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THE DETERMINATION OF IODIDES
IN STABILIZED IODIZED SALT

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THE DETERMINATION OF IODIDES IN
STABILIZED IODIZED SALT

by

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THESIS

The Determination of Iodides in Stabilized Iodized Salt*

INTRODUCTION

The abnormally high percentage of persons affected with goiter in the state of Michigan has long been a public health problem of considerable significance. The incidence of goiter in Michigan prior to the use of iodine supplements was probably higher than that in any other region of the world, with the exception of Switzerland. This incidence was emphasized by the selective service registration of 1918 which showed that goiter was so prevalent in some groups in northern Michigan and Wisconsin that as high as 30 per cent of the persons examined were incapacitated for army service, owing to disqualifying toxic goiters (1). This widespread prevalence of goiter had been a matter of common knowledge for years and was accepted by the populace as an environmental malady for which there was no relief. No serious consideration had been given the situation by the state medical profession until 1921 when the first stimulus for an active goiter campaign was occasioned by Levin's paper, reporting that of 1,783 persons in Lake Linden, Michigan, 1,146 had thyroid enlargement (2). This observation was immediately followed by a Michigan Department of Health survey in January, 1922, of residents of Iron Mountain, Michigan. This survey revealed that 54 per cent of the persons examined had perceptible enlargements of the thyroid (3). In another survey by Reed and Clay of Grand Rapids, Michigan, 30 per cent of the school children examined were found to have enlargement of the thyroid gland (4). The publication of

* This study was conducted at the Bureau of Laboratories, Michigan Department of Health, Lansing, Michigan, financed in part by a grant from the Salt Producers Association through their Committee on Simplified Practice and Standardization.

these data clearly pointed out the need for a public health program for the prophylaxis of simple goiter in Michigan.

While it is now almost universally agreed that iodine deficiency is the cause of simple goiter, there has been a great deal of discussion as to the methods of supplying iodine for the purpose of prophylaxis. References to the popular folk remedy of the time of giving burnt cork as a remedy for goiter and cretinism date back as far as 1280 A.D. Iodine was discovered by Courtois in 1811, and isolated from the sponge by Fyfe in 1819. In 1820 Coindet, a physician in Geneva where goiter was prevalent, postulated that iodine was the goiter-curative principle in burnt sponge and further obtained dramatic results using the new element for treating conditions of iodine deficiency. However, indiscriminate use of the new element produced toxic effects and it was not until the present century that iodine prophylaxis and therapy were accepted as sound medical and nutritional practice.

The first large scale experiment in goiter prevention was begun in this country in 1917 by Marine and Kimball in Akron, Ohio, where a survey of grade and high school girls revealed a goiter incidence of 56 per cent. Approximately one-half of the 4495 children examined were each given, in 0.2 gm doses distributed over a period of two weeks, a total of 2 gm of sodium iodide in drinking water, and the other half were given no iodine. This procedure was repeated twice a year and provided enough iodine to saturate the thyroid. At the end of four years all the children were reexamined. Of those who were normal when the administration was started, and took iodine as directed, none developed goiter, while of those who did not take iodine, 27.5 per cent developed goiter. Of those who had goiters and who took iodine systematically, the thyroid gland returned to

normal size in more than 60 per cent of the cases, while of those who did not take iodine, only in comparatively few did the thyroid gland show any tendency to decrease in size (5, 6, 7, 8, 9, 10).

Following Kimball's first publication in 1917, Klinger in Zürich, Switzerland, in 1918, began iodine therapy in the school children of that city, since it previously had been found that in some of these schools 100 per cent of the children were goiterous. Instead of the sodium iodide used by Marine and Kimball, Klinger used iodostarine chocolate tablets which were an iodized fatty acid (tariric acid diiodide) combined with chocolate and made into tablets, each containing 5 milligrams of iodine. This was a non-hygroscopic, stable preparation, and a far more pleasant medicant than sodium iodide. This method was employed in three cantons in Switzerland with even more striking results than those obtained by Marine and Kimball in this country. In one canton the goiter incidence was reduced from 87.6 per cent to 13.1 per cent in four years (10, 11). Thus the value of iodine prophylaxis has been unquestionably established for twenty-five years but further proof of its usefulness has been established by more recent investigations.

