a **xylem** brand

Determination of the Chemical Oxygen Demand COD according to DIN 38 409, part 41

date: 16.05.2013 page 1 from 7



a xylem brand

#### Use

Determination of the chemical oxygen demand above concentration of 15 mg/l. In the case of undiluted samples the procedure can be used up to 300 mg/l. The chloride contents of the undiluted sample must not exceed 1 g/l. Apart from that, the work should be performed according to the procedure laid down in Part 41-2.

### **Appliances**

- Titrator: TL 6000/7000 (TL 6000/7000 M1/20 or TL 6000/7000 M1/50) consists of
- Basic device
- Magnetic stirrer TM 235
- 20 mL exchange unit WA 20 or 50 mL exchange unit WA 50, with amber glass bottle for the titrant, complete

#### **Electrodes**

- Electrode: Pt 61
- Titration tip TZ 1623
- Option: longer stand rod with (450 mm) Z 601

### Reagents

- Titrant: Ferrous (II) ammonium sulphate solution 0.120 mol/l
- Sulphuric acid, potassium hydrogen phthalate solution as standard, potassium dichromate solution (containing mercuric sulphate) 0.02 mol/l

### **Description**

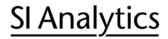
#### Preparation of the titration solution

47.1 g of ferrous (II) ammonium sulphate are dissolved in water in a 1 I volumetric flask. 20 ml of sulphuric acid  $\Box = 1.84 \text{g/l}$  are added, solution is topped up with water to 1000 ml.

### Titer standardisation of the titration solution

The titer of the solution is determined in use on a daily basis. To do so, 10.0 ml of the potassium dichromate solution 0.02 mol/l are diluted with water to approx. 100 ml. 30 ml of sulphuric acid are added carefully to this solution. After cooling down the solution is placed on the magnetic stirrer and titrated using 3 times the method "Titer COD". A triple determination is carried out. The average value is automatically calculated in mol/l and stored into the exchange unit.

date: 16.05.2013 page 2 from 7



a **xylem** brand

### **Method Titer COD:**

# Method data

 Method name:
 COD Titer
 Created at:
 05/16/13 16:18:56

 Method type:
 Automatic titration
 Last modification:
 05/16/13 16:22:11

Measured value: mV

Titration mode: Linear Documentation: GLP

Linear steps: 0.020 ml

Measuring speed / drift: User-defined: minimum holding time: 01 s

maximum holding time: 10 s
Measuring time: 01 s
Drift: 30 mV/min

25 s

Initial waiting time: 0 s

Titration direction: Decrease

Pretitration: 9.000 ml Delay time:

End value: Off EQ: On

Slope value: User-defined Value: 1200

Dosing parameter

Dosing speed: 100.00 % Filling speed: 30 s

Maximum dosing volume: 20.00 ml

Calculation formula

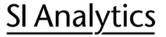
Titer: (V\*F2)/((EQ1-B)\*M\*F1) -> WA Mol (M): 1.00000

Unit: mol/I Decimal places: 4

Pattern (V): 1.000 ml Factor 2 (F2): 1.2000 Blank value (B): 0.0000 ml Factor 1 (F1): 1.0000

Statistics: 3

date: 16.05.2013 page 3 from 7



a **xylem** brand

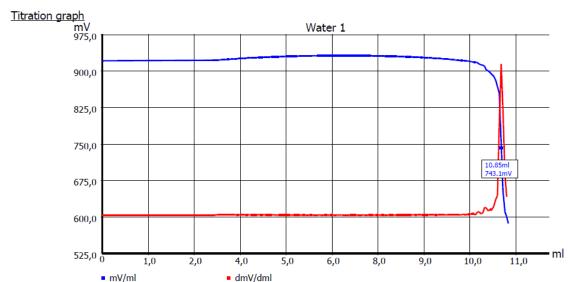
#### **Titration**

After the proper addition of potassium dichromate solution (containing mercuric sulphate) and sulphuric acid to the samples and the blank values, they are placed in a **suitable device** (e.g. from the company Behr) where the reaction mixture is heated up and boilded for 2 hours at 120 °C. Afterwards the samples and blank values are brought to room temperature and then placed on the magnetic stirrer. Carry out the method "COD blank" first. Repeat the method 2 times. The three blank values are used to automatically calculate an average value which is stored into the global memory (M01: COD blank). Now the sample titrations can be carried out.

