

## Title: Appearance

### 1. References / bibliography

Patterned acc. to DGF C-II 1

### 2. Definitions

Not documented

### 3. Scope

The appearance of a product is tested visually.  
The test temperature and the appearance are described in the product specification.

### 4. Interferences

Not documented

### 5. Reagents and Materials

As test-vessel translucent and spotless beakers and sample bottles are applied.

### 6. Procedure

The appearance-test is performed at the required temperature and compared with the description of the product specification.

Conformity is noted with an „OK“.

Is there no conformity with the description of the product specification it is noted with a „NOT OK“ and described detailed (e.g.: opaque, turbid, turbid with a settle, test sample is separated, test sample contains particles etc.).

In the case of doubts standard samples of the production are applied as a reference. The age of this reference-sample is limited on max. 6 months.

### 7. Calculation

The result is noted on the analysis form.

### 8. Remarks

Not documented

### 9. Changes

The analytical method GM\_0170\_00\_E\_E substitutes the analytical methods 48-01, SM0008 and EA 051.01 of Goldschmidt and Goldschmidt Rewo.

**10. Approvals**

step	org.-unit / location	name	date	signature <i>[on the original only]</i>
<i>prepared by</i>	CS P; QKL / Steinau	Christ	26.09.02	
<i>checked by</i>	B-CS P; QKL / Essen	Käseborn	26.09.02	
<i>checked by</i>	B-OS SE / Essen	Dr. Weibels	26.09.02	
<i>approved by</i>	CS P; QKL / Steinau	Kirschner	26.09.02	

**Reviewed regarding validity *[on the original only]***

<i>date</i>	<i>signature</i>	<i>date</i>	<i>signature</i>	<i>date</i>	<i>signature</i>

## Title: Determination of Color Indices

### 1. Normative References / Bibliography

For Gardner Color: DGF C-IV-4c; DIN-ISO 4630; DIN EN 1557; DGK F 010

For Lovibond-Color: DGF C-IV 4b; DIN 53995; DGK F 020

For Hazen-Color: DIN-ISO 6271; DIN EN 1557; ASTM D 1209 - 84; DGK F 040

For Hess-Ives Color: DGK F 050

Device manual for service

### 2. Terms / Definitions

Not verified.

### 3. Scope

Measurement of the colors of clear liquids or melted masses.

For cloudy liquids only the Gardner color index following the Komperator method (GM\_0140\_02) can be applied.

### 4. Interferences

Following this method clouds and air bubbles can lead to mistakes.

### 5. Materials and Reagents

Measurement device : Lico 200 (Dr. Lange) **or comparable device**; eventually with printer (LD 200)

Cuvettes :

1 cm rectangle cuvettes; (e.g. Dr. Lange LYY 214)

11 mm round cuvettes; (e.g. Dr. Lange LYY 621); especially for products, which have to be heated

For very light-colored products (Hazen < 120 : 5 cm rectangular cuvettes); (e.g. Dr. Lange LZM 130)

### 6. Procedure

#### 6.1. Calibration

A calibration has to be processed before starting any series of measurements with the particular type of cuvette.

#### 6.2. Measurement

The measurement is allowed to be done only after proper calibration.

The photometer is adjusted following the manual in that way, that the color mentioned in the test plan to be tested is measured.

The following color indices are possible **among other things**: (detailed information see manual)

Gardner – Color Index  
 Iodine Color Index  
 Hazen – Color Index  
 Hess-Ives Color Index  
 Lovibond - Color Indices

For some products the transmission at a given wave length e.g. 440, 460, and 550 nm is documented as well. This is measured strictly with a 1 cm rectangular cuvette.

The product is filled in the particular cuvette, if necessary heated and then put in the device.

### 7. Calculation

The color indices are given directly in the required scale.

### 8. Remarks

Not verified.

### 9. Changes

Replacing edition EA.008.06 and Rewo 05-01; 05-02, and 05-03

### 10. Enforcement

Step	Org.-Unit / Site	Name	Date	Signature [on original copy only]
<i>Prepared</i>	B-CS P (QKL) / Essen	Käseborn	23.09.2002	X X X X X
<i>Checked by</i>	CS P; QKL / Steinau	Hr. Kirschner	23.09.02	X X X X X
<i>Checked by</i>	B-OS P (QKL) / Essen	Dr. Weibels	23.09.02	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	19.11.02	

  

<b>Reviewed regarding validity</b> [on original copy only]					
Date	Signature	Date	Signature	Date	Signature

**Title: Odor****1. Normative References / Bibliography**

Not identified.

