Table III. Results of Recovery Determinations

Product	A Original Tin Content P.p.m.	B Tin Added P.p.m.	C[a] Tin Present P.p.m.	D Tin Found P.p.m.	E Recovery[b] %
Evaporated milk	25.8	25.0	50.8	52.3	103
	27.0	25.0	52.0	53.0	102
	39.1	25.0	64.1	63.9	100
	41.5	50.0	91.5	90.8	99
	46.7	25.0	71.7	72.4	101
	58.4	25.0	83.4	83.1	100
	62.3	25.0	87.3	88.0	101
	69.3	25.0	94.3	92.2	98
	82.6	25.0	107.6	107.6	100
Whole milk	0.4	5.0	5.4	5.4	100
Grapefruit juice	83.4	25.0	108.4	108.0	100
	98.0	25.0	123.0	122.0	99
Prunes	23.8	50.0	73.8	72.0	98
	21.2	15.0	36.2	36.0	101
	24.2	15.0	39.2	39.0	99
	2.1	10.0	12.1	12.0	99
Apricot preserves	1.4	10.0	11.4	11.4	100
Beans with pork	8.9	10.0	18.9	18.7	99
Beets	2.1	10.0	12.1	12.0	99
Mixed vegetable juices	15.6	15.0	30.6	31.8	104
	6.2	25.0	31.2	31.0	99
	3.1	25.0	28.1	27.8	99
Mixed vegetables	0.8	10.0	10.8	10.8	100
Black eyed peas	8.7	10.0	18.7	18.0	96
	6.0	10.0	16.0	16.5	103
Green peas	0.0	25.0	25.0	24.2	97
	0.0	25.0	25.0	23.9	96

[a] Values in C are equal to the sum of those in A and B.
[b] Recovery percentages are obtained from the ratio of figures shown in D to corresponding values in C.

initial tin content of the product was negligible and the fact that results of recovery determinations on these samples were uniformly acceptable more clearly indicates the precision which may be expected. Satisfactory recoveries have been obtained on samples containing as much as 400 parts per million of tin.

The data presented in Table III show the tin content in the original sample, the amount of tin added, and the amount found, all expressed in terms of parts per million. The ratio of the amount actually found to the sum of the original tin content and the added tin is given as the recovery.

DISCUSSION

By means of the procedure described it is possible to determine the tin content of a 10-gram sample of food product containing as little as 0.5 part per million. This represents a considerable increase in sensitivity over the volumetric iodometric method, which requires 50 to 100 grams of material. The increased sensitivity of the polarographic procedure in permitting the use of small samples makes possible a considerable saving in time and a marked economy of reagents. The reduction in sample weight, for example, reduces the quantity of nitric and sulfuric acids consumed in the digestion to less than one fifth that required in the volumetric method. The use of hydrogen sulfide with the accompanying problem of fume removal is eliminated, as well as the time-consuming filtration of the sulfide precipitate. The reduction in sample size also permits the use of smaller vessels and enables the operator to handle three to four times as many determinations concurrently.

LITERATURE CITED

(1) Alimarin, I. P., Ivanov-Emin, B. N., and Pevzner, S. M., *Trudy Vsesoyuz. Konferentsii Anal. Khim.*, **2**, 471–92 (1943).
(2) Assoc. Official Agr. Chem., "Official and Tentative Methods of Analysis", 5th ed., p. 413, 1940.
(3) Kolthoff, I. M., and Lingane, J. J., "Polarography", New York, Interscience Publishers, 1941.
(4) Lingane, J. J., Ind. Eng. Chem., Anal. Ed., **15**, 583 (1943).
(5) *Ibid.*, **18**, 429 (1946).
(6) Lingane, J. J., *J. Am. Chem. Soc.*, **65**, 866 (1943).
(7) *Ibid.*, **67**, 919 (1945).
(8) Smrz, J., *Rec. trav. chim.*, **44**, 580 (1925).

RAW SUGAR

Sampling, Mixing, and Testing

EDWARD F. KENNEY, Customs Laboratory, U. S. Treasury Department, Baltimore, Md.

