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CERTIFICATION OF REFERENCE IRON ORE SCH-1 FOR SODIUM AND POTASSIUM

R. Sutarno, D.J. Charette, W.S. Bowman and G.H.Faye

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CERTIFICATION OF REFERENCE IRON ORE SCH-1 FOR SODIUM AND POTASSIUM

by

R. Sutarno*, D.J. Charette**, W.S. Bowman*** and G.H. Faye*

SYNOPSIS

Reference iron ore SCH-1, previously certified for nine constituents, has been certified for sodium and potassium. Analytical results were obtained from 53 contributors in Canada and abroad, most of whom used an atomic absorption procedure that is a candidate method of the International Organization for Standardization. A statistical treatment of the data yielded recommended values which are 0.019% and 0.026% for sodium and potassium respectively.

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RAPPORT DE CANMET 78-5

HOMOLOGATION DU MINERAI DE FER DE REFERENCE SCH-1 POUR LE SODIUM ET LE POTASSIUM

par

R. Sutarno*, D.J. Charette**, W.S. Bowman***, et G.H. Faye*

SYNOPSIS

Le minerai de fer de référence SCH-1 ayant été homologué auparavant pour neuf composants est maintenant homologué pour le sodium et le potassium. Des résultats d'analyse ont été obtenus de 53 contribuants au Canada et à l'étranger, dont la plupart emploie un procédé d'absorption atomique reconnu par l'Organisation internationnale de normalisation. Après le traitement statistique des données, on a obtenu des valeurs recommandées de 0.019% pour le sodium et de 0.026% pour le potassium.

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INTRODUCTION

In 1975, reference iron ore SCH-1 was certified for nine constituents[1]. However, only provisional values for sodium and potassium were assigned because of the lack of consensus in the results obtained by atomic absorption and flame emission methods in a "free-choice" interlaboratory analytical program. The certification factors for these elements were 3.9 and 4.1 for sodium and potassium respectively, both close to 4, the maximum value for certifiability[2].

Because of the importance of even low concentrations of sodium and potassium in iron ore metallurgy, it was decided to pursue the certification of SCH-1 for these metals. Between 1972 and 1976, a working group of Sub-Committee 2 (Chemical Analysis) of ISO Technical Committee 102, Iron Ores, established that a particular procedure for the determination of sodium and potassium by atomic absorption spectrophotometry was reliable when applied to iron ores. Therefore, in 1977, a Canadian interlaboratory program was arranged in which 10 independent analysts were requested to analyze SCH-1 for sodium and potassium by the a.a. method, for certification purposes. Fortuitously, at the same time, Sub-Committee 2 of ISO Technical Committee 102 organized an extensive international trial involving up to 48 laboratories which applied the candidate a.a. method to a number of reference iron ores, including SCH-1. Because CCRMP (Canadian Certified Reference Materials Project) personnel were involved in the international program, it was possible to obtain the raw analytical results for SCH-1 and combine them with data from the concurrent Canadian program as well as with the original data from 1975[1] ultimately for the computation of recommended values for sodium and potassium.

This report presents details of the interlaboratory programs and the statistical treatment of all analytical results from 1975 through 1977. It should be noted that for continuity and clarity, laboratories (contributors) participating in the two 1977 programs have been assigned code numbers that follow consecutively those used in the original report of certification of SCH-1 in 1975[1].

Original interlaboratory program, 1975[1]

Contributors were requested to analyze two randomly-selected bottles of SCH-1 in quintuplicate for 13 constituents, including sodium and potassium, by methods of their choice. The names of the participating laboratories were given previously[1] and their sodium and potassium results appear in Tables 1a and 2a respectively.

Canadian interlaboratory program, 1977

As in the 1975 program, contributors were requested to analyze in quintuplicate two randomly-selected bottles of SCH-1 for sodium and potassium. They were asked to follow in detail the procedure of the ISO-evaluated a.a. method as described in the appendix. As will be seen, the results of this program shown in Tables 1b and 2b were used to confirm homogeneity of SCH-1.

