Solids Content (IR-Dryer Balance)

1 Normative references / bibliography

Following DGF B-II 3 / C-III 12

2 Definitions

The solids content is the remaining fraction by weight of a product under the defined test conditions.

3 Scope

This fast humidity determination method is a combination of a balance and an IR-heat source, therefore it is possible to process drying and calculation in one equipment.

4 Interference's

Thermally instable samples cannot be investigated.

5 Reagents and materials

- Electronic fast humidity determination with IR- heat source, like Sartorius MA 100, MA 150 C. The end of drying is detected automatically by weight reduction per time. This is preset in the equipment, for example 1 mg/60 sec.

- Aluminium dishes 90 mm
- Glassfiber filter

6 Procedure

The correlation between product, dryer temperature and equipment settings is described in a laboratory specific work instruction.
The typical temperatures are between 110 to 120 °C, the weights are between 1 to 3 g.
This drying temperature classification is necessary to maximize the conformance with the solid content, which was determined before. The drying temperature was optimized to avoid sample burning and to shorten the drying time.

An aluminium dish and a glass-fiber filter are put on the dish carrier and tared to zero. The product is equally spead and weighed.
After starting the measurement the result is calculated automatically and shown on the display.

7 Calculation

The shown measurement value has to be read off and documented.

8 Remarks

The sample should be applied equally on the filter.

9 Changes

Revision of the method from 26.09.2002.

10 Enforcement

Step	Org. – Unit / site	Name	Date	Signature *[on the original only]*
Prepared by	CS-STN-QSU-QC / Steinau	G. Christ	29.10.12	X X X X X
Checked by	B-CS P; QKL / Essen	H.Käseborn	29.10.12	X X X X X
Checked by	CS/EES / Granolers	D. Montllo	29.10.12	X X X X X
Checked by	CS-IM / Essen	Dr. S. Keune	29.10.12	X X X X X
Checked by	CS-NA-PT / Hopewell	M. Taylor	29.10.12	X X X X X
Checked by	CS-NA-PT / Janesville	J. Schultz	29.10.12	X X X X X
Checked by	B-OS SE / Essen	Dr. M. Weibels	29.10.12	X X X X X
Approved by	CS-STN-QSU-QC / Steinau	M. Hufnagel	29.10.12	

Reviewed regarding validity *[on the original only]*

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

Title: Active Content, Hyamin-WAS

1. Normative References / Bibliography

According to DGF H-III 10 (94)

2. Terms / Definitions

Not identified.

3. Scope

A red cationic and a blue anionic color are given to a sulphuric acid solution of an anion active surfactant. Shake after adding chloroform.
By reaction of the anion active surfactant with the cationic color the corresponding colored salt is created and due to his solubility properties transferred into the chloroform phase.
During the subsequent titration with the adjusted cation active surfactant solution (Hyamin 1622) the salt, dissolved in chloroform is transferred by a substitution reaction into the colorless salt of the anion active and the cation active surfactant.
After reaching the equivalence point the excess of cation active surfactant is creating with the anionic blue color the corresponding salt, which is soluble in chloroform. Therefore the color of the chloroform phase changes to gray with a little bit of blue.
This method serves to determine anion active surfactants in detergent raw materials.

4. Interferences

Quaternary compounds interfere.

5. Reagents and Materials

- Analytical balance
- Beakers
- Measuring flasks 100 ml, 250 ml, 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
- 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

An adequate sample weight (GM_0001_01) is dissolved in a 100 ml measuring flask. Fill up to the check mark with distilled water and mix well. Pipette 10 ml, 25 ml resp. (depending on content and molecular weight) in a 100 ml shaking cylinder.
After adding 10 ml distilled water, 10 ml acid indicator solution, and 15 ml chloroform titrate with hyamin solution to the dead stop.
The dead stop is reached, when the chloroform phase color is not pink anymore. This phase color is now gray-blue.

7. Calculation

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

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

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

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

V = Consumption hyamin solution (ml)
f = Factor hyamin solution
MW = Molecular weight of WAS
n = Normality of standard solution
ap = Aliquot part

8. Remarks

Not identified.

9. Changes

Replacing analytical method 03-01 (Goldschmidt-Rewo).

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	CS P; QKL / Steinau	Christ	09.12.02	X X X X X
Checked by	B CS-P / Essen	Käseborn	09.12.02	
Checked by	CS P; QKL / Steinau	Kirschner	09.12.02	
Approved by	CS P; QKL / Steinau	Kirschner	09.12.02	

Reviewed regarding validity *[on original copy only]*

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

Title: pH Value in Water; Electrode

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

2. Terms / Definitions
The pH value is the negative decade logarithm of the hydrogen-ion concentration.

3. Scope
Applicative for water solutions / dispersions of products.

4. Interference's
Not verified.

5. Materials and Reagents
pH-meter with glass electrode
Deionized water or distilled water
For testing of surfactants the deionized water has to be according DIN 53909 (free of CO_2 – by boiling; pH value has to be between 6.8 and 7.2).
Baker, 100 – 400 ml
Glass rod or spatula
Magnetic stirrer with stirring rod
Balance with min. 0.1 g accuracy
Thermostat

6. Procedure
According to the test plan the product to be investigated is stirred homogeneously in distilled water, if necessary under warming up on 60 - 70 °C. 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 given in the test solution. One minute after reaching a constant measuring value this is red off.
Dilutions from 0.5 to 50% are possible.

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

8. Remarks
The concentrations are deposited in the specifications.

9. Changes
Replacing old testing methods SM0036 , EA.006.xx, and 02-01 (Goldschmidt Rewo)

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	B-OS P SE / Essen	Hörnlein	18.11.2002	X X X X X
Checked by	B-OS P SE / Essen	Dr. Weibels	18.11.2002	X X X X X
Checked by	B-CS-STN-QKL	Kirschner	18.11.2002	X X X X X
Approved by	B-OS P SE / Essen	Dr. Weibels	18.11.2002	

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*