New practices relating to the sampling, mixing, and testing of undamaged raw sugar imported in bags became effective in the Bureau of Customs in 1943. At the same time a new procedure for controlling the mixing and testing was introduced. The new practices include reduction in the sampling ratio from 100% to one bag in seven and the optional use of a new method of mixing. Experimental work on which the new practices are based is described. Data on the efficacy of the mixing and testing, as revealed by the new control procedure, are given.

THE customs duty on imported raw sugar is based on the direct polarization. In the case of raw sugar in bags, the samples for analysis are obtained from mixtures of portions taken by means of a trier (*6*) inserted into the bags. The Customs Regulations of 1907, 1923, 1931, and 1937 provided that 100% of the bags be sampled.

In 1942, as the result of requests of several government agencies that all possible steps be taken to conserve jute bags because of war conditions, W. R. Johnson, Commissioner of Customs, initiated a systematic study of the sampling, mixing, and testing of raw sugar to ascertain if the percentage of bags sampled per cargo could be reduced without jeopardy to the revenue and the number of bags damaged by customs samplers thereby lessened. The study was developed and conducted by the bureau's Division of Laboratories under the direction of H. J. Wollner (then consulting chemist to the Secretary of the Treasury). The evaluation of the results was made by the application of statistical principles employing standard methods (*1*).

SAMPLING

Precision Using Customs Trier. The preliminary study involved the determination of the degree of variability of the sugar, from bag to bag, in two different cargoes, A and B. Similar information was subsequently obtained on a third cargo, C. The study was designed to reveal information regarding the number of bags which would have to be sampled to ensure that the maximum gain or loss in revenue per cargo due to sampling errors, with a given degree of probability, would not be excessive.

Cargo A appeared to contain a considerable amount of sugar which had been stored for some time prior to export; it was not very uniform in appearance. The sugar in cargo B was generally uniform in appearance. The sugar in cargo C appeared to represent an abnormal case, varying from moist dark brown to dry almost-white sugar.

The three cargoes were sampled and tested by customs officers at Philadelphia, the samples being taken with the customs short trier (*6*) and the tests being made by the customs laboratory method (*8*). One core was taken from each bag sampled and placed in a 2-ounce glass container, which was then sealed with Scotch tape. The cores were taken at regular intervals from

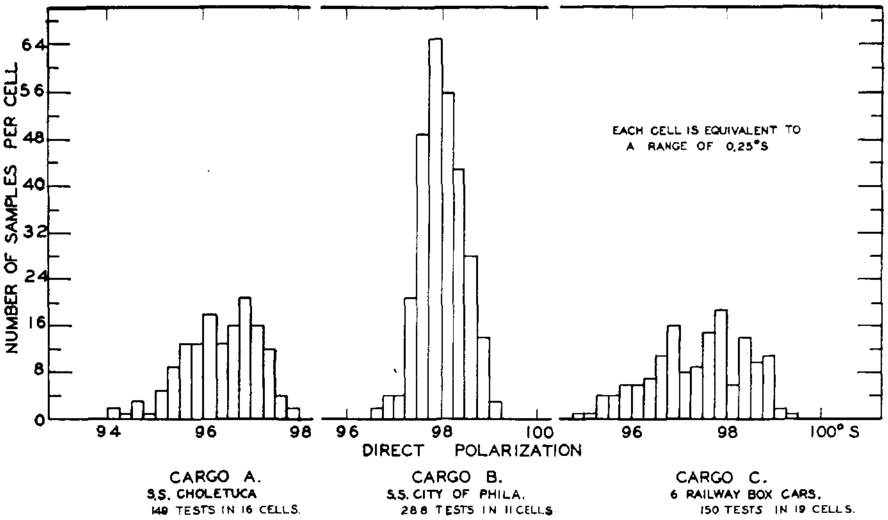

Figure 1. Histograms of Frequency Distribution in Sampling

Table I. Summary of Sampling Data for Three Different Cargoes Using Customs Short Trier

Cargo	No. of Bags	No. of Cores[a]	No. of Groups	Average Test, ° S.	σ, ° S.	Difference Between Highest and Lowest Individual Test, ° S. Actual	Statistical[b]
A	8,996	149	17	97.360	0.718	3.82	4.31
B	19,530	288	32	98.025	0.474	2.47	2.84
C	1,800	150	30	97.157	1.059	4.36	6.35

[a] Also number of tests.
[b] Based on $\pm 3\sigma$.

