Water determination in dimethyl sulfoxide (DMSO)

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It is not possible to reproduce water determination in DMSO because it alters the stoichiometry of the Karl Fischer (KF) reaction. This shortcoming becomes even greater as the number of samples increases.

We tested the water content in different DMSO sample quantities according to the following procedure. 30 mL of Hydranal-Methanol Rapid, Hydranal-Methanol dry or Hydranal-CompoSolver E were introduced into the titration vessel and titrated to dryness with Hydranal-Composite 5. The sample (or sample plus water) was then added and titrated with Hydranal-Composite 5.

Results are compared in the tables below.

Table 1. Water determination in various DMSO sample quantities

DMSO sample volume	Hydranal-Composite 5 consumption	Water quantity detected	Water content detected
1.0 mL	0.08 mL	0.44 mg	0.044%
2.0 mL	0.14 mL	0.77 mg	0.038%
5.0 mL	0.19 mL	1.05 mg	0.021%
10.0 mL	0.35 mL	1.93 mg	0.019%
20.0 mL	0.62 mL	3.41 mg	0.017%

Table 2. Different sample quantities of DMSO added to a defined water quantity (20.0 mg)

Sample	Hydranal-Composite 5 consumption	Water quantity detected	Recovery rate
20 mg H_2O	3.81 mL	20.00 mg	100.0%
20 mg H_2O + 1.0 mL DMSO	3.80 mL	19.95 mg	99.75%
20 mg H_2O + 2.0 mL DMSO	3.77 mL	19.79 mg	98.95%
20 mg H_2O + 5.0 mL DMSO	3.71 mL	19.48 mg	97.40%
20 mg H_2O + 10.0 mL DMSO	3.49 mL	18.32 mg	91.60%
20 mg H_2O + 20.0 mL DMSO	3.38 mL	17.74 mg	88.70%

Coulometric tests gave comparable findings:

- Water content detected in 1 mL DMSO: 311 ppm
- Water content detected in 20 mL DMSO: 266 ppm

Control conditions with Hydranal-Water Standard 1.0 (added water quantity: 1000 ppm) in the presence of DMSO produced the following results:

- Water content in the presence of 1 mL DMSO: 994 ppm
- Water content in the presence of 20 mL DMSO: 910 ppm

The volumetric and coulometric water content determination tests were carried out with two different samples.

The problem was picked up again in a series of subsequent tests. We ascertained that it is also impossible to perform indirect determination in a KF oven. A temperature ramp from 50°C to 250°C showed that the water is released by azeotropic distillation at temperatures between 130°C and 190°C. It is impossible to separate the water from the DMSO. The titration vessel is influenced in the same way as by direct injection of the sample. The error is comparable to the effect described for direct injection.

Conclusion

As a result of the various facts, the recommendation must be made that no more than 1 mL of DMSO should be analyzed. The injection of several samples in a coulometric cell should be avoided. Addition of a 1000 ppm standard shows the current effect on the titration vessel.

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