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: Solids Content

1. Normative References /Bibliography
Not verified.

2. Terms / Definitions
The solids content by this method is defined as : 100 % - % water content

3. Scope
Not verified

4. Interferences
Not verified.

5. Materials and Reagents
Not verified.

6. Procedure
See methods for the individual parameters

7. Calculation
% Solids content = 100 % - % H2O

8. Remarks
Not verified.

9. Changes
New edition.

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	B-CS P (QKL) / Essen	Käseborn	02.12.2002	X X X X X
Checked by	CS P; QKL / Steinau	Hr. Kirschner	11.12.2002	X X X X X
Approved by	B-CS P (QKL) / Essen	Käseborn	11.12.2002	

Reviewed regarding validity *[on original copy only]*

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

Title: Determination of the Betaine Content in Alkylamidobetaines

1. Normative References / Bibliography

Not identified.

2. Terms / Definitions

The concentration is mentioned in weight percent. Therefore the molecular mass of the betaine has to be known, otherwise a typical molecular mass should be assumed. For a cocoamidopropylbetaine the determination can be based on a molecular mass of 349.

3. Scope

The betaine sample is adjusted to an alkalic level with sodium hydroxide solution. Through this procedure all present compounds in the betaine are transferred into a defined form:
a) The betaine occurs as an inner molecular neutral form,
b) amido amine occurs as free amido amine,
c) acids (e.g. hydrochloric acid, fatty acids, mono and dichloro acetic acid, formic acid, and glycol acid) are present as sodium salts. During titration with perchloric acid solution in an anhydrous medium the
a1) betaine is transferred into the protonized form,
b1) amido amine is transferred into an amido amineperchlorate
c1) excess of sodium hydroxide solution and sodium salts of the different acids are transferred into the less dissociated sodium perchlorate. By choosing a solvent mixture which enables to distinguish between different pK (b) – values on a very high level, it is possible to separate the betaine from all accompanying compounds.

4. Interferences

Not verified.

5. Reagents and Materials

Titration apparatus (Metrohm; 716 DMS Titrino, version 716.0021 ! or higher or 736 GP Titrino with corresponding printer)

For both devices the same titration conditions are valid! (see 6.11.3 and 7.1)

Changing unit 20 ml with ceramic cock, syringe with micro valve (Metrohm 6.3013.223)
or changing unit 50 ml (Metrohm 6.3013.253)
Using the 50 ml changing unit has the following advantages regarding handling:
a) Shorter titration time;
b) Higher accuracy regarding dosing without second fill up out of 20 ml burette during titration.

Separate pH-electrode (Metrohm 6.0133.100)
LL-Reference electrode (Ag/AgCl-electrode Metrohm 6.0733.100)
Measuring flask, 1 l (brown glass)
Heating plate / magnetic stirrer
Titration stand (Metrohm Titration stand 727 ; 2.727.0010)

Rod stirrer (Metrohm 2.722.0010)

Potassium hydrogen phtalate primary standard (primary standard Merck Art.Nr. 1.02400.0080)

1.4 Dioxan reagent grade quality } alternatively: 0.1 molar perchloric acid in dioxan
Fixanal 0.1 M perchloric acid } Fa. Bernd Kraft; Art.Nr. 11370.3700;

Preparation of 0.1 M Perchloric Acid Solution in Dioxan

Prepare approx. 500 ml dioxan in a 1000 ml **brown glass-**measuring flask. Put on the fixanal - ampoule and open it using a glass rod, flush it and dilute up to 1000 ml with dioxan.

A solution prepared like this is stable for minimum 2 months.

Titer determination of 0.1 m perchloric acid solution in dioxan

The titer has to be determined for each new preparation of a new solution or using a new delivered batch.

0.2 g potassium hydrogen phtalate are weighed into the titration beaker with 0.1 mg accuracy. Add 40 ml methanol and 60 ml EGMME and stir with slight heating until the compound is solved completely. Subsequently cool down to room temperature and titrate with the Titrino against 0.1 m perchloric acid solution in dioxan.

A Titrator Settings for 20 ml Changing Unit

Use the following settings for the Titrino:

Titration parameter		**Smpl.data**	
Measuring point density:	**6**	Id1 or C21	
Min. increment	10.0µl	Id2 or C22	
Titr. rate	max.ml/min	Id3 or C23	
Signal drift	50mV/min	sample weight	xxxxxg
Holding time	26s	sample weight unit	g
StartV	off		
StartV	off ml	C-fmla	
Dos. rate.	max.ml/min	C01 :	0.02042
Break	15s		
Temperature	25°C		
Break off conditions		Formula	
StopV:	abs	Titer=C00/(EP1*C01)	
StopV	20ml	RS1text	Titer
StopU	off	RS1decimal places	4
StopEP	9	RS1unit	
Filling rate	Max.ml/min		
Calculation:		Periphery Devices	
EP criteria	**5**	send to	Citizen
EP recognition:	all		
Fix-EP1 at	off	RS232-Settings	
pk/HNP:	off	Baud rate	9600
		Data bit	8

