Republika e Kosovës
Republika Kosova-Republic of Kosovo
Qeveria-Vlada-Government

Ministria e Bujqësisë, Pylltarisë dhe Zhvillimit Rural
Ministarstvo Poljoprivrede, Šumarstva i Ruralnog Razvoja/ Ministry of Agriculture, Forestry and Rural Development

Ministry of Agriculture, Forestry and Rural Development,
Pursuant to Article 93 (4) of the Constitution of the Republic of Kosovo,
In accordance with Article article 5, paragraph 3, Law No. 04/L-114 on Enriched Flour (Official Gazette of the Republic of Kosovo, No. 26/21 September 2012), and Article 19 (6.2) of the Government Rules of Procedure No. 01/2011 (OG, No. 15, 12.09.2011),

Approves:

ADMINISTRATIVE INSTRUCTION MAFRD NO. 07/2013

ON STANDARD FOR ENRICHED FLOUR, CONTROL AND SAFETY ON THE QUALITY OF ENRICHED FLOUR

Article 1
Goal

By this Administrative Instruction is determined the standard on enriching the flour with iron and folic acid, control and safety and quality, as well as specification on labelling enriched flour.

Article 2
Definitions

1. Terms and expressions used in this Administrative Instruction have the following meaning:

1.1. **Premix** – means additional substance added for enriched floor;

1.2. **Label** – means visible written graphic content that contains specific data of the product;

1.3. **NIPHK**– National Institute for Public Health in Kosovo;

1.4. **Flour dosing equipment** – means technical equipment which is utilized to ad premix in the flour;

1.5. **Standard** – Voluntary document compiled with consensus and adopted by a recognized standardization authority, which for joint and repeating utility provided rules, instructions or characteristics for activities or its results, for an optimal rate of the rule, in a given context;

1.6. **400 and 500 Flour type**– is flour with percentage of ash up to DDD - Disinfection, Deratization and Disinsection.

Article 3
Standards for enriched flour

1. Enrichment of 400, 500 and other types of flour that consist of the ash less than 0.65 mg/kg, added to (1) ton of flour 200 gr of premix that contains Ferro Sulphate and Folic Acid.

2. Flour type from the paragraph 1 of this Article, must contain iron of 25.2 mg/kg in a form of Ferro Sulfate and Folic Acid 1.5 mg/kg.

Article 4
Flour label

Flour producer and importer, in addition to the specific details, according to the applicable legislation, shall contain also a logo for flour enrichment and premix content.

Article 5
Conditions for flour storage

1. The flour, should be stored in a dry place, with no moisture, not touching the floor and the wall, to have ventilation, bags should be piled up to 2m height and should have sufficient space for unobstructed movement.
2. Each producer, trader or warehouse of flour, should carry out DDD by authorized authorities, at least once within (6) six months and shall maintain evidence.

Article 6
Premix Label

1. Premix label, should contain the following data:
   1.1. Origin;
   1.2. Producer;
   1.3. Premix name and content;
   1.4. Date of production and expiry;
   1.5. Serial number of the production - Lot.

Article 7
Premix registration

Premix Producer – Importer should be registered with the Ministry of Agriculture, Forestry and Rural Development.

Article 8
Premix Storing Conditions

1. Premix should be stored in a dry place, not to be directly exposed to sunbeams and should have appropriate ventilation.
2. Premix should be deposited on top of each other, and there should be an arrangement of usage according to the supply and expiry date.

Article 9
Dosage equipment

1. Flour Producer is obliged to possess enriched flour dosage equipment.
2. Producer should control dosage equipment every hour.
3. Producer should maintain evidence on the date, time, and quantity of produced flour and used premix.

Article 10
Safety and control of enriched flour

1. Control of the enriched flour shall be conducted in accordance with tests and procedures which are integral part of this Administrative Instruction.
2. Testing of enriched flour shall be conducted in the following methods:
   2.1. With quick test about the iron, and
   2.2. Quality and quantity analysis in an accredited lab.
3. Flour testing from paragraph 2, subparagraph 2.1, of this Article is conducted by the producers, inspectors at the flour production site, warehouse and trading, in compliance with Annex 1 of this Administrative Instruction.
4. Flour testing from paragraph 2, subparagraph 2.2, of this Article is conducted in an accredited lab, in compliance with Annex 2 of this Administrative Instruction.
5. Expenses for the tests requested by the producer shall be covered by the producer, trader or the owner of the warehouse.
6. Expenses for tests during official controls by agriculture and phytosanitary inspectors shall be covered by Food and Vet Agency.

