50 mg/L (n=7). The LOQ sample was prepared at a concentration of 0.1 mg/L.

Two stock standards were prepared for DMF and DMSO at a concentration of 1000 mg/L in ethanol. They were both diluted up to 0.5 mg/L.

The sample preparation of the copper electrode plates was conducted as follows, 0.8 g of the plates were weighed and placed in a centrifuge tube. 10 mL of ethanol was added and then vigorously shaken and sonicated for 15 minutes. Approximately 1 mL of the sample was filtered using 0.45 µm PTFE syringe filters. This was determined as the blank QC samples (n=3).

For determining the recovery, QC spiked samples were prepared (n=6). The same sample preparation was used as described above but then the samples were spiked at different concentrations levels. Each level was prepared 6 times. The final concentrations of the QC spiked samples were, 0.5, 1.0, 20 and 50 mg/L.

Results

The calibration curves for the NMP standard were prepared at 5 low and 7 high levels from 0.5 mg/L up to 100 mg/L. The linearity results (R^2) obtained from the calibration curves are 0.9999 for both the low and high curve. Figures 2 and 3 show the results obtained using the SCION Instruments software CompassCDS.

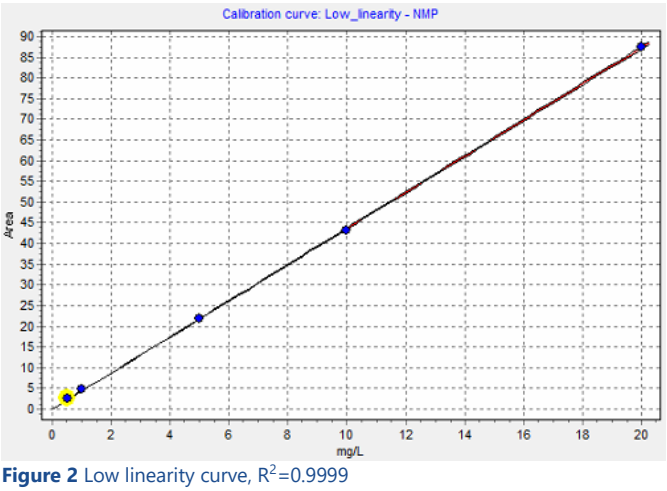

Figure 2 Low linearity curve, R^2=0.9999

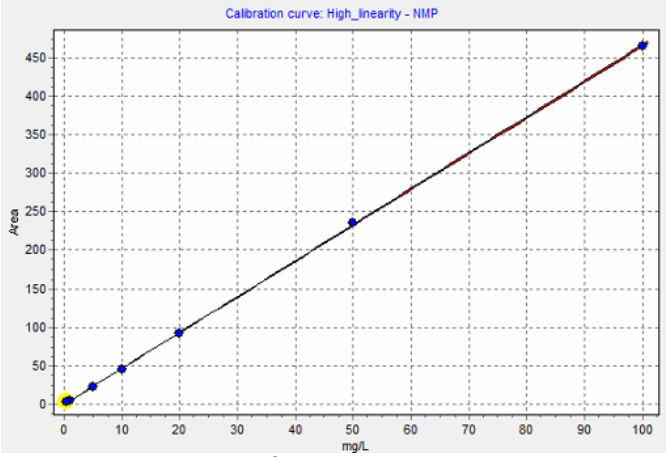

Figure 3 High linearity curve R^2=0.9999

The system precision of the method was obtained by 7 consecutive injections of the NMP standard. Repeatability results in Table 2 show that for the NMP compound, the relative standard deviations (RSD%) ranged from 0.46% to 3.66%. This shows a good precision across the different concentrations.

Table 2 Repeatability with different concentrations

Repeatability	0.5 mg/L	1.0 mg/L	10 mg/L	50 mg/L
Injection 1	135.3	281.7	2683.5	14180.2
Injection 2	138.9	261.5	2722.8	14053.7
Injection 3	146.4	269.6	2710.4	14046.8
Injection 4	140.3	262.2	2717.6	14078.7
Injection 5	130.3	263.9	2699.3	14204.2
Injection 6	143.2	258.2	2624.0	14171.8
Injection 7	134.6	273	2701.6	14197
Mean	138.4	267.2	2694.2	14133.2
RSD %	**3.66**	**2.83**	**1.15**	**0.46**

The LOQ sample was injected at a concentration of 0.1 mg/L. Figure 4 shows the NMP peak with an area of 60.2 µV.sec, a height of 45.6 µV and an RMS noise of 3.0 µV. Resulting in a S/N of 15.2.