Laboratories participating in the Canadian-organized program for 1977 were:

- Broken Hill Proprietary Limited, Australia
- Dominion Foundries and Steel Limited, Hamilton, Ontario
- Falconbridge Nickel Mines Limited, Metallurgical Laboratories, Thornhill, Ontario
- Geological Survey of Canada, Ottawa, Ontario
- Hudson Bay Mining and Smelting Company Limited, Flin Flon, Manitoba
- Iron Ore Company of Canada, Sept-Iles, Quebec
- Lakefield Research of Canada Limited, Lakefield, Ontario
- Lerch Brothers, U.S.A.
- Ontario Ministry of Natural Resources, Toronto, Ontario
- Sherritt Gordon Mines Limited, Mining and Milling Division, Lynn Lake, Manitoba

ISO interlaboratory program, 1977

As mentioned, the international program was designed by ISO/TC 102/SC 2 to determine the precision and relative accuracy of a candidate a.a. method for the determination of sodium and potassium in iron ores. Each participating laboratory analyzed in duplicate two randomly-selected bottles of a number of reference samples including SCH-1[3]. The results for the latter were made available to the CCRMP by special arrangement with ISO. Although 48 laboratories accepted samples and presumably would contribute results to ISO/TC 102/SC 2, it was considered sufficient for the certification of SCH-1 to utilize results from only the first 22 laboratories reporting as given in Tables 1c and 2c. The countries and the number of laboratories that contributed were: Canada 2, France 5, India 3, Japan 6, Sweden 3 and U.K. 3.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

As in the case of the 1975 interlaboratory program[1], the analytical data were evaluated to confirm the homogeneity of SCH-1 with respect to sodium and potassium and to estimate consensus values and their confidence intervals for use as the recommended values.

Confirmation of homogeneity

In addition to the lack of consensus among analytical results obtained for sodium and potassium in 1975, the between-bottle homogeneity of the potassium content was questionable. Table 4a, reproduced from the previous report[1], shows that 4 out of 18 laboratories (excluding those with zero within-laboratory variance) rejected the null hypothesis. It was therefore considered necessary

to verify the between-bottle homogeneity. Table 4b shows there is no rejection of the null hypothesis inherent in the analytical results from the 1977 interlaboratory program. Because the ISO interlaboratory program produced analytical results only in duplicate, the same t-test would not be meaningful. Therefore, a two-way analysis of variance was performed, i.e. between-laboratories, between-bottles within-laboratories, and within-bottles. After excluding those data with zero within-laboratory variance and those with more than two duplicates per bottle[3], 17 sets were available for this purpose. The results confirmed that the sample is homogeneous.

This was also the case with sodium results; Table 3a shows that three sets were rejected, while 3b shows no rejection. The two-way analysis of variance with 19 sets of results from the ISO international test confirms homogeneity of the sodium content of SCH-1.

Estimation of consensus values and related statistical parameters

The analytical results comprising a total of 53 sets for both sodium and potassium are given in Table 1 and illustrated in Figure 1. The improvement in precision of the 1977 results over those of 1975 is clearly evident.

As is normal practice in avoiding bias, sets of results whose means differ from the overall mean by more than twice the standard deviation of the individual results were excluded from the one-way analysis variance used to estimate the consensus values and their confidence intervals (Table 5).

It is evident that the 1977 means for sodium and potassium given in Table 5 are essentially the same as the provisional values assigned after the original interlaboratory program of 1975[1]. However, the confidence intervals and average within-laboratory coefficients of variation are appreciably smaller than the corresponding ones of 1975. The improved quality of results is reflected in the new certification factors which are 2.7 and 2.6 respectively for sodium and potassium. Because these factors are much lower than the critical value of 4 [2], the consensus means are accepted as recommended values for the alkali metals (Table 6). Success of the 1977 program was due to the exclusive use of the "standardized" ISO method (see Appendix) which is in contrast to the "free-choice" scheme of 1975.

Discussion of analytical method

In view of the newly assigned recommended values for sodium and potassium, it is of interest to reconsider the original 1975 values for methodological significance. Out of 21 laboratories reporting, six and four used flame emission spectroscopy for sodium and potassium respectively, and their means ranged from 0.01% to 0.06% for sodium and 0.02% to 0.04% for potassium. The emission method is evidently much less satisfactory than the atomic absorption method for a material such as SCH-1 and this is in line with theoretical expectations[4].

Although details of procedures used by the various contributors are not available, it is probable that the greater overall range and poorer within-laboratory precision of the 1975 atomic absorption results were due to incomplete decomposition, giving low results on the one hand, and on the other hand contamination from use of glassware rather than the recommended plastic-ware, giving high results.

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APPENDIX

OUTLINE FOR ISO-PROPOSED ATOMIC ABSORPTION METHOD FOR SODIUM IN IRON ORES

Subsamples, weighing approximately 0.5 g, of ore previously dried at 105°C for two hours, are decomposed by repeated treatment with a 1:1 mixture of concentrated hydrochloric and hydrofluoric acids. The test solution, essentially free of insoluble matter, is diluted to an appropriate volume in a plastic volumetric flask with dilute hydrochloric acid. Plastic pipettes are used if aliquoting is necessary.

