TITRATION METHODS FOR SALT IODINE ANALYSIS

INTRODUCTION

There are a number of methods for testing the iodine content of salt, ranging from qualitative "spot" tests which are useful in field settings (see Chapter 10, Rapid Salt Testing Kits, for details), to more quantitative methods, such as iodometric titrations performed in laboratories for validation purposes.

The technical information on salt iodine titration provided in this chapter should assist those wishing to establish laboratories for salt monitoring purposes. While iodine titration methods are reasonably simple, they are still quantitative chemical tests, and therefore demand a certain degree of analytical skill, as well 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 will primarily be aimed at-

- Medium to large scale salt producers (e.g.> 1,000 tonnes per year), as part of their factory quality control.
- Government agencies responsible for quantifying the iodine content of salt obtained from producers, and perhaps other sites, such as households, markets, warehouses and importers.

The technical requirements of iodine titration analysis may limit its use for some, such as small scale producers or field workers who also need to verify salt iodine content. In these situations, use of simpler semi-quantitative, or qualitative spot tests, as described in Chapter 10, would be much more appropriate.

A person with experience in laboratory chemistry techniques would be preferable for performing these tests and maintaining adequate quality assurance records. Such a person could be trained in less than a week. Less experienced persons could be considered to perform the actual titration procedure, but would require a longer training period and greater levels of routine supervision.

Different salt iodine test methods need to be used depending on the form of iodine (iodate or iodide) used in fortification. The iodometric method for iodate will not detect the iodine content of a salt sample fortified with potassium iodide, and vice versa. If the form of iodine in the salt sample is unknown, a simple spot check method can be employed for verification (see Chapter 10 for relevant details).

Information regarding the testing of salt fortified with potassium iodate (KIO_3), which is recommended in developing countries due to its greater stability than potassium iodide (KI), is detailed below. Information includes the chemical basis for the titration-method, reagent preparation and stability, step by step procedure and precautions, and cost details.

The second part of this chapter provide details regarding quality control practices necessary for laboratories to ensure that reliable data are generated. This includes steps required for the initial method validation, with worked examples, as well as more general routine quality control and quality assurance issues.

Appendices are also provided with information about laboratory water requirements, a listing of all necessary equipment, and information about an alternative titration method which can be used if salt is known to be fortified with potassium iodide instead of potassium iodate.

TITRATION METHOD FOR IODATE CONTENT

Description of Reaction

The iodine content of iodated salt samples is measured using an iodometric titration, as described by DeMaeyer, Lowenstein, and Thilly, (1979). The reaction mechanism can be considered in two steps (See Box 1):

Reaction 1: Liberation of free Iodine from salt

- Addition of H_2SO_4 liberates free iodine from the iodate in the salt sample.
- Excess KI is added to help solubilise the free iodine, which is quite insoluble in pure water under normal conditions.

Reaction 2: Titration of free Iodine with thiosulfate.

- Free iodine is consumed by sodium thiosulfate in the titration step. The amount of thiosulfate used is proportional to the amount of free iodine liberated from the salt.

- Starch is added as an external (indirect) indicator of this reaction, and reacts with free iodine to produce a blue colour. When added towards the end of the titration (that is, when only a trace amount of free iodine is left) the loss of blue colour, or endpoint, which occurs with further filtration, indicates that all remaining free iodine has been consumed by thiosulfate.

Box 1:

Reaction Steps for Iodometric Titration of Iodate

1. $IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O$

   (from salt) (from KI) (from H_2SO_4)

2. $2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$

   Sodium thiosulfate — Iodine — Sodium iodide — Sodium terathionate

REAGENT PREPARATION

Water Requirements for Reagent Preparation

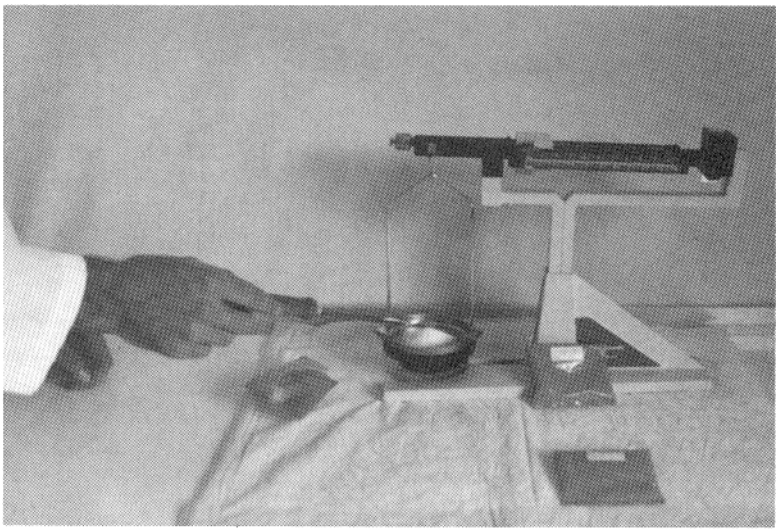
Water required for this method should be boiled, distilled water, which requires provision of a distillation unit. As a simpler alternative, regular tap water treated with a mixed bed deionizing resin can be used, thus avoiding the need for an expensive distillation unit (See Appendix 11-2 for further details on preparation of this water.)

- **0.005M Sodium thiosulfate ($Na_2S_20_3$):** Dissolve 1.24g $Na_2S_20_3$-$5H_20$ in 1000mL water. Store in a cool, dark place. This volume is sufficient for 100-200 samples, depending on the iodine content of samples. The solution is stable at least 1 month, if stored properly.
- **2N Sulfuric acid (H_2SO_4)** Slowly add 6mL concentrated H_2SO_4 to 90mL water. Make to l00mLwith water. This volume is sufficient for 100 samples. The solution is stable indefinitely.