The most notable recent investigations are the extensive studies made in Michigan and Ohio. In 1923 and 1924 the Michigan State Department of Health conducted a survey in various sections of the state to determine the correlation between the incidence of goiter in school children, and the amount of iodine in the water supply. The four counties of Macomb, Midland, Wexford, and Houghton were selected as showing the greatest variations in iodine content of the ground waters and as being representative

of a cross section of the population of Michigan. The iodine content of the ground waters in these four counties varied from 0 to 8.7 parts per billion. The total number of children examined in the four counties was 66,537 of whom 38.6 per cent had goiter. The incidence of goiter in each of these four regions was inversely proportional to the iodine content of the water. In the county having the highest iodine content of the water supply, the incidence of goiter was only 26 per cent, as contrasted to 64.4 per cent in the area having no iodine in its ground water (12).

Immediately following this survey a state-wide campaign was begun by the Michigan State Department of Health to emphasize the fundamental causes of endemic goiter and the principles of its prevention. In the spring of 1924 a special committee was appointed by the Michigan State Medical Society to work with the State Department of Health on this problem. These two groups, representing the medical profession of the state, cooperated with the Wholesale Grocers' Association and the Salt Manufacturers' Association with the result that beginning May 1, 1924, the salt manufacturers produced and put on the market throughout Michigan an iodized salt containing one part of potassium iodide to 5,000 parts of salt (0.02%). There was no law compelling people to use the iodized salt but the wholesale grocers did all they could to promote the sale of only this salt, and the State Department of Health carried on an active campaign to educate the public to its use. The program carried on by these groups included an estimation of goiter incidence during the ten-year period from 1924 to 1934, as indicated by records of goiter operations, and a re-survey of goiter incidence in school children.

In 1928, four years after the introduction of iodized salt, 50,134 of the school children examined in the 1923-24 survey were re-examined and

the incidence of goiter was found to have dropped from 38.6 per cent to 9.9 per cent. At the end of the ten-year period a final survey showed a further reduction to 8.2 per cent throughout Michigan. Among those using iodized salt during the entire ten-year study the incidence of goiter was only 2.98 per cent (13). The results of the entire ten-year study are best summarized by Dr. R. D. McClure, Chief Surgeon of Henry Ford Hospital, and a member of the State Medical Society Committee on Endemic Goiter (14).

1. Iodized salt as used in Michigan did at first apparently increase the number of thyroid operations.
2. The increase was in the nodular goiter or adenoma group, and we believe the iodized salt may have activated a group of quiescent adenomata, producing toxic goiter symptoms.
3. The increase reached its peak in the second year after the introduction of iodized salt.
4. An increase in the death rate from goiter as shown by the Board of Health statistics reached its peak in the second year after the introduction of iodized salt.
5. There was no increase in hyperthyroidism, excepting in the nodular goiter or adenomata group.
6. The number of operations for toxic, diffuse and toxic nodular goiter has rapidly and steadily decreased after the apex of the second year increase had been reached.
7. The incidence of endemic goiter or enlarged thyroid has been reduced almost to nil since iodized salt has been so widely used.
8. We now see no cases which show the slightest ill effects from the use of iodized salt.

9. Toxic nodular goiter and toxic diffuse goiter are less apt to occur when there has been no previous enlargement of the thyroid (endemic goiter); at least this would seem a safe conclusion based on experience.

In 1936 Kimball made a survey in the Cleveland, Ohio, parochial schools to determine the effect of the use of iodized salt on the incidence of goiter. Of those using iodized salt 7 per cent were affected with goiter as compared with 30.7 per cent of those not using it. The incidence of goiter in the latter group had been found to be essentially the same in a survey 12 years earlier (13).

The foregoing studies present ample evidence that iodized salt is effective as a prophylactic measure for goiter. In this connection McCollum stated "Moreover, there are other aspects of health than the prevention of goiter. Hypothyroidism or conditions of hyperplastic activity, owing to iodine deficiency, are accompanied by defects other than deformity of the neck. Mental development, sexual maturation, and the development of positive personality are retarded. Hence, iodine supplementation, insofar as it corrects or prevents a goiterous condition, promotes these functions, and contributes to the maintenance of good health." (15).

The excellent results obtained in the 1924-35 survey in Michigan stimulated an interest among public health leaders to allow other states to share the benefits of Michigan's experience through the services of a national organization. Consequently, a Study Committee on Endemic Goiter was organized in and under the direction of the American Public Health Association. This committee, together with the Michigan State Medical Society's Committee on Iodized Salt, and the National Research Council, Committee on Food and Nutrition, presented the following resolution

which was adopted by the Michigan Medical Society Committee in May 1940, and by the American Public Health Association Committee in June 1941.