Preselection		Stop bit	1
Require identity	**Id1**	Parity	none
Require sample size	Value	Handshake	Hwein
Activation pulse :	**off**	**Control via RS**	**on**

Common Variable		**Mean**	
C30 =	**MN1**	**MN1=**	**RS1**

Statistics		**Report**	
Status :	**on**	**Report:**	**full;comb;ff;**
Mean	**n = 5**		
Res.Tab:	**delete n**		

B Titrator Settings for 50 ml Changing Unit
The following parameters have to be changed for the 50 ml changing unit:

Titration parameter

Min. increment:	25µl
Titr. rate:	60ml/min

Break off conditions

Filling rate	*100ml/min*

Evaluation and Calculation;
Considering the consumption of perchloric acid and the sample weight the prechloric acid solution titer is calculated as follows:

$$\text{Titer} = \frac{E}{V * C01}$$

with:

E Sample weight potassium hydrogen phtalate in g
V Consumption perchloric acid solution in ml
C01 Calculation constant: 0.02042

Calculation of the Calculation Constant C01 :

$$C01 = \frac{M * c}{1000}$$

with :
M Molar mass of potassium hydrogen phtalate (204.23 g/mol)
C Nominal concentration of perchloric acid solution (0.1 mol/L)

The titer is reported with 4 decimal places. The relative standard deviation should not be greater than 0.5%.
The titer is documented in the betaine determination as “Common Variable C30” and used for the calculation.

Methylglycol reagent grade quality (analysis of amino acids; e.g. Merck 1.15118.xxxx or Fluka 64719

Methanol reagent grade quality

Fixanal 0.5 M; sodium hydroxide
Sodium acetate * 3 H_2O reagent grade quality

Preparation of Sodium hydroxide Solution / Sodium Acetate Solution

40g sodium acetate * 3 H_2O are dissolved in approx. 150 ml water and flushed into a 500 ml measuring flask. After adding the content of a Fixanalampoule 0.5 M sodium hydroxide solution fill up to the mark with water.

A solution prepared like this should be used for max. 1 month!

Dosing devices:

e.g. Fortuna Optifix 100ml for EGME-ether (Fleischhacker 9.287.907)
e.g. Fortuna Optifix 30 ml for methanol (Fleischhacker 9.287.905)
e.g. Fortuna Optifix 1,0 ml for sodium hydroxide solution/sodium acetate solution (Fleischhacker 9.287.901)

6. Procedure

1.3 g Sample are weighed directly in an titration beaker with 0.1 mg accuracy and dissolved in 20 ml methanol. After adding 0.5 ml sodium hydroxide solution / sodium acetate solution let react for 5 minutes at room temperature. Now add additional 20 ml methanol and 60 ml methylglycol and titrate at the Titrino against 0.1 m perchloric acid in dioxan.

A) Titrator Settings for 20 ml Changing Unit

Use the following settings for the Titrino:

Titration parameter		**Smpl.data**	
Measuring point density:	**6**	Id1 or C21	
Min. increment	10.0µl	Id2 or C22	
Titr. rate	max.ml/min	Id3 or C23	349
Signal drift	50mV/min	Sample weight	xxxxxg
Holding time	26s	Sample weight unit	g
StartV	abs.		
StartV	**6 ml**	C-fmla	
Dos. rate.	max.ml/min	C01	100
Break	15s	C30	XXX
Temperature	25°C		
Break off conditions		Formula	
StopV:	abs	Betaine=(EP2-EP1)*C23*C30/C01/C00	
StopV	**30ml**	RS1text	Betaine
StopU	off	RS1decimal places	2
StopEP	9	RS1unit	%
Filling rate	Max.ml/min		
Calculation:		Periphery Devices	
EP Criteria	**5**	send to	Citizen
EP recognition:	all		
Fix-EP1 at	off	RS232-Settings	

pk/HNP:	off	Baud Rate	9600
		Data Bit	8
Preselection		Stop Bit	1
Require identity	all	Parity	none
Require sample size	Value	Handshake	Hweinf
Activation pulse:	off	Control via RS	on

Report		Statistics	
Report :	full;comb.;ff;	Status :	off

B) Titrator Settings for 50 ml Changing Unit
The following parameters have to be changed for the 50 ml changing unit:

Titration parameter
Min. increment : 25µl
Titr. rate: 60ml/min

Break off conditions
Filling rate 100ml/min

In any case a titration has to be processed in dynamic mode. Other titrators have to be configured in that way, that dosing and shift controlled measurement value transfer enable to distinguish between the pK (b) – values safely.

If other conditions should be used, these have to be documented in a laboratory specific work instruction!
These cases require a validation of the conditions!