Table II. Summary of Sampling Data for Cargo C Using Three Different Types of Triers

Trier	No. of Cores[a]	Average Test, ° S.	σ, ° S.
Customs short form	150	97.157	1.059
Modified customs short form	150	97.136	1.022
Canadian	150	97.141	1.053

[a] Also number of tests.

different parts of the bags, and each sample was thoroughly mixed before testing. The data are summarized in Table I. Histograms, showing frequency distributions, appear in Figure 1.

The universe standard deviations, σ, shown in Table I represent the variation of the sugar in each cargo and are referred to as sampling precisions. A relatively minor part of the indicated variation is incidental to the testing (see Table IX).

Precision Using Different Triers. The data for cargo C were obtained in a study of the relative efficiency of three different types of triers, the customs short trier, a modified customs short trier, and a Canadian sugar trier. The modification of the customs trier consisted of building up one of its sides by welding on a strip of sheet metal, in the hope of obtaining a more representative sample. The Canadian trier was furnished to the bureau by one of the government war agencies interested in the conservation of jute. It had a solid pointed tip, was longer and narrower than the customs trier, and was said to cause less damage to the bags during sampling. In this study one trier was assigned to a sampler and three cores were taken from each bag sampled—one core for each trier. In terms of sampling precision, the three triers appeared to be about equal (Table II).

While the Canadian trier caused less damage to the bags, it was found unsuitable for use in sampling hard, gummy sugar, which was being imported in quantity at the time. An endeavor to eliminate this objection was made by cutting off the solid tip. While cores could be obtained from all types of raw sugar, using the modified Canadian trier, dry sugar had a tendency to slip off when the trier was removed from the bag, and some sugars caked in the spoon of the trier and were rather difficult to detach. Accordingly this phase of the sampling study was terminated, and all subsequent tests were made with the customs short trier.

Maximum Precision. The maximum universe standard deviation found in the study of cargoes A, B, and C was 1.059° S. The cargo for which this figure was obtained undoubtedly comprised abnormally nonuniform sugars. In view of this, and since customs experience over a period of years indicates that cargoes of raw sugar in normal times are generally more uniform, 1.1° S. is considered a conservatively high figure for the variation among bags in a cargo of undamaged raw sugar. The only published information on this subject which has come to the attention of the writer is that developed by Vondrak (*11*) and by Browne and Zerban (*2*).

Probable Effects of Reduced Sampling. A universe standard deviation of 1.1° S. was used in calculating the number of bags of undamaged raw sugar which would have to be sampled at random (one core per bag) to ensure that the maximum sampling error, with a given degree of probability, would not exceed certain figures. The calculated values appear in Table III.

Table III indicates that in order to ensure a sampling error of not more than 0.05° S. in 99 cases out of 100, at least 3212 bags should be sampled, one core per bag. For an average cargo of 20,000 bags this would require the sampling of about one bag in seven. If we assume that the cargo consisted of 20,000 bags of 96° S. Cuban sugar, containing 325 pounds per bag, a sampling error of 0.05° S. would be equivalent to about $37 at the present rate of duty (*10*), or about 0.075% of the total duty involved. According to Table III, if the same cargo were sampled 100%, the sampling error would not exceed 0.02° S. in 99 cases out of 100. This would be equivalent to about $15 at the present rate or about 0.03% of the total duty involved.

The possibility of applying a sampling ratio of one bag in seven to all cargoes of undamaged raw sugar was considered. In this connection the maximum gain or loss in revenue for different sized cargoes of 96° S. Cuban raw sugar due to sampling errors, assuming a sampling ratio of one bag in seven and a probability of 0.99, was calculated (Table IV).