**2. Terms / Definitions**

Not identified.

**3. Scope**

General control of products by odor according to this method.

**4. Interferences**

Not identified.

**5. Reagents and Materials**

Standard samples in standard sample glasses

**6. Procedure**

The test is processed with the standard sample glasses. During filling the glass take care, that there is enough air volume to create odor for odor proofing.  
(Optimum: 2/3 sample ; 1/3 air volume)

The odor is proofed at samples which are cooled down to room temperature if not different instructions are given in the test plan.

If the decision of the first test is insecure, the decision is checked with a stored sample. If there are deviations, the laboratory head has to be informed.

**7. Calculation**

The result is documented on the sample accompanying paper, resp. in LIMS/SAP as "ok" and "not ok" resp.

**8. Remarks**

Not identified.

**9. Changes**

Replacing EA.137.02, Goldschmidt-Rewo methods 22-15 and SM0096

**10. Enforcement**

Step	Org.-Unit / Site	Name	Date	Signature <i>[on original copy only]</i>
<i>Prepared by</i>	B-CS P , QKL/ Essen	Käseborn	28.02.2004	X X X X X
<i>Checked by</i>	B-OS P; QKL / Essen	Dr. Weibels	01.03.04	X X X X X
<i>Checked by</i>	CS P; QKL / Steinau	Kirschner	01.03.04	X X X X X
<i>Approved by</i>	B-CS P , QKL/ Essen	Käseborn	01.03.04	

**Reviewed regarding validity *[on original copy only]***

<i>Date</i>	<i>Signature</i>	<i>Date</i>	<i>Signature</i>	<i>Date</i>	<i>Signature</i>

**Title: Determination of Hydroxyl Value****1. Normative References / Bibliography**

DGF C-V 17 a (53)  
Ph. Eur. 2.5.3 Method A

**2. Terms / Definitions**

The hydroxyl value (OHV) indicates the necessary amount of KOH (in mg) to neutralize the consummated amount of acetic acid of 1 g fat during acetylation.

**3. Scope**

The sample is acetylated with acetic anhydride in presence of pyridine. Each hydroxyl group creates one mole acetic acid, the excessive acetic anhydride delivers two mole acetic acid. The consumption of acetic acid is determined titrimetric by the difference between main and blind value, which has to be determined parallel.

This method is applicable for all fat chemical products, even for sorbitan esters. Other methods can lead to different results!

**4. Interferences**

Primary and secondary amines are detected together with the OH-value determination. To high water content disturbs! (Notice excessive amount of acetylation reactant!).

**5. Materials and Reagents**

Pyridine e.g., Baker Art.-Nr. 8073  
Acetic anhydride >95%, Baker Art.-Nr. 6068

Acetylation mixture :

575ml acetic anhydride and 1925ml pyridine are filled in a 2500ml brown glass bottle for liquids. This prepared solution has to be stirred for 24h before usage.

Phenolphthalein solution (e.g.: Merck Art.-Nr. 7233.0100); 1% in ethanol

Potassium hydroxide solution 0.5 mole/l in ethanol, e.g. Fa. B. Kraft Art.-Nr. 12168.xxxx or 05554.xxxx

Ethanol tech. >98%

Ethanol tech. >98%; neutralized with phenolphthalein against 0.5mol/l potassium hydroxide solution in ethanol

dest. water

Dosing mechanism, e.g. Dosimaten, (Fa. Metrohm) with

50ml changing unit, e.g. Fa. Metrohm

10ml changing unit, e.g. Fa. Metrohm

Appropriate heating system, e.g.:

Heating bath (e.g. Fa. Haake) with silicone oil 100 AW, e.g. Fa. Wacker

and finger type thermostat e.g. DC 3 Fa. Haake with external temperature control e.g. Fa. Nolden Typ NL 1240

250ml round bottom flask ( neck length 130mm, total length 210mm, neck width 30mm.) e.g. Fa. Fischer Art-Nr.7416711  
 Dropping bottle 100ml  
 Glass funnel width 40mm  
 Analytical balance MC 210P e.g. Fa. Sartorius  
 1 ml measuring pipette  
 1 l cooling vessel  
 5 ml measuring pipette

## 6. Procedure

To achieve precise and repeatable results, the sample weight and the amount of acetylation mixture has to be chosen in that way, that you get 4 mole acetic anhydride per each mole OH. The corresponding amount of fat to the supposed OHV is weight precisely on  $\pm 0.1\text{mg}$  with an analytical balance.