Article 11
Official Control

1. Official control shall be carried out by the agriculture and phytosanitary inspectors in order to control and verify completion of the criteria for enriched flour in compliance with procedures determined in this Administrative Instruction.
2. Official control shall be carried out according to the instructions and Forms attached to this Administrative Instruction.
3. Official control shall be conducted by Food and Vet Agency.

Article 12

1. An integral part of this Administrative Instruction are:
   1.1. Annex 1: With quick test about the iron;
   1.2. Annex 2: Quality and quantity analysis;
   1.3. Annex 3: Determining Folic Acid based on HPLC analysis, and
   1.4. Annex 4: Logo of enriched flour.

Article 13

Ministry may issue bylaws on implementation of this Administrative Instruction.

Article 14

All incomes generated from implementation of this Administrative Instruction shall be deposited in Kosovo budget.

Article 15
Non-application

Non-application with this Administrative Instruction is sanctioned pursuant to Articles 11 of the Law No. 04/L-114 on Enrichment of Flour, (Official Gazette of the Republic of Kosovo No. 28/21 September 2012).

Article 16
Entry into force

This Administrative Instruction enters into force seven (7) days following its' signing by the Minister.

Blerand Stavileci

Minister of Agriculture, Forestry and Rural Development

02.08.2013

Annex 1: Quick test on iron

Reagents:

- Cilium Tiocianid KSCN or Ammonium Tiocianid NH_4SCN
- Chlorohydrin Acid 37%
- Hydrogen Peroxide 30%

Solutions:

- KSCN 10% in distillate water
- NH_4SCN 10%: in distillate water
- HCl acid 2M: 100ml water, 17ml HCl acid, 83 ml water total 200 ml solution
- H_2O_2 3%: in distillate water
- Keep the solution cold and away from light exposure
- Reagent 1

50% KSCN solvent, 50% HCl solvent
Prepare it everyday

- Reagent 2

H_2O_2 3% solution
Prepare it everyday

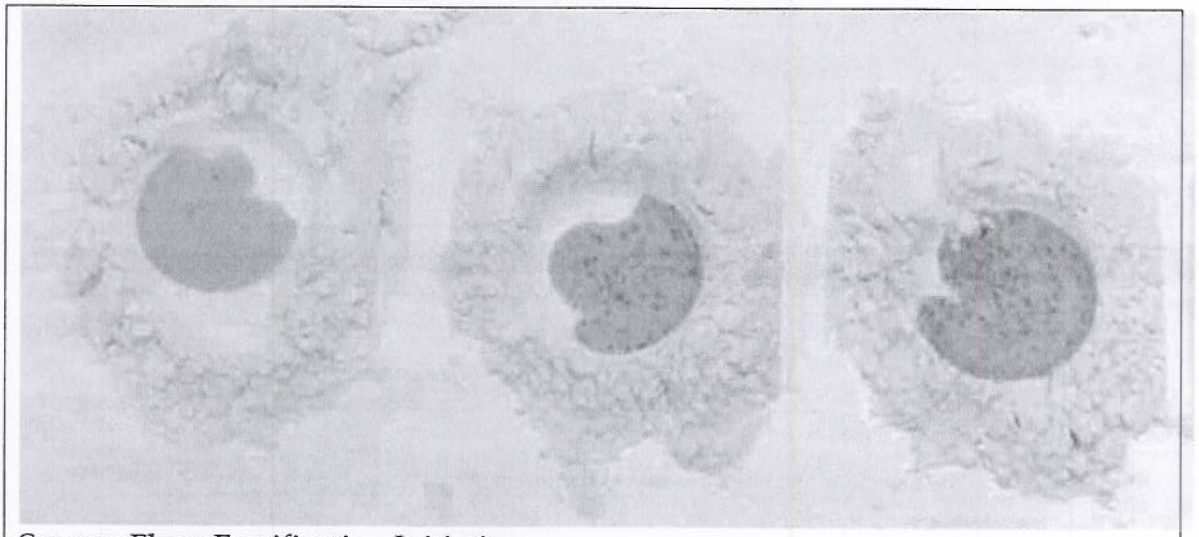
Procedure:

