

Specifications for Fluoride Field Test Kit



October 2002

Contents

Preamble	3
Scope	4
Chemistry of the kit	4
Constituents of the kit	4
Reagents preparation	5
Quality test	5
Conformity of acceptance	5
Packaging and Marking	6
Guarantee	6
Instructions for use	6

Annex - I

Specifications of Chemicals Used in the Manufacture of Kit Reagents

- Aluminum powder	7
- Sulfanilamide	8
- Color Reagent	9
- Sodium Chloride	10
- Potassium Nitrate	11
- Powdered Sugar	11
- Citric Acid	12
- Ethylene Diamine Tetra acetic Acid	13

Annex - II

Pictorial photographs of field-test kit and testing procedure.	15
--	----

Preamble

1. Fluoride is a naturally occurring ion in geological formations. When water comes in contact with high fluoride containing rocks or geological formations, the fluoride may get dissolved in water under certain conditions and in some cases it may be beyond the permissible limit of 1.5 mg/L.
2. Long term ingestion of high fluoride water (> 1.5 mg/L) can result in dental, skeletal and non-skeletal fluorosis. In the advanced stage, it can even cripple a person. Dental and skeletal fluorosis is non-reversible.
3. Due to inadequate infrastructure and resource constraints, the district laboratories have not been able to test most private water sources for fluoride concentration. It is estimated that number of household water supply sources is 10 to 15 times that of community water supply sources. Reliable and easy-to-use field test kit can facilitate monitoring of their water sources by themselves.
4. A study on evaluation of water test kits commissioned in 1998 by UNICEF, indicated that the kits available in the local market were not very user friendly and reliable.
5. UNICEF in consultation with the Rajiv Gandhi National Drinking Water Mission, Department of Drinking Water Supply, Ministry of Rural Development, Government of India initiated an R&D project for the development of a reliable and user-friendly field test kits for arsenic, fluoride and nitrate. The National Chemical Laboratory (NCL), Pune. Under an R&D project sponsored by UNICEF, NCL has completed development of a reliable and easy to use fluoride field test kit.
6. With a view to place technology details in the public domain, this specification has been prepared. It is a qualitative field test kit with capacity to test water samples containing fluoride (0-3 mg/L). It is expected that this specification will be useful to manufacturers, inspection agencies and buyers.

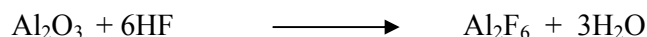
I. Scope:

This specification prescribes requirements of field test kits for assessing fluoride (1, 1.5, 2, 3 mg/L) levels in drinking water. This includes requirements related to preparation of reagents, quality of reagents used, construction details of kit, internal and external quality control, acceptance criteria, packaging and guarantee.

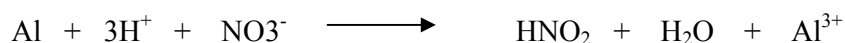
II. Chemistry:

A new and completely innovative chemistry is employed in the detection of Fluoride. Different reaction sequences involved in the development of the color are:

- a) Reaction of Fluoride with the oxide of aluminium present on the surface of the aluminum metal powder:



- b) The protective coating when depleted by fluoride, exposes the virgin metal. The aluminum metal, which is devoid of the oxide coating, is extremely active and is exposed to the various constituents present in the solution. One of the constituents present in the solution is nitrate ions. Aluminum in the presence of acid reacts with the nitrate producing nitrous acid.



- c) The HNO_2 generated by the reaction reacts with the $-\text{NH}_2$ group of the sulfanilamide producing the diazo compound which immediately diazotises with 1-N-naphthyl ethylene diamine dihydrochloride producing the pink-red colour.

III. Constituents of the Kit

The fluoride field test kit will comprise of the following:

- | | | |
|------|---|---------|
| i. | 15 ML test bottle (glass/acrylic without color)
plastic cap | 4 Nos. |
| ii. | 0.5 gm of reagent "A" as per 4.1 and packed in conical
centrifuge plastic tube with an airtight stopper | 100 Nos |
| iii. | 0.5 gm of reagent "B" as per 4.2 and packed in conical
centrifuge plastic tube with an air-tight stopper | 100 Nos |
| iv. | Color chart for comparison of color developed during test. | 1 No. |

- v. All the above placed in a 5 mm thick polypropylene corrugated board box with a lid, a nylon shoulder strap and separate compartments for each reagent and bottles and color chart etc.

