

## 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 <i>[on original copy only]</i>
<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	

**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 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 = Consumed 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.

**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	13.2.13	X X X X X
<i>Checked by</i>	CS P; QKL / Steinau	Hufnagel	14.2.13	X X X X X
<i>Checked by</i>	B-OS P (QKL) / Essen	Dr. Weibels	19.2.13	X X X X X
	/ Pandino	Dr. Sabrina Mazzoli	19.2.13	X X X X X
	/ Milton Keynes	Butler	13.02.13	X X X X X
	/ Granollers	Dolors Montilo	14.02.13	X X X X X
	/ Hopewell	Mac Taylor	13.02.13	X X X X X
	/ Mapleton	Charles Davis	22.02.13	X X X X X
	/ Jansville	Joanne Schultz	13.02.13	X X X X X
	/ Flimby	Thompson	13.02.13	X X X X X
	/ Jakarta	Almartoni Toni	20.02.12	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	23.02.13	

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

Date	Signature	Date	Signature	Date	Signature

## 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 or GM\_0030\_04.

Perhaps they are special regulations in laboratory working instructions.

**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 or similar automatic Burettes, e.g. Dosimates

Burette, 50 ml, scale 0.1 ml or similar automatic Burettes, e.g. Dosimates

Boiling stones

### 6. Procedure

Considering the supposed saponification value (see GM\_0001\_01) weigh a certain amount 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.

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.  
For complete neutralization it may be necessary, to heat up the solution again and again, until the color-change is no longer reversible.

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

Description for color changing

### 10. Enforcement

Step	Org.-unit / Site	Name	Date	Signature [on original copy only]
Prepared by	B-CS P (QKL) / Essen	Käseborn	15.04.10	X X X X X
Checked by	B-OS P (QKL) / Essen	Dr. Weibels	15.04.10	X X X X X
Approved by	B-CS P (QKL) / Essen	Käseborn	18.05.10	

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

Date	Signature	Date	Signature	Date	Signature

## 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 heighth 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

**Title: Determination of free i-Amylalcohol in Tegosoft AC****1. Normative References / Bibliography**

not covered

**2. Terms / Definitions**

not covered

**3. Scope**

The content is determined with the gas-chromatographic Headspace-Technique with FID detection. The quantification is processed with a one-point kalibration

**4. Interferences**

not covered

**5. Reagents and Materials**Iso-Amylalcohol  $\geq 99,00\%$ 

Stock solution EO/DX:

Kapillary gas chromatograph with FID and Headspace – sampling device e.g. AutoSystem XL with TurboMatrix 40 by Fa. Perkin-Elmer, Instrumentelle Analytik or comparable devices.

The device has to be equipped with a split system. The Headspace – sampling device must be conceived in that way, that every headspace sample can be tempered for a defined time period.

Analytical column: 30 m Fused-Silicagel-Kapillary Column DB-1 ( cross-linked DMPS )  
e.g. Fa. J. & W. Scientific or ZB-1 of Phenomenex  
Inner diameter: 0.25mm  
Film thickness: 1 $\mu$ m

Ampoules, to close with septum, e.g. 20 ml, suitable for Headspace-GC-device.  
PTFE-coated septi and aluminium caps with safety ring to close the ampoules, e.g. 20 ml  
Bördelkappen by Fa. Perkin-Elmer.  
Closing tongs for ampoules

Analytical balance  
Ultrasonic bath

Integrator or computer with appropriate calculation software.

## 6. Procedure

### 6.1. Calibration

A sample solution will provide of Tegosoft AC or Tegosoft M with 25 ppm Iso-Amylalcohol, which was previously tested on absence of Iso-Amylalcohol.

Before the start of a campaign, this solution is 3\* tested. The average of the peak area will be used for the calculation. The allowed maximum deviation of the 3 runs is 10 %.

The preparation of the sample solution is as follows:

To 100 g Product (free of Iso-Amylalcohol) add 0,01 g Iso-Amylalcohol (=100 ppm). This mixture has to be sealed and well homogenized.

25 g of the product which contains 100 ppm Iso-Amylalcohol is diluted to 100 g with Iso-Amylalcohol free product. (25 ppm) Good homogenization is recommended.

- Standard Solution (25 ppm)

### 6.2. Process of Determination

Weigh 0,1 sample in a Headspace – bottle weigh with 1 mg accuracy.