"Resolved, That all salt for table use of human beings and the salt used for feeding to domestic animals in the United States should contain one hundredth of one per cent of Potassium Iodide or its equivalent viz., forty-five milligrams of Potassium Iodide to each pound of salt, provided that an effective stabilizer be used."

The lowering of the level of potassium iodide from the two hundredths per cent previously used was made possible by the incorporation of a suitable stabilizer for the potassium iodide. An effective stabilizer for the potassium iodide content of iodized salt had been formulated through work at the University of Wisconsin resulting in the issuance of a patent to the Wisconsin Alumni Research Foundation (16). In non-stabilized iodized salt losses as high as 100 per cent of the potassium iodide were not uncommon. These losses are due to the formation of hydriotic acid and the subsequent liberation of elemental iodine and are accelerated by storage under warm, moist conditions or the presence of slight impurities in the salt such as traces of copper, iron, or manganese salts. Acid salts such as magnesium chloride or calcium chloride also will act on the potassium iodide producing hydriotic acid. To counteract the formation of hydriotic acid, alkaline salts such as magnesium carbonate, sodium bicarbonate, calcium phosphate, and calcium oxide are now employed as stabilizers. The oxidation of hydriotic acid to elemental iodine is prevented by the use of reducing agents such as sodium thiosulfate or dextrose. These alkaline salts and reducing agents are used in various combinations in commercial stabilizers depending to some extent on the natural impurities present in the salt.

The decreased iodine content of iodized salt and the use of various reducing agents as stabilizers made questionable the reliability of existing methods for the determination of small quantities of iodine in salts. Therefore, this study was initiated to evaluate analytical procedures for the determination of iodides in stabilized iodized salt for the purpose of selecting methods which could be made official by the Association of Official Agricultural Chemists and would be acceptable to the Federal Food and Drug Administration.

In October, 1943, the author was made Associate Referee on Iodides and Bromides for the Association of Official Agricultural Chemists, replacing Major J. T. Tripp, Michigan Department of Health, who had previously held the position.

The appointment of the A.O.A.C. Associate Referee on Iodides and Bromides to the American Public Health Association Committee on Standard Methods prevented duplication of work. Further coordination was secured by the appointment of members of the American Medical Association and National Research Council Committees to the American Public Health Association Committee on Endemic Goiter. Members of the salt producing industry were invited to present the technical difficulties incident to the newly adopted reduction in potassium iodide content and to suggest the technical needs of the industry regarding control methods. The salt producing industries cooperated by offering the services of their analysts as collaborators and by supplying salt samples and details of manufacture.

The work to date has been divided into two phases. Phase I was a collaborative study designed to evaluate the methods routinely used by control analysts in the laboratories of seventeen collaborating salt

producers. Each analyst was asked to use a method of his own choice in analyzing the samples submitted to him. Phase II also was a collaborative study designed to evaluate further two methods which gave satisfactory results and which were most frequently selected by the collaborators in Phase I of the study. Each analyst was asked to use the two specific methods in analyzing the samples submitted to him in the second phase of the study.

INVESTIGATION

PHASE I. Comparison of results obtained with methods selected and used by commercial analysts.

Source of samples; companies and locations.

Nine salt producing companies located in various areas throughout the United States and employing different stabilizers collected 24 cartons of iodized salt at random from their production lines.

Preparation of samples.

Code numbers from 1 to 9 were assigned to the salt samples from the nine different producers. These nine salt samples were prepared as follows for distribution to seventeen collaborating analysts: The contents of ten of the cartons from each company were pooled and thoroughly mixed. Aliquots of the salt were accurately weighed, and 20 liters of a 20 per cent solution were prepared from each of the nine salt samples submitted. Sufficient acid (concentrated H_2SO_4 or concentrated HCl , depending on the nature of the stabilizers) was added to dissolve the stabilizers. The quantities of acids were as follows:

<u>Sample No.</u>	<u>Stabilizers</u>	<u>Acid added per liter solution</u>
1	MgCO ₃	0.75 ml conc. H ₂ SO ₄
2	MgCO ₃	0.50 " " "
3	MgCO ₃	1.00 " " "
4	Ca ₃ (PO ₄) ₂ NaHCO ₃ Dextrose	0.89 " " HCl
5	MgCO ₃	0.75 " " H ₂ SO ₄
6	Ca ₃ (PO ₄) ₂	2.00 " " HCl
7	MgCO ₃ CaO Na ₂ S ₂ O ₃	0.80 " " H ₂ SO ₄
8	Ca ₃ (PO ₄) ₂ NaHCO ₃	2.00 " " HCl
9	Ca ₃ (PO ₄) ₂ NaHCO ₃	2.75 " " "

The solutions were dispensed in amber glass bottles, sealed with "Celoseal" caps, and labeled to show sample number, concentration of salt, and kind and amount of acid added.