Note: Always add acid to water, **not** water to acid, to avoid excess heat formation and spitting of acid. Stir solution while adding acid.

- **10% Potassium iodide (KI):** Dissolve l00g KI in 1000mL water. Store in a cool, dark place. This volume is sufficient for 200 samples. Properly stored the solution is stable for six months.
- **Starch indicator solution:** Make l00mL of a saturated NaCl solution, by adding NaCl to approximately 80mL water in a beaker, with stirring and/or heating, until no further solid will dissolve. This solution is stable for at least one year. Weigh 1g soluble starch into a l00mL beaker, add l0mL water, heat to dissolve. Add saturated NaCl solution to the hot starch solution to make up to l00mL Store in a cool, dark place. This volume is sufficient for 50 samples. The solution is stable for up to one month, and should be heated (not boiled) each day it is used to re-suspend any solids.

Figure 11-1: *Weighing salt sample*



Procedural Steps

Step 1. Weigh l0g of the salt sample into a 250rnL Erlenmeyer flask with a stopper.

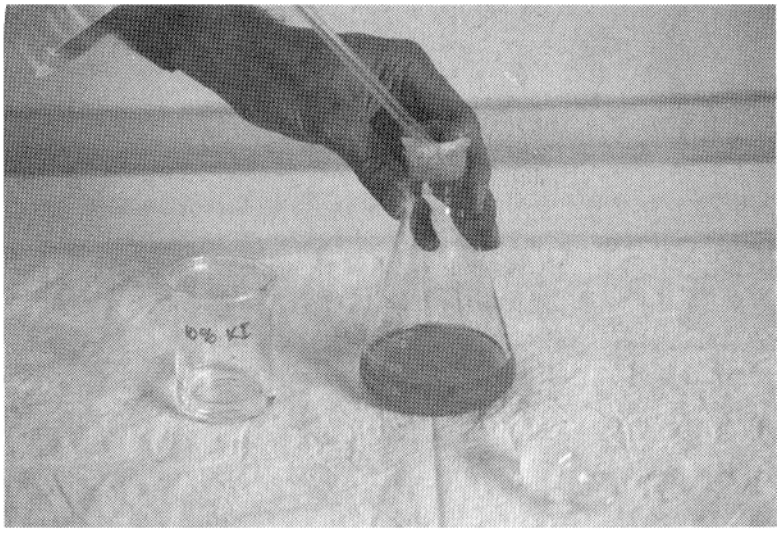
Step 2. Add approximately 30mL water, swirl to dissolve salt sample.

Step 3. Add water to make volume up to 50mL.

Step 4. Add 1mL 2N H_2SO_4.
CAUTION - Do not pipette by mouth.

Step 5. Add 5mL 10% KI. The solution should turn yellow if iodine is present.
CAUTION - Do not pipette by mouth.

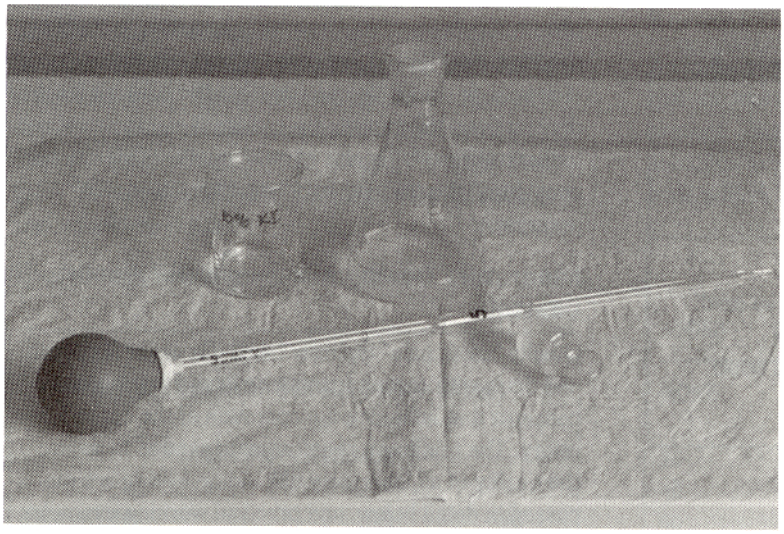
Figure 1 1-2: Addition *of 10%* potassium iodide solution



Step 6. Stopper the flask and put in the dark (cupboard or drawer) for 10 minutes.

Step 7. Rinse and fill burette with 0.005M $Na_2S_20_3$, and adjust level to zero.

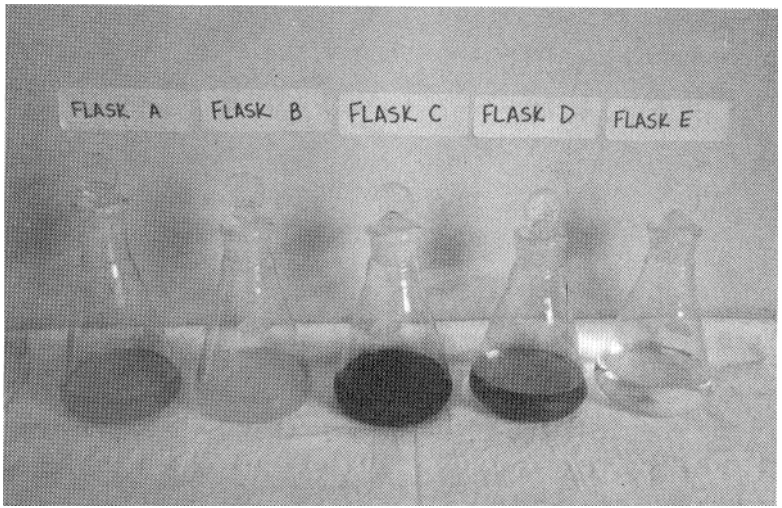
Figure 11-3: Filling the burette with sodium thiosulfate solution.