Actual Effects of Reduced Sampling. The customs duties which would result from 100% sampling and reduced sampling of Cuban raw sugar in bags were determined from tests made of actual samples from five different cargoes. The sampling, mix-

Table III. Relation between Maximum Sampling Error and Number of Bags to Be Sampled, One Core per Bag[a]

Maximum Sampling Error, ° S.	No. of Bags to Be Sampled (Cores)[b]
0.5	32
0.2	201
0.1	803
0.05	3,212
0.02	20,073
0.01	80,293

[a] Based on σ of 1.1° S. and probability of 0.99.
[b] Calculated by use of formula: $n = \frac{t^2 \times \sigma^2}{E^2}$

where n is number of bags to be sampled (cores), t is factor for probability, σ is universe standard deviation, and E is maximum sampling error.

ing, and testing were done by customs officers at Philadelphia. In the case of 100% sampling, the usual customs practices (3, 8) were followed, except that the average test of each cargo was calculated on a weight basis rather than on the usual basis of the number of bags. If the mixing practices in effect at the time were applied to the sugar obtained in the reduced sampling experiments, fewer mixes would result and the amount of duty represented by each mix consequently would be greater. This indicated the importance of ensuring thorough mixing in connection with reduced sampling practices—more so than in the case of 100% sampling. With this in mind, the following modifications in the existing practices (3, 8) were introduced in the case of the reduced sampling experiments: each bucket was mixed separately; one laboratory sample was prepared from each bucket; each laboratory sample was obtained by taking portions from different parts of the mixed sugar, using a measuring spoon; the quantity of sugar in each sample was in proportion to the quantity of sugar in each bucket; and the laboratory mixed together the samples from the half-day's sampling of the cargo and, with one exception, made ten tests on the resulting mixture. The results obtained for the five cargoes appear in Table V.

SAMPLING RATIO RECOMMENDED. It was decided to recommend that for customs purposes the sampling of one bag in seven, using the customs short trier, be authorized in the case of undamaged raw sugar. This recommendation was based primarily on the data derived from the statistical study (Table IV) and on actual cargoes (Table V). Data were subsequently obtained on a sixth cargo, sampling 100% and one bag in seven, but were not included in Table V.

MIXING

The over-all error in the average test for a cargo of raw sugar includes, in addition to the sampling error, errors incidental to the mixing and testing operations. Effects of sampling errors have previously been discussed. With regard to mixing and testing errors, for practical reasons these are considered as a combined error in the following discussion.

PRECISION OF HAND MIXING METHOD. The first step in the study of mixing consisted of determining the precision of the hand method of mixing (5) which was in effect at the time. Five cargoes of Cuban raw sugar were involved, one each at Baltimore, New Orleans, New York, Philadelphia, and Savannah. The sampling, mixing, and testing were done by customs officers in the usual manner, except that five tests instead of two were made on each mix of undamaged sugar. The data are summarized in Table VI.

The differences between the highest and lowest average tests of the mixes (Table VI) are an indication of the relative variability of the cargoes. While it might be expected that a definite relationship between the variability of the cargoes and the mixing precisions, $\acute{\sigma}$, would be found, this does not appear to be the case, probably because the testing precision is included in and represents a significant part of the precision of the mixing method.

Table IV. Relation between Maximum Sampling Error and Maximum Gain or Loss in Revenue for Different Sized Cargoes of 96° S. Cuban Raw Sugar (Sampling Ratio 1 in 7)[a]

No. of Bags in Cargo	No. of Bags Sampled (Cores)	Total Revenue, $	Maximum Sampling Error[b], ° S.	Maximum Gain or Loss in Revenue $	%
50,000	7,143	121,875	0.034	61	0.05
20,000	2,857	48,750	0.053	39	0.08
10,000	1,429	24,375	0.075	27	0.11
5,000	714	12,188	0.106	19	0.16
2,000	286	4,875	0.168	12	0.25
1,000	143	2,438	0.237	9	0.37
500	71	1,219	0.335	6	0.49
300	43	731	0.433	5	0.68

[a] Based on $\acute{\sigma}$ of 1.1° S., probability of 0.99 and 325 pounds of sugar per bag dutiable at $0.0075 per pound.
[b] Calculated by use of formula appearing under Table III.