The absorbance of the test solution is measured together with a blank solution having essentially the same acid and iron concentrations as the test solution. Depending upon the concentrations of the alkali metals, a wavelength of 589.0 or 589.6 nm is used for sodium, and 766.5 or 769.9 nm is used for potassium.

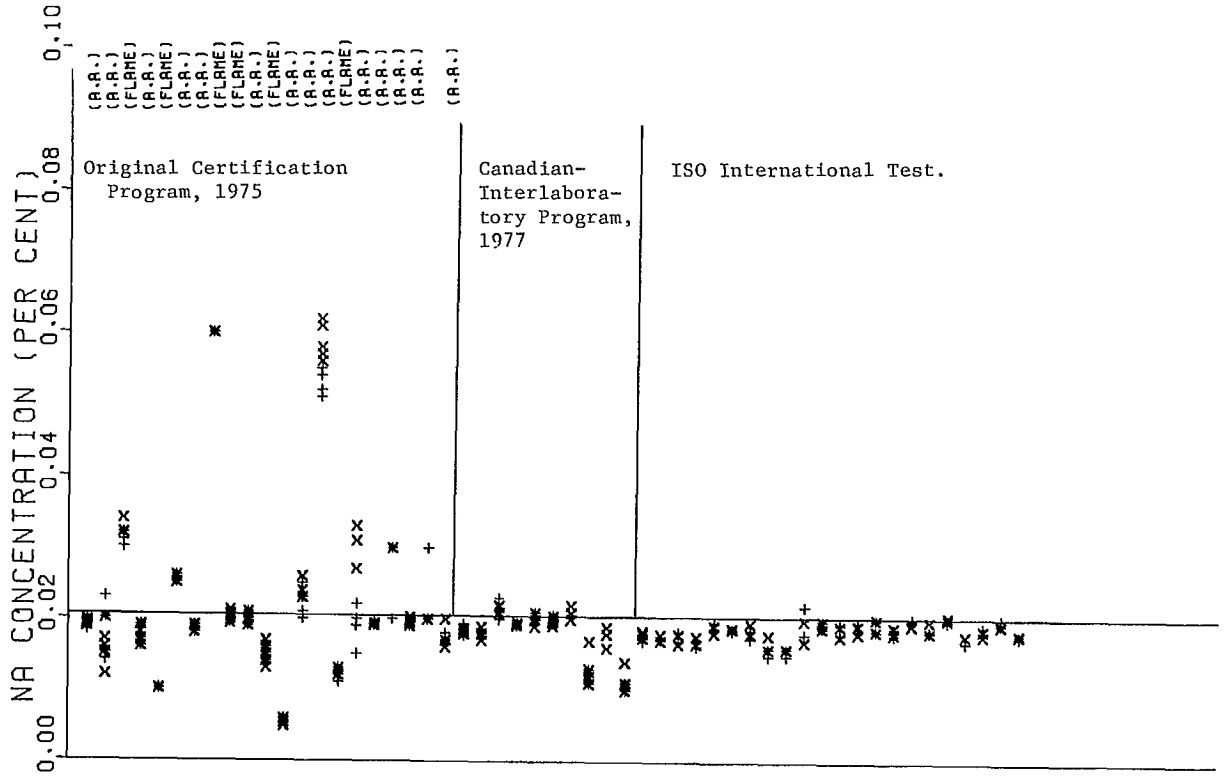

Figure 1a. Sodium results, laboratory sets

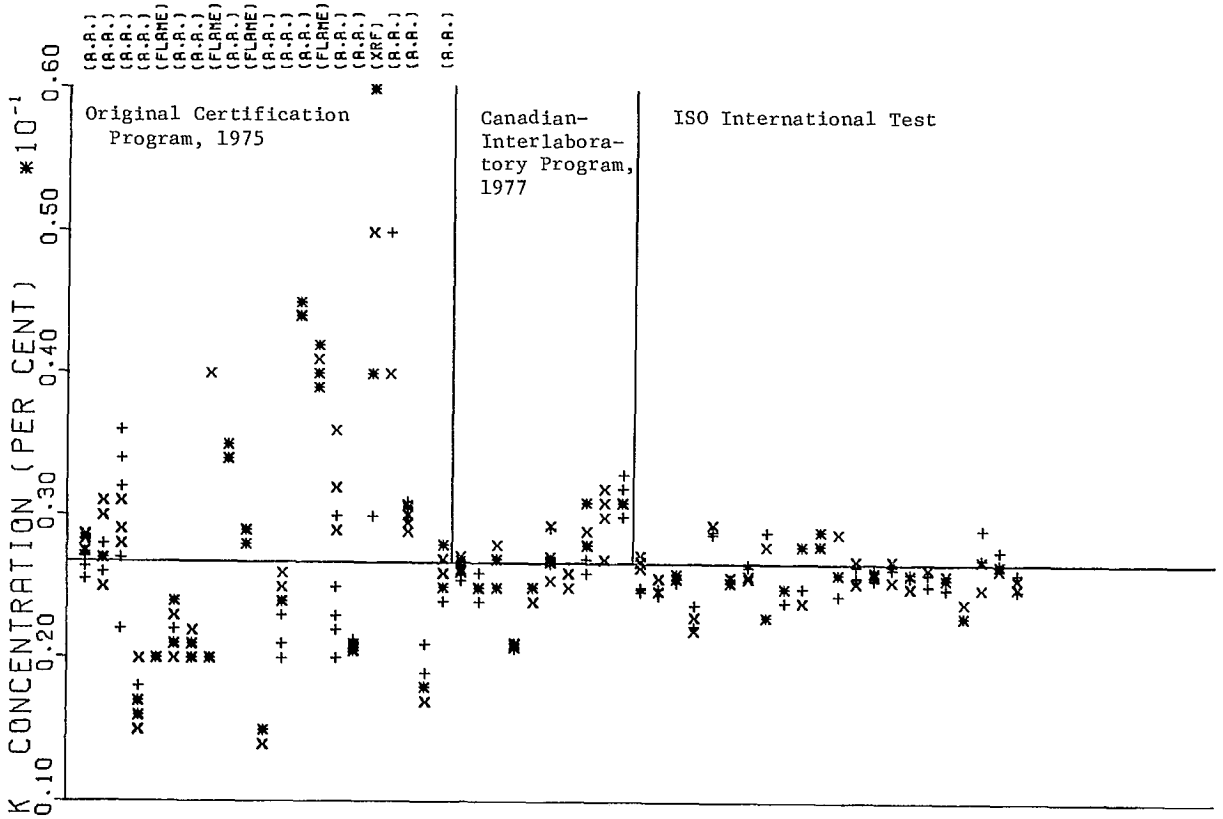

Figure 1b. Potassium results, laboratory sets

TABLE 1

Sodium results of three Interlaboratory Programs; (wt %)

(a) Original Certification Program, 1975

LAB- 1 (A.A.)	.0191	.0192	.0196	.0188	.0188	.0194	.0187	.0183	.0196	.0186
LAB- 3 (A.A.)	.0150	.0160	.0170	.0120	.0200	.0150	.0140	.0230	.0200	.0150
LAB- 5 (FLAME)	.0320	.0320	.0340	.0340	.0340	.0320	.0320	.0310	.0300	.0300
LAB- 6 (A.A.)	.0190	.0190	.0190	.0180	.0170	.0160	.0190	.0190	.0190	.0160
LAB- 7 (FLAME)	.0170	.0160								
LAB- 9 (A.A.)	.0100	.0100	.0100	.0100	.0100	.0100				
LAB-10 (A.A.)	.0260	.0250	.0250	.0260	.0250	.0260	.0250	.0260	.0250	.0250
