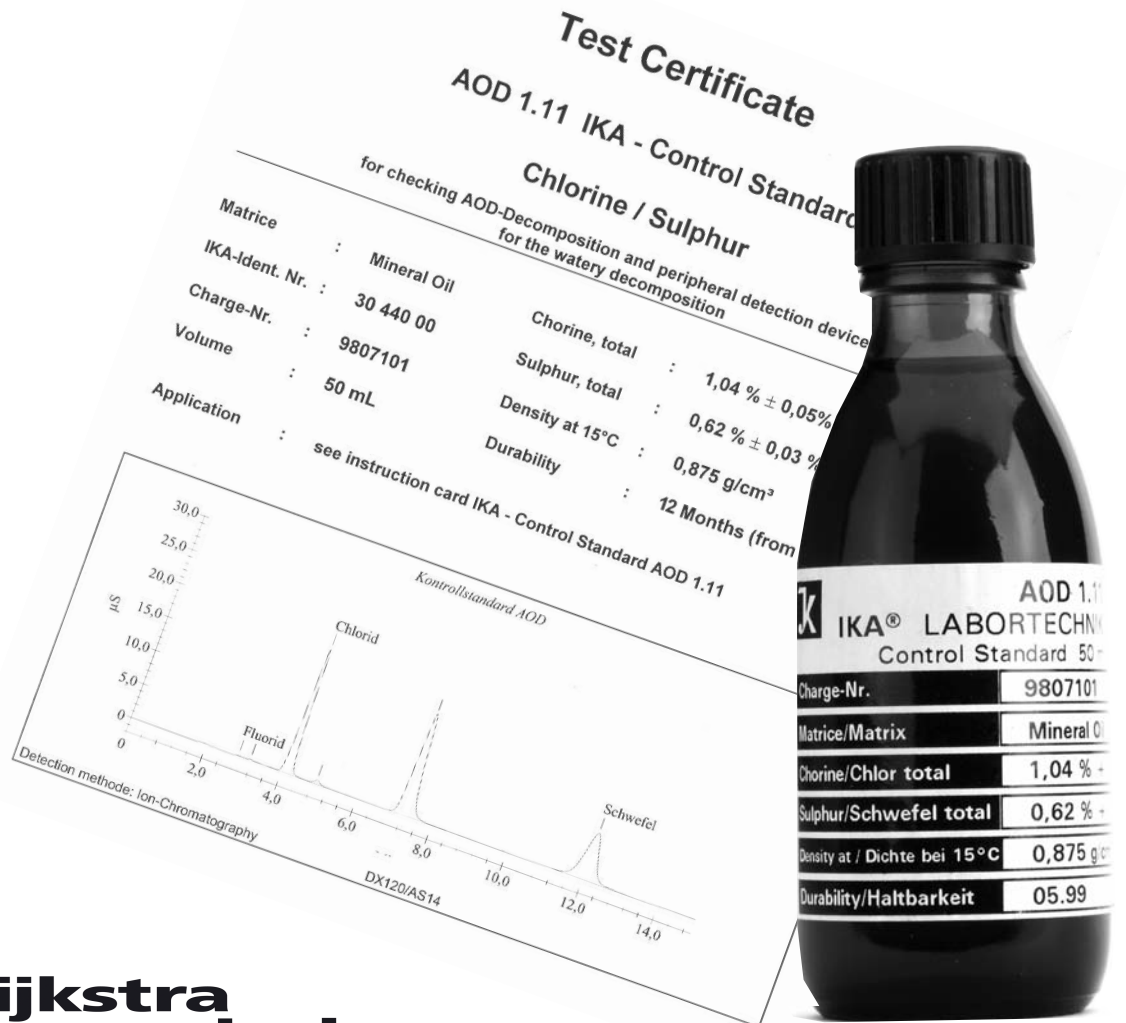


## IKA-Control standard AOD 1.11 AOD 1.12



**dijkstra  
vereenigde**

Postbus 2151  
8203 AD Lelystad  
Tel: 0320-266171

Pascallaan 9  
8218 NJ Lelystad  
Fax: 0320-257354

email: [laboratorium@dijkstra.net](mailto:laboratorium@dijkstra.net)  
[www.dijkstra.net](http://www.dijkstra.net)

Arbeitsanweisung  
Work instruction

DE  
USA

# WORK INSTRUCTION FOR ION CHROMATOGRAPHIC AND TITRIMETRIC DETERMINATION OF IKA-CONTROL STANDARD AOD 1.11 AND AOD 1.12

## Sample designation / description:

IKA – Control standard AOD 1.11 for chlorine and sulphur  
IKA – Control standard AOD 1.12 for fluorine and bromine

Ion(s) sought: Chlorid and sulfate  
Fluorid and bromid

Durability: In the case of normal keeping the durability amounts  
12 months (see label)  
Please you consider the following points:  
- always only to taking open, afterwards again lock and  
under 10°C store  
- protect against sun exposure

Analysis by means of / with:

- 1) Titration processor of the company (Mettler and/or Metrohm)  
Titration of chloride with silver nitrate
- 2) Ion chromatograph of the firm Dionex (DX- 1000/120)  
Determination of chloride and sulfate by means of conductivity measurement

Absorption solution:

1 liter consists of:  
50 ml stock solution (see below)  
25 ml 30% H<sub>2</sub>O<sub>2</sub>  
7 - 8 NaOH – pellets

1 Liter stock solution is made up of:  
21.2 g Na<sub>2</sub>CO<sub>3</sub> and 6.30 g NaHCO<sub>3</sub>

## Calorific value of the sample:

Can be determined in parallel with employment of a C 2000; C 5000 or C 7000 calorimeter

Combustion aid(s): Acetobutyrate capsules may be used

O<sub>2</sub> - pressure: 30 bar

## Description of the combustion residues:

Small portion of ash and soot residues in the crucible.