Collaborating analysts.

Aliquots of all nine samples were submitted to each of seventeen collaborating analysts who was asked to use a method of his choice in analyzing each sample for per cent iodine.

Methods employed.

Seven different methods of analysis were selected by the collaborating analysts. The methods employed are listed below.

- I. Elmslie-Caldwell (17)
- II. Sadusk-Ball (18)
- III. Potassium cyanide.
Original reference not available.

- IV. Tentative method for Waters, Brine and Salt, Association of Official Agricultural Chemists (19).
- V. Woodward (20).
- VI. Fresenius (21).
- VII. Modified Viebock and Breckner (22, 23).
Modified by New York Station of Food and Drug Administration.

Results and conclusions.

The results submitted by the seventeen collaborating analysts are presented in Table 1. The author is coded as analyst L.

The two most popular methods as evidenced by the number of analysts who selected them were the Elmslie-Caldwell (17) (Method I, Table 1) and the Sadusk-Ball (18) (Method II, Table 1) methods. Both procedures gave satisfactory results as judged by their distribution about the mean values. The other five methods (Methods III, IV, V, VI, and VII, Table 1) were less frequently used and while the results in some cases were apparently erroneous there are not data sufficient to prove or disprove their value as analytical procedures. By the same token there were not data sufficient to justify the acceptance of either Method I or Method II as completely satisfactory procedures. These data only indicate that Method I and Method II were employed most frequently by this collaborating group of analysts and that the results obtained showed deviations that were within reasonable limits.

PHASE II. Comparison of Elmslie-Caldwell and Sadusk-Ball methods.

Source of samples: Companies and location.

Four salt producing companies located in various areas and employing different stabilizers collected cartons of iodized salt from their production lines at specified time intervals during two consecutive days.

Preparation of samples.

The salt samples were prepared as follows for distribution to the twelve collaborating analysts: Code letters A, B, C, and D were assigned to the salt samples from the four different producers. To each of the code letters was added the sample number given by the individual producers denoting order of collection (e.g., A-1, A-2, A-3, etc.). Each sample was prepared by pooling and thoroughly mixing the contents of four cartons bearing the same code letter and number (e.g., A-1). Aliquots of salt were accurately weighed, and 6 liters of a 20 per cent solution prepared from each of the samples collected at specified intervals by the four companies. One ml of concentrated H_2SO_4 per liter of solution was added to each sample to dissolve the stabilizers. The solutions were dispensed in amber glass bottles, sealed with "Celoseal" caps, and labeled to show sample number, concentration of salt, and amount of H_2SO_4 added.

Collaborating analysts.

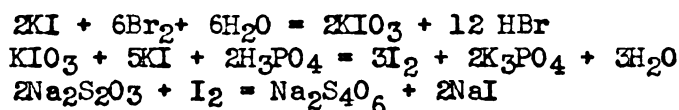
Six collaborating analysts were sent samples A-1 to A-9 and B-1 to B-9, and six analysts were sent samples C-1 to C-9 and D-1 to D-6. However one analyst failed to submit results, and another analyst experienced technical difficulties and was unable to submit an acceptable report. It was unfortunate that both analysts had received the C and D series of samples.

Methods employed.

The collaborating analysts were asked to analyze the samples for per cent iodine by both the Elmslie-Caldwell (17) and Sadusk-Ball (18) procedures, which in Phase I of the study gave satisfactory results, and were most frequently selected by the collaborators. The procedures are as follows:

Method I. Elmslie-Caldwell (17)Reagents

- (a) Sodium thiosulfate solution.-- 0.005 N. Dissolve 12.4 g of reagent-grade $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in freshly distilled H_2O and dilute to 1 liter giving an approximately 0.05 N stock solution. Prepare 0.005 N solutions by dilution of this stock solution with freshly distilled H_2O .
- (b) Standard Potassium Iodide solution.-- Prepare a solution containing exactly 0.1308 g of reagent-grade KI (0.1000 g I_2) per 1 liter of freshly distilled H_2O .