LAB-11 (FLAME)	.0190	.0190	.0190	.0190	.0180	.0190	.0180	.0190	.0190	.0190
LAB-12 (FLAME)	.0600	.0600	.0600	.0600	.0600	.0600	.0600	.0600	.0600	.0600
LAB-14 (A.A.)	.0193	.0206	.0212	.0198	.0204	.0197	.0204	.0208	.0194	.0198
LAB-16 (FLAME)	.0190	.0200	.0200	.0210	.0200	.0200	.0210	.0200	.0200	.0190
LAB-18 (A.A.)	.0140	.0130	.0150	.0160	.0170	.0150	.0160	.0140	.0150	.0160
LAB-20 (A.A.)	.0058	.0058	.0048	.0053	.0058	.0058	.0058	.0058	.0058	.0058
LAB-21 (A.A.)	.0260	.0260	.0240	.0230	.0230	.0230	.0250	.0230	.0200	.0210
LAB-23 (FLAME)	.0620	.0580	.0610	.0570	.0560	.0540	.0520	.0540	.0510	.0550
LAB-24 (A.A.)	.0120	.0130	.0120	.0120	.0120	.0120	.0120	.0110	.0120	.0130
LAB-26 (A.A.)	.0310	.0330	.0270	.0330	.0220	.0190	.0150	.0190	.0200	
LAB-28 (A.A.)	.0191	.0194	.0193	.0194	.0193	.0191	.0193	.0196	.0192	.0193
LAB-30 (A.A.)	.0300	.0300	.0200	.0300						
LAB-37	.0203	.0190	.0198	.0194	.0196	.0198	.0192	.0198	.0194	.0190
LAB-39 (A.A.)	.0200	.0200	.0200	.0200	.0200	.0300	.0200	.0200	.0300	.0200
	.0170	.0170	.0160	.0200	.0170	.0170	.0180	.0170	.0170	.0170

