A New Colorimetric Test for FeNa-EDTA in Flour

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Recommended Fe Fortification Levels

WHO 2009: Recommendations on Wheat and Maize Flour Fortification
Meeting Report: Interim Consensus Statement

http://www.who.int/nutrition/publications/micronutrients/wheat_maize_fort.pdf

Type of flour	Fortificant	Average daily consumption in g/d		
		> 300	150 – 300	< 150
LOW-extraction	**FeNa-EDTA**	**15**	**20**	**40**
	Ferrous sulfate or Ferrous fumarate	20	30	60
	Electrolytic iron	40	60	not recommended
HIGH-extraction	**FeNa-EDTA**	**15**	**20**	**40**

Intrinsic Fe Content in Flours

High-extraction wheat flour

- average: ~ 30 ppm
- range: 10 – 100 ppm (?)

Low-extraction wheat flour

- average: ~ 10 ppm
- range: 5 – 50 ppm (?)

Fe content may vary considerably from batch to batch

- and even within batches!

Total Fe Content

Determination

- dissolution of the flour in HNO_3/H_2O_2
- ultrahigh temperature in microwave
- all insoluble parts ➔ fully soluble ➔ clear solution
- injection into the flame of AAX / ICP

Total [Fe] can vary from 20 – 160 ppm

- [Fe] from fortification: 15 – 60 ppm
  - depending on iron compound and consumption level
- [Fe] of intrinsic origin: 5 – 100 ppm

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Intrinsic vs. Fortification Fe?

How to differentiate intrinsic vs. fortification Fe?

- both types fully dissolve

Suppose: AAS/ICP ➔ 40 ppm Fe

- 10 ppm from fortification, 30 ppm intrinsic?
- or the other way round?
- or 20 ppm + 20 ppm?

Type of fortificant (bio-availability) is critically important

Total Fe content (AAS/ICP) is not suitable

Other Methods for Flour

Red Spot Test

- directly on the flour as such
- only for elemental iron types

Extraction of flour + colorimetric determination

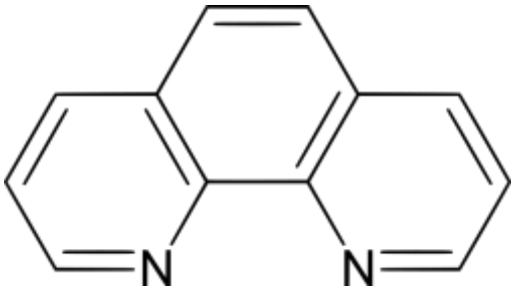
- flour is extracted with water/methanol ➔ suspension
- flour is separated from water layer
- coloring agents are added to filtrate solution

Phenanthroline (orange color) method

Determination of [Fe] in Water

Phenanthroline (Phen)

also: ortho-phenanthroline



Gives a highly intense, orange color with ferrous ions

Fe^{2+} + 3 Phen ➔ $\{Fe(Phen)_3\}^{2+}$

Ferric ions should be reduced first (e.g. by ascorbic acid)

Fe^{3+} ➔ Fe^{2+}

Analytical method already known from 1930's

Widely-used for [Fe] determination in water

Semi-Quantitative Procedure (DEMO)

Mix 10 g of flour with 30 mL of water/methanol (80/20)

- shake vigorously during 1 minute

Separate water from flour over paper filter (~ 15 minutes)

Transfer 15 mL to 50-mL volumetric flask

Add the following reagents

- 5 mL vitamin C solution (1 g/L)
- 5 mL phenanthroline solution (1 g/L)

Fill up to 50-mL mark with water

Wait for 10 minutes

Assess color intensity by visual inspection with calibration samples

Mix with Water/Methanol

Transfer 10 g of fortified flour into a 50-mL test tube
Add 30 mL water/methanol (80/20) and mix thoroughly to suspend all flour
Shake resulting suspension vigorously for 1 minute

Filter Solution

1. Separate water from flour through a folded paper filter
2. Collect 15 mL filtrate in a 50-mL test tube

1.

