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Development of a profitable method for salt iodine estimation

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ABSTRACT

The conventional method (CM) uses 500 mg potassium iodide (KI) per test irrespective of the iodine content of salt (1 µg or 1000 µg). Detailed experiments revealed that 3-5mg KI per test was sufficient to estimate even 1000 µg iodine. Therefore, 5 mg KI per test was fixed for iodine estimation by the profitable method (PM), which yielded accurate and reproducible results for the target range of 0-1000 µg iodine.The minimum detectable iodine was 0.997 ± 0.005 µg (mean ± 2SD, 95% CI). The precision, sensitivity, reproducibility and performance of PM was excellent and agreed well with CM (R^2 = 0.999). Extensive studies on PM with potassium iodate standard, iodated salts produced by different methods and market iodated salts showed results on par with CM. Substantial reduction in the estimation cost could be achieved because, 100 salt samples are tested in PM from the quantity of KI used in CM.

KEYWORDS

Iodated salt;
Conventional method;
Profitable method;
Precision;
Reproducibility.

INTRODUCTION

Universal salt iodization (USI) is the best intervention in eliminating iodine deficiency disorders (IDD) in a population[1]. Many countries have provided resources for IDD elimination in their national financial budgets and are progressing towards the goal of USI. Globally, 75 per cent of households have adequate iodine in salt. East Asia and the Pacific had the highest coverage, 87 per cent in 2011, and as a region had nearly reached the USI target of 90 per cent[2].

Salt iodine is monitored by the conventional method (CM) in which sulphuric acid (H_2SO_4) liberates iodine from potassium iodate (KIO_3) in salt; potassium iodide (KI) is added to keep the iodine in dissolved state; KI forms KI_3 when combined with I_2; five iodine atoms from five KI molecules are needed to solubilize one iodine atom released from one KIO_3 molecule in salt; unlike I_2, I_3 is highly watersoluble and consumed by sodium thiosulphate ($Na_2S_2O_3$) during titration[3]:

$$KIO_3 + H_2SO_4 \rightarrow K_2SO_4 + 2H^+ + IO_3^-$$

$$KI + I_2 \rightarrow KI_3$$

$$KIO_3 + 3H_2SO_4 + 5KI \rightarrow 3K_2SO_4 + 3H_2O + 3I_2$$

$$3I_2 + 6Na_2S_2O_3 \rightarrow 6NaI + 3Na_2S_4O_6$$

For iodine estimationin the past, 50 g salt dissolved in 250 ml distilled water was mixed with 1 ml 2N H_2SO_4 and 500 mg KI[4,5]. Subsequently, 10 g salt dissolved in 50 ml distilled water was mixed with 1 ml 2N H_2SO_4 and 500 mg KI[6]. Alas, 99% of KI was wasted in these estimations TABLE 1. Therefore, 500 mg KI per test is not justified.

It has been reported that iodine in double fortified

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salt is determined using less amount of KI[7]. Our preliminary studies showed that this method can be adopted for iodated salt also. Therefore, detailed studies were carried out to find the quantity of KI actually required for salt iodine estimation. The precision, sensitivity, reproducibility and performance of the profitable method (PM) were validated against CM. Iodated salts produced by different methods and iodated salts purchased from the local market were also tested for iodine by PM.Reduction in the quantity of KI would reduce the estimation cost.

TABLE 1 : Potassium iodide wastage insalt iodine estimation with time

Time *(Year)	Per estimation					
	Quantity			Potassium iodide (mg)		
	Salt (g)	Iodine		Required	Added	Wasted
		ppm	mg			
Past	50	30	1.5	7.5	500	492.5
From 1995	10	30	0.3	1.5	500	498.5

***Ref.4-6**

EXPERIMENTAL

All reagents used were of analytical grade and distilled water (conductivity:39-40 mho) was used. For iodine estimation by CM[6], 10 g iodated salt was dissolved in 46 ml distilled water or 1000 µg iodine from potassium iodate (KIO_3) standard (1 mg iodine/ml) was dissolved in 49 ml distilled water. Then 1 ml 2N H_2SO_4 and 5 ml 10% KI (500 mg KI) were added. The contents were kept in the dark for 10minutes and titrated against 0.005M thiosulphate.

In order to find the required quantity of KI that can be used in PM,1000 µg iodine was tested against a wide range of KI (1-500 mg) by replicate analyses (n= 6/KI level). According to Kolthoff, iodine should be liberated only when sufficient iodide is present in the solution to minimize the loss of iodine by volatilization[8]. Therefore, KI was added first to 10 g iodated salt or KIO_3 standard (1 mg iodine/ml) in PM before adding distilled water (46 ml for salt or 49 ml for KIO_3) and 2N H_2SO_4. The contents were kept in the dark for 10 minutes and titrated against 0.005M thiosulphate.