(b) Canadian Interlaboratory Program, 1977

LAB-41 (A.A.)	.0185	.0179	.0186	.0179	.0181	.0180	.0177	.0193	.0177	.0181
LAB-42 (A.A.)	.0180	.0190	.0180	.0170	.0180	.0180	.0180	.0180	.0180	.0180
LAB-43 (A.A.)	.0210	.0210	.0220	.0220	.0220	.0200	.0230	.0200	.0200	.0210
LAB-44 (A.A.)	.0193	.0195	.0191	.0193	.0193	.0195	.0190	.0191	.0195	.0194
LAB-45 (A.A.)	.0200	.0210	.0200	.0190	.0200	.0200	.0200	.0200	.0200	.0210
LAB-46 (A.A.)	.0196	.0198	.0191	.0205	.0199	.0195	.0196	.0195	.0206	.0196
LAB-47 (A.A.)	.0220	.0200	.0210	.0200	.0200					
LAB-48 (A.A.)	.0130	.0110	.0110	.0170	.0120	.0120	.0120	.0130	.0110	.0130
LAB-49 (A.A.)	.0120	.0120								
LAB-50 (A.A.)	.0160	.0180	.0180	.0190	.0140	.0100	.0110	.0100	.0100	.0110
	.0100	.0110	.0110	.0110						

(c) ISO International Test

LAB-51	.0184	.0180	.0174	.0182	.0178	.0178	.0176	.0169
LAB-52	.0171	.0180	.0172	.0174				
LAB-53	.0166	.0180	.0170	.0183				
LAB-54	.0177	.0166	.0172	.0163				
LAB-55	.0182	.0193	.0189	.0196				
LAB-56	.0189	.0188	.0188	.0185				
LAB-57	.0196	.0181	.0186	.0174				
LAB-58	.0180	.0160	.0150	.0160				
LAB-59	.0160	.0160	.0160	.0150				
LAB-60	.0200	.0170	.0220	.0180				
LAB-61	.0190	.0200	.0190	.0200				
LAB-62	.0193	.0178	.0181	.0195				
LAB-63	.0194	.0182	.0198	.0186				
LAB-64	.0186	.0203	.0186	.0202				
LAB-65	.0192	.0182	.0190	.0180				
LAB-66	.0194	.0195	.0202	.0204				
LAB-67	.0200	.0184	.0186	.0184				
LAB-68	.0207	.0203	.0203	.0200				
LAB-69	.0180	.0180	.0170	.0170				
LAB-70	.0188	.0180	.0192	.0185				
LAB-71	.0196	.0195	.0196	.0204				
LAB-72	.0181	.0182	.0178	.0181				

TABLE 2

Potassium results of three Interlaboratory Programs; (wt %)