Step 8. Remove flask from drawer, and add some $Na_2S_20_3$ from the titration burette until the solution turns pale yellow (Flask B shown in Figure 11.4).

Step 9. Add approximately 2mL of starch indicator solution (the solution should turn dark purple) and continue titrating until the solution becomes pink, and finally colourless. (Colour sequence of titration is shown in flasks C, D and E, figure 11.4)

Figure 1 1-4: *This photo shows the various color changes that will be seen* during the titration.
Flask A - after addition *of KI (Step 5);*
Flask B -just prior to addition of starch (Step 8);
Flask C - after starch has been added (Step 9);
Flask D -just prior to titration end-point (Step 9),
Flask E - titration end-point (step 9).



Stop 10. Record the level of thiosulfate in the burette and convert to parts per million (ppm) using the conversion table in Appendix 11-3.

NOTE: Analysis time is approximately 20 minutes per sample.

Precautions

- The reaction mixture should be kept in the dark before titration because a side reaction can occur when the solution is exposed to fight that causes iodide ions to be oxidized to iodine.
- Inaccurate results may occur if starch solution is used while still warm.
- If starch indicator is added too early, a strong iodine-starch complex is formed, which reacts slowly, and gives falsely elevated results.
- The reaction should be performed at mild room temperature (<30°C), since the iodine is volatile, and the indicator solution loses sensitivity when exposed to high temperatures.

Box 2

Salt Iodine Laboratory Costs

Note — A complete laboratory equipment and reagent list is given in Appendix 11–1.

The equipment required to set up the salt iodine titration method described would cost approximately the following, based on US scientific company prices:

US $3,280
if distilled water is to be used

US $2,005
if tap water treated with deionizing resin is to be used

Reagents sufficient for at least 1,000 samples would cost: US$ 330

SALT IODINE METHOD VALIDATION AND QUALITY ASSURANCE

It is of the utmost importance that salt iodine test results be reliable, accurate and timely. This is especially the case if the salt iodine test data is to be used for iodine deficiency programme evaluation and monitoring.

Establishing a salt iodine monitoring system that gives information about how well the salt is fortified is the "first level" in salt iodine quality assurance. However, we must also be sure that the information derived from the monitoring system (i.e., the actual salt iodine test results) is also of good quality. This can be considered the "second level" of salt iodine quality assurance.

Laboratory Quality Assurance and Quality Control

Quality "assurance" typically takes a broader approach, and deals with certain management and organisation concepts that influence the operation of the entire laboratory. The minimum requirements needed to assure the quality of all laboratory salt iodine testing are discussed in detail below, and practical working examples are provided. Figure 11-5 details some of the key elements of salt iodine laboratory quality assurance.

Figure 11-5

Key elements of Total Laboratory Quality Assurance for Salt Iodine

- Salt Sample Recording
- Reagent inventory/batch Checks
- Equipment Checks
- Method Validation
  - Sensitivity, recovery, cross-checks
- Internal Quality Control
  - Establish QC materials
  - Routine QC testing
  - Monitor test Precision
- External Quality Control
  - Establish laboratory network
  - Link industry and Government labs

VALIDATION

During the initial set-up phase of salt iodine titration methods, these four performance characteristics should be thoroughly validated: precision, sensitivity, recovery, and comparison and cross-checking. Each is briefly described below.

Precision

Calculate the percentage Coefficient of Variation (%CV) for repeat analysis of the same sample (at least ten separate estimates). If possible, this should be done on a number of different salt samples that have a range of iodine concentrations, e.g., 25, 50 and 100 ppm. With good technique, and reliable methodology, the precision should be <15% CV.

The following gives a worked example:

SALT IODINE TITRATION (ppm)

SALT SAMPLE NUMBER

RESULT No.	**#1**	**#2**	**#3**
1	18	48	75
2	20	52	68
3	19	50	73
4	16	47	67
5	22	55	70
6	17	48	72
7	21	43	75
8	23	51	66
9	19	55	72
10	20	49	78
M EAN	19.5	49.8	71.6
STD DEV	2.17	3.68	3.86
%CV*	**11.1**	**7.4**	**5.4**

$$* \%\ CV = \frac{\text{Standard deviation}}{\text{mean}}$$

Sensitivity

Establish an estimate of the lowest iodine level that can be reasonably detected by the test method used (i.e., its operational sensitivity). An example of the criterion that might be used to calculate this is the point at which the mean salt iodine concentration (ppm) of samples consistently yields results with a CV >20%.

Recovery

An initial percent recovery should be made to ensure that the test system is capable of detecting all iodine present. This can be done by analysing a series of salt solutions to which known concentrations of iodine have been added. The following is a worked example:

	IODINE		
ADDED	**OBSERVED**	**MEASURED***	**%RECOVERY****
NONE	15		
20	32	17	85
40	53	38	95
60	77	62	103
AVERAGE			94*

MEASURED = OBSERVED value corrected for BASELINE (i.e., value obtained with NO iodine added)

** % Recovery = (MEASURED ppm/ADDED ppm) * 100%

As a guideline, the average recovery, allowing for expected test imprecision, should be between 85 and 115 percent.

Comparison & cross-checking

If possible, an initial sample cross-check should be performed with others as a means of assessing method bias. This could be done either with a laboratory using the same method or compared to alternative techniques e.g., correlation between titration method and spectrophotometric method.

