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# Application Bulletin

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Of interest to:      Plastics industry

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## Determination of low water contents in plastics using the KF oven method

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### *Summary*

In many cases it is essential to know the water content of plastics. This is of particular interest in connection with their processing or their use as insulators in electrical engineering. This bulletin describes the coulometric determination of water according to the Karl Fischer method, which is both simple to carry out with the instruments mentioned and much less time-consuming than the other methods normally used.

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### *Instruments and accessories*

- 756 or 737 KF Coulometer with titration cell with or without diaphragm
- 768 KF Drying Oven
- Remote cable 6.2141.010 (for 756 KF Coulometer) or 6.2141.000 (for 737 KF Coulometer) to connect the 768 KF Oven
- Possibly additional 6.2125.110 connecting cable (to print out the oven parameters in the result report of the 756 KF Coulometer)
- Possibly compressed gas cylinder with nitrogen

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### *Reagents*

Commercially available Karl Fischer reagents for the coulometric determination of water using titration cells with or without diaphragm.

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### *Preparation*

- Connect the instruments according to the Instructions for use. Fill the titration cell with the KF reagent(s), then switch on the instruments.
- Program the parameters on the 768 KF Oven.
- Program the method on the KF coulometer (parameter, definitions, operands).
- Heat the oven to the respective temperature. Switch the valve to position «TRANSFER» (using the «VALVE» key on the 768 KF Oven). When the desired temperature has been reached set the gas flow (e.g. nitrogen) at ca. 100 mL/min (gas flow controller on the 768 KF Oven).

- Equip the sample boat with an aluminum insert and place it in the hot part of the oven («BOAT IN» key).
- Start conditioning on the KF coulometer («START» key). As soon as the drift is below 20 µg/min (typically 6 ... 8 µg/min) and stable, the system is ready and the analysis can be started.

### Analysis

- Withdraw the sample boat from the hot part of the oven («BOAT OUT» key).
- Start the determination on the 768 KF Oven («START» key). Insert the sample (sample size 0.1 ... 3 g, depending on expected water content) as fast as possible through the opening of the septum screw cap in the sample boat and close the opening again immediately.  
After a short purge and conditioning time the determination is automatically started on the KF coulometer and the sample boat is transferred to the hot part of the oven.
- Enter the sample size on the coulometer.
- Upon completion of the analysis, the result report is automatically printed out and the sample boat is withdrawn from the hot part of the oven.
- To remove the sample, open the side screw cap of the 768 KF Oven and pull out the guide rod and the sample boat with the holding clamp. Using a pair of tweezers, remove the aluminum insert (with the old sample), place a new insert in the boat and close again. (Never touch the guide rod or sample boat with your hands! Always use the holding clamp!)
- Insert the sample boat again in the hot part of the oven («BOAT IN» key) and condition the system until the drift is again below 20 µg/min (typically 6 ... 8 µg/min) and stable.
- Analyze the next sample ...

### Remarks

- 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 (the higher the temperature, the faster the analysis). Using the obtained curve, the optimum extraction can be determined and then entered on the KF coulometer.
- If oven temperatures higher than 160 °C are applied, nitrogen instead of air should be used as drying/carrier gas.
- Here a few examples of plastics analyzed in our laboratories:

Designation	Oven temperature	Remarks
Acrylonitrile/butadiene/styrene copolymer (ABS)	160 °C	
Epoxy powder	120 °C	
Urea/formaldehyde resin	100 ... 150 °C	depending on the type
Urea/phenol resin	100 ... 150 °C	depending on the type
Polymethylmethacrylate (PMMA)	200 °C	

Designation	Oven temperature	Remarks
Polyamide	160 ... 230 °C	depending on the type [polyamide 6 (perlon), polyamide 66 (nylon)]
Polycarbonate	140 °C	
Polyester	140 ... 240 °C	depending on the type
Polyethylene	200 °C	
Polyisobutylene	300 °C	
Polyoxymethylene	160 °C	
Polypropylene	160 ... 200 °C	depending on the type
Polystyrene	120 °C	
Polyurethane	180 °C	
Silicone rubber	250 °C	
Terephthalic acid ester	150 °C	

- Plastics samples with low water content are extremely hygroscopic! They quickly take up water from the ambient air and therefore have to be analyzed immediately after opening the vessel.
- Used molecular sieve can be regenerated by drying at 300 ... 350 °C.
- The gas passing through the electrolyte in the titration cell leads to the evaporation of liquid. It is advisable to mark the liquid level on the vessel at the beginning of the analyses and to refill with methanol from time to time.
- 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 higher than 20 µg/min, check to see whether the carrier gas is dry enough by separating the connecting tube to the oven.