## Work instruction:

1. Sample preparation

From the homogeneous standard oil 0.2 - 0.5 g of the sample are filled directly into the quartz crucible and/or into a acetobutyrate capsule (C 10).

The originally weighted-in quantity of the sample material is noted and required later for the content determination. The weight of the cap is not important for the measurement as it contains neither halogens nor sulfur. The weight is important only during parallel calorific value determination, since the energy additionally introduced thereby must be deducted from the gross calorific value as foreign energy.

The capsule is for better sample handling as well as to ensure that no volatile substances can escape in the case of unknown samples. Furthermore, the capsule prevents a spattering of the sample when igniting and supports decomposition through their additional energy of approximately 20100 J/g.

## 2. Cleaning the decomposition bomb

It must be guaranteed that no contamination is present in the decomposition bomb, and the latter shall be cleaned in such a way that the parameters which may be examined are not effected.

Suitable cleaning agents are acetone, isopropyl alcohol and distilled water. The decomposition vessel is dried using a cellulose cloth or the like and can be used for the decomposition.

## 3. Preparing the decomposition bomb

First the ignition wire is fastened onto the ignition electrodes. After this one fills approximately 10 ml of the absorption solution (see above for composition) into the decomposition vessel. Alternatively, it is possible to use  $0,25 \frac{\text{mol}}{\text{l}}$  NaOH and 100µl hydrogen peroxide as the absorption solution.

The quartz crucible is inserted into the mounting plate of the bomb. Then the ignition wire must be hung into the crucible in such a way that it can ignite the acetobutyrate capsule or oil directly with its firing during the ignition.

The ignition insert is now placed into the decomposition bomb and screwed on tightly. Now the decomposition bomb at the oxygen filling station is filled with 20 bar excess pressure and emptied again two times by means of the filling station so that the entire nitrogen / oxygen of the room air (approximately 210 ml) is driven out. For combustion the bomb is filled with 3.5 oxygen or purer with 30 bar excess pressure and is now ready for ignition. After this the washing bottle at the oxygen station is filled with 50 ml of the absorption solution.

## 4. The AOD 1 procedure

A calorimeter for igniting the sample in the decomposition vessel is not needed for this procedure, instead one ignites the sample with an electrical impulse which is directly applied to the electrodes of the decomposition vessel. One places the ignition cap on the decomposition vessel filled with oxygen and starts combustion by pressing the igniter. Successful ignition is characterized by the heating up of the decomposition vessel. After ignition the vessel can be cooled down directly in cooling unit (cooling unit as an option to the system). If the vessel is cooled off then the incineration gases are discharged from the decomposition vessel at venting station C 7030 / C 5030 via a washing bottle.

This section replaces point 4.a of this work instruction in the event that one uses with the AOD 1 procedure.

### 4.a Combustion in the calorimeter

One enters the base values of the measurement at the calorimeter. Then one places the decomposition vessel into the calorimeter and closes the cover. Now one starts the measurement and waits for the ignition. After the ignition calorific value as well as the rise in temperature are indicated by the calorimeter. The decomposition vessel can be taken out of the calorimeter and be placed into the cooling unit of the system.

If the vessel is cooled off then the incineration gases are discharged from the decomposition vessel at venting station C 7030 / C 5030 via a washing bottle.

5. Rinsing the decomposition vessel for potentiometric content determination

The decomposition vessel is opened and thoroughly rinsed out with distilled water. Care should be taken to ensure that all parts of the vessel are accounted for.

Then the decomposition solution as well as the absorption solution are filled from the washing bottle into a 250 ml flask and fills this up to the calibration mark. The aliquot removed for the measurement (50.0 ml or 100.0 ml) is transferred into a titration cup.

The aliquot factor of 5 or 2.5 is noted and needed for calculation of the result. The titration cup is then filled approximately halfway with distilled water and 2 ml concentrated nitric acid is added.

6. Potentiometric measurement

The titration cup is then placed into the analysis processor and the corresponding procedure is started. After several minutes the result can be read off in milligrams. The halogen concentration can be calculated from this taking the originally weighted-in quantity and the aliquot factor into consideration.

7. Rinsing the decomposition vessel for analysis in the ion chromatograph

The procedure for this step is the same as under point 5 with the difference that one uses eluent for rinsing out instead of distilled water. 1000 ml eluent consists of:

In accordance with the data supplied by the equipment manufacturer and/or the column supplier.

Calculation is performed by taking the originally weighted-in quantity into consideration as well as the flask contents in which the decomposition solution and the absorption solution were combined from the washing bottle.

 **dijkstra  
vereenigde**  
Postbus 2151                      Pascallaan 9  
8203 AD Lelystad                8218 NJ Lelystad  
Tel: 0320-266171                Fax: 0320-257354  
email: [laboratorium@dijkstra.net](mailto:laboratorium@dijkstra.net)  
[www.dijkstra.net](http://www.dijkstra.net)