DRAFT TANZANIA STANDARD

TBS/CDC 7(5419) P3 Guideline for internal monitoring of Iodation of salt in small scale operations

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TANZANIA BUREAU OF STANDARDS

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Foreword

This Draft Tanzania Standard is being developed by the Industrial and Laboratory Chemicals Technical Committee under supervision of the Chemicals Divisional Standards Committee and it is in accordance with the procedures of the Bureau.

A need to prepare guideline for internal monitoring of Iodated salt in small scale operations has risen because of importance of iodine as a micronutrient needed for human health. The problems of iodine deficiency disorders include mental retardation, brain damage, growth impairment to children and goiter, which can be prevented by use of iodized salt and effective monitoring of iodine nutrition.

The guide is meant directly contributing the overall effort to strengthen food fortification in our country. The use of this guide should help strengthen food control activities in our country in order to deliver safe and quality fortified foods to the population.

In preparation of this guide, assistance was obtained from *Manual for internal monitoring of iodized salt in small scale operation* Published by East, Central and Southern Africa Health Community (ECSA-HC).

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Introduction

Iodine deficiency is the most common cause of preventable mental retardation and brain damage in the world. Also, it decreases child survival, causes goiters, and impairs growth and development. Iodine deficiency in pregnant women causes miscarriages, stillbirths, and other complications. Children with iodine deficiency disorder (IDD) can grow up stunted, mentally retarded, and incapable of normal movements, speech, or hearing.

Iodine deficiency and the problems it causes can be prevented by the use of adequately iodated salt and effective monitoring of iodine nutrition. However, the level of iodation of salt should be carefully regulated as inadequate levels in the diet will have no noticeable benefit, while excessive doses are wasteful, and may actually result in detrimental health effects. A reliable, simple, and rapid method is therefore needed for routine monitoring of iodine levels in iodated salt at the manufacturing, distribution, retail and household levels.

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DRAFT TANZANIA STANDARD **TBS/CDC 7(5419) P1**

Guideline for internal monitoring of iodation of salt in small scale operations

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1. Scope

This guideline describes the internal monitoring for salt iodation carried out in small scale operations to assure that, a produced salt is safe and is adequately fortified using potassium iodate.

2. Normative reference

TZS 132/EAS 35 *Edible common salt – Specification*

3. Terms and definitions

3.1

Iodation

process of adding iodine as potassium iodate to raw salt

3.2

Iodated salt

salt obtained from iodation of raw salt with potassium iodate

3.2

Premix

substance or product consisting of ready-mixed material

4. Requirement

3.1 General

Raw salt should only be iodized using potassium iodate because iodate compounds are less soluble than iodide in water, less reactive than iodide and therefore are more stable in contact with food. Iodate has another advantage over iodide that, it is easier to detect through simple chemical reaction that involves formation of a purple colour when in contact with starch. This simple detection method has allowed the use of a simple rapid test kit (RTK) to detect the presence of iodine in salt.

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3.2 Quality assurance of the potassium iodate (or premix) receipt, storage and use

3.2.1 *Objectives and Accountability*

The purpose of the Quality Assurance of the Potassium Iodate (or premix) receipt, storage and delivery is to ensure that:

3.2.1.1 The salt iodation unit always has enough iodate inventories.

3.2.1.2 Potassium iodate (or premix) is stored under adequate conditions and is used based on the "first-in, first-out" basis.

3.2.1.3 Personnel in charge of the salt iodation units shall be responsible for achieving the above objectives.

3.2.2 *Procedures*

3.2.2.1 *Receipt and Storage of potassium iodate or premix of iodate*

Every time a new lot of pure potassium iodate or a premix of iodate is received in the unit, check that the containers are sealed, and that a certification stating the amount of iodine per kilogram be included. Record in a form similar to Tables A-1 and A-2 the amounts of potassium iodate or premix of iodate, respectively, that is received, the lot number, and the name of the supplier.
Store the iodate or the premix in a clean dry area and away from chemical products or other potential contaminants. Stack the iodate or the premix in such a way that the first that arrived is used first, following the "first-in, first-out" system.

3.2.2.2 *Preparation of premix*

If the iodation unit produces its own premix of iodate, prepare it as follows:

Weigh 10 kilogram of potassium iodate and 90 kilograms of salt (or calcium carbonate), or other amounts as required. Use Table A-1 for recording. Shake well the mixture, and pack it into clean bags. Label the premix of iodate with the following information: Iodate Premix, Content of iodine = 60 g/kg, Lot number, and Date. Write down the amount of the premix produced both in Table A-1 as well as in Table A-2.

Note:
Potassium iodate as salt usually contains 600 g of iodine per kilogram. Dry premixes of iodate, where it is combined either with salt or calcium carbonate, are usually prepared to contain 50-60 g of iodine per kilogram; *i.e*. a dilution 1 to 12 and 1 to 10, respectively, of potassium Iodate.