7. Calculation

Considering the consumption of perchloric acid and the sample weight the concentration of betainic nitrogen is calculated.

$$\% N = \frac{V (EP\ 2\text{-}EP\ 1) (HClO\ 4) * t * M (Betaine) * n (HClO\ 4)}{m * 10}$$

with :

V (HClO 4)	Consumption of perchloric acid (ml)
EP 2	End point 2
EP 1	End point 1
t	Titer of perchloric acid solution (out of paragraph 6.11)
M (Betaine)	Molecular weight betaine
n (HClO 4)	Normality of perchloric acid (0.1)
m	Sample weight of betaine (g)

The betaine content is reported with one decimal place. The used basic molecular mass has to be reproted as well.

Cocoamidopropylbetaine	= 349
Betaine L 7	= 349
Betaine F	= 349
Betaine F 50	= 349
Betaine CK	= 360
Betaine 810	= 297

8. Remarks

Not identified.

9. Changes

Replacing EA.:113.01

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	B-CS P (QKL) / Essen	Käseborn	09.02.04	X X X X X
Checked by	S2-AL, Essen	Dr. Keune	16.02.04	X X X X X
Checked by	CS -STN- QKL, Steinau	Kirschner	11.02.04	X X X X X
Approved by	B-CS P (QKL) / Essen	Käseborn	16.02.04	

Reviewed regarding validity *[on original copy only]*

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

Title: Potentiometric Determination of Chloride / Sodium Chloride Content

1. Normative References / Bibliography

DGF H-III 9

2. Terms / Definitions

Not identified.

3. Scope

The chloride / sodium chloride content of surfactants and the active content of methyl chloride quats determined by potentiometric titration with silver nitrate solution.

4. Interferences

Not verified.

5. Reagents and Materials

All reagents have to be, if not mentioned differently, for analysis.
Titroprocessor e.g. Modell 686 or Titrino 716 by Metrohm
Combined silver electrode e.g. Metrohm 6.0404.100
Analytical balance; Beakers; etc.
Silver nitrate solution 0.1 N (e.g. Fa. Kraft: 50263, Fa. Baker: 7096)
Nitric acid 5 N
Distilled water
Sodium chloride for analysis
Propanol – 2 for analysis

6. Procedure

An adequate sample weight (GM_0001_01) is dissolved in a beaker in distilled water (hard to dissolve quats are pre-dissolved in approx. 20 ml propanol – 2 with heating). Approx 10 ml nitric acid 5 N are added to create an acid level. Titrate potentiometrically with 0.1 N silver nitrate solution.

7. Calculation

$$\frac{V \times 0.355 \times F}{E} = \text{Chloride [\%]}$$

$$\frac{V \times 0.585 \times F}{E} = \text{Sodium chloride [\%]}$$

$$\frac{V \times MW \times F}{E \times 10} = \text{Active content [\%]}$$

V = Consumption 0.1 N silver nitrate solution (ml)
E = Sample weight (g)
F = Factor silver nitrate solution
MW = Molecular mass

8. Remarks

Titrator settings are to document in a laboratory specific work instruction.

9. Changes

Replacing analytical methods EA.044.01, 06-01, 04-11 (Goldschmidt Rewo) and JV-13A (Janesville)

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	CS; QKL / Steinau	Kirschner	27.11.2002	
Checked by	B-CS P; QKL / Essen	Käseborn	28.11.2002	
Approved by	CS; QKL / Steinau	Kirschner	05.12.2002	

Reviewed regarding validity *[on original copy only]*

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

Title: pH-Value (100%); as is

1. Normative References / Bibliography

According DIN EN 1262
DGF H – III 1
ISO 4316
Ph.Eur. 2.2.3

2. Terms / Definitions

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

3. Scope

With this method the pH-value of a product can be measured.

4. Interferences

Not verified.

5. Reagents and Materials

pH-Measurement device with glass electrode
Beaker
Magnetic stirrer with heating plate

6. Procedure

The measurement has to be processed at room temperature between 20 – 25 °C.
The clean glass electrode is put into the sample. One minute after the measurement value has reached a constant level this value is red off.

7. Calculation

The pH-value is documented with one decimal place in SAP.

8. Remarks

Not applicable.

9. Changes

Replacing EA.006.08 and 02-01 Goldschmidt Rewo

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	B-CS P (QKL) / Essen	Käseborn	07.12.02	X X X X X
Checked by	CS P; QKL / Steinau	Hr. Kirschner	11.12.02	X X X X X
Checked by	B-OS P (QKL) / Essen	Dr. Weibels	09.12.02	X X X X X
Approved by	B-CS P (QKL) / Essen	Käseborn	11.12.02	

Reviewed regarding validity *[on original copy only]*

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

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

$J_2 + SO_2 + H_2O = 2\ HJ + SO_3$

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** *[on original copy only]*
Prepared by	CS; QKL / Steinau	Kirschner	28.03.2003	
Checked by	B-CS P; QKL / Essen	Käseborn	28.03.2003	
Checked by	S2 AL / Essen	Dr. Keune	28.03.2003	
Checked by	B-OS P SE / Essen	Hörnlein	28.03.2003	
Approved by	CS; QKL / Steinau	Kirschner	28.03.2003	

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*