NOTE: PAMM (Program Against Micronutrient Malnutrition) provides a service for those laboratories wishing to cross-check samples for their initial validation. For further information, please contact:

PAMM Laboratory
Centers for Disease Control
Mailstop F20
4770 Buford Highway Ne
Atlanta, Ga, 30341-3724, USA
Phone: 1 404 488 4088
Fax. 1 404 488 4609

ROUTINE QUALITY CONTROL

Once the laboratory method has been validated as above, it must establish and maintain ongoing quality control (QC) data, both internally (or 'in-house') and externally (inter-laboratory), as described below.

Internal or "in-house" Quality Control

Known positive iodized salt sample(s) should be obtained by the laboratory and stored in sufficient quantity for analysis every time salt titrations on unknown samples are run e.g., daily or weekly. By performing multiple analyses on these positive salt samples, a concentration range can be established and used for operational quality control purposes. The following provides a description and worked example of how to calculate and establish a quality control range and a quality control chart.

Establishing and Interpreting a Quality Control Range:

Multiple salt iodine analyses on a known positive salt sample should be performed until approximately 15 to 20 titration results have been obtained. To establish the control range, it is best if these results are obtained over a period of time (e.g., three to four tests per day), rather than all at once (e.g. twenty tests in one day), as this will give a more realistic estimate of true day-to-day and analytic variability.

Once a sufficient number of these test results have been obtained, use a hand calculator or standard statistical formulae to calculate the sample mean concentration (X) in ppm, and standard deviation (SD). The 95% confidence interval can then be calculated and used as the operating control range, as follows:

$$\textbf{Sample Mean } (\bar{X}) \pm 2 \times SD$$

The X - 2 (SD) = the lower confidence limit (L), and X + 2 (SD) = the upper confidence limit (U.) The operating control range is (L, U).

Unless serious technical or performance errors occur during these initial analyses, the above range should reasonably reflect the normal amount of variation expected when using this method over time. Therefore, any future analysis of the same sample should produce a result between the lower and upper limits (i.e., the L - U range), for 95% of test results. Values falling within this range are considered to be 'in control." Only 5% of subsequent test values for this sample should fall outside the established range, provided the method (and technician) is operating normally. Results falling outside the established range are considered potentially suspicious and therefore classed as 'out-of-control.'

Example: A known iodized salt sample was analysed by titration twenty times. For the 20 result values obtained, the calculated sample mean was 32 ppm, and the standard deviation was 2.5. The operating control range (OCR) for this example can be established as:

$$OCR = 32 \pm 2\ (2.5)$$
$$32 \pm 5$$
$$(27, 37)$$

Therefore, the lower control limit is set at 27 ppm, the upper control limit is 37 ppm, and the control range is 27 to 37 ppm. Subsequent test results falling between 27 and 37 ppm are to be considered in control, while any results <27, or >37 ppm are outside the control range, and therefore out-of-control.

Quality Control Charts

Once the above operating control range has been established, standard quality control charts and rules should be used to interpret these control values, decide acceptability of test results, and be kept as a permanent record to verify all unknown sample results.

The quality control chart is prepared as follows:

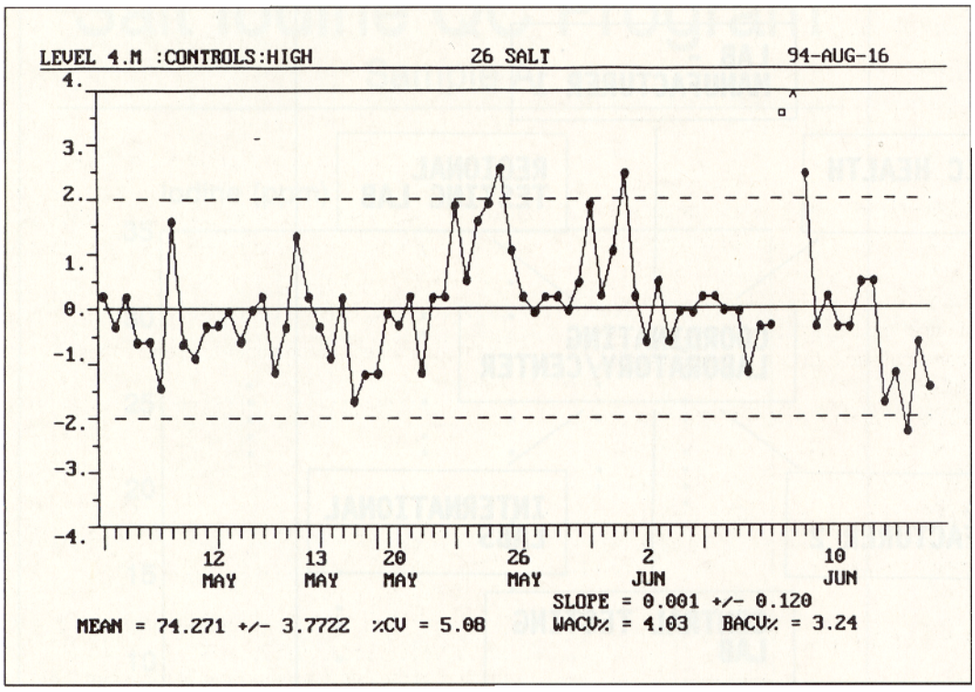
- Use regular linear graph paper to prepare these plots.
- Enter the salt iodine concentration (in ppm) scale for the control on the Y-axis. Make sure the concentration range plotted on this axis extends from less than the lower limit (i.e., <L), to above the upper limit (i.e., >U). For the example given above, which has a calculated range of 27 to 37 ppm, the Y-axis could be scaled from 20 to 40 ppm.
- Find the sample mean concentration value (i.e.,) on the Y-axis scale, and draw a continuous horizontal line across the entire graph paper at this point For the example this would be at 32 ppm.
- Find the lower limit concentration value (i.e., L) on the Y-axis scale, and draw a continuous horizontal line across the entire graph paper at this point For the example this would be 27 ppm.
- Find the upper limit concentration value (i.e., U) on the Y-axis scale, and draw a continuous horizontal line across the entire graph paper at this point For the example this would be 37 ppm.
- The X-axis is used to plot time, i.e., the date on which the control sample is analysed.