(a) Original Certification Program, 1975

LAB- 1 (A.A.)	.0287	.0283	.0275	.0286	.0283	.0274	.0274	.0264	.0271	.0255
LAB- 3 (A.A.)	.0300	.0250	.0250	.0310	.0270	.0260	.0260	.0270	.0270	.0280
LAB- 5 (A.A.)	.0310	.0290	.0280	.0310	.0310	.0340	.0320	.0270	.0360	.0220
LAB- 6 (A.A.)	.0160	.0150	.0160	.0170	.0200	.0170	.0160	.0160	.0160	.0170
LAB- 7 (FLAME)	.0180	.0180								
LAB- 9 (A.A.)	.0200	.0200	.0200	.0200	.0200	.0200				
LAB-10 (A.A.)	.0210	.0200	.0240	.0210	.0230	.0210	.0220	.0210	.0240	.0220
LAB-11 (FLAME)	.0220	.0200	.0210	.0210	.0210	.0210	.0200	.0210	.0200	.0200
LAB-14 (A.A.)	.0200	.0200	.0200	.0400	.0200	.0200	.0260	.0200	.0200	.0200
LAB-16 (FLAME)	.0340	.0340	.0340	.0350	.0340	.0340	.0340	.0350	.0350	.0340
LAB-18 (A.A.)	.0290	.0290	.0280	.0290	.0290	.0280	.0280	.0280	.0280	.0290
LAB-20 (A.A.)	.0140	.0150	.0140	.0140	.0140	.0150	.0150	.0150	.0150	.0150
LAB-21 (A.A.)	.0240	.0250	.0260	.0240	.0240	.0230	.0240	.0230	.0200	.0210
LAB-23 (FLAME)	.0440	.0440	.0450	.0440	.0440	.0440	.0450	.0450	.0450	.0440
LAB-24 (A.A.)	.0410	.0400	.0400	.0420	.0390	.0420	.0420	.0400	.0420	.0390
LAB-26 (A.A.)	.0360	.0320	.0290	.0290	.0300	.0250	.0220	.0230	.0200	
LAB-28 (XRF)	.0210	.0205	.0208	.0210	.0208	.0213	.0209	.0210	.0209	.0205
LAB-28 (A.A.)	.0500	.0500	.0400	.0500	.0600	.0400	.0400	.0300	.0600	.0400
LAB-30 (A.A.)	.0400	.0400	.0500	.0500						
LAB-37	.0301	.0308	.0289	.0307	.0295	.0309	.0306	.0299	.0307	.0310
LAB-39 (A.A.)	.0170	.0170	.0170	.0170	.0180	.0180	.0190	.0180	.0180	.0210
	.0250	.0260	.0260	.0270	.0280	.0240	.0280	.0240	.0250	.0240

(b) Canadian Interlaboratory Program, 1977

LAB-41 (A.A.)	.0261	.0272	.0269	.0262	.0263	.0267	.0255	.0265	.0259	.0266
LAB-42 (A.A.)	.0250	.0250	.0250	.0250	.0250	.0240	.0260	.0240	.0250	.0250
LAB-43 (A.A.)	.0270	.0250	.0260	.0270	.0270	.0250	.0270	.0250	.0270	.0270
LAB-44 (A.A.)	.0208	.0208	.0210	.0210	.0211	.0207	.0210	.0211	.0210	.0211
LAB-45 (A.A.)	.0250	.0250	.0250	.0240	.0250	.0250	.0250	.0250	.0250	.0250
LAB-46 (A.A.)	.0255	.0272	.0294	.0268	.0269	.0267	.0270	.0292	.0268	.0267
LAB-47 (A.A.)	.0260	.0260	.0250	.0260	.0250					
LAB-48 (A.A.)	.0280	.0290	.0280	.0310	.0280	.0280	.0270	.0260	.0280	.0310
LAB-49 (A.A.)	.0280	.0310								
LAB-50 (A.A.)	.0310	.0270	.0300	.0320						
	.0310	.0310	.0310	.0310	.0310	.0300	.0310	.0310	.0320	.0330

(c) ISO International Test

LAB-51	.0264	.0268	.0273	.0269	.0249	.0250	.0247	.0249
LAB-52	.0248	.0257	.0250	.0245				
LAB-53	.0257	.0260	.0254	.0260				
LAB-54	.0220	.0230	.0223	.0238				
LAB-55	.0295	.0295	.0288	.0290				
LAB-56	.0254	.0258	.0257	.0254				
LAB-57	.0259	.0257	.0265	.0267				
LAB-58	.0280	.0230	.0290	.0230				
LAB-59	.0250	.0250	.0240	.0250				
LAB-60	.0280	.0240	.0280	.0250				
LAB-61	.0280	.0290	.0290	.0280				
LAB-62	.0289	.0260	.0245	.0260				
LAB-63	.0254	.0270	.0258	.0266				
LAB-64	.0260	.0262	.0256	.0263				
LAB-65	.0270	.0255	.0264	.0266				
LAB-66	.0251	.0260	.0261	.0258				
LAB-67	.0264	.0264	.0260	.0252				
LAB-68	.0257	.0260	.0257	.0250				
LAB-69	.0230	.0240	.0230	.0230				
LAB-70	.0250	.0270	.0292	.0271				
LAB-71	.0264	.0267	.0268	.0277				
LAB-72	.0257	.0251	.0261	.0249				

TABLE 3

Results of t-test on between-bottle means of analytical results for sodium in SCH-1