3.2.2.3 *Use of the premix to iodate salt*

Estimate the amount of salt to be iodated in kilograms, and divide that amount by 1 000. This is the amount of premix of iodate to be used. Record both the amount of premix that is used as well as the amount of salt that is iodated in Table A-2.

Apply the premix of iodate as usual, during grinding of the salt, or to be mixed as batch either manually or using appropriate equipment.

3.2.2.4 *Packaging and planning*

Iodated salt should be packed in bags labeled with the name of the Iodation unit, address, brand, iodine level, and date of production and expiration.

3.2.2.5 *Records and reporting*

Keep the records in Table A-1 and A-2 and show them to the inspectors when required.

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Table A-1 Inventory control log of potassium iodate in stock and premix production

Harvest Year……………………

Date	Received				Amount Used (B)	In Stock (C) (C)=(A) – (B)	Amount of Premix Produced (Kg)
	Supplier name	Kg (A)	Lot #	Content of Iodine (g/kg)			

Table A-2 Inventory control log of potassium iodate premix in stock and salt production

Harvest Year …………………

Date	Received/Produced locally				Amount Used (B)	In Stock (C) (C)=(A) – (B)	Amount Of salt Produced (Kg)
	Supplier name	Kg (A)	Lot #	Content of Iodine (g/kg)			

3.3 Quality control of iodated salt

The quality control of salt iodization through testing is critical to the overall success of any iodine deficiency disorders (IDD) elimination program.

3.3.1 *Objectives*

The purpose of Quality Control of the fortified salt is to ensure that:
All salt samples contain iodine levels >30 mg/kg (or whatever is the regulatory minimum).
80% samples have iodine levels within the factory level (e.g. 40 mg/kg to 60 mg/kg) and the average concentration is close to the addition level at the factory (e.g. 45 mg/kg).
Fortified salt is packaged and labeled as required in TZS 132/EAS 35

3.3.2 *Methods for measuring iodine in salt*

The quality control of salt iodization through testing is critical to the overall success of any iodine deficiency disorders (IDD) elimination program

Two techniques available for measuring iodine levels in salt are; -

i. Rapid salt testing kits (RTK)
ii. Titration method for salt iodine analysis

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3.3.2.1 *Rapid salt testing kits*

Rapid "spot" tests are highly sensitive tests that can be performed rapidly to detect levels of iodine in salt, and play an important role in salt monitoring programs.

Spot tests are technically simple, rapid check methods for detecting salt iodine, and can be readily performed outside the laboratory

3.3.2.1.1 *Categories of the rapid salt iodine tests:*

i. *Qualitative tests:*
These indicate only the presence or absence of iodine over a broad range, e.g., a positive test result may simply indicate a salt sample with iodine of content somewhere between 5 to 100mg/kg.

ii. *Semi-quantitative tests:*
These give an approximate concentration of the iodine content in salt. These tests generally use some forms of colour chart by which the iodine levels in a salt sample are estimated, e.g., <10 mg/kg, 10-24 mg/kg, 25-40 mg/kg.

Various spot test methods basically use the same general reaction mechanism: a starch-based reagent solution which produces a blue colour when iodine is present in the salt sample.

3.3.2.1.2 *Purpose of Salt Iodine Spot Tests*

Because spot tests are simple, rapid, and are easily applied in field settings, individuals without specific
chemistry training can easily verify whether a salt sample has been iodized. Spot tests can be used at the production, distribution, retail, and household levels. They are particularly appropriate for small scale salt producers who may not be able to achieve the level of sophistication needed to establish more quantitative laboratory titration methods.

3.3.2.1.3 *Quality control aspects*

While spot tests and kits are relatively simple, it is still important that routine testing of a "control" salt sample be performed whenever other samples are being checked. This control can be a salt sample known to have a positive iodine content, which is taken into the field with each kit and tested along with the unknown samples at each site. Should this known positive sample give a negative or lower than expected result, then one may suspect reagent deterioration or expiry of kit.

3.3.2.1.4 *How to use the Salt Rapid Test kit*

Fill a cup with a few spoons of the salt for testing and spread open the test solution ampoule by piercing it with a pin and add the drops of the test solution on the salt surface till the surface is flooded. The reaction liberates iodine in the salt, and depending on the content of iodine, the solution will change the colour of the salt. The intensity of the colour varies with the amount of iodine and by matching it with the colour chart the range of iodine can be ascertained.

3.3.2.1.5 *Records and reporting*

Keep the records in Table B and show it to the inspectors when required.

