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カール・フィッシャー容量滴定法 / 電量滴定法の組み合わせによる水分測定法

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Validity Study for Combination of Volumetric and Coulometric Methods in Karl Fischer Water Titration

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Summary

An automatic Karl Fischer (KF) moisture titrator that employs a new titration method has been developed. In this method, designated as Hybrid KF titration, the water amount of a sample can be determined by means of automatically combined volumetric and coulometric KF titrations in a specially configured single titration flask equipped with both a titration nozzle and a pair of electrodes separated by a diaphragm for electrolysis. In addition, by taking advantage of the titration flask, the titrant factor of a KF test solution can be standardized by controlled potential coulometry with no need for any water reference materials.

In this work, these titration and standardization methods have been experimentally validated and their practical usefulness has been examined. Analytical samples as well as reference water standards were measured by Hybrid titration. The results show a good agreement over a wide range from 0.1 to 12.0 mgH_2O with those obtained by the pharmacopeial standard methods. The relative error and RSD are within ± 2% and less than 1%, respectively. The high correlation coefficient of R^2=0.998 was obtained between the titrant factors of KF test solutions standardized by coulometry and those determined by the pharmacopeial standard method.

These results indicate that the proposed methods can be practically used for ordinary laboratory measurements and are equivalent to the pharmacopeial standard methods. Moreover, owing to its applicability over a wide range of the water amount, the Hybrid KF titration would best suited for analytical samples that contain unknown amounts of water. The coulometric standardization of a KF test solution offers a totally automatic titrant factor standardization procedure in which errors due to weighing and dispensing would be completely eliminated.

Key words

Karl Fischer titration, Water determination, Volumetric titration, Coulometric titration, Titrant factor standardization, Controlled potential coulometry