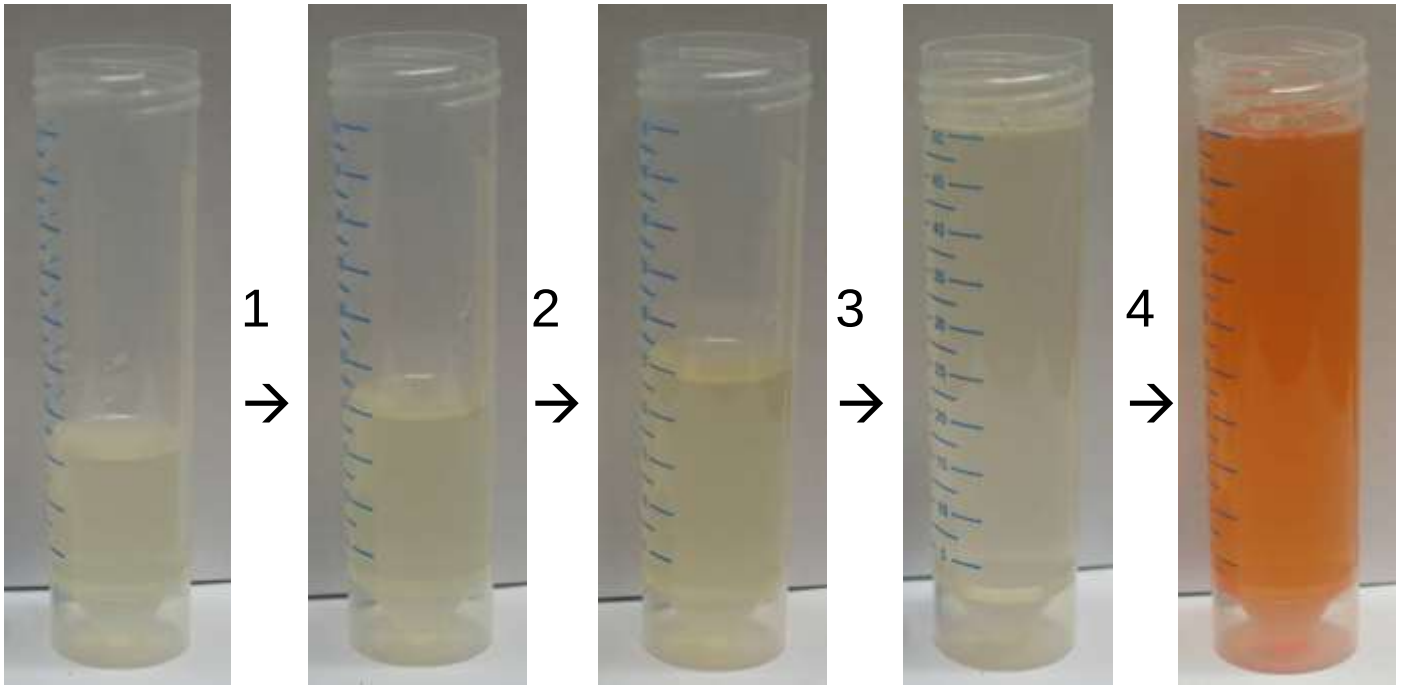
→

2.

→

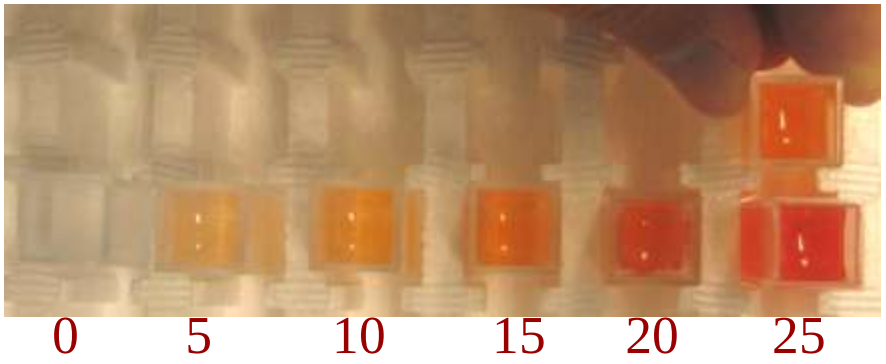
Add Reagents

1. 5.0 ml of vitamin C solution (1 g/L)
2. 5.0 ml of phenanthroline solution (1 g/L)
3. Fill up to 50 mL and mix
4. After 10 minutes the color reaction has completed



Transfer to Cuvette

and Compare Visually



Other Iron Fortificants

In flour

- Fe as Ferrazone: can be determined reliably (2 – 30 ppm)
- Fe as *dried* $FeSO_4$: is NOT detected
- Fe as ferrous fumarate: is not detected either
- Fe as electrolytic iron: does not dissolve in water

Conclusion: phenanthroline method is suitable for discriminating Ferrazone vs. other (recommended) iron fortificants in wheat flour

Current Applications FeNa-EDTA

China: soy sauce, wheat flour

Vietnam: fish sauce

Pakistan: atta flour

Brazil, Mexico, Philippines, …

powdered beverage

Regulatory Status FeNa-EDTA

JECFA (2007)