IV. Reagents Preparation:

i. Reagent 'A' Composition:

Aluminum powder	0.5%
Naphthyl ethylene diamine dihydrochloride ?	0.05%
Sulfanilamide	0.2%
Sucrose powder	q.s. 100%

Weigh accurately the constituents as per the formula. Mix thoroughly using a suitable mixing device (mortar pestle or mixer). Pack the powder in 0.5ml capacity stoppered vials.

ii. Reagent 'B' Composition

Sodium Chloride	60%
Citric acid	20%
Potassium Nitrate	10%
Ethylene diamine tetra acetic acid	10%

Mix thoroughly the constituents as per the formulation and pack the powdered mixture in 0.5ml capacity stoppered vials.

4.2 Quality Control of the Reagent A Mixture:

Performance test should be carried out with the standard samples. Prepare standard fluoride solutions in the range of 1, 1.5, 2 and 3 mg/L compare the color developed by using the newly developed reagent mixture, with the color chart.

Certify OK if the color matches in all tests with the corresponding standard color of the color chart provide with the instruction sheet

V. Quality Tests:

- i. Performance tests to be carried out with standard fluoride samples (1, 1.5, 2, 3 and 5 mg/L) and compare color developed during these tests. There should be 100% agreement between color developed during tests and colors given in the color chart.
- ii. In five percent kits subject to minimum of five including glass/plastic bottles, plastic caps and polypropylene corrugated sheet box etc. shall be visually examined for visible defects. There shall be no manufacturing defects like air leakage or cracks or any other defects in the above components.

VI. Conformity for acceptance:

- i. Unless otherwise specified in the contract or order, the procedure given in IS: 2500 (Part I) “Sampling Inspection Tables: Part I Inspection by Attributes and by Count of Defects” shall be followed for sampling inspection. For characteristics (color matching at 1, 1.5, 2, 3 and 5 mg/L fluoride levels) the single sampling plan with inspection level S3 and AQL 1.5 shall be followed.
- ii. The lot shall be considered conforming to requirements of the specifications if the kits satisfy the following requirements.
 - a. All 5% randomly selected field test kits meet the requirement meet the requirement of 5.2.
 - b. The number of test kits, selected as per 5.1 conform, not meeting the requirements of characteristics in 5.1 does not exceed the corresponding acceptance number.

7) Packaging and Marking:

- 7.1 The test kit components shall be packed in a box with compartments for each reagent and glass tubes and a lid and made from polypropylene corrugated board. This box will be placed in a snug-fit nylon cloth bag with a shoulder strap.
- 7.2 Each kit box will have the following markings: Name of the product, name and address of the manufacturer, batch and code number, date of manufacture, expiry date and precaution in handling reagents. The user instruction card shall be fixed inside of the box lid.

8) Guarantee:

The kits will be guaranteed for three years from the date of manufacture.

9) Instructions for use (see also Annex II):

- i. Take 15 ml of water sample in the sample vial
- ii. Add contents of the capsule A in to the sample
- iii. Add contents of capsule B into the sample
- iv. Stopper the vial
- v. Shake the contents of the bottle occasionally
- vi. After 10 minutes compare the pink color developed with standard color chart provided with the kit to assess the fluoride level. Please note that colour comparison should be done between 10-12 minutes time.

Specifications of Chemicals Used in the Manufacture of Kit Reagents**1. Aluminium (Fine Powder)**

Mol wt. 26.98 g/mol
 Melting point 660⁰ C
 Boiling point 2550⁰ C
 Insoluble in water
 Ignition temperature 440⁰ C
 Highly inflammable

Specifications:

Assay (by complexometry) ----- > 93%
 Heavy metals (as Pb) ----- <0.03%
 Arsenic (As) ----- < 0.0005%
 Iron (Fe) ----- <0.5%
 Fats ----- <1%

2. Sulfanilamide:

GR/AR Grade Sulfanilamide should be used.

