REPORT OF

ANALYTICAL EVALUATION PROGRAM

STANDARD WATER SAMPLES NUMBERS 3 and 4

Comparison of results obtained using SDA 3A with results using tax-free ethanol

Total hardness, Sulfate,

Chromium, Copper, Nickel

U. S. GEOLOGICAL SURVEY
WATER RESOURCES DIVISION
Quality of Water Branch
Denver, Colorado

1963

CONTENTS

PURPOSE AND PLAN

Tax-free ethanol is used in many analytical procedures; however, there are certain mandatory controls on its use which are both time consuming and costly. It has been proposed that, if possible, some non-controlled reagent be substituted for tax-free ethanol. Denatured alcohol is not a controlled item; and Specially Denatured Alcohol No. 3A (SDA 3A) would appear to be a satisfactory substitute because of its formulation (5 gallons commercially pure methyl alcohol to 100 gallons 190-proof ethyl alcohol) and its low cost. It may be purchased [1/] in either 5-gallon containers ($5.95) or in 55-gallon drums ($43.45). These prices are approximately the same as for tax-free ethanol. A permit is required for the purchase of SDA 3A, and the same procedure for ordering tax-free ethanol must be used for ordering SDA 3A. The Internal Revenue Code governing the use of specially denatured spirits by a government agency does not require that a record of use be kept (Part 211 of Title 26, Code of Federal Regulations, Subpart L, paragraphs 211.231-211.237).

There are presently four analytical procedures in Water-Supply Paper 1454 which specify the use of 95 percent ethanol. Of these, the most frequently run determinations are (1) total hardness, and (2) sulfate; the other two are chromium and copper. A nickel procedure which was distributed to District laboratories about two years ago also specifies the use of 95 percent ethanol.

In order to evaluate the feasibility of substituting SDA 3A, Specially Denatured Alcohol, for 95 percent ethanol where specified in the analytical procedures for determining total hardness, sulfate, chromium, copper, and nickel, all QW District laboratories were invited to participate in the analysis of standard reference samples, using both alcohols for comparison. The evaluation, while providing a basis for comparison of results obtained using denatured alcohol and undenatured alcohol, the program also provided a simultaneous evaluation of the methods and the laboratories performing the analyses. This report is a summary of the data submitted by the participating laboratories.

1/ Supplier: U. S. Industrial Chemicals, 624 South Michigan Avenue, Chicago 5, Illinois.

PREPARATION OF THE SAMPLES

Each sample was prepared from accurately weighed amounts of analytical reagent-grade chemicals dissolved in an accurately measured volume of distilled water which had been further purified by passage through a mixed-bed exchanger. When necessary to affect solution of the reagent, a slight excess of reagent-quality nitric acid was added to the sample.

The following compounds were used for the preparation of the samples:

$MgSO_4 \cdot 7H_2O$	$NiCl_2 \cdot 6H_2O$
K_2CrO_4	Cu (metal)

Concentrated stock solutions were prepared to contain the following concentrations of the substances indicated:

Stock solution 3

Total hardness (as $CaCO_3$)	992 ppm
Sulfate (SO_4)	953 ppm
Chromium (Cr)	5.8 ppm
Copper (Cu)	6.8 ppm
Nickel (Ni)	1.8 ppm

Stock solution 4

Total hardness (as $CaCO_3$)	4438 ppm
Sulfate (SO_4)	4265 ppm
Chromium (Cr)	32.8 ppm
Copper (Cu)	29.5 ppm
Nickel (Ni)	7.8 ppm

Individual 1-liter samples of Standard Water Sample No.3 were prepared by taking 25.0 ml of Stock solution 3 and diluting to exactly one liter. Standard Water Sample No. 4 was prepared in a similar way by diluting a 25.0-ml portion of Stock solution 4 to exactly one liter. The calculated concentrations of the two samples thus prepared were as follows:

	Standard Water Sample	
	No. 3	No. 4
Total hardness (as $CaCO_3$)	25	111
Sulfate (SO_4)	24	107
Chromium (Cr)	0.14	0.82
Copper (Cu)	0.17	0.74
Nickel (Ni)	0.04	0.20

After preparation, each sample was analyzed in duplicate using both tax-free ethanol and SDA 3A at two different times over a period of about four weeks. The results of these analyses are given in the following table. Neither sample showed any change in the concentration of the substances determined over the period of storage.