**Figures**

```

'pa
756 KF Coulometer      02141 5.756.0010
date 1999-03-10      time 17:58      23
KFC-B                  Oven-Det
parameters
>control parameters
  EP at U              50 mV
  dynamics             70 mV
  max.rate             max. µg/min
  min.rate             15 µg/min
  stop crit:          rel.drift
  rel.drift            5 µg/min
>titration parameters
  pause                0 s
  extr.time            300 s
  start drift          20 µg/min
  I(pol):              10 µA
  electrode test:     ON
  temperature          25.0 °C
  time interval        5 s
  max.titr.time        OFF s
>statistics
  status:              OFF
>preselections
  drift corr:          auto
  req.ident:           id1
  req.smpl size:       value
  request and titr:    ON
  smpl unit:           mg
  limit smpl size:    OFF
  text id1             id1 or C21
  text id2             id2 or C22
  text id3             id3 or C23
  cell:                no diaph.
  generator I:         400 mA
  oven:                COM2
  activate pulse:      OFF
  -----

'de
756 KF Coulometer      02141 5.756.0010
date 1999-03-10      time 17:59
KFC-B                  Oven-Det
def
>formula
  blank=C39
  RS1 text             blank
  RS1 decimal places  1
  RS1 unit:            µg
  RS1 limit control:   OFF
  content=(H2O-C39)*C01/C00/C02
  RS2 text             content
  RS2 decimal places  3
  RS2 unit:            %
  RS2 limit control:   OFF
>silo calculations
  match id:            OFF
>common variables
>report
  internal:result;
  report COM1:result;water crv;mplist;
>mean
  MN1=RS2
  -----

'cf
756 KF Coulometer      02141 5.756.0010
date 1999-03-10      time 17:59
KFC-B                  Oven-Det
C-fmla
  C01                  100
  C02                  1000
  -----
    
```

**Fig. 1:** Settings on the 756 KF Coulometer: parameters, definitions, operands.

```

'co
768 KF Oven            07194 5.768.0010
configuration
>oven settings
  auto preparation:    ON
  valve control:       OFF
  start if cond.ok:    OFF
  start temp.range     5 °C
  temp.correcture      0.0 °C
  send to:             IBM
  report:              OFF
>auxiliaries
  dialog:              english
  run number           0
  auto start           OFF
  start delay          0 s
  beeper               1
  device label
  program              5.768.0010
>RS232 settings
  baud rate:           9600
  data bit:            8
  stop bit:            1
  parity:              none
  handshake:           Hws
  RS control:          ON
  -----

'pa
768 KF Oven            07194 5.768.0010
parameters
  temperature          250 °C
  unit gas flow:       mL/min
  min.gas flow         85 mL/min
  gas type:            N2
  purge time           5 s
  cond.time            2 s
  -----
    
```

**Fig. 2:** Settings on the 768 KF Oven: configuration, parameters.

**Literature**

- Metrohm Monograph No. 8.026.5003, Water Determination by Karl Fischer Titration, 2003
- Metrohm Application Bulletin No. 109  
Karl Fischer water determination with the KF drying oven
- Metrohm Application Bulletin No. 255  
Validation of Metrohm KF titrators and KF ovens according to GLP/ISO 9001
- Metrohm Application Bulletin No. 273  
Validation of Metrohm KF coulometers using Standard Operating Procedures
- DIN EN ISO 960: 1997  
Kunststoffe – Polyamide (PA) – Bestimmung des Wassergehalts
- Norme Française NF T 52-115 (1978)  
Matières plastiques. Matières de base pour polyuréthanes, polyéthers et polyesters. Dosage de l'eau