Quality control: In order to ensure the reliability of the results, quality control measures were strictly adhered to for PM. Non-iodated salt was used as a blank. Iodine content of a known 'Reference Salt' was determined by multiple analyses (n = 20). The 95% confidence interval (CI) of mean iodine values was calculated along with the control operating range (mean ± 2SD) for preparing the quality control charts and to test the reproducibility of results.

The performance of PM was checked against CM over a wide range of iodine (0-1000 µg) from KIO_3 standard (1 mg iodine/ml). Salt iodine values are generally expressed as *parts per million* (ppm), which is nothing but microgram iodine per gram of salt. Therefore,10 g non-iodated salt was mixed with iodine (1-1000 µg) from KIO_3 standard (1 mg iodine/ml) and the results (ppm) were determined by PM. Iodated salts produced by spray mixing/dry mixing/submersion methods[9] and iodated salts purchased from the local market were also tested by PM for iodine.

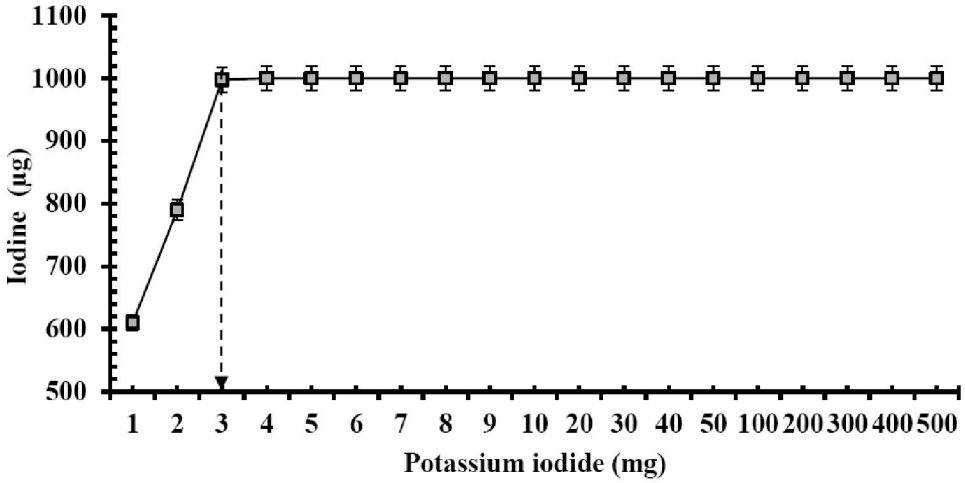
Statistical analysis: Mean, standard deviation (SD), coefficient of variation (CV) and regression analysis were done.

RESULTS AND DISCUSSION

The results of iodine estimation were corrected for a blank reading from non-iodated salt, which was zero. Replicate analyses of 1000 µg iodine against KI showed a linear response upto 3 mg KI and then plateaued. The results revealed that 3 mg KI was sufficient to estimate 1000 µg iodine Figure 1. Any further addition of KI was a wasteful exercise because no improvement in the results was observed Figure 1. Therefore, 5 mg KI per estimation was fixed for iodine estimation in PM for practical reasons.

The performance of PM checked against CM for the target range of 0-1000 µg iodine TABLE 2. The minimum detectable iodine was 0.997 ± 0.005 µg (mean ± 2SD, 95% CI). PM and CM corresponded very well for iodine estimation.

Potassium iodate standard showed 1.005 ± 0.008mg and 1.005 ± 0.006mg iodine per ml (mean ± SD) for PM and CM respectively. The CV of PM was not different from CM at every iodine level (TAVLE 2). The overall mean CV of PM and CM was the same (1.37%). The two methods agreed well at all levels of iodine ($R^2 = 0.999$). Thus, iodine estimation of KIO_3



(mean ± SD, n = 6 at each level of KI)

Figure 1 : Potassium iodide requirement for 1000 µg iodine

TABLE 2 : Iodine estimation of KIO_3 standard (1 mg iodine/ml) for the target range of 0-1000µg iodine by the conventional method (CM) and the profitable method (PM)

CM*			PM*		
Mean iodine (µg)	SD	CV (%)	Mean Iodine (µg)	SD	CV (%)
0	0	0	0	0	0
29.9	0.43	1.3	29.8	0.36	1.2
99.9	2.0	1.4	100	1.6	1.6
202	4.2	2.1	201	4.4	2.2
300	5.1	1.7	299	4.8	1.6
398	6.0	1.5	400	6.3	1.6
500	7.6	1.5	498	7.5	1.5
598	8.4	1.4	600	7.9	1.3
700	9.0	1.3	701	8.4	1.2
800	8.1	1.0	799	7.9	0.99
898	9	1.0	901	9.1	1.01
1000	9.5	0.95	1001	9.1	0.91

***mean of six estimations**

standard showed that PM was as good as CM.

