Karl Fischer water determination in non-explosive gases

Of interest to: General analytical laboratories

Summary

This Application Bulletin describes the determination of water in non-explosive and non-flammable gaseous samples with the coulometric Karl Fischer method. This method is ideal for very low water contents.

Apparatus and accessories

- 2.756.0010 KF Coulometer, cell with diaphragm, including 728 Magnetic Stirrer
- 2.731.0010 Relay Box
- 6.2148.010 Remote Box
- 2.846.0010 Dosing interface
- 6.1439.020 Gas inlet tube with frit
- 6.1805.010 FEP tubing/ M6/ 13 cm
- 6.2811.000 Molecular sieve 0.3 nm
- 6.2151.000 Cable (Dosing Interface PC)
- 6.2125.100 Cable (Relay Box)
- 2 x Magnetic valve, Lucifer
- T-distribution connector 6 mm MS, Swagelock
- Copper tubing 2 m, 4 x 6 mm
- 4 x Connector 6 mm x ¹/₄ " MS, Swagelock
- Connector 1/4 " x 1/8", Swagelock
- Flow controller Red-y smart controller GSC (Vögtlin Instruments AG, Aesch (CH))
- Pressure meter Bioblock Scientific MP 340A
 0...2000 mbarA
- 6.6056.112 *tiamo* full 1.1
- Remark: For gases with a high water content (> 50 ppm), a generator electrode without diaphragm can be used. For gases with a water content below 50 ppm, a generator electrode with diaphragm has to be used, otherwise the results will be too high.

Reagents

- Anolyte: Hydranal-Coulomat AG Oven, Sigma-Aldrich 34739
- Catholyte: Hydranal-Coulomat CG, Sigma-Aldrich 34840

Setup

See Appendix

Parameters

Start drift	:	5 µg/min
Extraction time	:	15 min
Drift correction	:	automatic
Stop criterion	:	rel. drift
Relative stop dri	ift :	5 µg/min

Stirrer

Speed : 6

The stirrer speed should be set high enough to obtain thorough mixing of the gas bubbles with the KF reagent. Too strong stirring of the solution may, however, affect the drift value. For vessel contents between 110 and 200 mL, a stirring speed of 6 is recommended.

A Metrohm

Karl Fischer reagent

110...200 mL

Volumes larger than 200 mL KF reagent make it difficult to reach a low start drift. Less than 110 mL is not enough to make all the gas react with the solution.

Gas settings

Gas pressure at the cylinder: at least 2 bar (200 kPa) Gas flow rate: at least 200 mL/min

Analysis

Prior to the analysis, the whole system should be purged with the gas to be measured. Several liters of gas may be needed for a complete rinsing. The gas tubing and valves should be rinsed until a stable water content is reached.

During the extraction time, valve 1 is closed and valve 2 is opened to let the gas enter the titration cell. At the end of the extraction time, valve 2 is closed and valve 1 is opened. In this way the gas lines are constantly rinsed with gas and no water enters the tubing.

Remark: If measurements are performed over several days, conditioning of the reagent should not be interrupted during the whole period.

No blank measurements were carried out as the blank values are equal to the drift. Therefore, only a drift correction was applied. (2)

Metrohm

Calculation

$$V_1 = t \cdot \Phi \tag{1}$$

$$\mathsf{m}_{\mathsf{Gas}} \,[\mathsf{mg}] = 1000 \cdot \frac{V_1 \cdot M \cdot T_0 \cdot P_1}{V_0 \cdot (T_0 + \theta_1) \cdot P_0}$$

H₂O [ppm] = $1000 \cdot \frac{m_1}{m_{Gas}}$ (3)

- m₁ = absolute water content [μg]
- Φ = gas flow rate [L/min]
- t = extraction time [min]
- V₁ = added gas volume [L]
- V₀ = gas volume at 0 °C = 22.4 L/mol
- T₀ = 273.15 K
- θ_1 = gas temperature [°C]
- M = molar mass of gas [g/mol]
- P_1 = absolute gas pressure at θ_1 [kPa]
- P_0 = gas pressure at T_0 = 101.325 kPa
- Remark: For a correct calculation of the water content, a pressure correction should be applied, especially for gases with a low water content.

Results

The detection limit of this method was found to be around 2 ppm. The determination limit was approximately 5 ppm.

Relative standard deviations with nitrogen gas were found to lie between 5 and 6%.

Remarks

- As magnetic valves are used, this setup is not suitable for explosive gases.
- A good repeatability can only be obtained with a precise flow meter and pressure meter.
- The setting used is different from the one stipulated in the ISO 10101-3:1993 standard. The flowmeter used in the ISO norm contains water, which means that the gas must be dried before entering the flow meter. Accordingly, the flow rate has to be measured after the cell. However, the measured flow rate is reduced by a factor of 2 compared to a measurement before the titration cell.

Literature

ISO 10101-3:1993

Natural gas – Determination of water by the Karl Fischer method – Part 3: Coulometric procedure

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Appendix

Instrument setup



Caution: the valves heat up to 75 °C!

Metrohm









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