### 6.3. Chromatographic Conditions

#### Temperature of the Solutions to investigate and Dosages:

Thermostatting time: 45 min.  
Thermostatting temperature: 70 °C

#### Head-Space Conditions:

Needle temperature: 100°C  
Temperature of transfer pipe: 140°C  
Pressure build up time: 0.5 min.  
Analytical time: 0.06 min.  
High pressure pre-pressure: 100 kpa

#### Gas chromatographic Conditions:

Oven: 60°C (5 min. isoth.), then from 60 till 120°C with 8°C/min  
Hold for 5 minutes, then with 25°C/min to 255°C and hold temperature for 5 minutes.

Detector: FID: 300°C  
Injector: 170°C

Carrier gas: Helium, approx. 1 ml/min  
 Column pre-pressure approx. 70 kpa  
 Split: 1 : 5 (2 ml/min)  
 Hydrogen and air ( for FID ) optimized  
 Attenuation -6

**7. Calculation**

The measurement results for the individual signals are reported as peak area in integrator units, e.g. in mV.

The content of free Iso-Amylalcohol is calculated as follows:

$$W \text{ (Iso-Amylalcohol)} = \frac{25 \times \text{Peak area B}}{\text{Peak area A}}$$

Peak area A = Peak area of Iso-Amylalcohol Standard Solution (25 ppm)  
 Peak area B = Peak area of Iso-Amylalcohol of the sample

W (Iso-Amylalcohol ) = Amount free Iso-Amylalcohol in ppm ( µg/g )

**8. Remarks**

not covered

**9. Changes**

not covered

**10. Enforcement**

Step	Org.-Unit / Site	Name	Date	Signature <i>[on original copy only]</i>
<i>Prepared by</i>	CU-CS-PT-ESS-TS	Eulenbach	23.07.2013	X X X X X
<i>Checked by</i>	CU-CS-PT-ESS-TS	Käseborn	23.07.2013	X X X X X
<i>Approved by</i>	CU-CS-PT-ESS-TS	Käseborn	23.07.2013	see german edition

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

Date	Signature	Date	Signature	Date	Signature

GM\_0623\_01\_E\_E

## Title: Refraction Index

### 1. Normative References / Bibliography

Following DIN 51423 and DGF C-IV 5 and USP (831) Chemical Tests.

### 2. Terms / Definitions

The relative index of refraction  $n$  is the relation of the speed of the light in the air and in the material to investigate. He can be also defined as a relation of the sine of light angle of the incidence to the sine of the light angle of refraction when the light enters from the air into the material to investigate. The absolute index of refraction of a substance is referred to the empty space. He can be calculated from the relative value by multiplication with the factor 1.00027.

### 3. Scope

Some drops of a liquid product are brought on the measuring prism of the refractometer. Wait until measuring prism and product have the same temperature and read off the index.

### 4. Interferences

Not verified.

### 5. Reagents and Materials

1. Abbe- Refractometer

Water bath, controlled with thermostat

Light source: Sodium-spectral lamp, tungsten filament lamp, day light

2. PTR-Refractometer (e.g. Fa. Index Instruments Ltd.)  
with build-in Peltier- element

### 6. Procedure

1. 1. The sample to investigate is applied on the clean measuring prism. The lighting prism is closed carefully. In the Abbe- Refractometer prism and sample are irradiated with the source of light. The temperature balance is carried out with the thermostat.

After one till two minutes the bright-darkness border is adjusted achromatically and sharply in the observation field on the crosshair of the refractometer. If the so adjusted scale value does not change anymore, this is red off.

2. At first the PTR- refractometer is adjusted at the temperature given in the test plan. When this is reached, some drops of the sample to investigate are applied on the prism and the lids are closed. If the value of the index of refraction on the display does not change anymore it is red off.

The prism and the lid of the refractometer are cleaned after every measurement with a suitable solvent (e.g., ethanol or water) and a soft cloth.

### 7. Calculation

The value red off is documented with 4 decimal places in SAP.

### 8. Remarks

Not verified.

### 9. Changes

New Evonik Layout.

### 10. Enforcement

Step	Org.-Unit / Site	Name	Date	Signature <i>[on original copy only]</i>
<i>Prepared by</i>	CU-CS-PT-ESS-SE	Dr. Weibels	14.04.2010	X X X X X
<i>Checked by</i>	CU-CS-PT-ESS-SE	Hr. Goetz	14.04.2010	X X X X X
<i>Approved by</i>	CU-CS-PT-ESS-SE	Dr. Weibels	14.04.2010	

#### 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>