Table V. Comparison of Revenue for Five Cargoes Based on Actual Tests

Cargo	No. of Bags in Cargo	100% Sampling: No. of tests	Average test, ° S.[a]	1 Revenue	Reduced Sampling: Sampling ratio	No. of tests	Average test, ° S.[b]	2 Revenue	Difference in Revenue, 1 − 2: $	%
1	7043	24	96.92028	$ 17,578	3 in 7	40	96.913	$ 17,576	+ 2	0.01
2	15709	48	97.31611	39,397	2 in 7	63	97.342	39,412	−15	0.04
3	24993	74	97.10752	62,557	1 in 7	80	97.106	62,555	+ 2	0.00
4	7124	24	97.01166	17,864	3 in 7	50	96.999	17,861	+ 3	0.02
5	49483	138	97.18038	124,038	1 in 7	80	97.218	124,106	−68	0.05

[a] Calculated to 5 decimals in accordance with practice in effect at the time.
[b] Calculated to 3 decimals in accordance with present practice.

Table VI. Comparison of Mixing and Testing Precisions, $\acute{\sigma}$, for Different Ports (Hand Method of Mixing)

Port Symbol	No. of Mixes	Average Test of Cargo, ° S.	Difference between Highest and Lowest Average Tests of Mixes, ° S.	$\acute{\sigma}$, ° S.
A	36	97.528	0.484	0.0409
B	33	97.324	0.828	0.0658
C	20	97.549	0.142	0.0536
D	21	97.188	0.484	0.0479
E	15	96.864	0.160	0.0438

PRECISION OF NEW MIXING METHOD. The customs regulations in effect at the time (6) required that the average test be determined for the sugar represented by each half-day's sampling of a cargo and that not more than 3 full buckets of sugar should be mixed together (7). This meant that under 100% sampling a number of mixes were generally necessary for each half-day's sampling of a cargo. It was anticipated that a similar condition might also apply when one bag in seven was sampled. Since by limiting the number of mixes per cargo to one each half-day the over-all work would be simplified, it was decided to develop a method of mixing which would serve this purpose in most cases and yet be sufficiently precise.

While the modifications in existing practices referred to in connection with Table V resulted in one mix per half-day, it was believed practicable to devise a simpler method. Accordingly, a number of mixing experiments were subsequently conducted by customs officers at Philadelphia. These experiments culminated in the development of special equipment and a new method of mixing, which was called the box and screen method (5). (The method employs a 0.375-inch mesh wire screen, fitted over a 4-box receiver, each box being detachable. With the aid of a similar receiver all the sugar is screened and quartered three times.) The most important experiment is described in the following:

Two abnormal mixtures, each consisting of about 37.5 pounds of low test raw sugar (95° S.) and about 37.5 pounds of high test raw sugar (97.7° S.) were used. In each case 5 bucketfuls (about 75 pounds) were prepared, each containing approximately equal layers of the low and high test sugar. Each lot was mixed by the box and screen method, with the exception that one lot was screened twice instead of three times. In each case 30 groups of samples, each group consisting of one sample from each of the four boxes, were taken from the final mixture. The four samples in each group were taken at the same time. Each sample consisted of about 2 ounces. All the samples in each mixture were tested as a unit, without mixing, by the same chemist, under the same conditions. In the case of two screenings the elapsed time for the mixing was 3.33 minutes and the mixing precision, $\acute{\sigma}$, was 0.1701. In the accepted mixing procedure involving three screenings 5.67 minutes were consumed and the precision was found to be 0.1102.

The precision, $\acute{\sigma}$, of 0.1102 for the box and screen method of mixing represents an extreme condition. In view of this and the results obtained in the study of the hand method of mixing, 0.11° S. may be considered a conservatively high figure for the

Table VII. Relation between Number of Tests per Mix and Maximum Mixing and Testing Error per Mix[a]

No. of Tests per Mix	Maximum Mixing and Testing Error per Mix, ° S.[b]
2	0.200
4	0.142
6	0.116
8	0.100
16	0.071
32	0.050
64	0.035
128	0.025
256	0.018

[a] Based on $\acute{\sigma}$ of 0.11 and probability of 0.99.
[b] Calculated by use of formula appearing under Table III.

combined mixing and testing precision, $\acute{\sigma}$, when either method is properly used on undamaged raw sugar. An idea of the relative efficiency of the two methods can be obtained by reference to Table X.

TESTING

MIXING AND TESTING ERRORS. Using 0.11 as the mixing and testing precision, the maximum mixing and testing errors per mix for different numbers of tests per mix, for a probability of 0.99, were calculated (Table VII).