(a) Original Certification Program, 1975

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(A.A.)	5	.0283	.0005	5	.0268	.0008	REJECT	10	.0275	.0010	3.70
LAB- 3	(A.A.)	5	.0276	.0028	5	.0268	.0008	A	10	.0272	.0020	7.31
LAB- 5	(A.A.)	5	.0300	.0014	5	.0302	.0057	A	10	.0301	.0039	12.96
LAB- 6	(A.A.)	6	.0168	.0017	6	.0168	.0010	A	12	.0168	.0013	7.94
LAB- 7	(FLAME)	3	.0200	0.0000	3	.0200	0.0000	***R**	6	.0200	0.0000	0.00
LAB- 9	(A.A.)	5	.0218	.0016	5	.0220	.0012	A	10	.0219	.0014	6.26
LAB-10	(A.A.)	5	.0210	.0007	5	.0204	.0005	A	10	.0207	.0007	3.26
LAB-11	(FLAME)	5	.0240	.0089	5	.0200	0.0000	A	10	.0220	.0063	28.75
LAB-14	(A.A.)	5	.0342	.0004	5	.0344	.0005	A	10	.0343	.0005	1.41
LAB-16	(FLAME)	5	.0288	.0004	5	.0282	.0004	A	10	.0285	.0005	1.85
LAB-18	(A.A.)	5	.0142	.0004	5	.0150	.0000	***R**	10	.0146	.0005	3.54
LAB-20	(A.A.)	5	.0246	.0009	5	.0222	.0016	REJECT	10	.0234	.0018	7.59
LAB-21	(A.A.)	5	.0442	.0004	5	.0446	.0005	A	10	.0444	.0005	1.16
LAB-23	(FLAME)	5	.0404	.0011	5	.0410	.0014	A	10	.0407	.0013	3.08
LAB-24	(A.A.)	4	.0315	.0033	5	.0240	.0038	REJECT	9	.0273	.0052	19.01
LAB-26	(A.A.)	5	.0208	.0002	5	.0209	.0003	A	10	.0209	.0002	1.15
LAB-28	(XRF)	5	.0500	.0071	5	.0420	.0110	A	10	.0460	.0097	21.00
LAB-28	(A.A.)	2	.0400	0.0000	2	.0500	0.0000	***R**	4	.0450	.0058	12.83
LAB-30	(A.A.)	5	.0300	.0008	5	.0306	.0004	A	10	.0303	.0007	2.28
LAB-37		5	.0172	.0004	5	.0188	.0013	REJECT	10	.0180	.0012	6.93
LAB-39	(A.A.)	5	.0264	.0011	5	.0250	.0017	A	10	.0257	.0016	6.10
								TOTAL	201	.0274	.0094	34.30

(b) Canadian Interlaboratory Program, 1977

LAB-41	(A.A.)	5	.0265	.0005	5	.0262	.0005	A	10	.0264	.0005	1.89
LAB-42	(A.A.)	5	.0250	.0000	5	.0248	.0008	A	10	.0249	.0006	2.28
LAB-43	(A.A.)	5	.0268	.0011	5	.0262	.0011	A	10	.0265	.0011	4.08
LAB-44	(A.A.)	5	.0209	.0001	5	.0210	.0002	A	10	.0210	.0001	.68
LAB-45	(A.A.)	5	.0248	.0004	5	.0250	.0000	A	10	.0249	.0003	1.27
LAB-46	(A.A.)	5	.0272	.0014	5	.0273	.0011	A	10	.0272	.0012	4.36
LAB-47	(A.A.)	THERE IS ONLY 1 BOTTLE							5	.0256	.0005	2.14
LAB-48	(A.A.)	6	.0287	.0012	6	.0285	.0021	A	12	.0286	.0016	5.67
LAB-49	(A.A.)	THERE IS ONLY 1 BOTTLE							4	.0300	.0022	7.20
LAB-50	(A.A.)	5	.0310	.0000	5	.0314	.0011	A	10	.0312	.0008	2.53
								TOTAL	91	.0265	.0029	11.03

TABLE 4

Results of t-test on between-bottle means of analytical results for potassium in SCH-1

