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]*
Prepared	B-CS P (QKL) / Essen	Käseborn	23.09.2002	X X X X X
Checked by	CS P; QKL / Steinau	Hr. Kirschner	23.09.02	X X X X X
Checked by	B-OS P (QKL) / Essen	Dr. Weibels	23.09.02	X X X X X
Approved by	B-CS P (QKL) / Essen	Käseborn	19.11.02	

Reviewed regarding validity *[on original copy only]*

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

Title: pH-Value in Isopropanol / Water

1. Normative references / bibliography

Following DIN EN 1262, DIN 53909, DGF H-III 1, ISO 4316 und Ph.Eur. 2.2.3

2. Definitions

The pH value is the negative decade logarithm of the hydrogen-ion concentration.

3. Scope

As indicated in the test plan

4. Interferences

not covered

5. Reagents and materials

pH-meter with glass electrode
Deionized water or distilled water (boiled)
Isopropanol
Baker, 100 – 400 ml
Magnetic stirrer with stirring rod
Balance with min. 0.1 g accuracy

Isopropanol / Water Mixture 50 / 50 (w/w)

6. Procedure

According to the test plan the product to be investigated is stirred homogeneously in Isopropanol / Water if necessary under warming. After that it is cooled down on room temperature in the chill washbasin under stirring.
The measurement has to be processed at 20 - 25 °C.
The clean glass electrode is immersed in the test solution. One minute after reaching a constant measuring value, record result
Dilutions from 0.5 to 50% are possible.

7 Calculation

8. Remarks

Not covered

9. Changes

Replacing old testing method EA.006.15

10. Enforcement

Step	**Org. – Unit / site**	**Name**	**Date**	**Signature** *[on the original only]*
Prepared by	B-CS P / Essen	Käseborn	25.08.04	X X X X X
Checked by	PC-CS-QKL-STN	Kirschner	27.08.2004	X X X X X
	S2 AL - Essen	Fr. Dr. Keune	27.08.04	X X X X X
Approved by	B-CS P / Essen	Käseborn	27.08.04	See GM_0133_01_G_E

Reviewed regarding validity *[on the original only]*

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

Title: Active Content, Cat. SO_3 Acid Level

1. Normative References / Bibliography

According to DGF H-III 10a (92)

2. Terms / Definitions

Not identified.

3. Scope

The cation active compound is titrated with a sodium lauryl sulphate standard solution in a 2-phase system created from water and chloroform using a mixed indicator made of dimidium bromide and disulfine blue.
The anionic color disulfine blue creates a Ion pair with the cation active compound, and the cationic color dimidium bromide with sodium lauryl sulphate as well.
At the beginning the blue color of the ion pair, created by the cation active compound with disulfine blue dominates the chloroform phase. Since the Ion pair created by lauryl sulphate and the cation active compound is stronger, the disulfine blue-anion is displaced in the ion pair with the cation active compound with ongoing progression of the titration. The excess of sodium lauryl sulphate reacts with the present dimidium bromide cation at the equivalence point to a ion pair with pink color in chloroform.
The equivalence point is indicated by a gray combination color created by disappearing blue color and upcoming pink color, created by the increasing excess of sodium lauryl sulphate.
A clear pink color indicates over titration!
With this method the cation active compounds in raw materials and formulations are determined.

4. Interferences

All materials, which can create a stable ion pair with quaternary compounds, interfere. The most powerful known ion pair creators are anionic surfactants.

5. Reagents and Materials

- Analytical balance
- Beakers
- Measuring flasks 100, 250, and 1000 ml
- Shaking cylinder 100 ml
- Pipettes 10, 20, and 25 ml
- Dispenser 10 and 15 ml
- Dosimat e.g.: Metrohm 775
- Hyamin 1622, 0.004 M solution – the solution factor is guaranteed between 0.995 and 1.005.
- Dodecyl sulphate sodium salt min 99% - NLS
- NLS solution, 0.004 M - 1.154 g NLS weigh in exactly, flush quantitatively in a 1000 ml measuring flask and fill up to the check mark with distilled water or ready-to-use solution resp.
  The solution factor is determined against the hyamin solution.
- Dimidium bromide
- Disulfine blue VN 150
- Ethanol
- Distilled water
- Sodium chloride

- Mixed indicator solution: Weigh in exactly 0.5 g dimidium bromide in a 50 ml beaker. Weigh in exactly 0.25 g disulfine blue in a second 50 ml beaker. Dissolve both indicators in approx. 25 ml 10 % ethanol V/V and transfer by flushing into a 250 ml measuring flask. Fill up to the check mark with 10 % ethanol V/V.
- Sulphuric acid 98 % for analysis
- Sulphuric acid 2.5 m – 245 g, 134 ml resp. sulphuric acid are stirred in 300 ml distilled water, and transferred into a 1 l measuring flask. Fill up to the check mark with distilled water.
- Acid indicator solution: 200 ml distilled water, 20 ml mixed indicator solution and 20 ml 2.5 m sulphuric acid are given in a 500 ml measuring flask, mixed and filled up with water to the check mark.
  Protect against direct sunlight!
- Chloroform for analysis

6. Procedure

6.1 Standard procedure

Both procedures, A and B are possible and have the same result.