Once prepared, this chart is used to plot the specific analysis date, and salt iodine concentration obtained for the control every time it is tested. If the control point obtained is between the two limit lines, then the test is deemed in control, and all results are accepted. Any control values that are plotted outside the two limit lines should be considered as out-of-control, and the results of any corresponding unknown salt samples tested at the same time should also be rejected as unacceptable due to possible method error.

When an out-of-control value is obtained, steps should be taken to identify the possible reason for this event (e.g., use of old reagent, incorrect procedure used or reagent mix-up), and correct the problem. Once resolved, and control values have returned to normal, repeat the previously rejected unknown salt samples to obtain acceptable results.

Figure 11-6 gives a real example of a typical salt iodine quality control chart for a control with a mean salt iodine concentration of 74 ppm, a standard deviation of 3.8, and an operating control range of 66.4 to 81.6 ppm. (Note: The computer software used to generate this chart plots the y-axis in units of standard deviation, as opposed to ppm units, but this will not change the general overall shape of the chart.) As can be seen, such charts are very useful in identifying when problems occur within the test system, and allow corrective action to be taken. In Figure 11-6 the extremely high values above the upper limit (called outliers) were due to a deterioration in the sodium thiosulfate solution which give falsely elevated test results.

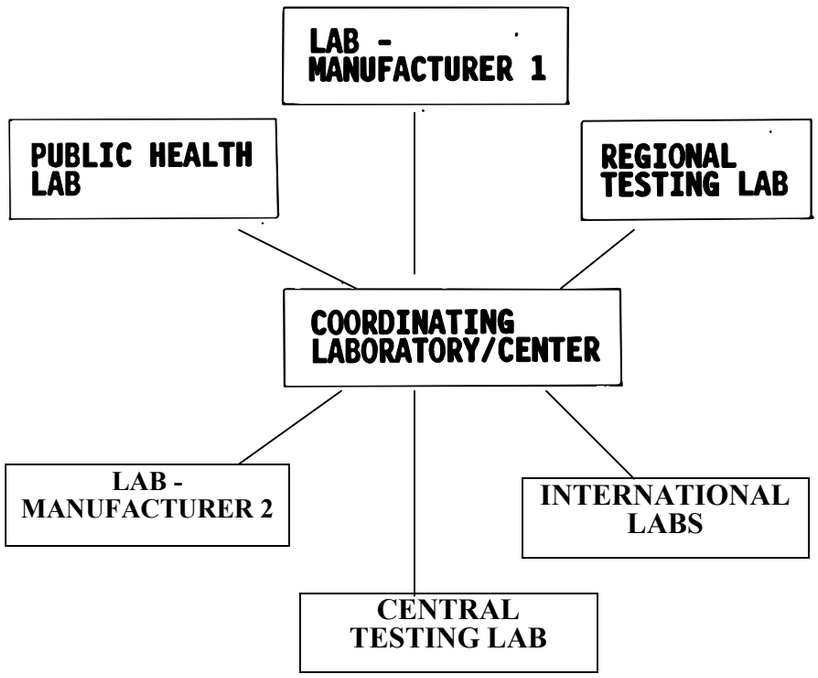
Figure 11-6: *Example of a Salt Iodine QC Chart*



External or Inter-laboratory Quality Control

External cross-checking of samples is the best way for each laboratory to assess its own performance compared to other laboratories, and detect potential method bias or inaccuracy. This type of inter-laboratory exchange should be seen as a supplement to internal QC, not as its replacement! Each salt iodine testing laboratory (government and industry) should be encouraged to form or participate with others in an on-going salt sample exchange network (see Figure 11-7).

Figure 1 1-7: *Extemal Salt Iodine QC Network*



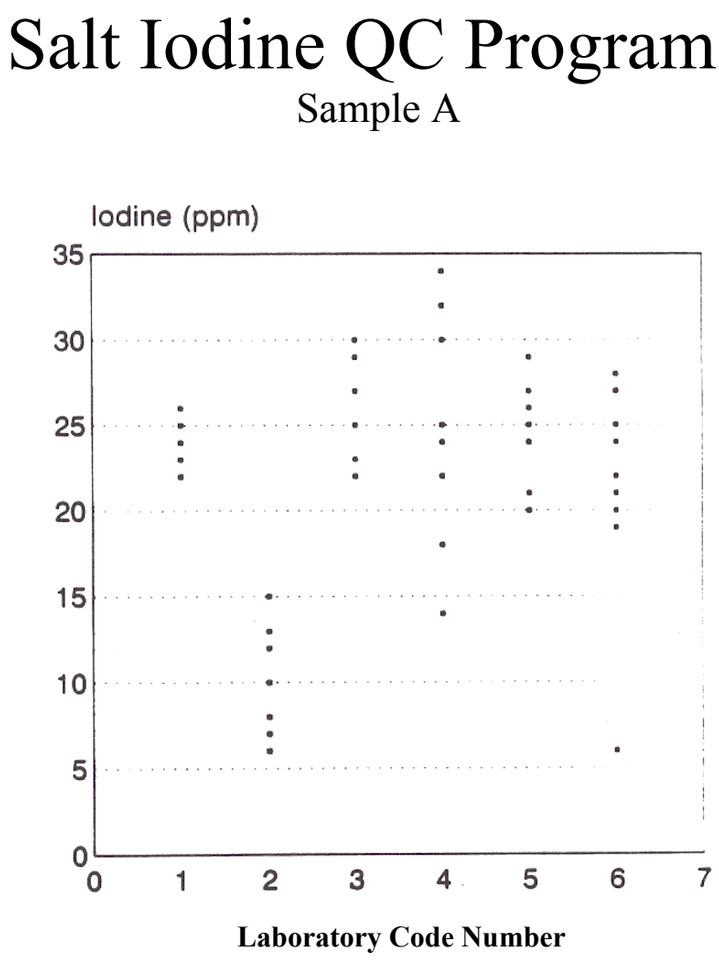
These 'external' comparisons should occur at regular intervals (e.g., two to three times per year). Each time participants in the QC programme are sent unknown salt samples for analysis, and test results should be returned to the QC programme coordinator by a specific date, collated, reviewed, and reported to each participant as soon as possible. Feedback should show how results from each laboratory compare to all others participating in the same programme. However, it is most important that the results be presented anonymously. This is easilv achieved by giving all laboratories a special code number known only by the coordinator and participating laboratory.