ReactionsDetermination

The following method of analysis is the Elmslie-Caldwell method with the ignition step omitted.

Take 25 ml aliquots of the 20 per cent salt solution, add 5 g of Na_2CO_3 , cover with watch-glass and boil gently for 10 min. Transfer contents to 18 cm filter paper and wash with boiling H_2O , catching filtrate and washings in 500 ml Erlenmeyer flask (solution should total ca 300 ml). Neutralize to methyl orange with 85% H_3PO_4 , and add 1 ml in excess.

Add excess of Br water and boil solution gently until colorless, and then 5 min. longer. Add a few crystals of salicylic acid and cool to ca 20° . Add 1 ml of 85% H_3PO_4 and ca 0.5 g of KI and titrate I with 0.005 N $\text{Na}_2\text{S}_2\text{O}_3$ in the usual way, using starch solution as indicator.

Standardize the $\text{Na}_2\text{S}_2\text{O}_3$ solution by measuring into a 500 ml Erlenmeyer exactly 25 ml of the standard solution containing 0.1308 g of KI per liter,

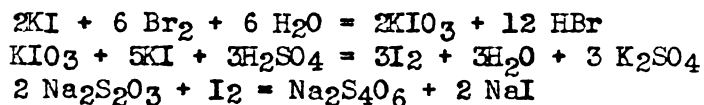
adding 300 ml of H₂O and 5 g of Na₂CO₃, neutralizing, and proceeding as directed above. (It is advisable to standardize the Na₂S₂O₃ solution same day determinations are conducted).

Method II. Sadusk-Ball (18).

Reagents

- (a) Sulfuric acid.-- 2 N. 1 volume of concentrated H₂SO₄ plus 17 volumes of freshly distilled H₂O.
- (b) Bromine vapor.-- Blow air (washed with H₂O and dried by CaCl₂) through a gas washing tube containing C. P. bromine. Deliver vapors just above the liquid in the flask.
- (c) Potassium iodide solution.-- 10%. Prepare a KI solution containing 10 g reagent-grade KI per 100 ml (freshly prepared).
- (d) Sodium thiosulfate solution.-- 0.005 N. Dissolve 1.24 g of reagent-grade Na₂S₂O₃·5H₂O and 3.8 g crystallized borax (Na₂B₄O₇·10H₂O) in freshly distilled H₂O and dilute to 1 liter. The solution is standardized immediately before use by titration against standard KIO₃ solution.
- (e) Standard potassium iodate solution-- 0.005 N. Prepare a stock solution containing exactly 3.567 g reagent-grade KIO₃ per liter, giving a 0.1 N solution. The stock solution is further diluted to give a 0.005 N reagent.

Reactions



Determination

Transfer a 5 g portion of the salt and 100 ml of freshly distilled H₂O to a 200 ml Erlenmeyer flask and mix the contents of the vessel until

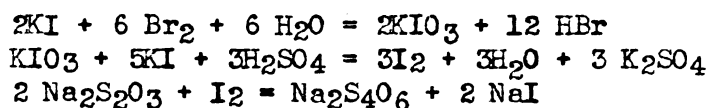
adding 300 ml of H₂O and 5 g of Na₂CO₃, neutralizing, and proceeding as directed above. (It is advisable to standardize the Na₂S₂O₃ solution same day determinations are conducted).

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Reagents

- (a) Sulfuric acid.-- 2 N. 1 volume of concentrated H₂SO₄ plus 17 volumes of freshly distilled H₂O.
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- (d) Sodium thiosulfate solution.-- 0.005 N. Dissolve 1.24 g of reagent-grade Na₂S₂O₃·5H₂O and 3.8 g crystallized borax (Na₂B₄O₇·10H₂O) in freshly distilled H₂O and dilute to 1 liter. The solution is standardized immediately before use by titration against standard KIO₃ solution.
- (e) Standard potassium iodate solution-- 0.005 N. Prepare a stock solution containing exactly 3.567 g reagent-grade KIO₃ per liter, giving a 0.1 N solution. The stock solution is further diluted to give a 0.005 N reagent.

