

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. Interference's

Not verified.

5. Materials and Reagents

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

additional reference in point 1.

10. Enforcement

Step	Org.-Unit / Site	Name	Date	Signature <i>[on original copy only]</i>
<i>Prepared by</i>	B-OS P SE / Essen	Hr. Götz	10.03.2005	X X X X X
<i>Checked by</i>	B-CS P / Essen	Hr. Käseborn	11.03.2005	X X X X X
<i>Approved by</i>	B-OS P SE / Essen	Dr. Weibels	11.03.2005	

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: Density Determination

1. Normative References / Bibliography

similar to DIN 51757 ; Ph. Eur: 2.2.5 and USP (841) Chemical Tests

2. Terms / Definitions

The density of a material is the quotient from masses and volume.

3. Scope

Determination of the density of liquid products

The determination can be carried out with three different procedures:

- a) with the flexible swing
- b) with the spindle (Aerometer)
- c) with the pycnometer

4. Interferences

Not verified.

5. Materials and Reagents

5.1. For determination with the flexible swing:

Density measuring instrument e.g. DMA 48, Fa. Anton Paar KG
One-way syringe 10 ml with Luer – Konus,
Abatement container e.g. beaker 600 ml,
Water p. a. Fa. Merck Art. 16754
Alcohol for cleaning

5.2. For determination with the spindle:

Spindle with density scale, calibrated at 20 °C
Plain cylinder
Thermometer

5.3. For determination with the pycnometer:

Pycnometer
Thermometer
Analytical balance

6. Procedure

6.1. For determination with the flexible swing:

To get valid measuring results, the density measuring instrument is to be calibrated basically.

The sample to be measured is brought by a plastic syringe free of air bubbles through the pour in device in the swing. Also in the syringe no air bubbles should be. The necessary volume amounts are approximately 0.7 ml. The filling process can be observed by the show glass with switched on lighting. The filling is finished when the liquid thread has transgressed the opposite recumbent thickening of the swing pipe. Leave the syringe in the pour in device. The lighting has to be switched off to hold the temperature on a constant level. After reaching the period duration stability (light beam expires) the value is indicated on the display.

After every measurement the measuring cell is to be cleaned carefully and to dry. The drying is processed with the built-in air pump.

6.2. For determination with the spindle:

The product is tempered on 20 °C, filled in a plain cylinder free of bubbles and the spindle is dipped carefully. So that the spindle does not get stuck in the plain cylinder, move her in slow rotation and then read the scale, while looking levelly above the surface of the liquid.

6.3. For determination with the pyknometer:

The clean and well dehydrated pyknometer is weighed on the analytical balance with 1 mg accuracy (weight C), is filled with the sample carefully and free of bubbles, tempered on 20 °C, and weighed out (weight A). After thorough cleaning of the pyknometer it is filled with water, tempered on 20 °C, and weighed out (weight B).

7. Calculation

7.1. For determination with the flexible swing

The density can be read off directly at the device.

7.2. For determination with the spindle

Red off scale value = Density [g/ml]

7.3. For determination with the pyknometer

$$\frac{\text{Mass (m)}}{\text{Volume (V)}} = \text{Density [g/ml]}$$

m = Weight A - weight C

V = Weight B - weight C; with density of water at 20° = 1.00 g/ml dimension is ml!

8. Remarks

- 8.1. The information of the density following this method occurs with max. 4 decimal places.
8.2. To avoid the risk of glass break of the measuring cell of the flexible swing by improper filling in of the sample, the provided pour in device must be used always. Basically only syringes with Luer-Konus are to be used.

9. Changes

Adding some references in Point 1

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	10.03.05	X X X X X
<i>Checked by</i>	S2 AL / Essen	Fr. Dr. Keune	10.03.05	X X X X X
<i>Checked by</i>	CS P; QKL / Steinau	Hr. Kirschner	10.03.05	X X X X X
<i>Checked by</i>	B-OS P (QKL) / Essen	Dr. Weibels	10.03.05	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	10.03.05	

Reviewed regarding validity *[on original copy only]*

Date	Signature	Date	Signature	Date	Signature

Title: Solidification Point at the Rotating Thermometer**1. Normative References/ Bibliography**

ISO 2207
DGF M-III 4a

2. Terms / Definitions

Solidification point: The temperature, at which a melted product starts to solidificate.

3. Scope

The solidification point at the rotating thermometer is the temperature, at which a melted sample stops to flow under the conditions of the method described below. It is an important characteristic for the investigation of petrolatum's and ceresins, i.e. of microcrystalline paraffin's and paraffin slack wax's as well, but can also be used to investigate all the rest of the wax's.

Abbreviation: SP. (rot.)

4. Interferences

Not identified.

5. Reagents and Material

To determine the solidification point up to 80 °C use a thermometer with a temperature range from 0 till 100 °C, divided in steps of 0.5 °.

To determine solidification points above 80 °C use a thermometer with a measurement range of 50 till 150 °C.

Additional: Length 300 mm (+- 0.5 mm), diameter 6.5 mm (+- 0.5 mm).

A one-sided closed glass protective cover with a diameter of 25 mm and 55 mm length can be put over the lower part of the thermometer. It is fixed with a penetrated cork, placed in the appropriate height at the thermometer.

6. Procedure

The sample is heated carefully up to a clear melting and slowly adjusted to a temperature approx. 20°C above the solidification point while stirring. If there is no clear melting up to temperatures, where decomposition could start, report this in the results.