NEW CUSTOMS METHOD

SAMPLING AND MIXING PROCEDURES. Detailed instructions on new procedures for the sampling and mixing of raw sugar for customs purposes were prepared as an amendment to the Bureau of Customs Sampling Guide. This amendment (*5*), in the form of method 17, entitled "Raw Sugar", became effective July 14, 1943.

INSTRUCTIONS ON TESTING. The maximum gain or loss in revenue for a cargo of raw sugar due to the mixing and testing error depends in part on the number of mixes, the quantity of sugar in each mix, and the number of tests per mix. In view of this, and since the amount of revenue represented by a cargo depends partly on the quantity of sugar involved, it was deemed impractical to require that the number of tests per mix or per cargo be such that the maximum mixing and testing error, with a given degree of probability, would not exceed a specified figure. The instructions (*9*) finally issued in this connection were based on a practical application of statistical principles. The pertinent part of these instructions is as follows:

> The sample submitted to the laboratory for a mix may consist of four full cans, two full cans, one full can, or even one partially full can, depending upon the quantity of sugar present in the mix. Two tests shall be made on each can submitted from each mix of undamaged raw sugar, except that in the case of mixes representing 5000 or more bags, four tests shall be made on each can.
>
> Inasmuch as it is desired to obtain data on the efficacy of the previous mixing of the sample, special care shall be taken to avoid mixing the sugar in the can either before or when removing a portion for test. All reasonable efforts shall be made to take each test portion from a different position in the can. For example, after the upper surface layer has been discarded, the first test portion shall be taken from the resulting exposed surface. The succeeding surface layer shall then be discarded before the next portion is removed for weighing.

The requirement as to the number of tests per can was designed to keep the errors incidental to the mixing and testing as low as practicable. For this purpose it was desirable that each test portion come from a different part of the mix, preferably by having the number of cans sent to the laboratory for each mix correspond to the number of tests to be made on each mix. It was deemed impractical at the time to submit one can per test because of the difficulty in getting proper containers. In order to accomplish the desired purpose and at the same time permit a study of the efficiency of the routine mixing and testing operations, instructions regarding the nonmixing of the samples in the laboratory were included.

ESTIMATED OVER-ALL PRECISION. It was anticipated that, following the practical application of method 17, the mixing and testing error for an average cargo generally would not exceed the maximum sampling error for the cargo. For example, according to Table IV, the sampling error for a 20,000-bag cargo, sampling ratio one bag in seven, would not be expected to exceed 0.053° S. in 99 cases out of 100. Assuming five as the average number of mixes, each representing 4000 bags, and eight tests per mix, the mixing and testing error for the cargo would not be expected to exceed 0.045° S. in 99 cases out of 100. This figure is obtained by dividing the maximum mixing and testing error per mix (from Table VII) by the square root of the number of mixes. It may also be assumed that the sampling, mixing, and testing error for the cargo would not exceed 0.069° S. in 99 cases out of 100. This figure is obtained by taking the square root of the sum of the squares of the maximum sampling error and the maximum mixing and testing error.

In view of the foregoing, it would appear reasonable to assume that the maximum sampling, mixing, and testing errors due to chance, when method 17 is followed, generally will not be excessive.

CONTROL OF MIXING AND TESTING

CONTROL PROCEDURE. Under the reduced sampling procedure authorized by method 17 each mix represents a greater proportion of the cargo and the total number of mixes required for the cargo are materially less than when 100% sampling is practiced. This indicated the desirability of maintaining some kind of statistical control in connection therewith. Accordingly a procedure designed to reveal inadequacies in the routine mixing and testing of each importation of undamaged raw sugar was devised (*4*), and made official for customs purposes on July 14, 1943. The following are the pertinent portions of the procedure:

> When imported raw sugar is sampled for test at each port, the appraiser forwards to the director of the National Bureau of Standards a duplicate of one of the samples sent to the customs laboratory.
>
> Only one sample is submitted to the National Bureau of Standards for each importation. It is taken from one of the regular mixes of undamaged sugar and consists of the same number of cans as the regular laboratory sample from that mix.
>
> The samples sent to the customs laboratory and the National Bureau of Standards are tested immediately on receipt. The average tests and the individual tests obtained on the duplicate samples are forwarded to the U. S. Customs Laboratory, Baltimore, Md., where they are studied by statistical methods.