Sodium iron EDTA is suitable for use as a source of iron for food fortification to fulfill nutritional iron requirements …

US FDA

Gras Notices GRN 152 and 178

EU EFSA

Dossier submitted, final statement pending

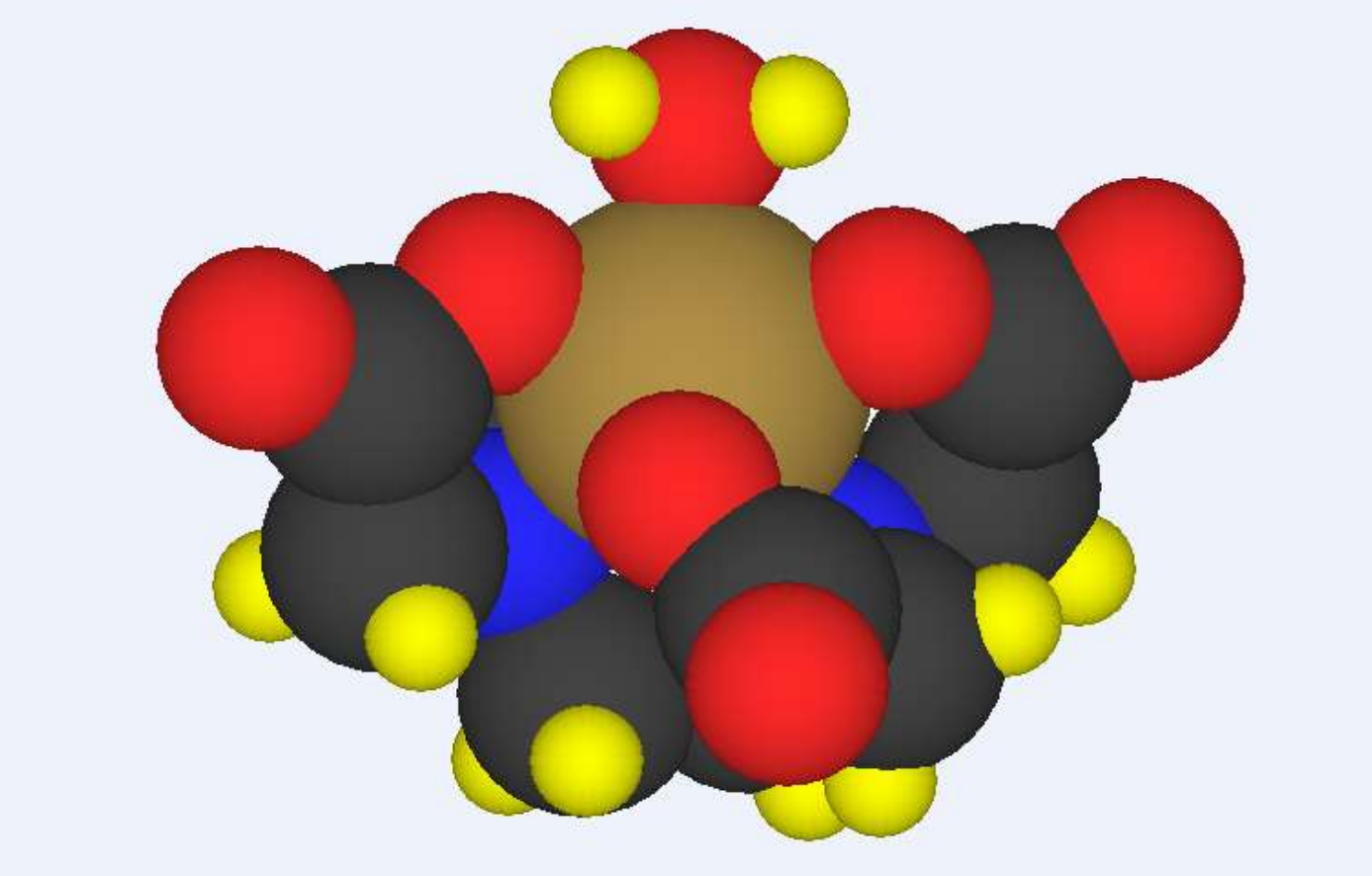
India

Approved for atta flour and drinks by PFA

Approval also in Brazil, China, Mexico, Pakistan, Philippines, Vietnam, …

No formal approval yet in African countries

3-Dimensional Model FeNa-EDTA



Ferric-EDTA monohydrate complex-ion
Wageningen University 2007

A Colorimetric Test for FeNa-EDTA in Flour

AkzoNobel
Tomorrow's Answers Today

Thank You for Your Attention

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