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 achromaticly 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** *[on original copy only]*
Prepared by	B-OS P SE / Essen	Hr. Götz	10.03.2005	X X X X X
Checked by	B-CS P / Essen	Hr. Käseborn	11.03.2005	X X X X X
Approved by	B-OS P SE / Essen	Dr. Weibels	11.03.2005	

Reviewed regarding validity *[on original copy only]*

Date	*Signature*	*Date*	*Signature*	*Date*	*Signature*

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 pyknometer

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 pyknometer:

Pyknometer
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 red 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** *[on original copy only]*
Prepared by	B-CS P (QKL) / Essen	Käseborn	10.03.05	X X X X X
Checked by	S2 AL / Essen	Fr. Dr. Keune	10.03.05	X X X X X
Checked by	CS P; QKL / Steinau	Hr. Kirschner	10.03.05	X X X X X
Checked by	B-OS P (QKL) / Essen	Dr. Weibels	10.03.05	X X X X X
Approved by	B-CS P (QKL) / Essen	Käseborn	10.03.05	

Reviewed regarding validity *[on original copy only]*

Date	*Signature*	*Date*	*Signature*	*Date*	*Signature*

Title: Gardner – Color Index (Komperator)

1. Normative References / Bibliography

DGF - C - IV - 4c; DIN ISO 4630

2. Terms / Definitions

The color index is defined as number of that color glass, which is most similar to the color of the sample.

3. Scope

The following method serves for the determination of products which can be measured as liquids. Small opacities are allowed to occur.
The color of the sample is compared in translucent light to standard stained glasses. The comparison series consists of 18 consecutively numbered stained glasses whose color depth increases with rising identity number.

4. Interferences

Not verified.

5. Materials and Reagents

Komperator with lightning and color panes with standard stained glasses according the Gardner series.
Lovibond 2000 (Fa. Tintometer) or comparable devices.

11 mm round cuvettes (e.g. Fa. Lange) or adequate rectangle 10 mm cuvettes

5. Procedure

Solid fats are investigated melted.

The sample to investigate is filled in an **adequate cuvette**. By comparison with the stained glasses in the translucent light that one is determined, which shows the highest conformity with the sample color.

6. Calculation

As Gardner – Color Index the number of the determined standard stained glass is documented. If the sample has a color depth deeper than the measurement range of the scale, the color index is noted as deeper than 18.

8. Remarks

Not verified.

9. Changes

Replacing EA. 08.02 and 05-01 (Goldschmidt Rewo)

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	B-CS P (QKL) / Essen	Käseborn	10.11.03	X X X X X
Checked by	CS -STN- QKL, Steinau	Kirschner	10.11.03	X X X X X
Checked by	B-OS P SE / Essen	Dr. Weibels	11.11.03	X X X X X
Approved by	B-CS P (QKL) / Essen	Käseborn	11.11.03	

Reviewed regarding validity *[on original copy only]*

Date	*Signature*	*Date*	*Signature*	*Date*	*Signature*

Title: Recrystallization

1. Normative References / Bibliography

According to DIN ISO 3841

2. Terms / Definitions

Recrystallization or melting point (cooling curve) is that temperature, at which the melted sample shows a temperature holding line for the first time during cooling down.

3. Scope

The determination is applicable for mineral oil paraffin's and silicon waxes.

4. Interferences

The method is not applicable for microcrystalline paraffin's, mixtures of these compounds with macro crystalline paraffin waxes or scale wax.

5. Reagents and Materials

Test vessel; chemically resistant glass with outer diameter 25 - 30 mm, length of approx. 200 mm, bottom end half-rounded.
Stir thermometer up to 100 °C with 1 °C scale division

5. Procedure

The sample to investigate is filled in the glass vessel up to a height of approx. 4 cm.
Now heat from outside with hot water while stirring with the thermometer up to approx. 10 °C above the expected recrystallization point. Subsequently cool down with tap water while stirring. When the recrystallization point is reached, the temperature stops.

7. Calculation

The temperature of the holding point is documented as recrystallization in SAP.

8. Remarks

Not identified.

9. Changes

First edition.

10. Enforcement

Step	Org.-Unit / Site	Name	Date	Signature *[on original copy only]*
Prepared by	B-OS P SE / Essen	Hörnlein	05.11.2002	X X X X X
Checked by	B-OS P SE / Essen	Dr. Weibels	05.11.2002	X X X X X
Approved by	B-OS P SE / Essen	Dr. Weibels	05.11.2002	

Reviewed regarding validity *[on original copy only]*

Date	*Signature*	*Date*	*Signature*	*Date*	*Signature*