The customs laboratories do not know which mixes are being studied until after their reports have been submitted to the appraisers. Accordingly, their results represent routine conditions for the testing.

DATA, CRITERIA, AND CONCLUSIONS. The more important of the statistical values derived from the control procedure will be found in Tables VIII, IX, and X.

Some of the criteria which were set up in connection with the study of the data and the conclusions which were drawn from the study are as follows:

> If the standard deviation, σ, for both the customs and the National Bureau of Standards tests for a given sample are out of control, existence of disturbing factors outside the laboratory, presumably in the mixing operation, is indicated. Only three such cases were noted among the 399 samples which could be considered.
>
> If the difference between the average tests (customs and N. B. S.) on the same sample exceeds the range of the control limits for averages, $2A\acute{\sigma}$, an assignable cause in the nature of a constant testing error or a change in the moisture content is indicated. In determining the range of the control limits for averages the higher of the two $\acute{\sigma}$'s was used. Assignable causes were indicated in 28% of the cases when the hand method of mixing was involved, and

Table VIII. Comparison of Average Tests for Individual Customs Chemists and Individual Customs Laboratories with Those for N.B.S.[a]

Customs Laboratory Symbol	Customs Chemist Symbol	No. of Samples	Average Test, ° S. 1 Customs	2 N.B.S.	Difference 1 − 2
A		57	97.158	97.185	−0.027
	H	8	97.404	97.345	+0.059
	I	49	97.118	97.159	−0.041
F		99	97.401	97.343	+0.058
	J	86	97.403	97.334	+0.069
	K	2	97.000	96.936	+0.064
	L	11	97.460	97.487	−0.027
B		128[b]	97.138	97.139	−0.001
	M	6	96.754	96.738	+0.016
	N	46	97.296	97.284	+0.012
	O	76	97.073	97.082	−0.009
C		120	97.218	97.149	+0.069
	P	14	97.058	96.941	+0.117
	Q	106	97.239	97.177	+0.062
D		150	97.369	97.356	+0.013
	R	144	97.370	97.358	+0.012
	S	6	97.353	97.298	+0.055
G		7	98.576	98.710	−0.134
	T	6	98.556	98.705	−0.149
	U	1	98.696	98.738	−0.042
E		64	96.997	96.940	+0.057
	V	21	96.934	96.857	+0.077
	W	24	96.962	96.904	+0.058
	X	19	97.112	97.078	+0.034
Total		625			
Averages			97.254	97.227	+0.027

[a] Table is based on data for samples obtained by both methods of mixing. Number of tests per sample varies from 2 to 8.
[b] Includes samples from 2 ports.

in 34% of the cases when the box and screen method was involved. For these, 70% of the differences were 0.2° S. or less, 95% were 0.3° S. or less, 98% were 0.4° S. or less, 99% were 0.5° S. or less, and none exceeded 0.583° S.

The following conclusions can be drawn from Table X: There is little difference in the precision of the mixing by customs officers at the several ports; and in terms of mixing precision there is little difference between the two methods of mixing as practiced by customs officers.

The $\hat{\sigma}$'s in Tables IX and X can be used in calculating the maximum mixing and testing error per mix for a given probability. However, since the $\hat{\sigma}$'s are based on two tests per can per sample, there may be some question as to the reliability of the error thus determined. Accordingly, a number of the $\hat{\sigma}$'s in Table IX were recalculated, using the data for the first test on each can per sample. Table XI indicates that, as anticipated, the effect of making two tests per can under the prescribed conditions is equivalent to that of making one test on each of two cans.

Table IX. Comparison of Mixing and Testing Precisions, $\hat{\sigma}$, for Each Method of Mixing Based on Tests by N.B.S. and Customs[a]