Analysis by Preparations Lab[a/]

	Standard Water Sample			
	No. 3		No. 4	
	Tax-free ethanol	SDA 3A	Tax-free ethanol	SDA 3A
Total hardness (as $CaCO_3$)	24 ppm	24 ppm	110 ppm	110 ppm
Sulfate (SO_4)	25	25	108	107
Chromium (Cr)	0.13	0.14	0.80	0.79
Copper (Cu)	0.19	0.20	0.78	0.79
Nickel (Ni)	0.05	0.04	0.18	0.18

a/ Each result represents the average of 2 duplicate determinations made over a period of about 4 weeks.

PREPARATION OF DENATURED ALCOHOL

Specially denatured alcohol No. 3A was prepared by adding one liter of commercially pure methyl alcohol to 20 liters of 190-proof ethyl alcohol. One liter of SDA 3A was sent to each of the participating laboratories.

PARTICIPATING LABORATORIES

Alaska, Palmer

California, Menlo Park

California, Sacramento

Colorado, Denver

Florida, Ocala

Louisiana, Baton Rouge

Nebraska, Lincoln

New Mexico, Albuquerque

New York, Albany

North Carolina, Raleigh

Ohio, Columbus

Oregon, Portland

Pennsylvania, Philadelphia

Texas, Austin

Utah, Salt Lake City

Wyoming, Worland

REPORTED RESULTS: TOTAL HARDNESS (ppm)

Standard Sample No. 3

Code No.	Tax-free ethanol			SDA 3A		
	(1)	(2)	Avge.	(1)	(2)	Avge.
101	25	26	26	26	26	26
102	5.9	6.1	6.0[a/]	6.3	6.1	6.2[a/]
103	25	26	26	26	26	26
104	25	25	25	25	25	25
105	23	24	24	24	24	24
106	25	25	25	25	25	25
107	--	--	--	--	--	--
108	25.0	25.0	25.0	24.0	25.0	24.5
109	26	26	26	26	26	26
110	25	25	25	25	25	25
111	25	25	25	25	25	25
112	26 26	27 27	26	26 26	27 26	26
113	27	28	27[b/]	28	27	27[b/]
114	26	26	26	26	26	26
115	25.5	26.0	26	25.5	26.0	26
116	25	25	25	25	25	25

a/ Reported as magnesium; ≅ 25 ppm T.H. and ≅ 26 ppm T.H., respectively.

b/ Should have been reported as 28 ppm.

REPORTED RESULTS: TOTAL HARDNESS (ppm)