Reactions



Determination

Transfer a 5 g portion of the salt and 100 ml of freshly distilled H₂O to a 200 ml Erlenmeyer flask and mix the contents of the vessel until

solution of the sodium chloride has taken place. Add methyl orange indicator and introduce 2 N H_2SO_4 drop by drop until a pink color results which persists on standing. After the addition of 2 ml of 2N H_2SO_4 and Br vapor, add a few glass beads and boil the solution until the yellow color due to bromine disappears and then for 2 minutes longer. Upon cooling, wash down the sides of the flask with approximately the amount of water lost by boiling. Add 2 ml of a 10% KI solution (freshly prepared), gently swirl the contents of the flask, and titrate liberated iodine immediately with 0.005 N thiosulfate, adding 2 ml of starch indicator near the end point.

Results and conclusions.

The results of the twelve collaborating analysts are given in Table 2. The author is coded as analyst 6. These data show that with one exception, there was close agreement between the results submitted by individual analysts, and that the Elmslie-Caldwell and Sedusk-Ball methods are equally suitable as analytical procedures for the determination of iodine in stabilized iodized salt. These data have been subjected to graphic and variance analyses which confirmed these conclusions.

Recovery experiments.

In order to evaluate further the two methods for the determination of iodine in stabilized iodized salt, recovery experiments were performed. In both procedures reagent-grade KIO_3 (Bakers analyzed) was used to standardize the 0.005 N $Na_2S_2O_3$ solution. The iodine contents of the iodized salt solution and of the stock KI solution were determined by a series of 6 analyses on each, and mean values were used in the calculation of the

recoveries. The results of these recovery series are given in Table 3. These data are in agreement with those previously presented, and further indicate that both methods are sufficiently accurate for the determination of iodine in stabilized iodized salt.

Summary and recommendations made to the Committee on Waters, Brine and Salt, Association of Official Agricultural Chemists.

In a collaborative study it was found that the Elmslie-Caldwell and Sadusk-Ball methods of analysis for the determination of iodine in stabilized iodized salt were most frequently employed for routine analyses in the laboratories of salt producers. The results of the collaborative study and the recovery experiments performed show that both methods are sufficiently accurate and reproducible for the purpose designated.

On the basis of these findings, the author as Associate Referee on Iodides and Bromides, A.O.A.C. presented the Referee for Waters, Brine, and Salt with the following recommendations:

1. That the Elmslie-Caldwell method be considered for adoption as an official method for the determination of iodides in stabilized iodized salt.
2. That the Sadusk-Ball method be considered for adoption as a tentative method for the determination of iodides in stabilized iodized salt.

As a result of these recommendations the Association of Official Agricultural Chemists has adopted the Elmslie-Caldwell method as official (first action) for the determination of iodine in salt (24). Official action on the Sadusk-Ball method has been deferred pending further collaborative work (25).

Collaborating Analysts

<u>Organization</u>	<u>Analyst</u>
American Salt Company, Kansas City, Missouri	Kansas City Testing Laboratory
Barton Salt Company, Hutchinson, Kansas	R. S. Humphreys
Carey Salt Company, Hutchinson Kansas	L. A. Enberg
*Colonial Salt Company, Akron, Ohio	L. C. Judy
*Diamond Crystal Salt Company, St. Clair, Michigan	H. W. Diamond F. G. Miller J. E. LaPorte
*Hardy (Manistee) Salt Company, Manistee, Michigan	C. G. Lindstrom
*International Salt Company, Watkins Glen, N. Y.	A. Marciniak
Jefferson Island Salt Company, Jefferson Island, La.	J. Grant-Mackay
Leslie Salt Company, Neward, California	D. S. See
Michigan Department of Health, Lansing, Mich.	Laura Huntley
*Morton Salt Company, Chicago, Ill.	C. H. Martin
Myles Salt Company, Weeks, Louisiana	Staff chemists
*Ohio Salt Company, Rittman, Ohio	T. R. Rader
*Union Salt Company, Cleveland, Ohio	The Textor Laboratories
U.S. Department of Agriculture, Washington, D. C.	R. A. Osborn
*Watkins Salt Company, Watkins Glen, N. Y.	
Wisconsin Department of Agriculture, Madison, Wis.	E. Lemense
*Worcester Salt Company, Silver Springs, New York	F. D. Everett

* Salt companies supplying samples of commercial iodized salt for the study

Acknowledgments

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Standardized salts as determined by collaborating analysts employing methods of their own choice