- Prepare part of the flour sample of known standard (30 ppm, 60 ppm) along with a plain sample in a visible cup.
- Mix the sample and the standard with reagent 1. Let it stay for 15-30 seconds.
- Add reagent 2
- Let the sample stay 2 minutes until read – brown dots appear.
- Then read the sample compare to the standard.

Other dots:

- During compare of the samples with the standards **MUST** be used the same type of iron components in the sample and in the standard, i.e. $FeSO_4$ for $FeSO_4$
- Differences because of the component types and size of granules.

Examples of the test for added layers in the flour:



Source: Flour Fortification Initiative

Interpretation

Presentation of spots with red colour indicates iron presence. Number of dot sis a broad assessment of quantity and iron homogeneity in the sample. If more precise assessment is required, test with known iron concentrations (30, 60, and 90 ppm) is recommended in order to compare results of these samples with those of the tested flour.

Annex 2: Quality and quantity analysis

Spectrophotometric method for determination of Fe
AACC Method 40-41A adapted

Definition:
Representing sample shall be mineralized with dry burning (2-5g). Ash remains shall be mixed with acid solution and thinned up to a certain volume (100 ml). Aliquant part of the solution is taken for a reaction with orto-phenentroline in which case is formed a red colour complex. Solution absorbance is measured with spectrophotometer in a wave length of 510 um and is converted to concentric using calibration curve made by standards.

Equipment:
Platinum, silica or porcelain pot
Spectra photo meter or colour meter.

Reagents:
0.1 % Ortho-phenanthroline solution.
Standard Solutions of Fe Iron 2, 4, 6, 8, 10 mg Fe/l
3. Hydroxylamine hydrochloride solution 10 %.
4. Buffer Acetate. Solution 8.3 g Acetate Natron anhider previously dried shall be mixed in 100^0C) water, 12 ml acid acetic should be added, and thinned with water in 100^0C.

Product: Wheat flour 400, and 500 type
Parameter: mineral matter Ca, Fe, Cu - dry mineralization. Internal method 01.01.0050

Method description: Dry mineralization is carried out by burning the sample in 900^0C and mixing the burnt solution with acid Chlorine hydric 1:4.

Tools and equipment:
Burning chamber 1100^0C
Analytic balance
Porcelain cup
Spoon for sample measuring

Reagents:
Chloric hydric Acid 1:4

Work procedure: one porcelain cup previously dried in 200^0C (30 min) and cooled in a room temperature, empty cup is measured in analytic balance (A_0) adding the flour sample of 2-5 g (A_1), initially the cup filled with the sample will be dried 130^0C, burnt on a flamer and placed in the chamber initially in low temperature 200-300^0C which gradually should be increased up to 560 ^{0}C. In this temperature the sample shall stay for 6 hrs. If the is not burnt completely after the cooling the same can be added few drops of Nitric Acid and the burning can proceed for two more hours. Hot sample shall be placed in exycator and after the cooling it shall be mixed with solution of 25 ml acid chloric hydric 1:4. The solution shall be put in the meskolben 100 ml and filled with water up to the level indicator.

Upon mineralization based on the method from the solution it will be determined the Fe through AAS method, using standard series on Fe determination (4, 8, 12 ppm)

Calculation:

Calculation of absorption factor

$F = (C_1 + C_2 + C_3) / (E_1 + E_2 + E_3)$

Calculation of Ca content in the solution

$Ca\ (ppm) = F \times E_{sample}$

Calculation of Ca content in the sample

$Ca\ (mg/kg) = (F \times E_{sample} \times 100)/ m$

In which case:

f- Absorption factor

C_1, C_2, C_3 - standards concentration

E_1, E_2, E_3 – standards absorptions

E_m – Sample absorptions

m – Sample measure.

Annex 3: DETERMINATION OF FOLIC ACID BY HPLC ANALYSIS

1. SCOPE

This method describes the procedure for the determination of folic acid in flour samples using the Dionex 500 Series HPLC.