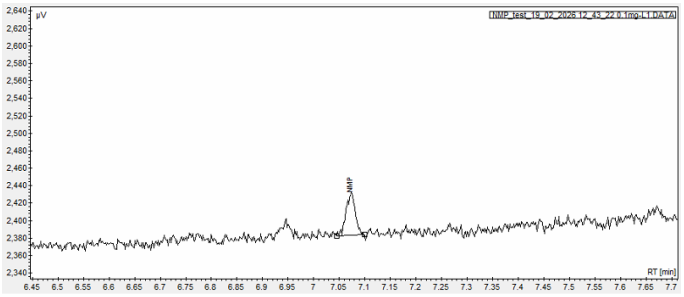

Figure 4 NMP peak at LOQ level

DMF and DMSO are considered to be alternatives to NMP therefore these compounds were analysed to see how they would behave in this method. All compounds were eluted and separated from one another. DMF and DMSO were positively detected at a concentration of 0.5 mg/L.

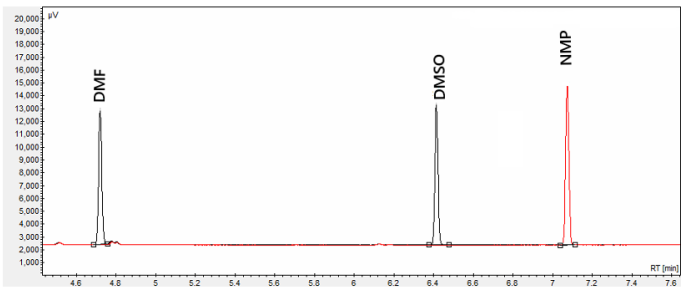

Figure 5 Zoomed chromatogram of DMF, DMSO and NMP

The recovery results (Table 3) indicate that no NMP was detected in the QC blank samples. The recovery percentages for the QC spiked samples ranged from 94.86% up to 115.18%, with repeatability (RSD) values between 0.13% and 8.35%. A linearity was calculated from the QC spiked recovery samples results resulting in an R^2 value of 0.994.

Table 3 Recovery and repeatability results for QC samples

Sample	NMP added (mg/L)	NMP found (mg/L)	Recovery	RSD
QC Blank	0.00	0.00	N/A	N/A
QC Spike Level 1	0.50	0.52	104.84	8.35
QC Spike Level 2	1.00	1.08	107.78	2.96
QC Spike Level 3	20.00	23.04	115.18	0.13
QC Spike Level 4	50.00	47.43	94.87	0.46

Conclusion

The SCION 8500/8300 GC platform equipped with a split/spitless injector, SCION-WAXMS column, FID detector and 8400PRO Autosampler is a perfect solution for analyzing NMP in batteries.

Good system precision, linearity and recovery results were achieved for this application. The recovery was achieved with multiple QC spiked recovery samples showing good linearity, confirming good working of the analytical method.

The samples were prepared using an extraction sample preparation technique to prepare NMP samples for GC analysis.

References

1. NMP Solvent in Lithium-Ion Battery Production: A Key Enabler (accessed April 2026)
2. Risk Management for n-Methylpyrrolidone (NMP) | US EPA (accessed April 2026)
3. Helium Shortage 4.0: What caused it and when will it end? (accessed April 2026)

Order Information

Ordering Information for the GC	
Part	**Part Number**
8500-GC, S/SL-EFC 21, FID-DEFC 11, 230V	859001702
8500-GC, S/SL-EFC 21, FID-DEFC 11, 120V	859001701
8300-GC, S/SL-EFC 21, FID-DEFC 11, 230V	839001702
8300-GC, S/SL-EFC 21, FID-DEFC 11, 120V	839001701
CompassCDS Data Acquisition Software	BR502002
Suggested consumables	
Part	**Part Number**
15% Graphite/85% Vespel Ferrule 1/16" with 0.4 mm hole pk/10	41312148
BTO Septa 9 mm, pk/50	CR298713
10µL fixed needle syringe, 5 cm, 0.47 mm OD, 26 g conical needle	41312133
SCION-WAXMS column 30m x 0,25mm x 0,25 µm	SC32423
1177 4mm SPLT LINER / FRT-SILTEK PK/5EA	RT210462145

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