Title: Determination of Assay by titration

1. Normative references / bibliography

not covered

2. Definitions

Neutral and basic amino acids as well as salts of amino acids are titrated potentiometrically with perchloric acid in anhydrous pure acetic acid.
The addition of formic acid improves the solubility of amino acids.

3. Scope

Natural Betain

4. Interferences

not covered

5. Reagents and materials

- acetic acid, min. 99,8% p.A. (e.g. Merck Art. 33209)
- perchloric acid, 0,1 mol/L in anhydrous acetic acid (e.g. Merck Art. 9065)
- formic acid 98-100 % p.A. (e.g. Riedel de Häen Art. 33015)

- Automatic Titrator e.g. Titrino 716 DMS
- Stirrer e.g. E 649
- pH- electrode 6.0123.100; storage in water
- reference electrode 6.0726.100 (inner electrolyte: LiCl sat. in ethanol, outer elektrolyte; $LiClO_4$ c=1 mol/L) storage in acetic acid

6. Procedure

Determination of blank value:
A solution of 3 mL formic acid and 50 mL pure acetic acid is titrated with 0,1 mol/L perchloric acid by potentiometric dead stop method.

Measuring of samples:

Defined quantity according below mentioned table is solved in 3 mL formic acid in a 100 mL beaker. 50 mL pure acetic acid is added. The sample is titrated potentiometric with 0,1 mol/L perchloric acid. Special data about batch number, net weight and loss of drying are put in separately in titroprocessor.

Product	Net weight [mg]	Molecular weight [g/mol]
Betaine	110	117,15

The determination of the assay of Betaine monohydrate is carried out according to the betaine method. The crystal water is included in the calculation by input of C23 (loss of drying).

7 Calculation

$$\text{Assay (\%)} = \frac{V_{sample}[mL] - V_{blank\ value}[mL] * F * M[g/mol] * c[g/mol]}{10 * \text{net weight [g]}}$$

V – titrated volume of $HClO_4$ in mL
M – molecular weight of amino acid or betaine in g/mol
c - concentration of $HClO_4$ = 0,1 mol/L
F – factor $HClO_4$

8. Remarks

Not covered

9. Changes

Not covered

10. Enforcement

Step	**Org. – Unit / site**	**Name**	**Date**	**Signature** *[on the original only]*
Prepared by	B-CS P / Essen	Käseborn	22.03.2006	X X X X X
Checked by	B-CS P / Essen	Käseborn	22.03.2006	X X X X X
Approved by	B-CS P / Essen	Käseborn	22.03.2006	See GM_1505_01_G_E

Reviewed regarding validity *[on the original 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*

Title: pH Value in Water; Electrode: 0,02 m KCl-Solution

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. Interferences

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).
Potassium chloride (p.A.)

0,02 molar KCl-solution : dissolve 1,492 g KCl in a.m. water

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 the 0,02 molar KCl-Solution, 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.

7. Calculation

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

8. Remarks

The concentrations are deposited in the specifications.

9. Changes

First edition

10. Enforcement

Step	**Org.-Unit / Site**	**Name**	**Date**	**Signature** *[on original copy only]*
Prepared by	B-CS P (QKL)	Käseborn	05.1.2010	X X X X X
Checked by	B-CS P (QKL)	Käseborn	05.1.2010	X X X X X
Approved by	B-CS P (QKL)	Käseborn	05.1.2010	See german edition

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*