Method I	Method II		Method III		Method IV		Method V		Method VI		Method VII	
	I ₂	Analyst	I ₂	Analyst	I ₂	Analyst	I ₂	Analyst	I ₂	Analyst	I ₂	Analyst
	.0107	B	.0105	D	.0107	F	.0109	H	.0102	I	.0118	Q
	.0109	C	.0109	K	.0121	M	.0109	J	.0107			
	.0105	G	.0116			N	.0118					
	.0117	O	.0108									
	.0107	P	.0107									
	.0108											
an	.0108		.0108		.0114		.0109		.0104		.0118	
	.0109		.0109		.0114		.0112		.0104		.0118	
	.0174	B	.0175	L	.0191	F	.0174	H	.0179	I	.0180	Q
	.0176	C	.0174									
I	.0185	O	.0170									
L	.0175	P	.0170									
O	.0146											
dian	.0174		.0170		.0206		.0180		.0164		.0183	
an	.0170		.0169		.0206		.0178		.0164		.0183	
A	.0203	B	.0194	D	.0222	F	.0207	H	.0186	I	.0229	Q
C	.0204	C	.0206	K	.0300	M	.0207	J	.0214			
E	.0200	G	.0180			N	.0211					
I	.0226	O	.0208									
L	.0200	P	.0200									
O	.0205											
dian	.0204		.0200		.0261		.0207		.0200		.0229	
an	.0206		.0198		.0261		.0208		.0200		.0229	

Table 2

the content of iodized salts as determined by collaborating analysts employing the (I) Kimmel-Caldwell and (II) Seduck-Bell methods of analysis.

samples A-1 to A-9, labeled as containing 0.010% KI and 0.6% MgCO₃;
 samples B-1 to B-9 labeled as containing 0.010% KI, 0.5% MgCO₃, 1.0% CaO, and 1.0% MgSO₄.

	Analyst 2		Analyst 3		Analyst 4		Analyst 5		Analyst 6		Mass		Range		Median
	Meth. I	Meth. II	Meth. I	Meth. II	Meth. I	Meth. II	Meth. I	Meth. II	Meth. I	Meth. II	Meth. I	Meth. II	Meth. I	Meth. II	
	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	% I ₂	
	.0103	.0104	.0104	.0106	.0097	.0105	.0102	.0097-.0106	.0102	.0097-.0106	.0102	.0097-.0106	.0102	.0097-.0106	.0102
	.0102	.0101	.0107	.0116	.0097	.0101	.0102	.0097-.0107	.0102	.0097-.0107	.0102	.0097-.0107	.0102	.0097-.0107	.0102
	.0112	.0112	.0116	.0116	.0112	.0112	.0112	.0110-.0116	.0112	.0110-.0116	.0112	.0110-.0116	.0112	.0110-.0116	.0112

B. Samples C-1 to C-9
 Samples D-1 to D-6

Sample No.	Time of collection	Analyst 7	Analyst 7
		Meth. I	Meth. I
		Meth. II	Meth. II
		% I ₂	% I ₂
C-1	First day 9 a.m.	.0063	.0058
		.0062	.0061
C-2	First day 10 a.m.	.0054	.0053
		.0054	.0054
C-3	First day 12 noon	.0044	.0045
		.0044	.0044
C-4	First day 2 p.m.	.0036	.0036
		.0034	.0034
C-5	Second day 9 a.m.	.0063	.0061
		.0063	.0063
C-6	Second day 10 a.m.	.0079	.0076
		.0080	.0080
C-7	Second day 12 noon	.0063	.0059
		.0063	.0063
C-8	Second day 1 p.m.	.0057	.0054
		.0056	.0056
C-9	Second day 2 p.m.	.0062	.0059
		.0063	.0063
D-1	First day 11:30 a.m.	.0070	.0066
		.0069	.0069
D-2	First day 1 p.m.	.0087	.0088
		.0086	.0086
D-3	First day 1:30 p.m.	.0074	.0077
		.0073	.0073
D-4	First day 2:30 p.m.	.0071	.0066
		.0071	.0071
D-5	First day 3:30 p.m.	.0083	.0088
		.0083	.0083
D-6	First day 4 p.m.	.0076	.0071
		.0075	.0075

* Obviously erroneous, not include

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Table 3

Recoveries of iodine added as KI to 25 ml of a 20% iodized salt solution

A. By the Elmslie-Caldwell method

Trial number	I ₂ content of iodized salt solution	I ₂ added as KI	Total I ₂	Determined I ₂	Recovery
	mgs	mgs	mgs	mgs	%
1	.6714	.2236	.8950	.8979	100.32
2	"	.2236	.8950	.8817	98.51
3	"	.4472	1.1186	1.1122	99.43
4	"	.4472	1.1186	1.1214	100.25
5	"	.8943	1.5657	1.5948	101.86
6	"	.8943	1.5657	1.5927	101.72