Standard Sample No. 4

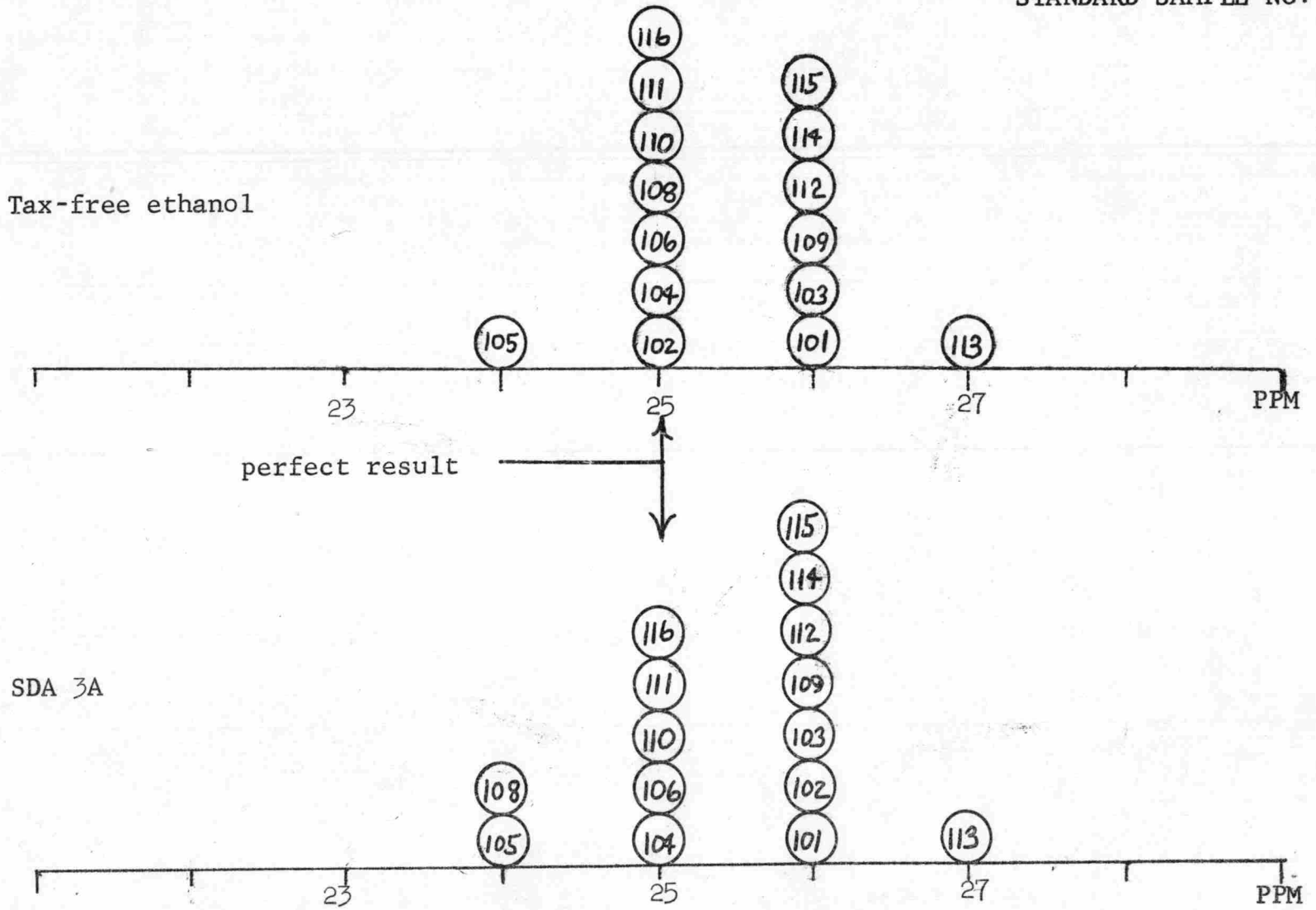
Code No.	Tax-free ethanol			SDA 3A		
	(1)	(2)	Avge.	(1)	(2)	Avge.
101	116	116	116	114	116	115
102	27	27	27[a/]	28	28	28[a/]
103	123	121	122	123	123	123
104	112	112	112	112	112	112
105	108	108	108	109	109	109
106	110	110	110	110	110	110
107	--	--	--	--	--	--
108	112	113	112.5	113	112	112.5
109	113	114	114	114	115	114
110	108	107	108	108	108	108
111	114	114	114	114	114	114
112	114 117	114 114	115	116 114	114 114	114
113	113	114	113[b/]	115	115	115
114	113	114	114	115	114	114
115	113	114	114	113	114	114
116	113	113	113	113	113	113

a/ Reported as magnesium; ≅ 111 ppm and 115 ppm T.H., respectively.