The results of KIO_3 standard (1 mg iodine/ml) plus 10 g non-iodated salt showed that the precision of PM was excellent and agreed well with that of CM for the target range of 0-100 ppm iodine Figure 2. The performance of PM was on par with CM.

Quality control exercise on PM with 'Reference Salt' revealed excellent reproducibility Figure 3. The iodine (mean ± SD) was 33.1 ± 0.09 ppm and the CV was 0.27%. The control operating range (mean ± 2SD, 95% CI) was 32.92–33.28 ppm iodine. These observations are in agreement with others for the quality control of salt iodine estimation[7,9]. The day-to-day results of 'Reference Salt' were well within the control operating range throughout the study period. Thus, effective quality control was ensured for the reliability of results.

Iodine content of iodated salts produced by spray mixing, dry mixing and submersion methods showed no difference between CM and PM TABLE 3.

The iodine values were >30 ppm and therefore, well within the regulatory stipulations[10]. Though dry mixing and spray mixing methods showed 33 ppm iodine, it was 54 ppm for submersion method. Nevertheless, the agreement between PM and CM was excellent ($R^2 = 0.999$) irrespective of the method of preparation (dry mixing/spray mixing/submersion) and the type of salt (ordinary/refined) used.

Iodated refined salt (n =10) and iodated ordinary salt (n = 10) were procured from the local market for iodine estimation by PM and CM. The results of replicate analyses of market iodated salts showed no difference between PM and CM TABLE 4.

The production of iodated salt is increasing day by day. As a case analysis in India, the annual production of iodated salt increased from 5.37 million metric ton (MT) in 2008-09 to 6.18 million MT in 2012-13 with an average annual production of 5.958 million MT during this period[11]. In accordance with the sampling norms, if 10 samples per MT of iodated salt were to be tested, the number of estimations for 5.958 million MT in duplicates would be 119.16 million ($5.958 \times 10^6 \times 10 \times 2$). KI required for 119.16 million estimations

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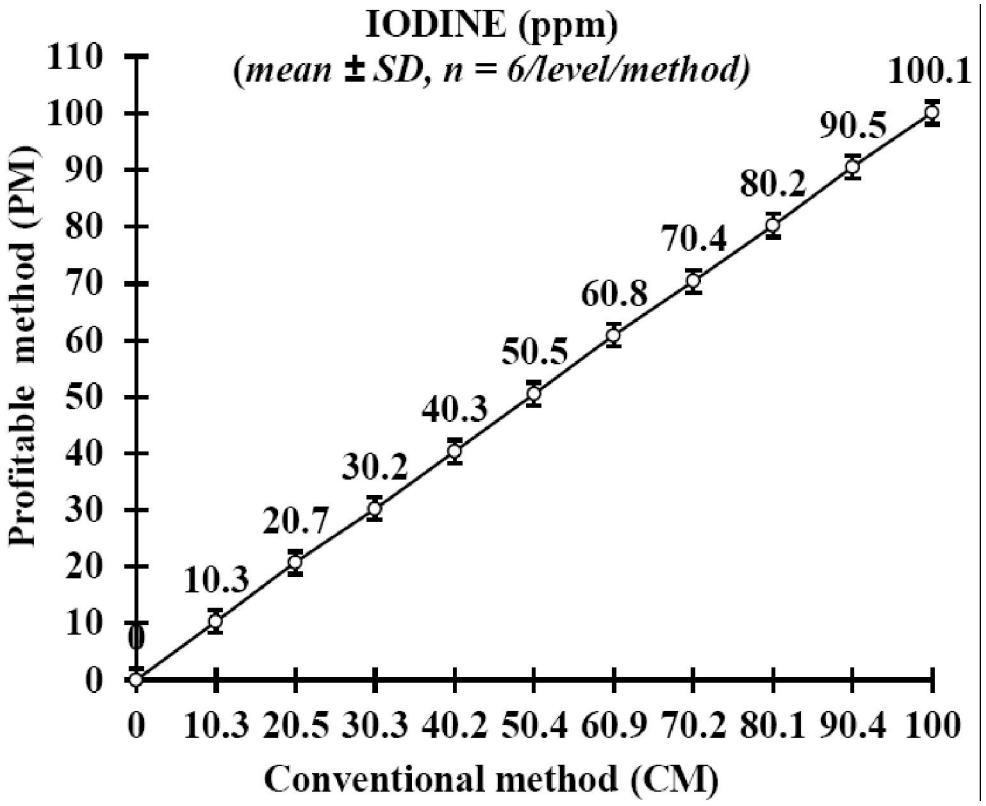