Laboratory Symbol	Customs Chemist Symbol	Hand Method No. of samples	$\hat{\sigma}$	Box and Screen Method No. of samples	$\hat{\sigma}$
A	I	32	0.064	..	...
N.B.S.		32	0.039	..	...
F	J	76	0.060	..	...
N.B.S.		76	0.040	..	...
F	L	30	0.064	..	...
B	O	..	...	41	0.061
N.B.S.		..	...	41	0.050
B	O	..	...	58[b]	0.060
N.B.S.		..	...	58[b]	0.049
B	N	..	...	34[b]	0.044
N.B.S.		..	...	34[b]	0.042
C	Q	..	...	106	0.065
N.B.S.		..	...	106	0.046
C	P	..	...	37	0.088
D	R	41	0.047	103	0.044
N.B.S.		41	0.043	103	0.042
D	S	..	...	30	0.055
G	T	34	0.041	..	...
E	V	..	...	34	0.041
	W	..	...	47	0.051
	X	..	...	49	0.066

[a] Same samples used in each case where N.B.S. data are given. In such cases, each sample came from a different cargo. Where no N.B.S. data are given, all samples were not tested by N.B.S. and more than one sample may have come from the same cargo.
[b] Includes samples from 2 ports.

Table X. Comparison of Mixing and Testing Precisions, $\hat{\sigma}$, for Each Method of Mixing Based on N.B.S. Tests

Port	Hand Method No. of samples	N.B.S., $\hat{\sigma}$	Box and Screen Method No. of samples	N.B.S., $\hat{\sigma}$
A	38	0.038	...	...
F	87	0.040	...	...
Y	...	...	33	0.045
B	...	...	63	0.049
C	...	...	120	0.046
D	43	0.043	107	0.041
E	...	...	63	0.047
All	202	0.040	423	0.044

Table XI. Comparison of Mixing and Testing Precisions, $\hat{\sigma}$, for Each Method of Mixing Based on Customs Tests

(2 tests per can and one test per can per sample)

Laboratory Symbol	Customs Chemist Symbol	No. of Tests per Can	Hand Method No. of samples	$\hat{\sigma}$	Box and Screen Method No. of samples	$\hat{\sigma}$
A	I	2	32	0.064	..	...
A	I	1	32	0.056	..	...
F	J	2	76	0.060	..	...
F	J	1	76	0.063	..	...
B	O	2	..	...	41	0.061
B	O	1	..	...	41	0.063
B	O	2	..	...	58[a]	0.060
B	O	1	..	...	58[a]	0.062
B	N	2	..	...	34[a]	0.044
B	N	1	..	...	34[a]	0.045
C	Q	2	..	...	106	0.065
C	Q	1	..	...	106	0.069
D	R	2	41	0.047	103	0.044
D	R	1	41	0.055	103	0.049

[a] Includes samples from 2 ports.

ACKNOWLEDGMENT

The author is indebted to John F. Williams, chief, Division of Laboratories, Bureau of Customs, for valuable suggestions, to Joseph Lamb, Customs Laboratory, Philadelphia, Pa., and many other customs officers who cooperated in this investigation, and to Carl F. Snyder of the Polarimetry Section, National Bureau of Standards, for assisting in the control procedure

LITERATURE CITED

(1) Am. Soc. Testing Materials, Manual on Presentation of Data. Including Supplements A and B, Philadelphia, American Society for Testing Materials, 1943.
(2) Browne and Zerban, "Handbook of Physical and Chemical Methods of Sugar Analysis", 3rd ed., p. 2, New York, John Wiley & Sons, 1941.
(3) Bureau of Customs, U. S. Treasury Department, "Appraisement Circular Letter 25", Washington, D. C., Oct. 23, 1939.
(4) *Ibid.*, No. 46, July 14, 1943.
(5) Bureau of Customs, U. S. Treasury Department, "Circular Letter 2416", sampling method 17 attached, Washington, D. C., July 14, 1943.
(6) Bureau of Customs, U. S. Treasury Department, "Customs Regulations", article 721, p. 416, Washington, D. C., U. S. Government Printing Office, 1937.
(7) *Ibid.*, article 735, p. 418.
(8) Bureau of Customs, U. S. Treasury Department, "Official Laboratory Method 501.1–39", Washington, D. C., 1939.
(9) Bureau of Customs, U. S. Treasury Department, Washington, D. C., letter from chief, Division of Laboratories, to each customs chief chemist, July 14, 1943.
(10) Supplementary trade agreement between United States and Cuba, signed Dec. 23, 1941, effective Jan. 5, 1942.
(11) Vondrak, Jiri, *Z. Zuckerind. čechoslovak. Rep.*, 55, 371–6 (1931)