Application Bulletin

Of interest to:	General analytical laboratories	D F 1, 3, 4, 6, 7, 11, 12, 15

Karl Fischer water determination with the KF drying oven

Summary

The KF drying oven makes it possible to determine the water content of samples that either undergo unwanted side reactions with the Karl Fischer reagent or are unsuitable for direct introduction into the titration vessel. With this method the sample is heated up in the oven and the released water is transferred by a stream of dry carrier gas (e.g. nitrogen or air) to the titration vessel, where it is titrated.

Instruments and accessories

- 701 KF Titrino, 758 KFD Titrino or 784 KFP Titrino or 737 or 756 KF Coulometer
- 688 or 768 KF Drying Oven
- Possibly 661 Pump Unit and flow meter (when using the 688 KF Drying oven)
- Possibly compressed gas cylinder with nitrogen

Reagents

Karl Fischer reagents, depending on whether volumetric or coulometric titrators are used.

Method

- Install and connect the instruments according to the Instructions for use, then turn them on.
- Set the oven temperature. This should be set as high as the respective sample permits. The higher the oven temperature, the less time the analysis will take.
- Adjust the gas flow. Standard value is ca. 100 mL/min (6 L/h).
- Select «conditioning» on the titrator and condition the entire system for ca. 30 min.
- Determine the blank. Several sample boats are equipped with aluminum inserts and then left in contact with the laboratory air in order to condition them. Open the KF drying oven, place one boat in the cold part of the oven and immediately close again. Then transfer the sample boat to the hot part of the drying oven and carry out the water determination (e.g. during 20 min). On completion of the analysis, take out the boat and condition it again in the laboratory air. Now proceed in the same way with the next sample boat, etc. Be careful to only touch

the sample boats with a pair of tweezers. Calculate the mean value of the multiple determination and enter this as blank on the titrator.

- If the samples are unknown, a heating curve should first be recorded. To do this, set the oven temperature as high as the respective sample permits. Depending on the water content, 0.1 ... 3 g sample are weighed into the conditioned sample boat. Place the boat, as described, in the oven and start the determination. Plot the consumption of KF reagent or the released amount of water as a function of time. Using this curve, the optimum extraction time can be determined.
- Drift has proved to be the best stop criterion (to stop the titration).

Application examples

- Bicarbonates, hydroxides, carbonates, oxides
- Salts containing water of crystallization
- Coal dust, soot
- Effervescent powder/effervescent tablets
- Pharmaceutical raw materials and preparations
- Plastics, cartons, insulation materials
- Pasta, potato chips, etc.
- Washing powder
- Organic substances that react with the Karl Fischer reagent and have a sufficiently high melting point

Remarks

- It usually takes 1 ... 2 min until the first water reaches the titration vessel.
- For the analysis of samples that are sensitive to oxygen or air, nitrogen has to be used as carrier gas.
- The analysis system as a whole can be checked using a water standard (e.g. Hydranal Water Standard KF Oven no. 34748 from Riedel-de Haën).
- If the drift is too high, a test can be made to determine whether the carrier gas is dry enough by separating the connecting tube to the oven.
- The molecular sieve can be regenerated by drying at 300 ... 350 °C.

Figure

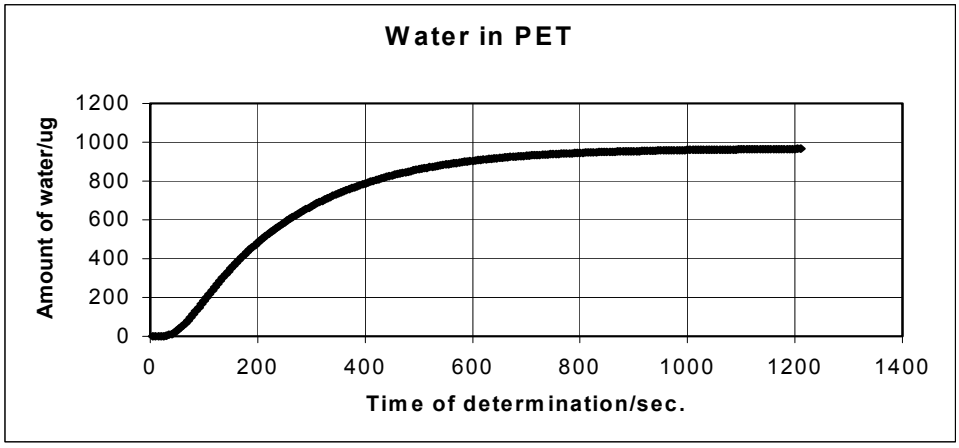

Fig. 1: *Heating curve, recorded with the 756 KF Coulometer and 768 KF Drying Oven.*

Literature

- Metrohm Monograph No. 8.026.5003, Water Determination by Karl Fischer Titration, 2003
- Metrohm Application Bulletin No. 145
  Determination of low water contents in plastics using the KF oven method
- Metrohm Application Note K-1
  Water in potassium chlorate ($KClO_3$)
- Metrohm Application Note K-8
  Water in plastic chips
- Metrohm Application Note K-10
  Water in coal dust
- Metrohm Application Note K-25
  Water in potato crisps
- Metrohm Application Note K-27
  Water in lime ($CaCO_3$)
- ASTM D 5460-93 (1998)
  Standard Test Method for Rubber Compounding Materials – Water in Rubber Additives
- British Standard BS 6829: 1.5 (1990)
  Analysis of surface active agents (raw materials). Part 1 General methods. Section 1.5 Methods for determination of water content
- Norme Française NF T 52-115 (1978)
  Matières plastiques. Matières de base pour polyuréthanes, polyéthers et polyesters. Dosage de l'eau