Figure 2 : Precision of PM (R^2 =0.999)

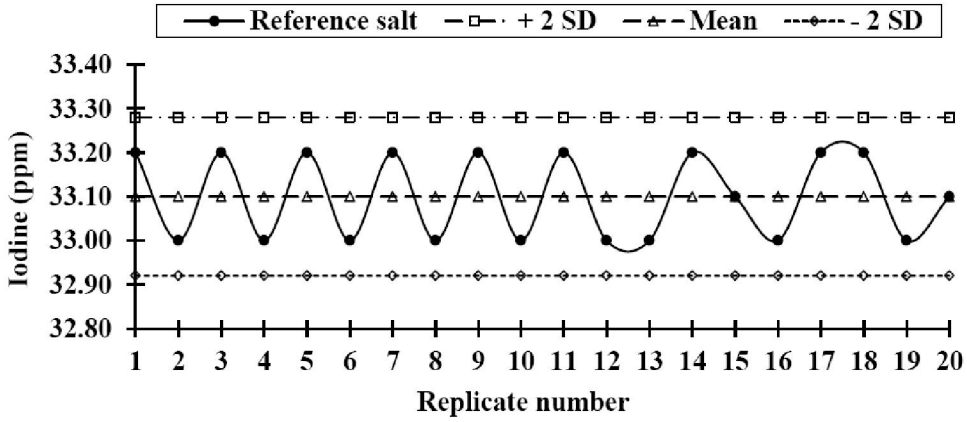

Figure 3 : Reproducibility of PM

TABLE 3 : Iodine content of iodated salts (prepared by different methods) determined by the conventional method (CM) and the profitable method (PM)

Iodated salt	Iodine (ppm)			
Method of preparation	Iodated ordinary salt		Iodated refined salt	
	CM*	PM*	CM*	PM*
Spray mixing	34.2 ± 1.2	34.2 ± 1.1	31.1 ± 0.5	31.1 ± 0.4
Dry mixing	34.7 ± 0.7	34.7 ± 0.7	31.0 ± 1.1	31.0 ± 1.1
Submersion	54.5 ± 2.1	54.5 ± 2.0	54.4 ± 2.0	54.4 ± 2.0

***mean ± SD, n = 6 salt/method**

would be 59.58 MT (119.16 x 10^6 x 0.5g) for CM and 0.5958 MT (119.16 x 10^6 x 0.005g) for PM.The average annual expenditure on KI,@ $60 per kg,would be $3.575 and $0.03575 million for CM and PM respectively and more than $17 million wasted on KIin CM would have been saved by PM TABLE 5. Such savings are applicable to other countries also where USI is in force.

It is ambiguous from the literature that how 500 mg KI was fixed per test in CM. Perhaps the axiom that excess KI is required to help solubilize the liberated iodine was responsible for this. The present study con-

TABLE 4 : Iodine content of market iodated salts determined by the conventional method (CM) and the profitable method (PM)

Iodated salt (Refined)	Iodine (ppm)*		Iodated salt (Ordinary)	Iodine (ppm)*	
	CM	PM		CM	PM
1	51.0 ± 1.3	51.0 ± 1.3	1	35.0 ± 0.9	35.1 ± 0.9
2	50.5 ± 1.5	50.5 ± 1.3	2	34.5 ± 0.7	34.6 ± 0.8
3	49.5 ± 1.3	49.5 ± 1.4	3	32.3 ± 0.9	32.1 ± 0.8
4	48.4 ± 1.3	48.4 ± 1.2	4	31.2 ± 0.7	31.1 ± 0.8
5	48.3 ± 1.4	48.3 ± 1.3	5	30.1 ± 0.8	30.0 ± 0.7
6	47.4 ± 1.8	47.5 ± 1.8	6	29.4 ± 0.7	29.3 ± 0.8
7	47.1 ± 1.2	47.1 ± 1.3	7	28.5 ± 0.9	28.5 ± 0.9
8	46.0 ± 1.1	46.0 ± 1.0	8	27.1 ± 0.7	27.1 ± 0.7
9	40.6 ± 1.1	40.6 ± 1.2	9	26.0 ± 0.5	26.0 ± 0.4
10	38.4 ± 0.7	38.4 ± 0.7	10	25.0 ± 0.6	25.0 ± 0.5

