

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: 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	0.60
	10	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: 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 <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: Rise Melting Point**1. Normative References / Bibliography**

DGF-C-3a (52)

Ph.Eur. 2.2.15

2. Terms / Definitions

Not identified.

3. Scope

This method determines the melting point of a product in an open capillary in a water bath.

4. Interferences

Not identified.

5. Reagents and Materials

Heated magnetic stirrer

Magnetic stirrer rod 4 cm * 7 mm

600 ml beaker, low form

Bi-stable thermometer

„Checked liquid thermometer, measurement range:-10°C till +150°C „

(Fa.Morgenbrot, Oberhausen)

Glass capillaries, open on both sides, diameter: 1.0 mm / length: 80 mm

(e.g. Fa. Hirschmann Laborgeräte)

Rubber ring, rubber hose, length 2-3 mm, inner diameter 6 mm

Distilled water

6. Procedure

Preparation of glass capillaries:

One end of the capillary is filled in a range of 0.5 - 1 cm with the fatty compound (in liquid state) and stored for 2 hours at +5°C.

The capillary is fixed with the rubber ring at the thermometer in that way, that the compound column (lower capillary end) is on the same level as the mercury ball of the thermometer.

Thermometer and capillary should dip so far into the determination bath, that the lower capillary end is 4 cm under the liquid surface.

The determination bath is a heated magnetic stirrer with beaker.

The magnetic stirrer creates an equal temperature distribution.

The bath is tempered in that way, that 10°C under the expected soar melting point the temperature increases by 1°C per minute.

The stirrer speed should be too low to create suction.

7. Calculation

The temperature is red off, when the sample becomes liquid and soars in the capillary.

8. Remarks

Not identified.

9. Changes

Replacing analytical method EA.09.01

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	02.12.2002	X X X X X
<i>Checked by</i>	B-CS P (QKL) / Essen	Käseborn	02.12.2002	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	02.12.2002	

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

Title: Saponification Value of Hard to Saponify Products; Butanol

1. Normative References / Bibliography

According to DGF C-V 3 and DIN EN ISO 3681

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 into 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 the remaining excess of potassium hydroxide is determined by titration with hydrochloric acid.

Adding of butanol as solvent facilitates the hydrolysis of hard to saponify products.

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 different results!**

5. Materials and Reagents

Alcoholic potassium hydroxide solution 1 n (in 90% ethanol !); .e.g. Fa. Kraft 05083.xxxx

Phenolphthalein 0.1 % in ethanol

Hydrochloric acid 1 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

n-Butanol

6. Procedure

Considering the expected saponification value (see GM_0001_01) approx. 2 g of the sample to investigate are weighed into a 100 ml round bottom flask with +/- 0.005 g accuracy. 25 ml 1 n alcoholic potassium hydroxide solution and 20 ml n-butanol are added. 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. Subsequently flush the flask with ethanol.

Some drops of phenolphthalein solution are added to the saponified hot solution. Then titrate with 1 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 1 n hydrochloric acid in main and blind test and the sample weight.

$$\text{S.V. (mg KOH / g)} = \frac{(b-a) * 56.109}{E}$$

E= Sample weight product in g

a= Consumption ml 1 n hydrochloric acid in main test

b= Consumption ml 1 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

Hint

10. Enforcement

Step	Org.-Unit / Site	Name	Date	Signature [on original copy only]
<i>Prepared by</i>	B-CS P (QKL) / Essen	Käseborn	25.02.04	X X X X X
<i>Checked by</i>	B-CS P (QKL) / Essen	Käseborn	25.02.04	X X X X X
<i>Approved by</i>	B-CS P (QKL) / Essen	Käseborn	02.03.04	

Reviewed regarding validity [on original copy only]

Date	Signature	Date	Signature	Date	Signature