This is not an official method.

4. CHEMICALS AND EQUIPMENT

4.1 CHEMICALS DESCRIPTION

Acetonitrile HPLC grade

Ascorbic acid Analytical reagent grade

Deionized (DI) water Nanopure, 18.2 megaohm

Flour Un-enriched

Folic acid 98% pure analytical reagent grade

Glacial acetic acid Analytical reagent grade

Hexane HPLC grade

Methanol HPLC grade

pH buffers 4.00 and 7.00

Phosphoric acid Analytical reagent grade

Potassium hydroxide Analytical reagent grade

Potassium phosphate, dibasic Analytical reagent grade

Reference flour

Sodium acetate, anhydrous Analytical reagent grade

Sodium chloride Analytical reagent grade

4.2 EQUIPMENT DESCRIPTION

Balance, analytical Capable of weighing to 0.0001 gm

Balance, top-loading Capable of weighing to 0.01 gm

Beakers 30 ml and 3,000 ml

Column Phenomenex Bondclone, 150 x 3.9 mm, 10 µm, C18

Eppendorf pipet 5 ml adjustable

Flask 250 ml Nalgene with screw lids

Filter paper Whatman #4, 12.5 cm

Food processor

Funnels 60 mm powder

HPLC system Dionex 500 series with AD20 absorbance detector, GP50 pump, AS40 auto-sampler

Injection loop 100 µL

Pails Plastic, one gallon, with lids

pH meter

Shaker Wrist action with timer

SPE tubes Varian SAX quaternary amine ion exchange, 500 mg/10 ml

Syringes 20 ml disposable

Syringe filters Acro-disc, 0.22 µm

Test tubes 16 x 100 mm

Volumetric pipets 40 ml Class A

Weigh boats

Weighing paper 8 x 8 cm

5 SAFETY

6 OPERATION

6.1 SOLUTION PREPARATION

6.1.1 Stock Standard

Weigh 453.5 ±0.1 gms of un-enriched flour into the food processor bowl. Onto weighing paper, weigh out 0.1103 ± 0.0003 gms of folic acid. Tare the analytical balance, transfer the folic acid to the food processor bowl, and reweigh the weighing paper. Record the weight loss in the standards workbook. Close up the food processor and mix for 5 minutes. Transfer stock standard to a one gallon pail. After adjusting for the water content and purity, the stock standard will contain 100.0 mg/pound folic acid. Label with contents and date prepared.

6.1.2 Working Standards

Five working standards are made by diluting the stock standard. The weights of stock standard for the five working standards are: 1.14, 2.13, 3.18, 4.54, and 7.26 gms. The weights of un-enriched flour for the five working standards are: 452.46, 451.47, 450.42, 449.06, and 446.34 respectively. Weigh the un-enriched flour into the food processor bowl and add the corresponding amount of stock standard. Close up the food processor and mix for 5 minutes. Transfer working standards to a one gallon pail. The current bag of un-enriched flour contains 0.156 mg/pound folic acid. The working standards will contain 0.407, 0.626, 0.857, 1.157, and 1.757 mg/pound folic acid, respectively. Label each container with contents and date prepared.

6.1.3 "A" Mobile Phase

Add 980 mls DI water to a 1,000 ml beaker. Add a magnetic stirring bar and place on a stir plate. Weigh out 8.20 ± 0.01 gms of sodium acetate. Transfer the sodium acetate to the beaker. Adjust the pH with acetic acid to a pH of 5.70 ± 0.05. Add 20 mls of acetonitrile. Pour into the mobile phase reservoir for delivery to HPLC system.

6.1.4 "B" Mobile Phase

Add 800 mls DI water to a 1,000 ml beaker. Add a magnetic stirring bar and place on a stir plate. Weigh out 8.20 ± 0.01 gms of sodium acetate. Transfer the sodium acetate to the beaker. Adjust the pH with acetic acid to a pH of 5.70 ± 0.05. Add 200 mls of acetonitrile. Pour into the mobile phase reservoir for delivery to HPLC system.