***mean ± SD, n = 6/salt/method**

firmed that 500 mg KI usage is exorbitant for salt iodine estimation and therefore, it is a colossal waste to continue to use 500 mg KI pertest. Under the National Iodine Deficiency Disorders Control Programme in India, salt fortified with KIO_3 is the recommended strategy, with the level of iodine fixed at not less than 30 ppm at production[10]. Hence iodated salt factories generally use 40 ppm iodine at the time of production. Thus, 10 g iodated salt will have about 400 µg iodine, which is well below the upper level (1000 µg) of PM. Therefore, it is not prudent to use 500 mg KI to estimate 400 µg iodine.

It is evident that one hundred salt samples could be tested by PM from the quantity of KI used for one test in CM. This leads to 100-fold reduction in the cost of iodine estimation. It is obvious that iodine is a precious commodity and widely imported to manufacture KI. As per the regulatory norms, iodated salt would be subjected to scrutiny at production, wholesale, retail and consumer levels. This may lead to testing of very large number of salt samples and therefore, PM is more suitable than CM. It would be wise to adopt PM for the estimation of salt iodine.

TABLE 5 : Projected savings on potassium iodide (KI) due to the profitable method (PM) over the conventional method (CM) for salt iodine estimation in India

Year	Iodated salt Produced (million MT)*	Projected number of iodine Estimations (million)**	Potassium iodide				Projectedsavings on KI due to PM (million $)
			Used (MT)		Price (million $)		
			CM	PM	CM	PM	
2008-09	5.37	107.4	53.7	0.537	3.22	0.0322	3.187
2009-10	5.82	116.4	58.2	0.582	3.49	0.0349	3.455
2010-11	6.22	124.4	62.2	0.622	3.73	0.0373	3.693
2011-12	6.20	124.0	62.0	0.620	3.72	0.0372	3.683
2012-13	6.18	123.6	61.8	0.618	3.71	0.0371	3.673
Total	29.79	595.8	297.9	2.979	17.87	0.1787	17.691

***Ref. 11; **10 samples in duplicates per MT of iodated salt**

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REFERENCES

[1] [WSC] World Summit for Children; Sustainable Elimination of Iodine Deficiency: Progress since the 1990. ISBN:978-92-806-4304-6, UNICEF Report, **(2008)**.

[2] [ICN] Improving Child Nutrition; The achievable imperative for global progress, UNICEF, April 2013, ISBN: 978-92-806-4686-3, **(2013)**.

[3] Revised Policy Guidelines on 'National Iodine Deficiency Disorders Control Programme', DGHS, Ministry of Health and family Welfare, New Delhi, Government of India, **(2006)**.

[4] J.C.Holman, W.McCartney; Iodated Salt, In: WHO Monograph Series No. 44, Endemic Goitre, Geneva:

World Health Organization, 437-438 **(1960)**.

[5] P.M.DeMaeyer, F.W.Lowenstein, C.H.Thilly; The Control of Endemic Goitre, World Health Organization, Geneva, 31 **(1979)**.

[6] K.M.Sullivan, R.Houston, J.Gorstein, J.Cervinskas; Titration Methods for Salt Iodine Analysis, In:UNICEF/PAMM/MI/ICCIDD/WHO, 'Monitoring Universal Salt Iodization Programmes', Atlanta, GA, USA, 86-89 **(1995)**.

[7] S.Ranganathan, M.G.Karmarkar; Indian J.Med.Res., **123**, 531-540 **(2006)**.

[8] I.M.Kolthoff, R.Belcher; Volumetric Analysis, Titration Methods, Interscience Publishers, Inc., New York, **3**, 235 **(1957)**.

[9] P.Pavan Kumar; Iodization of Common Salt with Available Techniques and Effective Quality Monitoring of Iodized Salt Using Appropriate Quality Control Measures, Master's Thesis, Centre for Food Technology, Oil Technological Research Institute, Jawaharlal Nehru Technological University, Anantapur, Andhra Pradesh, India, 12-14 **(2004)**.

[10] Food Safety and Standards Authority of India: Food Safety and Standard (Food Products Standards and Food Additives) Regulations, Govt. of India, **(2011)**.

[11] [SDAR] Salt Department Annual Report 2012-13:Annexure – 9.1, Ministry of Commerce & Industry, Govt. of India, 70 **(2013)**.