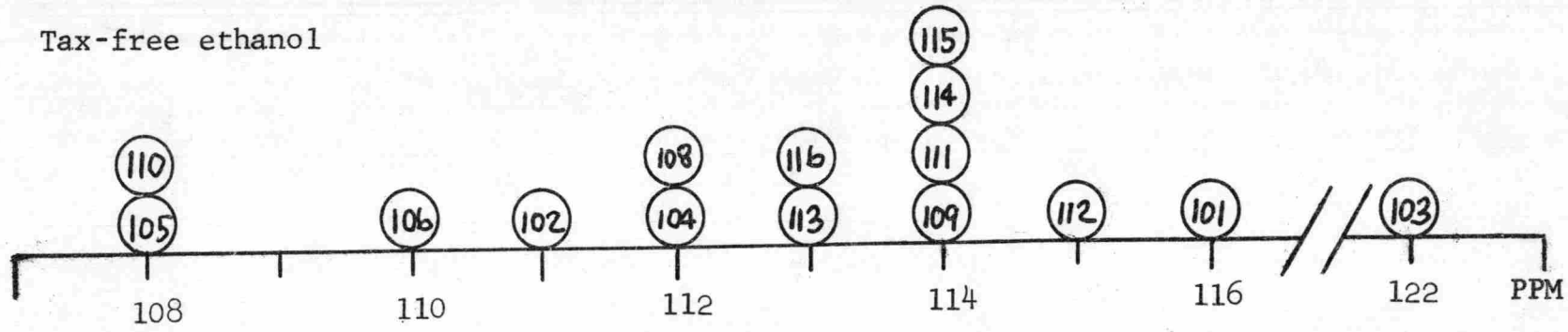
b/ Should have been reported as 114 ppm.

TOTAL HARDNESS

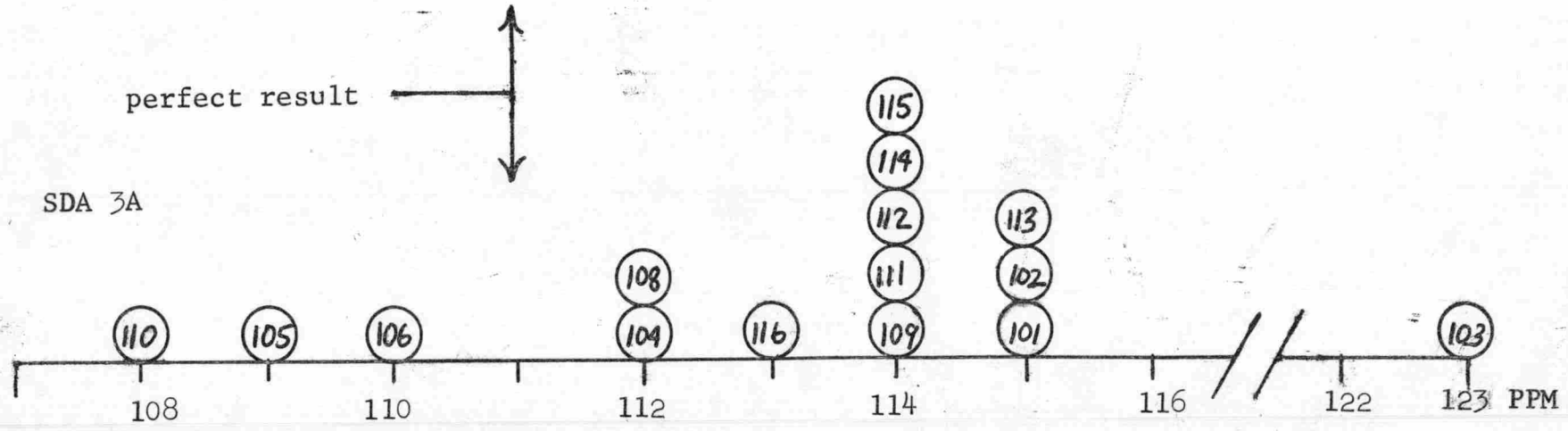
STANDARD SAMPLE NO. 3



TOTAL HARDNESS
STANDARD SAMPLE NO. 4



10



Methods used: Hardness (as $CaCO_3$)