The weighed portion and the required volume of acetylation mixture, which has to be added measured exactly with the dosing mechanism, can be taken from the table below:

Supposed OHV	Acetylation mixture in ml	Sample weight in g
10 - 100	5	2.00
100 - 150	5	1.50
150 - 200	5	1.00
200 - 250	5	0.75
250 - 300	5 or 10	0.60 1.20
300 - 350	10	1.00
Up to 700	15	0.75
Up to 950	15	0.50
Up to 1500	15	0.30
Up to 2000	15	0.20

Install a little funnel, serving as a reflux cooler, on the flask neck. Now put the flask in a silicon filled steel bath at 95 to 100°C. The flask has to be dipped in 1 cm deep. After 60 min. at temperature add 1 ml dest. water and wait another 10 min. at this temperature.

After that the flask is taken out of the bath and put in a water filled cooling vessel for cooling down to room temperature. Flash the condensed liquid at the funnel and the flask neck after cooling with 5 ml neutralized alcohol into the flask. Then titrate in the flask against phenolphthalein with 0.5 n potassium hydroxide solution in ethanol.

Conduct a blind test at the same time. The acid value of the investigated fat has to be determined separately.

### 7. Calculation

The hydroxyl value is calculated considering the consummated ml 0.5 n potassium hydroxide solution in main and blind test, the sample acid value, and the weighed portion.

$$OHV = \frac{(b - a) \times 28.05}{E} + AV$$

a = consummated ml 0.5 n potassium hydroxide solution in main test  
 b = consummated ml 0.5 n potassium hydroxide solution in blind test  
 E = sample weight in g

### 8. Remarks

Not verified.

### 9. Changes

Replacing EA.003.01

### 10. Enforcement

Step	Org.-Unit / Site	Name	Date	Signature [on original copy only]
<i>Prepared by</i>	B-CS P (QKL) / Essen	Käseborn	11.11.2002	X X X X X
<i>Checked by</i>	S2-AL / Essen	Fr. Dr. Keune	21.11.2002	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	07.12.2002	

#### Reviewed regarding validity [on original copy only]

Date	Signature	Date	Signature	Date	Signature

**Title: Determination of Acid Value / Free Fatty Acid****1. Normative References / Bibliography**

Similar to:  
DGF C-V 2  
Ph.Eur. 2.5.1  
ISO 3682  
ASTM D 974  
DIN EN ISO 2114

**2. Terms / Definitions**

The acid value (A.V.) indicates the necessary amount of KOH (in mg) to neutralize the content of free acids in 1g product.

**3. Scope**

Dissolve the sample in an appropriate solvent and titrate the present acid with potassium hydroxide solution.

This absolutely non-specific method is not able to distinguish between mineral acids, free and bonded fatty acids, or other organic acids.

Therefore this method includes other attendant mineral acids, which can be determined separately by DGF C-III 14.

**4. Interferences**

not verified

**5. Materials and Reagents**

Solvents: The usually used solvent is a mixture ethanol / toluene (1:1). Other possible solvents are e.g. ethanol or isopropanol.

**Other used solvents has to be documented in an internal working instruction.**

Ethanol (neutralized against phenolphthalein)

Toluene (neutralized against phenolphthalein)

Isopropanol (neutralized against phenolphthalein)

Ethanol / toluene mixture 1:1 (v/v) (neutralized against phenolphthalein)

0.5 N potassium or sodium hydroxide solution in ethanol or in water

0.1 N potassium or sodium hydroxide solution in ethanol or in water

0.02 N potassium or sodium hydroxide solution in ethanol or in water

Solution of phenolphthalein, 1 % in ethanol

(Solution of thymolphthalein, 0.04 % (g/v) in ethanol, 50 % (v/v) – see remarks)

Erlenmeyer flask ca. 200 ml / 300 ml

Appropriate titration apparatus, e.g. Metrohm Dosimat 725 or adequate burettes

Analytical balance

## 6. Procedure

Regarding sample weight see GM\_0001\_01

The sample to investigate is to weigh with 0.1 % accuracy in the Erlenmeyer flask. Add ca. 50 –100 ml of **neutralized solvent** and dissolve the sample, if necessary, with slight heating. Avoid boiling. After adding solution of phenolphthalein, titrate with adjusted potassium hydroxide solution while constant swirling until the color changes consistently.