Table B Quality control log of iodine test in salt using salt rapid test kit (RTK)

Date ……………………

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S/N	Time	Test result(s)	Remark(s)

3.3.2.2 *Titration method for salt iodine content analysis*

This method is applicable to those wishing to establish laboratories for salt monitoring purposes. While iodine titration methods are reasonably simple, they are quantitative chemical tests, and therefore demand a certain degree of analytical skills, as well as adequate funds to setup and maintain a modest laboratory. In addition, the analyst will need some expertise in order to maintain quality assurance records for method and result validation.

For the above reasons, these guidelines on salt iodometric titration are primarily aimed at-
Medium to large scale salt producers (e.g.> 1 000 tones per year), as part of their factory quality control, and government agencies responsible for quantifying the iodine content of salt obtained from production units, households, markets, warehouses and imports entry points.

Note: For description of the reaction see annex A.

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Annex A
(Normative)

Titration method for salt iodine content analysis

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A.1 Principle

Iodine is added to salt in the form of potassium iodate (KIO_3). To determine the concentration of the added iodate, salt is dissolved in slightly acidic solution to which excess potassium iodide (KI) is added. The iodate from the salt reacts with iodide (I^-) toform iodine (I_2) and triiodide ($I_3{}^-$), which is very soluble in water. A yellow color is formed. If a starch solution is added, a blue colored complex is then formed with triiodide. The amount of iodine in solution is determined by a colorimetric titration with a standard thiosulphate solution, which removes the iodine, and hence the disappearance of the blue color. The end point is visually determined by the disappearance of the blue color when no more iodine is present.

The related chemical equations are various steps are as follows:

Formation of iodine from the iodate in salt solution

$$IO_3{}^-{}_{(aq)} + 5I^-{}_{(aq)} + 6H_3O^+{}_{(aq)} \rightarrow 3I_{2(aq)} + 9H_2O \ldots\ldots\ldots\ldots\ldots\ldots.\ldots (1)$$

Formation of blue complex of starch and triiodide

$$I_{2(aq)} + I^-{}_{(aq)} \leftrightarrow I_3{}^-{}_{(aq)} + \text{Starch} \rightarrow \text{Blue Complex} \ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Reduction of iodine by thiosulphate

$$I_3{}^-{}_{(aq)} + \text{Starch} + 2S_2O_3{}^{2-}{}_{(aq)} \rightarrow S_4O_6{}^{2-}{}_{(aq)} + 3I^-{}_{(aq)} \ldots\ldots\ldots\ldots\ldots.(3)$$

(Blue solution) (Colorless)

The combination of equations implies that one equivalent of iodate ($IO_3{}^-$) reacts with 6 equivalents of thiosulphate. Therefore, in terms of weight, one equivalent of thiosulphate means 35.6667 grams of potassium iodate (FW/6 = 214/6), or 21.222 grams of iodine, knowing that potassium iodate contains 59.5% iodine.

A.2 Critical Points and Cautions

The starch solution should be freshly prepared because it is easily destroyed by microorganisms. In any case, each time that the method is used, a control sample of iodized salt with a known amount of iodine should be analyzed first to confirm its reliability.

A.3 Equipment and Materials

A.3.1 *Beaker* (250 mL - 500 mL)
A.3.2 *Burettes or graduated pipettes* (to measure 10 mL- 50 mL)
A.3.3 *Glass rods*
A.3.4 *Graduate cylinder* (50 mL)

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A.3.5 *Containers calibrated by volume to weigh approximately 10 g of salt.*
A.3.6 *Graduated pipettes*, 1 to 5 (5 mL)

A.4 Reagents

A.4.1 *Sodium Thiosulfate Solution (0.005 N)***:** Dissolve 1.24 g $Na_2S_2O_3.5H_2O$ (FW= 248) in 1 L of distilled water and store in a cool dry place. Solution is stable for 1 month. This amount is sufficient for about 200 samples.

A.4.2 *Sulfuric Acid Solution (2N)***:** Using concentrated sulfuric acid, slowly add 60 mL to 900 mL of distilled water and mix. The solution is cooled down and made up to 1 L. This amount is sufficient for about 1,000 samples.

A.4.3 *Potassium Iodide (10%)*: Dissolve 100 g of potassium iodide in water and make up to 1L. Store in a cool dark place. This solution is stable for 6 months provided there is no color change. This amount is sufficient for about 200 samples.

A.4.4 *Starch Solution*: Weigh 1 g of soluble starch into a 100 mL beaker and add 10 mL of water, heat to dissolve. Prepare a saturated solution of sodium chloride dissolving NaCl in 80 mL of distilled water, heat up the solution until no more NaCl dissolves. Cool the solution and add to the dissolved starch and make up to 100 mL; store in a cool dark place. This amount is sufficient for about 50 samples. Use freshly prepared starch solution. The saturated NaCl solution is stable for 12 months.

