

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 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 = 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.

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

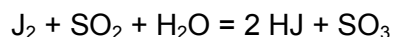
Following DIN 51777, DGF E-III 10, and DGF C-III 13a

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