The reported results are best presented graphically, as shown in figure 11-8. The value of external comparisons can be seen in this example. While most laboratories yielded similar salt iodine results (20 to 30 ppm), Laboratory 2 showed consistently lower values, while Laboratory 4 had greater imprecision compared to the other laboratories. Also note that laboratory 6 had generally satisfactory results, except for one obvious outlier.

Figure 11-8: *Example of Salt Iodine External QC* Chart

An alternative approach is to have all participating laboratories send salt samples along with their test results to some central coordinating laboratory for analysis and comparison. However this approach will increase the work load at the coordinating laboratory.

Coordination of the external QC programme is probably best done by an independent agency (e.g., Government), and every effort should be made to encourage voluntary participation by all salt testing laboratories, especially industry and producers. Use of awards or certificates sent to regular participants in the programme can be a helpful motivational tool.



OTHER ELEMENTS OF QUALITY ASSURANCE

Salt Sample Recording

Each laboratory must maintain a logbook with sample details recorded in ink, such as:

• Date/time collected
• Date/time received
• Sample specific details (code #, brand, batch, expiry date)
• Date analysed
• Technician performing test
• Test result
• Supervisor's signature
• Date result is reported

An example of a format that could be used in a salt sample analysis logbook is given at the end of the chapter, which could be copied and adapted for use as a "master" form.

Reagent Inventory Details

The laboratory supervisor should ensure all relevant details regarding test chemicals are recorded:

• Chemical brand, quantity, grade and batch/lot number
• Date ordered and received
• Date each "working" reagent is prepared, and by whom
• Give each working reagent an "in-house" lot number

An example of a salt iodine reagent inventory form is given at the end of the chapter, which could be copied and adapted for use as a "master" form.

Instrument Calibration

The exact details depend on the type of test method used, but these should be performed in some routine fashion (e.g., calibrate balance every month). For each calibration keep a record of the following details:

- Instrument tested
- Date calibrated
- Calibrated by whom?
- Outcome (pass/fail, specific reading.)

REFERENCES

1. De Maeyer EM, Lowenstein FW, Thilly CH. "The control of endemic goiter." World Health Organization, Geneva, 1979.

SALT SAMPLE ANALYSIS LOGBOOK

SAMPLE ID'*	DATE SAMPLE COLLECTED	DATE SAMPLE RECEIVED	DATE OF ANALYSIS	TECHNICIAN	RESULT (ppm)	SUPERVISOR'S SIGNATURE*
			ETC.			

** Sample ID = code number, brand, batch, expiry date etc.

SALT IODINE REAGENT INVENTORY

CHEMICAL:_____

*DATE ORDERED	DATE RECEIVED	BRAND, QUANTITY	BATCH/LOT NUMBER	DATE WORKING REAGENT PREPARED	TECHNICIAN	WORKING SOLUTION LOT No.
			ETC.			

APPENDIX 11-1

Equipment and Reagents Required to Establish a Salt Iodine Laboratory

Many items on the following list of equipment and reagents can be procured through UNICEF Copenhagen Supply Division, or various scientific supply companies.

QUANTITY		UNICEF CODE
1	Balance, Four-beam pan Sensitivity = 0.01g, Capacity = 410g (e.g., Fisher Scientific Cat. No. 02-020-411)**	
1	Set of weights for above (e.g., Fisher Scientific Cat. No. 02-314)	
4	Flask. volumetric, 1000mL (e.g., Fisher Scientific Cat. No. 10-210G)	
2	Flask, volumetric, l00mL (e.g., Fisher Scientific Cat. No. 10-119-10D)	
2	Measuring cylinder, l0mL (e.g., Fisher Scientific Cat. No. 08-552-4H)	
12	Measuring cylinder with stopper, l00mL	09 374 30
12	Beakers, Pyrex	09 160 00
	Flasks, Elenmeyer (conical) with stopper, 250mL (e.g., Fisher Scientific Cat. No. 10-098E)	
4	Pipette, volumetric, 1mL	09 676 00
4	Pipette, volumetric, 5mL	09 679 05
4	Burette w/straight stopcock l0mL	09 239 00
4	Burette stand	09 767 00
2	Laboratory safety glasses (e.g., Fisher Scientific Cat. No. 17-286-1C)	
1	Parafilm, for covering beakers (e.g., Fisher Scientific Cat. No. 13-374-12)	
12	Glass bottles with stoppers for reagents, 250mL	09 194 50
4	Funnel, lab, plain, diam. 65mm	09 450 00
4	Watch glass, 75mm diam.	09 888 00
6	Spatula Lab single blade 150mm SS lenqth	09 699 10
4	Dropper bottle, glass 25-60mL	09 190 00
1	Desiccator plain Scheibler l50xl50mm	09 374 60