## 7. Calculation

$$\text{Acid value [mg KOH/g]} = \frac{V \times 56.1 \times N}{E}$$

$$\text{Free fatty acid [\%]} = \frac{V \times \text{MG} \times N}{10 \times E}$$

V = Consummed volume titrant (ml)

N = Normality titrant

E = Sample weight (g)

MG = Molecular weight of fatty acid

## 8. Remarks

Detection of point of change is difficult with dark products, in this cases you can use thymolphthalein instead of phenolphthalein. Add 0.3 ml to the solution.

For very low acid values it is advantageous to flash the flask with nitrogen.

**Deviations to this method should be documented in an internal working instruction and should be validated for each product or product-group.**

Regarding potentiometric determination of acid value see: GM\_0010\_08

### 9. Changes

considered other solvents and alkali hydroxide solutions

### 10. Enforcement

Step	Org.-Unit / Site	Name	Date	Signature <i>[on original copy only]</i>
<i>Prepared by</i>	B-CS P (QKL) / Essen	Käseborn	1.11.05	X X X X X
<i>Checked by</i>	CS P; QKL / Steinau	Hr. Kirschner	3.11.05	X X X X X
<i>Checked by</i>	B-OS P (QKL) / Essen	Dr. Weibels	3.11.05	X X X X X
	/ Pandino	Dr. Sabrina Mazzoli	21.11.05	X X X X X
	/ Milton Keynes	Ratna Morjaria	7.11.05	X X X X X
	/ Granollers	Dolors Montilo	15.11.05	X X X X X
	/ Hopewell	James Calligeros	17.11.05	X X X X X
	/ Mapleton	Charles Davis	N.A.	X X X X X
	/ Jansville	Joanne Schultz	N.A.	X X X X X
	/ Flimby	Brian McAvoy	N. A.	X X X X X
	/ Jakarta	Dr. Sari Kusumawaty	18.11.05	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	7.12.05	
<b>Reviewed regarding validity <i>[on original copy only]</i></b>				
<i>Date</i>	<i>Signature</i>	<i>Date</i>	<i>Signature</i>	<i>Date</i>
				<i>Signature</i>

## Title: Determination of Saponification Value

### 1. Normative References / Bibliography

similar to: DGF C-V 3 and DIN EN ISO 3681  
Ph.Eur. 2.5.6; US P (401) Chemical Tests

### 2. Terms / Definitions

The saponification value (S.V.) is a metric for the amount of free and bonded (fatty) acids in a fat (ester), which can be transferred with lye by saponification in the corresponding soap.

It indicates the necessary amount of KOH (mg) to saponify 1g fat.

### 3. Scope

The sample to investigate is saponified with an excess of ethanolic potassium hydroxide solution. After finished saponification is the remaining excess of potassium hydroxide determined by titration with hydrochloric acid.

### 4. Interferences

Hard to saponify products can deliver too low results. As the case may be use method GM\_0030\_02.

**Usage of alcoholic potassium hydroxide solution in a different composition can deliver differences!**

### 5. Materials and Reagents

Alcoholic potassium hydroxide solution 0.5 n (in 80% ethanol !); .e.g. Fa. Kraft 12168.xxxx or 05554.xxxx  
Phenolphthalein 0.1 % in ethanol  
Hydrochloric acid 0.5 n  
Ethanol (neutralized against phenolphthalein)

Flat bottom flask (250 ml) / round bottom flask (100ml) / Erlenmeyer flask (300ml) with alkali-resistant ground-glass  
Reflux condenser  
Heating device  
Analytical balance  
Pipettes, 25 ml  
Burette, 50 ml, scale 0.1 ml  
Boiling stones

### 6. Procedure

Considering the supposed saponification value (see GM\_0001\_01) weigh a certain amount of ca. 2 g of the sample to investigate in an appropriate flask with +/- 0.005 g accuracy. Add 25 ml 0.5 n ethanolic potassium hydroxide solution. Put some boiling stones in the flask, connect with the reflux condenser and heat with light boiling for 60 minutes. Swirl from time to time.  
After that flash condenser with a small amount of ethanol.  
Some drops of phenolphthalein solution are added to the saponified hot solution. Then titrate with 0.5 n hydrochloric acid until color changing.  
Conduct a blind test under same conditions without sample.

### 7. Calculation

The saponification value is calculated considering the consummated 0.5 n hydrochloric acid in main and blind test and the sample weight.

$$\text{S.A. ( mg KOH / g )} = \frac{(b-a) * 28.05}{E}$$

E= Sample weight product in g

a= Consumption ml 0.5 n Hydrochloric acid in main test

b= Consumption ml 0.5 n Hydrochloric acid in blind test

### 8. Remarks

The titration can be processed in a titration apparatus with potentiometric dead stop determination as well.