6.1.5 Extraction Solvent

Add 2,000 mls DI water to a 3,000 ml beaker. Add a magnetic stirring bar and place on a stir plate. Weigh out 34.83 ± 0.01 gms of potassium phosphate and

transfer it to the beaker. Weigh out 1.00 ± 0.01 gms of ascorbic acid and transfer it to the beaker. Adjust the pH with phosphoric acid or potassium hydroxide to a pH of 8.50 ± 0.05.

6.1.6 Salt Eluent

To 250 ml of extraction solvent, add 25.00 ± 0.01 gms of sodium chloride. Stir until dissolved.

6.2 SAMPLE PREPARATION

NOTE: Normal analysis run consists of the 5 standards, 3 reference standards, and 24 samples.

6.2.1 Weigh out 4.00 ± 0.01 gms of sample. Transfer into a labelled screw capped flask using a funnel. Repeat for all samples and reference flours. Pipet 40 mls of extraction solvent into each flask, cap, and place on the wrist action shaker. When the shaker is full, shake the flask for 20 minutes. While the flasks are shaking, prepare for filtration by placing funnels in 30 ml flask then fold a #4 filter paper into quarters. When the shaker has stopped, remove the flask, swirl, open, and pour into filter paper. Allow a minimum of 20 mls to filter before proceeding.

6.2.2 Place a syringe filter on the end of the 20 ml syringe. Remove the plunger, pour the filtrate into the syringe, replace the plunger, discard the first 1 ml of filtrate, and collect about 6 mls of filtrate in a test tube. Repeat for the other 15 flasks.

6.2.3 Place unmarked test tubes into the vacuum chamber in positions 1-12, 14, 17, 20, and 23. Place the top on the vacuum chamber. Check to make sure the pointer on the top is pointing to waste, change if necessary. Place a SPE cartridge into each stopcock. Turn on the vacuum and close the manifold. Fill the SPE cartridge with hexane. Open the stopcocks to allow the hexane to flow through until a thin film remains. Close the stopcock. *DO NOT LET THE CARTRIDGES GO DRY.* Repeat with methanol, then DI water. Pipet 5 ml of the first sample into the first SPE cartridge. Repeat for all 16 test tubes. Open the stopcocks to allow the sample to flow through until a thin film remains. Pipet 5 ml of DI water into each SPE. Open the stopcocks to allow the water to flow through until a thin film remains. Open the manifold. When the vacuum is at zero psi, turn the top so the pointer is pointing to collect. Pipet 5 mls of salt eluent into each SPE. Open the stopcocks to allow the salt eluent to flow through until a thin film remains. Open the manifold and turn off the vacuum. Remove the tubes, but keep them in the correct order.

6.2.4 Vortex the contents of each test tube. Pour contents into a labeled polyvial. Cap the polyvial.

6.3 EQUIPMENT PREPARATION

6.3.1 Column Switching

6.3.1.1 Open the door of the column holder. Locate the line going from the injector to the columns. If the line goes to the Dionex column, the line will need to be flushed. Disconnect the line from the Dionex column and place into a small beaker or flask. On the pump module, move the cursor so it is in front of "Remote", press the Select key so the display changes to "Local". Move the cursor up to in front of the "% A", type 100, and press "Enter". Move the cursor to in front of "mls/min", type 1, and press "Enter". Start the pump, run for 5 minutes, and then stop the pump.

6.3.1.2 Connect the line to the inlet of the guard column. Use the blind in the guard column to seal the Dionex column. Connect the outlet of the column to the line going to the detector. Blind off the reaction tube outlet.

6.3.2 Sample Schedule

A sample schedule tells the computer what sample and type of sample is being analysed. The easiest way to build a sample schedule is to open the last one, make changes, then save as a new file name. The next to last line is for cleaning the column and the last line shuts everything down.

6.4 ANALYSIS

6.4.1 Open the run window of Peak Net. Click on the second icon from the left to load a schedule. Choose the schedule that was developed in Step 6.3.2. After clicking on the last "OK", the pump will start. Let the system run for 30 minutes before continuing.

6.4.2 Load samples into AS40 automated sampler in appropriate order and press the Run button.

6.4.3 The Dionex software will automatically calculate mg/lb folic acid and print out a report for each standard and sample.

Annex 4: Logo of enriched flour