QUANTITY		UNICEF CODE
	NOTE: If distilled water is to be used, the following equipment is required:	
1	Water Still 4L/day electric 220V	01 676 00
1	Hot plate 220V	20 004 02
1	Rod, stirring, flint glass assorted pkt	09 686 00
	OR	
	NOTE: If tap water treated with deionizing resin is to be used (see Appendix 11.2), the following equipment replaces the 3 previously listed items:	
2	Flask, Erlenmeyer, 4L (e.g., Fisher Scientific Cat. No. 10-040P)	
1	Whatman Filter Paper, 15cm diameter (e.g., Fisher Scientific Cat. No. 09-805G)	
1	4L Polyethylene carboy for water storage (e.g., Fisher Scientific Cat. No. 02-963AA)	
1 kg	Mixed bed deionizing resin 1.5meq/mL, 300-1,180µm, mesh size 20-50 (e.g., Fisher Scientific Cat. No. 31038, 0.1 cubic feet - this should be enough to provide sufficient water for at least one year).	
1	Hotplate/stirrer, 220V (e.g., Fisher Scientific Cat. No. 11-501-7SH)	
3	Magnetic stirring bars (e,g., Fisher Scientific Cat. No. 14-511-70)	

REAGENTS REQUIRED

Sodium Thiosulfate Pentahydrate, ANALAR, 500g
(e.g., Fisher Scientific Cat. No. S445-500)
(Sufficient for 50,000 samples)

Sulfuric Acid, concentrated, 2.5L
(e.g., Fisher Scientific Cat. No. A298-212)
(Sufficient for 40,000 samples)

Potassium Iodide, 500g

Sodium Chloride, 3kg
(e.g. Fisher Scientific Cat. No. S271-3)
(Sufficient for 3,000 samples)

Soluble starch, 500g
(e.g. Fisher Scientific Cat. No. S516-500)
(Sufficient for 25,000 samples)

** Fisher Scientific, 50 Fadem Road, Springfield, NJ, 07081, USA Fax: 201-379-7415, ATTENTION: Jackie Dubeau

APPENDIX 11-2

USE OF TREATED TAP WATER WITH DEIONIZING RESIN AS AN ALTERNATIVE TO DISTILLED WATER

The resin required (as per Appendix 11-1) is a mixed bed resin, containing cation and anion exchange beads. Deionization occurs by exchanging cations with hydrogen, and anions with hydroxyl on the resin. In this way, all ionic species are removed from the water.
e.g., Resin-H + Resin-OH + NaCl → Resin-Na + Resin-Cl + H_20

The resin contains a colored dye (e.g. purple) irreversibly bound to the anion exchange resin, which turns from purple to gold when the exchange capacity is exhausted.

To deionize water for use in the laboratory, follow these steps:

Step 1. Add resin to a conical flask or beaker, covering the base with approximately 1.5cm of resin. Usually a 2 - 5 L flask is used; the bigger the flask, the more resin needed.

Step 2. Fill the flask with good quality tap water (distilled water can also be used if available) and mix on laboratory stirrer for approximately one to three hours. Alternatively, water can simply be left in the flask with the resin for a longer period of time (24 hours), with occasional stirring and then let resin settle.

Step 3. Decant the water from the resin, making sure not to leave the resin dry. ALWAYS LEAVE AT LEAST 1cm OF WATER ON THE RESIN. If the resin is allowed to dry out it must be discarded, since the ion exchange capability may be greatly reduced.

Step 4. To ensure complete removal of resin particles that may float on the surface, simply pass resin-treated water through standard laboratory-grade filter paper.

Mixed bed resins are not normally regenerated after exhaustion because of the difficulty of separating the two resin components, and proper re-mixing. However, if you wish to regenerate the resin after it has changed colour, you must separate the anion and cation exchange resin beads. Shake the resin in twice its volume of water, let it settle, and decant the top layer containing the less dense anion exchanger. Repeat until separation is complete.

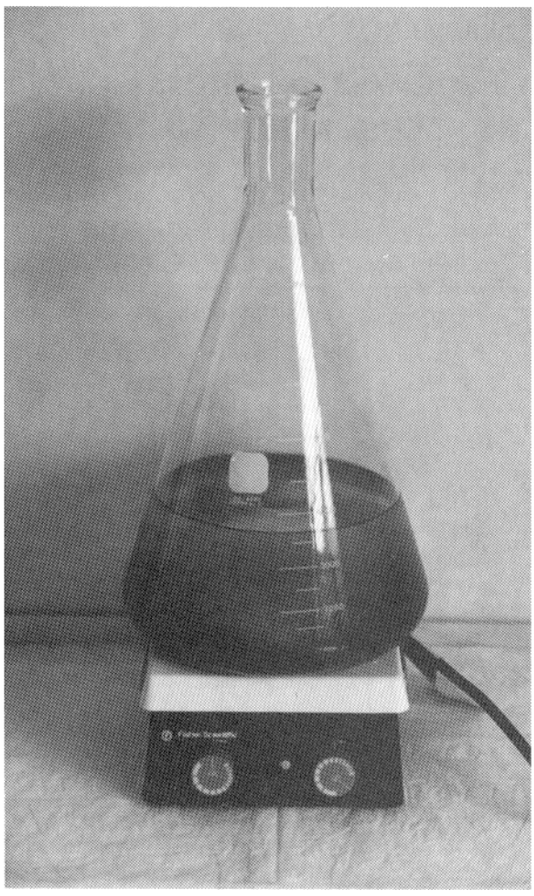


Figure 11-9 *Mixing resin procedure*

Regenerate the cation exchanger using three times the volume of 3N HCl and rinse with four volumes of deionized water. Check that the pH is <5. Regenerate the anion exchanger with at least ten volumes of 3N NaOH and rinse with deionized water until the pH <9. Mix the resins thoroughly by agitating with a stirring rod.