The protection cover is put so far over the lower part of the thermometer, that the end of the mercury ball has a distance of 10 to 15 mm to the ground. Then heat it in the water bath, until the thermometer displays approximately the same temperature as the melted sample. Now take a sample plug with the thermometer by dipping the mercury ball completely into the sample. After that put the pre-heated protection cover immediately over the thermometer again. For that the thermometer should be hold strictly horizontal. Avoid, that a part of the taken sample sticks at the thermometer shank. The thermometer is rotated once in two seconds in horizontally around its longitudinal axis. Hereby watch

the state of the drop, which starts to rotate after some time. As soon as this has started, read off the temperature. This is the solidification point, determined under these conditions.

The solidification point at the rotating thermometer is reported with 0.5°C accuracy.

7. Calculation

Not identified; see procedure

8. Remarks

Not identified.

9. Changes

Replacing EA.021.01

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	18.01.04	X X X X X
<i>Checked by</i>	B-CS P (QKL) / Essen	Käseborn	18.01.04	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	3.2.04	

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

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	

Reviewed regarding validity [on original copy only]					
Date	Signature	Date	Signature	Date	Signature

Title: Hanus Iodine Value**1. Normative References / Bibliography**

DGF C-V 11 a (53)
Ph. Eur. 2.5.4 (Method A)

2. Terms / Definitions

The iodine value (I.V.) indicates how many grams halogen, calculated as iodine, are bonded by 100 g sample material under the conditions of these method.

3. Scope

The iodine value is a metric for the amount of double bonds in a product. Halogen compounds (iodine monobromide) can be added to these double bonds. The excess of iodine monobromide is transferred to iodine by addition of potassium iodine. This is determined by titration.

4. Interference's

Different test conditions can lead to different results!

5. Materials and Reagents

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

Iodine monobromide solution (dissolve 10 g iodine monobromide in 500 ml pure acetic acid) ;
Hanus iodine solution (e.g. Fa. Kraft 05072.xxxx)

Chloroform

Iodine monobromide

Potassium iodine

Acetic acid (conc; min. pure)

Potassium iodide – solution 25 % in water

Distilled water

Sodium thiosulphate – standard solution $c = 0.1 \text{ mol/l}$ (0.1 N) (e.g. Fa. Kraft 03067.xxxx)

Soluble starch

Starch solution (ca. 1% in water)

Pipettes (appropriate dosing mechanism, resp.)

(Automatic) burettes

6. Procedure

The weight portion has to conform with GM_0001_01.

The product is weight in a 300 ml Erlenmeyer flask and dissolved in 10 - 20 ml chloroform, if necessary under warming up. After cooling to room temperature add of 25 ml iodine monobromide solution, close the flask and sway shortly. The flask is abandoned in the dark for 1 hour. Then add 10 ml potassium iodide solution, approx. 100ml water and titrate with sodium thiosulphate solution until light yellow color; after adding starch until white color.
A blind test is processed at the same time without sample.

7. Calculation

The iodine value is calculated considering the consummated ml 0.1 N sodium thiosulphate solution in main and blind test and the sample weight.

$$\text{I.V.} = \frac{(b-a) \times 1,269}{E}$$

a = Consumption ml 0.1 N sodium thiosulphate solution in main test

b = Consumption ml 0.1 N sodium thiosulphate solution in blind test

E = Sample weight in g

8. Remarks

not verified

9. Changes

it's necessary to cool to room temperature, before adding of iodine monobromide solution

10. Enforcement

Step	Org.-Einheit / Standort	Name	Date	Signature [on original copy only]
<i>Prepared by</i>	B-CS P (QKL) / Essen	Käseborn	16.12.04	X X X X X
<i>Checked by</i>	B-OS P (QKL) / Essen	Dr. Weibels	16.12.04	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	16.12.04	

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 [on original copy only]
<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	
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 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	

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

Title: GC-Fingerprint of Products (e.g. Tegosoft)**1. Normative References / Bibliography**

Not identified.

2. Terms / Definitions

Not identified.

3. Scope

A general gas chromatographic method to characterize products; Determination of side products, purity determinations resp.

4. Interferences

Not verified.

5. Reagents and Materials

Toluene reagent grade quality (e.g. Fa. Baker Ident.No. 8077)

Analytical balance

Sample bottles 1.5 ml (e.g. Fa. Zinsser Ident.No. 3088001)

Gas chromatograph e.g. Chrompack 9000 with adequate calculation system

6. Procedure

Approx. 0.03 g sample are dissolved in 1.5 ml toluene reagent grade quality and a chromatogram is measured with the conditions mentioned below.

Gas chromatographic conditions:

Injection temperature:	300 °C
Detector temperature:	365 °C
Oven temperature (start):	50 °C / hold for 2 min
Temperature program:	10 °C /min; hold on 365 °C/8 min
Column pre pressure:	50 kPa
Carrier gas:	Helium
Splitflow:	85 ml /min
Column gas flow:	4,23 ml/min
Splitflow proportion:	20,09
Separation column:	WCOT Fused silica Sim - Dist CB 10 m; 0.32 mm inner diameter (e.g. Fa. Chrompack Ident.No. 7521)
Detector type:	FID
Injection volume:	0.5 µl

7. Calculation

The calculation is processed using the area percents. Since response factors are not considered, this does not correspond to the actual composition. You only may compare chromatograms which are created with the same conditions.

8. Remarks

Not identified.

9. Changes

Correction Point 7: Gas flow parameters

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	17.09.2005	X X X X X
<i>Checked by</i>	B-CS P (QKL) / Essen	Käseborn	17.09.2005	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	17.09.2005	

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