### 9. Changes

### 10. Enforcement

Step	Org.-unit / Site	Name	Date	Signature <i>[on original copy only]</i>
<i>Prepared by</i>	B-CS P (QKL) / Essen	Käseborn	10.01.05	X X X X X
<i>Checked by</i>	B-OS P (QKL) / Essen	Dr. Weibels	10.01.05	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	10.01.05	
<b>Reviewed regarding validity <i>[on original copy only]</i></b>				
<i>Date</i>	<i>Signature</i>	<i>Date</i>	<i>Signature</i>	<i>Date</i>
				<i>Signature</i>

## Title: Water Determination by Karl Fischer (Standard Method)

### 1. Normative References / Bibliography

Following DIN 51777, DGF E-III 10, and DGF C-III 13a

### 2. Terms / Definitions

The water content, expressed in percentage by weight, is the amount of water calculated by the following method considering the iodine consumption.

The sample is titrated with a solution containing sulphur dioxide and iodine in presence of methanol. Since sulphur dioxide and iodine react to equivalent amounts of sulphur trioxide and hydrogen-iodine only in presence of water, the water content can be calculated in percentage by weight by the iodine consumption.

#### 1.1 Chemical Equation



### 3. Scope

This method is used to determine the water content of fats, oils, polyethers, and surfactants.

### 4. Interferences

This method can be used for all products without contamination's which could lead to side reactions. Such contamination's are alkaline compounds and peroxides. By conversion with the reagent solution they deliver too high values.

### 5. Materials and Reagents

Unless otherwise noted, use analytical-reagent-grade quality material.

Karl Fischer reagent : Hydranal Composite 5, Riedel de Haen; 34805

**The factor of KF-solution is determined by the laboratory specific testing agent supervision!**

Methanol reagent grade; e.g. Baker ; 2045

Chloroform reagent grade; e.g. Baker;7386

Karl Fischer titration apparatus; (e.g. Metrohm; KF-Titrino 701 or 758 )

Titration stand; Metrohm; 2.703.0010

Changing unit 20 ml with ceramic cock (**for water contents < 10 %** )

Changing unit 50 ml with ceramic cock (**for water contents > 10 %** )

Magnetic stirrer, 25mm; e.g. Metrohm: 6.1903.030

Double-Pt-electrode; Metrohm; 6.0338.100

Analytical balance; e.g. Sartorius AC210S

Drying pistol with activated molecular sieves ; Metrohm; 6.2811.000

The molecular sieves should be changed with every change of titration solution or monthly. The regeneration of the molecular sieves is processed in the drying-oven at 180 - 240°C for 48 hours.

### 6. Procedure

The sample is to homogenize by thoroughly stirring. For this solid fats are melted carefully, it should be taken care, that they are not heated higher than just necessary for melting.

Methanol or methanol / chloroform (1:1 ) are provided in a titration vessel ( fill heigth 1/3 to ¼ of the vessel) and titrated with Composite 5. An adequate sample (GM\_0001\_01 ) is weight with an analytical balance directly or with a disposable syringe by difference weighing in the titration vessel. Titrate to the electrometric dead stop.

**7. Calculation**

$$\frac{V \times F}{10 \times E} = \text{Water content [\%]}$$

V = Consumption Composite 5 solution (ml)  
 F = Factor of Composite 5 solution  
 E = Sample weight (g)

**8. Remarks**

Solvents and instrument parameter are to document in a laboratory specific work instruction.

**9. Changes**

Replacing method EA.04.01 and 28-02 (Goldschmidt Rewo)

**10. Enforcement**

Step	Org.-Unit / Site	Name	Date	Signature <i>[on original copy only]</i>
<i>Prepared by</i>	CS; QKL / Steinau	Kirschner	28.03.2003	
<i>Checked by</i>	B-CS P; QKL / Essen	Käseborn	28.03.2003	
<i>Checked by</i>	S2 AL / Essen	Dr. Keune	28.03.2003	
<i>Checked by</i>	B-OS P SE / Essen	Hörnlein	28.03.2003	
<i>Approved by</i>	CS; QKL / Steinau	Kirschner	28.03.2003	

**Reviewed regarding validity *[on original copy only]***

Date	Signature	Date	Signature	Date	Signature

GM\_0080\_01\_E\_E.doc