A. An adequate sample weight (GM_0001_01) is weighed in a 250 ml beaker and dissolved in 150 ml hot distilled water. After transfer into a 1000 ml measuring flask fill up to the check mark with distilled water at 20 °C. 25 ml of that solution are pipetted in a 100 ml shaking cylinder.
B. An adequate sample weight (GM_0001_01) is weighed in a 100 ml measuring flask and dissolved in 2 ml n-butanol with light heating. The measuring flask is filled up to the check mark with distilled water at 20 °C. 10 ml of that solution are pipetted in a 100 ml shaking cylinder.

10 ml distilled water, 10 ml acid indicator solution and 15 ml chloroform are added to the aliquot part.
Then titrate with 0.004 M NLS-solution until the dead stop.
The dead stop is reached, when the chloroform phase color is changing from gray-blue to pink.
A clear pink color indicates over titration!

6.2 Procedure with more acid indicator and sodium chloride

An adequate sample weight (GM_0001_01) is weighed in a 250 ml beaker and dissolved in 15 ml ethanol. Add 150 ml warm distilled water. Transfer to a 500 ml measuring flask fill up to the check mark with warm distilled water. 5 ml of that solution are pipetted in a 250 ml shaking cylinder.

10 ml distilled water, 25 ml acid indicator solution and 15 ml chloroform are added to the aliquot part.
If indicated in the product instructions, 0.7 – 1.3 g sodium chloride is added.
Then titrate with 0.004 M NLS-solution until the dead stop.
The dead stop is reached, when the chloroform phase color is changing from gray-blue to pink.
A clear pink color indicates over titration!

7. Calculation

$$\frac{V \times n \times f \times MW \times ap \times 100}{E \times 1000} = \text{Active content \%}$$

$$\frac{V \times n \times f \times ap}{E} = \text{Active content meq/g}$$

$$\frac{V \times n \times f \times 80 \times ap \times 100}{E \times 1000} = \text{Cat } SO_3 \text{ \% acid level}$$

$$\frac{\text{Active \\% } \times 10}{MW} = \text{Active content meq/g}$$

$$\frac{\text{Active content meq/g} \times MW}{10} = \text{Active \%}$$

V = Consumption 0.004 m NLS-solution (ml)
MW = Molecular weight of WAS
ap = Aliquot part
n = Normality of NLS-solution
f = Solution factor

8. Remarks

Not identified.

9. Changes

Second edition, new procedure.

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	CU-CS-STN-QKL/Steinau	Christ	20.06.06	X X X X X
Checked by	CU-CS-PT-TS/Essen	Käseborn	20.06.06	X X X X X
Checked by	CU-CS-NA/Janesville	Schultz	20.06.06	X X X X X
Checked by	CU-CS-STN-QKL/Steinau	Kirschner	20.06.06	X X X X X
Approved by	CU-CS-STN-QKL/Steinau	Kirschner	20.06.06	

Reviewed regarding validity *[on original copy only]*

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

Title: Appearance

1. References / bibliography
Patterned acc. to DGF C-II 1

2. Definitions
Not documented

3. Scope
The appearance of a product is tested visually.
The test temperature and the appearance are described in the product specification.

4. Interferences
Not documented

5. Reagents and Materials
As test-vessel translucent and spotless beakers and sample bottles are applied.

6. Procedure
The appearance-test is performed at the required temperature and compared with the description of the product specification.

Conformity is noted with an „OK“.

Is there no conformity with the description of the product specification it is noted with a „NOT OK“ and described detailed (e.g.: opaque, turbid, turbid with a settle, test sample is separated, test sample contains particles etc.).

In the case of doubts standard samples of the production are applied as a reference. The age of this reference-sample is limited on max. 6 months.

7. Calculation
The result is noted on the analysis form.

8. Remarks
Not documented

9. Changes
The analytical method GM_0170_00_E_E substitutes the analytical methods 48-01, SM0008 and EA 051.01 of Goldschmidt and Goldschmidt Rewo.

10. Approvals

step	**org.-unit / location**	**name**	**date**	**signature** *[on the original only]*
prepared by	CS P; QKL / Steinau	Christ	26.09.02	
checked by	B-CS P; QKL / Essen	Käseborn	26.09.02	
checked by	B-OS SE / Essen	Dr. Weibels	26.09.02	
approved by	CS P; QKL / Steinau	Kirschner	26.09.02	

Reviewed regarding validity *[on the original only]*

date	*signature*	*date*	*signature*	*date*	*signature*