A control determination can be made using potassium hydrogen phthalate solution. In this case one uses 20 ml of the potassium hydrogen phthalate solution instead of the sample as pattern. The theoretical COD value of this solution is 200 mg/l. Values between 192 and 208 mg/l are admissible.

### Result example

### GLP documentation



Method data

Method name: COD

End date: 31.10.11

End time: 16:28:46 Titration duration: 8 m 22 s

Titration data

Sample ID: Water 1 Pattern: 20.0000ml

Start mV: 919.7mV End mV: 586.4mV

EQ1: 10.854 ml/743.1mV

date: 16.05.2013 page 4 from 7

# SI Analytics

# **Application**

a **xylem** brand

#### Method

#### **COD Blank value:**

Linear steps:

# Method data

Method name: COD Blank Created at: 05/16/13 16:23:24
Method type: Automatic titration Last modification: 05/16/13 16:23:57

Measured value: mV

Titration mode: Linear Documentation: GLP

0.020 ml

Measuring speed / drift: User-defined: minimum holding time: 01 s

maximum holding time: 10 s

Measuring time: 01 s

Drift: 30 mV/min

Initial waiting time: 0 s
Titration direction: Decrease

Pretitration: 9.000 ml Delay time: 25 s

End value: Off EQ: On

Slope value: User-defined Value: 1200

#### Dosing parameter

Dosing speed: 100.00 % Filling speed: 30 s

Maximum dosing volume: 20.00 ml

### Calculation formula

Blank: EQ1 -> M01

Unit: ml Decimal places: 2

Statistics: 3

date: 16.05.2013 page 5 from 7



GLP

25 s

# **Application**

a **xylem** brand

# COD sample:

# Method data

Method name: COD sample Created at: 05/16/13 16:25:15
Method type: Automatic titration Last modification: 05/16/13 16:26:42

Measured value: mV
Titration mode: Linear

Linear steps: 0.040 ml

Measuring speed / drift: User-defined: minimum holding time: 01 s

maximum holding time: 10 s
Measuring time: 01 s
Drift: 30 mV/min

Documentation:

Delay time:

Initial waiting time: 0 s
Titration direction: Decrease

Pretitration: Decrease
Pretitration: 3.000 ml

End value: Off EQ: On

Slope value: User-defined Value: 1200

Dosing parameter

Dosing speed: 100.00 % Filling speed: 30 s

Maximum dosing volume: 20.00 ml

Calculation formula

COD: (B-EQ1)\*T\*M\*F1/(V\*F2) -> M01 Mol (M): 1.00000

Unit: mg/l Decimal places: 1

 Blank value (B):
 M01
 Titre (T):
 auto

 Factor 1 (F1):
 8000.0000
 Pattern (V):
 Vol

 Factor 2 (F2):
 1.0000
 Statistics:
 3

date: 16.05.2013 page 6 from 7



a xylem brand

# **Electrode handling**

After use, the Pt 61 electrode should be placed in KCl 3 mol/l immediately. It has to be made sure that there is always sufficient electrolyte solution in the electrode. For further details, please refer to the electrode's operating instructions.

#### **Notes**

If you have any questions on the application, you can feel free to contact us..

SI Analytics GmbH Hattenbergstr. 10 55122 Mainz Germany Phone: +49 (0) 6131 / 66 - 5062

+49 (0) 6131 / 66 - 5118

Fax: +49 (0) 6131 / 66 – 5001 E-Mail: titration@si-analytics.com Homepage: www.si-analytics.com

date: 16.05.2013 page 7 from 7