(a) Original Certification Program, 1975

		BOTTLE 1			BOTTLE 2			NULL HYPOTH.	OVERALL			
		N	MEAN	ST.DEV.	N	MEAN	ST.DEV.		N	MEAN	ST.DEV.	C.V.(%)
LAB- 1	(A.A.)	5	.0191	.0003	5	.0189	.0006	A	10	.0190	.0004	2.32
LAB- 3	(A.A.)	5	.0160	.0029	5	.0174	.0039	A	10	.0167	.0033	19.97
LAB- 5	(FLAME)	5	.0332	.0011	5	.0310	.0010	REJECT	10	.0321	.0015	4.75
LAB- 6	(A.A.)	6	.0180	.0013	6	.0177	.0015	A	12	.0178	.0013	7.50
LAB- 7	(FLAME)	3	.0100	0.0000	3	.0100	0.0000	***R**	6	.0100	0.0000	0.00
LAB- 9	(A.A.)	5	.0254	.0005	5	.0254	.0005	A	10	.0254	.0005	2.03
LAB-10	(A.A.)	5	.0188	.0004	5	.0188	.0004	A	10	.0188	.0004	2.24
LAB-11	(FLAME)	5	.0600	.0000	5	.0600	.0000	A	10	.0600	.0000	.00
LAB-12	(FLAME)	5	.0203	.0007	5	.0200	.0006	A	10	.0201	.0006	3.13
LAB-14	(A.A.)	5	.0200	.0007	5	.0200	.0007	A	10	.0200	.0007	3.33
LAB-16	(FLAME)	5	.0150	.0016	5	.0152	.0008	A	10	.0151	.0012	7.93
LAB-18	(A.A.)	5	.0055	.0004	5	.0058	.0000	A	10	.0056	.0003	5.97
LAB-20	(A.A.)	5	.0244	.0015	5	.0224	.0019	A	10	.0234	.0020	8.35
LAB-21	(A.A.)	5	.0588	.0026	5	.0532	.0016	REJECT	10	.0560	.0036	6.41
LAB-23	(FLAME)	5	.0122	.0004	5	.0120	.0007	A	10	.0121	.0006	4.69
LAB-24	(A.A.)	4	.0310	.0028	5	.0190	.0025	REJECT	9	.0243	.0068	27.95
LAB-26	(A.A.)	5	.0193	.0001	5	.0193	.0002	A	10	.0193	.0001	.77
LAB-28	(A.A.)	2	.0300	0.0000	2	.0250	.0071	A	4	.0275	.0050	18.18
LAB-30	(A.A.)	5	.0196	.0005	5	.0194	.0004	A	10	.0195	.0004	2.10
LAB-37		5	.0200	0.0000	5	.0240	.0055	A	10	.0220	.0042	19.17
LAB-39	(A.A.)	5	.0174	.0015	5	.0172	.0004	A	10	.0173	.0011	6.12
								TOTAL	201	.0230	.0131	56.75

(b) Canadian Interlaboratory Program, 1977

LAB-41	(A.A.)	5	.0182	.0003	5	.0182	.0007	A	10	.0182	.0005	2.72
LAB-42	(A.A.)	5	.0180	.0007	5	.0180	.0000	A	10	.0180	.0005	2.62
LAB-43	(A.A.)	5	.0216	.0005	5	.0208	.0013	A	10	.0212	.0010	4.87
LAB-44	(A.A.)	5	.0193	.0001	5	.0193	.0002	A	10	.0193	.0002	.95
LAB-45	(A.A.)	5	.0200	.0007	5	.0202	.0004	A	10	.0201	.0006	2.82
LAB-46	(A.A.)	5	.0198	.0005	5	.0198	.0005	A	10	.0198	.0005	2.34
LAB-47	(A.A.)	THERE IS ONLY 1 BOTTLE							5	.0206	.0009	4.34
LAB-48	(A.A.)	6	.0127	.0023	6	.0122	.0008	A	12	.0124	.0016	13.06
LAB-49	(A.A.)	THERE IS ONLY 1 BOTTLE							4	.0178	.0013	7.09
LAB-50	(A.A.)	5	.0114	.0015	5	.0104	.0005	A	10	.0109	.0012	10.98
								TOTAL	91	.0176	.0036	20.43

TABLE 5

Estimation of statistical parameters for sodium and potassium in SCH-1 from the results of 1975 and 1977 interlaboratory programs.

Statistical parameters	Na		K	
	1975	1977	1975	1977
Results, no. of sets				
Total	21	53	21	53
Used for ANOVA	19	51	21	50
No. of results				
Total	201	384	201	384
used for ANOVA	181	364	201	360
Median, %	0.0192	0.0190	0.0260	0.0258
Mean, %	0.0192	0.0186	0.0274	0.0256
95% Confidence limits of the means, %				
Low	0.0163	0.0172	0.0232	0.0241
High	0.0221	0.0200	0.0316	0.0271
Average within-laboratory cv,%	7.71	5.48	7.53	4.52
Certification factor	3.9	2.7	4.1	2.6

TABLE 6

Recommended values for sodium and potassium in SCH-1.

Constituent	Recommended value	95% confidence limits
Sodium	0.019	0.017 - 0.020
Potassium	0.026	0.024 - 0.027