Application Note AN-K-076

Vinylene carbonate water determination via coulometric Karl Fischer titration

Water determination in vinylene carbonate by Karl Fischer coulometric titration made simple with alcohol-free reagents

Vinylene carbonate (vc) is commonly used as a battery electrolyte additive in lithium-ion batteries. Its popularity is due to the positive impact it has on anode cyclability and reduction of irreversible capacity. Small amounts of moisture in Li-ion batteries can significantly decrease performance and lifetime. Therefore, moisture levels in electrolyte solutions and their additives are controlled for use in manufacturing. Until recent years, accurate moisture quantification in vinylene carbonate samples was difficult due to side reactions. With the commercial development of alcohol-free coulometric Karl Fischer reagents, accurate determination is now much more accessible to end-users.

SAMPLE AND SAMPLE PREPARATION

Vinylene carbonate was used in this study without any sample preparation steps.

EXPERIMENTAL

This analysis was performed using an 852 Titrando (2.852.0060) instrument, equipped with the electrodes listed in Table 1.

Table 1. Overview of electrodes used in this application.

Equipment	Article number
Generator electrode with diaphragm	6.00348.100
Double Pt-wire electrode for coulometry	6.0341.100

A small volume of vinylene carbonate was aspirated into a gas-tight syringe to coat its interior before being disposed of. Excess vinylene carbonate was then drawn into the gas-tight syringe. The prepared syringe was then tared on a balance with four decimal places.

The titration vessel was pre-titrated during conditioning to remove any moisture present from both the immediate environment and the reagents themselves. A volume of 0.5 mL of vinylene carbonate was directly injected into the titration vessel equipped with NEXTGEN Coulomat A-FA/C-FA reagents. The mass was measured by difference. Water from the sample was finally titrated in the coulometric vessel.

RESULTS

Results are recorded in Figure 1 and Table 2.

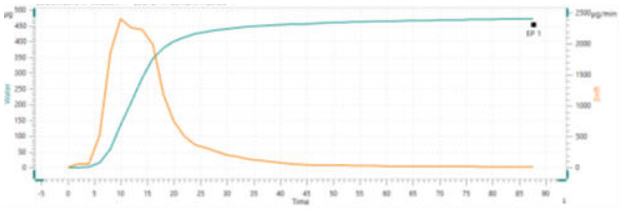

Figure 1. Example coulometric KF titration curve for vinylene carbonate.

Table 2. Results for the vinylene carbonate water content determination (n = 3) as evaluated by direct injection coulometric KF titration.

Result 1 (ppm)	Result 2 (ppm)	Result 3 (ppm)	Mean (ppm)	%RSD
787.73	795.67	788.79	790.73	0.55

CONCLUSION

During battery manufacturing, control of all moisture present is important to guarantee adequate performance in the finished product. More easily quantifying the moisture contribution from additives such as vinylene carbonate makes this a much simpler task. The introduction of alcohol-free Karl Fischer reagents accomplishes this effectively, allowing the generation of accurate and repeatable moisture data—even for a difficult sample matrix.

CONTACT

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CONFIGURATION

852 Titrando mit Touch Control und Generatorelektrode mit Diaphragma

Coulometer inklusive Generatorelektrode mit Diaphragma, Touch Control, 801 Magnetrührer und kompletter volumetrischer Titrierzelle.

Für die Wasserbestimmung im Spurenbereich (10 µg bis 10 mg Absolutwasser) ist die Coulometrie die ideale Methode zur Wasserbestimmung in Flüssigkeiten, Feststoffen und Gasen. Zudem ist die Coulometrie eine absolute Methode, weshalb keine Titerbestimmung notwendig ist.

Empfohlener Messbereich Coulometrie: 10 µg...200 mg Absolutwasser

Der 852 Titrando beherrscht neben der coulometrischen auch die volumetrische Karl-Fischer-Titration.

Verwendung mit OMNIS-Software, *tiamo*-Software oder Touch Control. Erfüllt GMP/GLP- und FDA-Anforderung wie 21 CFR Part 11, falls erforderlich.

Generatorelektrode mit Diaphragma

Generatorelektrode mit Diaphragma für coulometrische Karl-Fischer-Titrationen, Normschliff 29/22. Diese Elektrode hat einen nach oben abgewinkelten Kabelanschluss, welcher die Kabelführung und die Bedienung bei Verwendung mit dem OMNIS Sample Robot Oven vereinfacht.

Diese Generatorelektrode eignet sich speziell für:

- Proben mit sehr tiefen absoluten Wassergehalten 50 ppm
- Coulometrische Wassergehaltsbestimmung in Ölproben
- Anwendungen, bei denen Ketonreagenzien verwendet werden
- Anwendungen, bei denen schlecht leitende Reagenzien verwendet werden (Zusatz von Chloroform, Xylen >10%)

Doppel-Pt-Draht-Elektrode für die Coulometrie

Indikatorelektrode, die für coulometrische Karl-Fischer-Titration verwendet wird.