A.5 Procedure

A.5.1 *Solubilization of the salt sample*

Mix well the sample of salt and weigh accurately 50 g and dissolve in a 250 mL beaker. Transfer to 250 mL volumetric flask and fill to the mark. Transfer 50 mL of the salt solution to a 200 mL Erlenmeyer flask
Using a graduated pipette, add 1 mL of the 2 N sulfuric acid to the salt solution and mix thoroughly. Add 5 mL of the 10% potassium iodide solution using a measuring cylinder or a pipette; if iodine is present a yellow solution is formed. Cover the flask and put in the dark or in a cupboard for 10 min.

A.5.2 *Titrating iodine in the salt solution*

Fill the 50 mL burette with the thiosulphate solution in readiness for titration; titrate the iodine solution in the flask with the thiosulfate and stop the titration when the dark color of the solution turns to pale yellow. Agitate the salt solution continuously. Add 2 mL of the starch solution and the solution should turn blue. Resume titration with thiosulfate until the blue color disappears. Agitate the salt solution continuously and gently. Record the volume from the burette or serologic pipette as accurately as possible to the nearest 0.1 mL

A.6 Calculations

The amount of iodine in the salt is determined using the following equation.

Iodine in salt (mg/kg) = $\frac{(N_t \times 21.222\ V_t \times \frac{V_f}{v})}{W}$

If the procedure is strictly followed, the prior equation can be simplified to:

Iodine in salt (mg/kg) = $\frac{(0.005N \times 21.222\ V_t \times \frac{250}{50})}{0.05Kg}$

$= 10.61 \times V_t$

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Where;

N_t	is a Normality of thiosulphate
V_t	is the volume thiosulphate in mL
21.222	is an equivalent weight of iodine
V_f	is the final volume in mL
v	is the used volume in mL
W	is the weight of salt used in kg

The following chart (Table C) presents the equivalences between volume of the thiosulphate solution used and the amount of iodine in salt, under those conditions;

Table C: Conversion Chart for Iodine in Fortified Salt (mg/kg)

Salt fortified with Iodate or Iodide

Iodine (mg/kg)	Volume Thiosulfate (mL)	Iodine (mg/kg)	Volume Thiosulfate (mL)	Iodine (mg/kg)	Volume Thiosulfate (mL)	Iodine (mg/kg)	Volume Thiosulfate (mL)	Iodine (mg/kg)	Volume Thiosulfate (mL)	Iodine (mg/kg)	Volume Thiosulfate (mL)
0.1	1.1	2.0	21.2	3.9	41.3	5.8	61.5	7.7	81.6	9.6	101.8
0.2	2.1	2.1	22.2	4.0	42.4	5.9	62.5	7.8	82.7	9.7	102.8
0.3	3.2	2.2	23.3	4.1	43.5	6.0	63.6	7.9	83.4	9.8	103.9
0.4	4.2	2.3	24.4	4.2	44.5	6.1	64.7	8.0	84.8	9.9	104.9
0.5	5.3	2.4	25.4	4.3	45.6	6.2	65.7	8.1	85.9	10.0	106.0
0.6	6.4	2.5	26.5	4.4	46.4	6.3	66.8	8.2	86.9	10.1	107.1
0.7	7.4	2.6	27.6	4.5	47.7	6.4	67.8	8.3	88.0	10.2	108.1
0.8	8.5	2.7	28.6	4.6	48.8	6.5	68.9	8.4	89.0	10.3	109.2
0.9	9.4	2.8	29.7	4.7	49.8	6.6	70.0	8.5	90.1	10.4	110.2
1.0	10.6	2.9	30.7	4.8	50.9	6.7	71.0	8.6	91.2	10.5	111.3
1.1	11.7	3.0	31.8	4.9	51.9	6.8	72.1	8.7	92.2	10.6	112.4
1.2	12.2	3.1	32.9	5.0	53.0	6.9	73.1	8.8	93.3	10.7	113.4
1.3	13.8	3.2	33.9	5.1	54.1	7.0	74.2	8.9	94.3	10.8	114.5
1.4	14.8	3.3	35.0	5.2	55.1	7.1	75.3	9.0	95.4	10.9	115.5
1.5	15.9	3.4	36.0	5.3	56.2	7.2	76.3	9.1	96.5	11.0	116.6
1.6	17.0	3.5	37.1	5.4	57.2	7.3	77.4	9.2	97.5	11.1	117.7
1.7	18.0	3.6	38.2	5.5	58.3	7.4	78.4	9.3	98.6	11.2	118.7
1.8	19.1	3.7	39.2	5.6	59.4	7.5	79.5	9.4	99.7	11.3	119.8
1.9	20.1	3.8	40.3	5.7	60.4	7.6	80.6	9.5	100.7	11.4	120.8

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