The mixed bed resin has a shelf life of two years at room temperature. This shelf life may be extended by storing in the refrigerator.

APPENDIX 11-3

CONVERSION TABLE : IODINE CONTENT IN PARTS PER MILLION

BURETTE READING	PARTS PER MILLION (ppm)	BURETTE READING	PARTS PER MILLION (ppm)
0.0	0.0	5.0	52.9
0.1	1.1	5.1	54.0
0.2	2.1	5.2	55.0
0.3	3.2	5.3	56.1
0.4	4.2	5.4	57.1
0.5	5.3	5.5	58.2
0.6	6.3	5.6	59.2
0.7	7.4	5.7	60.3
0.8	8.5	5.8	61.4
0.9	9.5	5.9	62.4
1.0	10.6	6.0	63.5
1.1	11.6	6.1	64.5
1.2	12.7	6.2	65.6
1.3	13.8	6.3	66.7
1.4	14.8	6.4	67.7
1.5	15.9	6.5	68.8
1.6	16.9	6.6	69.8
1.7	18.0	6.7	70.9
1.8	19.0	6.8	71.9
1.9	20.1	6.9	73.0
2.0	21.2	7.0	74.1
2.1	22.2	7.1	75.1
2.2	23.3	7.2	76.2
2.3	24.3	7.3	77.2
2.4	25.4	7.4	78.3
2.5	26.5	7.5	79.4
2.6	27.5	7.6	80.4
2.7	28.6	7.7	81.5
2.8	29.6	7.8	82.5
2.9	30.7	7.9	83.6
3.0	31.7	8.0	84.6
3.1	32.8	8.1	85.7
3.2	33.9	8.2	86.8
3.3	34.9	8.3	87.8
3.4	36.0	8.4	88.9
3.5	37.0	8.5	89.9
3.6	38.1	8.6	91.0
3.7	39.1	8.7	92.0
3.8	40.2	8.8	93.1
3.9	41.3	8.9	94.2
4.0	42.3	9.0	95.2
4.1	43.4	9.1	96.3
4.2	44.4	9.2	97.3
4.3	45.5	9.3	98.4
4.4	46.6	9.4	99.5
4.5	47.6	9.5	100.5
4.6	48.7	9.6	101.6
4.7	49.7	9.7	102.6
4.8	50.8	9.8	103.7
4.9	51.9	9.9	104.7

APPENDIX 11-4:

ALTERNATIVE TITRATION FOR IODIDE CONTENT

Description of Reaction

While use of potassium iodide (KI) is not common in many developing countries for salt fortification, basic details of a titration method (De Maeyer EM, Lowenstein FW, Thilly CH, 1979) suitable for analysing salt iodized with KI are provided here.

The reaction mechanism for this iodometric titration is as follows:

Reaction 1: Potassium **iodide** is dissolved from the salt.

Reaction 2: Bromine water oxidizes iodide ions to free iodine. Sodium sulfite and phenol are added to destroy excess bromine so that no further oxidation of I- can occur before KI solution is added.

Reaction 3. : The titration reaction with thiosulfate is the same as that described in the iodate method earlier.

Box 3

Reaction steps for Iodometric Titration of Iodide

1. $KI \rightarrow K^+ + I^-$
2. $Br_2 + 2I^- \rightarrow 2Br^- + I_2$
3. $I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$

REAGENT PREPARATION

Water preparation is the same as for procedures described in section on "Titration Method for Iodate content," page 86.

1. Methyl Orange lndicator - Dissolve 0.0lg methyl orange in l00mLwater.

2. *2 N Sulfururic Acid* - Add 5.56mL concentrated H_2SO_4 to 90mL water, make to l00mL with water.

3. *Bromine Water* - Place 5mL in a small flask, (keep in fume hood due to dangerous fumes)

4. *Sodium Sulfite Solution* - Dissolve lg sodium sulfite in l00mLwater

5. *Phenol Solution* - Dissolve 5g phenol in l00mL water

6. Potassium Iodide Solution - Dissolve l0g potassium iodide in l00mL water

7. *Sodium Thiosulfate Solution* (0 005N - Dissolve 0.124g sodium thiosulfate pentahydrate in l00mL water

8. *Starch Solution* - Dissolve 1g soluble starch in l00mL water, with heating

(All the above need stoppered flasks and should be stored in the dark)

Procedure

Step 1. In a 250mL Erlenmeyer flask place l0g of salt sample and 50mL water. Swirl to dissolve.

Step 2. Add 6 drops of methyl orange indicator (solution turns pale orange). Add 2N H_2SO_4 dropwise (1 drop or until a pink colour change). This is done to neutralise the reaction mixture.

Step 3. Add 0.5mL bromine water (solution changes to yellow).

Step 4. Add sodium sulfite solution, dropwise, until solution turns pale yellow. Wash down the flask sides with H_20.

Step 5. Add 3 drops phenol solution (solution turns clear).

Step 6. Add 1mL 2N H_2SO_4

Step 7. Add 5mL potassium iodide solution. (Turns yellow).

Step 8. Add sodium thiosulfate solution from the titration burette until solution turns pale yellow. Add 1mL starch solution, leading to a dark purple colour. Continue titration until solution becomes colourless.

Step 9. Note the burette reading and convert to ppm using the table in Appendix 11-3.

Precautions - Please refer to precautions listed in the iodate method described earlier.