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.

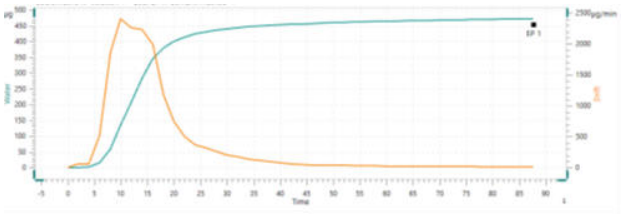

Figure 1. Example coulometric KF titration curve for vinylene carbonate.

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 with Touch Control and generator electrode with diaphragm

Coulometer including generator electrode with diaphragm, Touch Control, 801 Magnetic Stirrer, and complete volumetric titration cell.

Coulometry is the ideal method for water content determination in liquids, solids, and gases when it comes to water content determination in the trace range (10 µg to 10 mg absolute water). In addition, coulometry is an absolute method and thus no titer determination is necessary.

Recommended coulometry measuring range: 10 µg - 200 mg absolute water

The 852 Titrando controls not only coulometric, but also volumetric Karl Fischer titration.

For use with OMNIS Software, *tiamo* software, or Touch Control unit. Compliance with GMP/GLP and FDA regulations such as 21 CFR Part 11, if required.

Generator electrode with diaphragm

Generator electrode with diaphragm for coulometric Karl Fischer titrations, SGJ 29/22. This electrode has a cable connection angled upwards, which simplifies cable guiding and operation when used with the OMNIS Sample Robot Oven.

This generator electrode is well-suited for:

- Samples with very low absolute water content 50 ppm
- Coulometric water content determination in oil samples
- Applications where ketone reagents are used
- Applications where low-conducting reagents are used (addition of chloroform, xylene >10%)

Double Pt-wire electrode for coulometry

Indicator electrode used for coulometric Karl Fischer titration.