Specifications:***4-Aminobenzenesulfonamide***

$C_6H_8N_2O_2S$

Reagent Grade: GR /AR
 Preferred manufacturer: MERCK

Mol. Wt. 172.21g/mol
 Melting Range 163-166⁰C
 Solubility in water/20⁰C 5g/l

Assay (by bromometry, on dried substance) ----- min 99%
 Identity ----- IR Spectrum passes test
 Chloride (Cl) ----- max 0.01%
 Sulfate (SO₄) ----- max 0.02%
 Alkali-insoluble impurities ----- passes test
 Acid-insoluble impurities ----- passes test
 Heavy metals (as Pb) ----- max 0.002%
 Loss on drying (105⁰C) ----- max 0.5%
 Sulfated ash ----- max 0.1%

3 Color Reagent

GR/AR Grade Color Reagent should be used.

Specifications:

N- (1-naphthyl) ethylenediamine hydrochloride

$C_{12}H_{16}Cl_2N_2$

Reagent grade: GR /AR

Mol wt. 259.18g/mol

Assay (by argentometry, on anhydrous substance) ----- min 90%
Identity ----- IR Spectrum passes test
1-Naphthylamine (HPLC) ----- max 0.1 %
2-Naphthylamine (HPLC) ----- max 0.01%
Water ----- max 5.0%
Suitability for detection of sulfonamide ----- passes test
and determination of nitrite

4. Sodium Chloride

GR/AR Grade Sodium Chloride is used.

Specifications:

NaCl

Reagent grade: GR /AR

Preferred manufacturer: MERCK

Mol wt. 58.44 g/mol

Melting point 801⁰C

Boling point 1413⁰C

Solubility in water / 20⁰C 360g/l

Assay (by argentometry) ----- min 99.5%

pH (5%, water) ----- 5-8

5. Potassium Nitrate

Specifications:

KNO₃

Reagent grade: GR /AR

Mol wt. 101.11g/mol

Melting Point 334⁰C

Solubility in water / 20⁰C 320g/l

Thermal decomposition above 400⁰C

Assay (by acidimetry) ----- min. 99%

pH (5%, water) ----- 5.0-7.5

6. Powdered Sugar

Purified white sugar (sucrose) of size 80 mesh. (any quality requirement??)

7. Citric Acid

Specifications:

Citric Acid Monohydrate

C₆H₈O₇.H₂O

Reagent grade: GR /AR

Mol wt. 210.14 g/mol

Melting range 135-153⁰C

Solubility in water 20⁰C 1630 g/l

Assay (by acidimetry)----- 99.5 - 102 %

Substances insoluble in water ----- max 0.005 %

Chloride (Cl) ----- max 0.0005%

Phosphate (PO₄) ----- max 0.001%

Sulfate (SO₄) ----- max 0.002%

Copper (Cu) ----- max 0.0005%

Iron (Fe) ----- max 0.003%

Lead (Pb) ----- max 0.002%

Oxalates (as C₂H₂O) ----- max 0.05%

Reaction against sulfuric acid ----- passes test

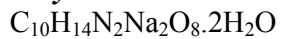
Residue on ignition (as sulfates) ----- max 0.01%

8. EDTA Na

Specifications:

Reagent grade: GR /AR

Ethylene diamine tetraacetic acid disodium salt



Mol wt. 372.24 g/mol

Assay (by complexometry on anhydrous substance) ----- min.99%

Identity ----- IR spectrum passes test

pH (5% in water) ----- 4-5

Annex – II

Figures and Photographs

Instructions For Use



Fluoride Test Kit



1. Take 15ml. of water sample



2. Add Reagent A.



*3. Add Reagent B.
Close the bottle.
Shake the bottle
gently. Compare the
colour developed
against the colour chart
after 15 minutes.*



Colours Developed With Different Concentrations Of Fluoride.

*Fluoride Monitoring Kit
Developed by
National Chemical laboratory
Pune-411008.*