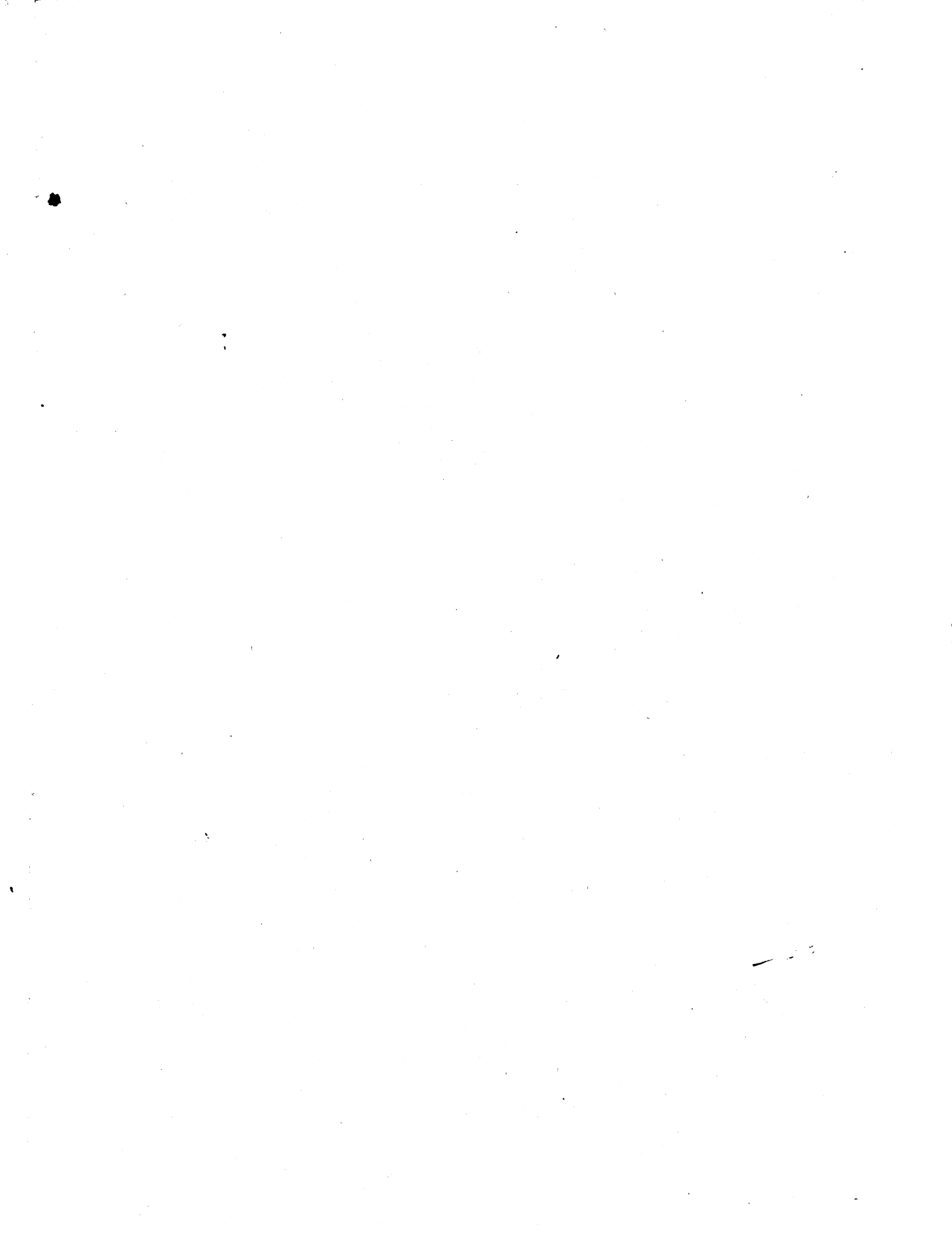
B. By the Sadusk-Ball method

Trial number	I ₂ content of iodized salt solution	I ₂ added as KI	Total I ₂	Determined I ₂	Recovery
	mgs	mgs	mgs	mgs	%
1	.6973	.2236	.9209	.9194	99.84
2	"	.2236	.9209	.9206	99.97
3	"	.4472	1.1445	1.1468	100.21
4	"	.4472	1.1445	1.1427	99.85
5	"	.8943	1.5916	Lost	-
6	"	.8943	1.5916	1.5974	100.36

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