Lab.	Method	Modifications
101	WSP 1454, D:17a-1	Porcelain evaporating dishes; no blank correction; glycerin solvent for EBT indicator.
102	WSP 1454, D:23a-3	Magnesium determined.
103	WSP 1454, D:17a-1	None
104	" "	"
105	" "	No. 2 evaporating dishes used.
106	" "	None
107	(not determined)	--
108	WSP 1454, D:17a-1	--
109	(not designated)	--
110	WSP 1454, D:17a-1	None
111	" "	$NH_2OH \cdot HCl$, NH_4OH added in order (1.5 ml each) with dropping pipet; 10-ml buret used for hardness titration.
112	" "	--
113	" "	Tax-free alcohol-EBT indicator was several months old.
114	" "	NaCN added before NH_4OH.
115	" "	None
116	" "	"

HARDNESS DETERMINATION

Standard Water Sample No. 3, 25 ppm

Errors

Error (absolute)	Number of laboratories reporting		Percentage of 15 laboratories reporting	
	Tax-free ethanol	SDA 3A	Tax-free ethanol	SDA 3A
0 ppm	7	5	47 percent	33 percent
±1 "	14	14	93 "	93 "
±2 "	15	15	100 "	100 "

Comparison of results obtained using SDA 3A with results obtained using tax-free ethanol

Laboratories reporting	-1 ppm	difference	- 1	
"	"	0 "	"	13 (87 percent)
"	"	+1 "	"	1

Standard Water Sample No. 4, 111 ppm

Errors

Error (absolute)	Number of laboratories reporting		Percentage of 15 laboratories reporting	
	Tax-free ethanol	SDA 3A	Tax-free ethanol	SDA 3A
0 ppm	1	0	7 percent	0 percent
±1 "	4	3	27 "	20 "
±2 "	6	5	40 "	33 "
±3 "	12	11	80 "	73 "
±4 "	13	14	87 "	93 "
±5 "	14	⋮	93 "	⋮
⋮	⋮	⋮	⋮	⋮
±11 "	15	⋮	100 "	⋮
±12 "		15		100 "

Comparison of results obtained using SDA 3A with results obtained using tax-free ethanol

Laboratories	reporting	-1	ppm	difference	- 2
"	"	0	"	"	9 (60 percent)
"	"	+1	"	"	2
"	"	+2	"	"	1
"	"	+3	"	"	0
"	"	+4	"	"	1

The evaluation of the data for total hardness shows no significant difference in results if denatured alcohol (SDA 3A) is substituted for tax-free ethanol. Eighty-seven percent of the laboratories reported identical values for Sample No. 3 and 60 percent reported identical values for Sample No. 4. The remaining laboratories, however, reported both higher and lower values using denatured alcohol. Nevertheless, all results are within the limits of the method.

The most probable values for hardness in Samples Nos. 3 and 4 are 25 ppm and 111 ppm, respectively. There were as many laboratories reporting 26 ppm hardness in Sample No. 3 as those reporting 25 ppm. This again indicates, as was shown in a previous study (Standard Water Samples Nos. 1 and 2), a bias in favor of reporting even-numbered values. Most of the values reported for Sample No. 4 are higher than the calculated value. Perhaps a blank correction is being neglected. The data for Sample No. 4 also indicates that reporting the results to 1 ppm probably is not justified.

REPORTED RESULTS: SULFATE (ppm)

Standard Sample No. 3

Code No.	Tax-free ethanol			SDA 3A		
	(1)	(2)	Avge.	(1)	(2)	Avge.
101	22	23	22	23	22	22
102	24	24	24	24	24	24
103	25	24	24	25	23	24
104	29	29	29	29	29	29
105	23	24	24	23	24	24
106	24	24	24	24	24	24
107	26	24	25	24	25	24
108	24.0	24.0	24.0	25.0	--[a]	25.0
109	24	24	24	24	24	24
110	24	24	24	24	22	23
111	23	24	24	24	24	24
112	24 23	24 23	24	24 23	25 24	24
113	27	26	26	25	25	25
114	24	24	24	24	24	24
115	22	23	22	22	23	22
116	24	23	24	24	23	24

a/ Omitted; bad indicator solution.

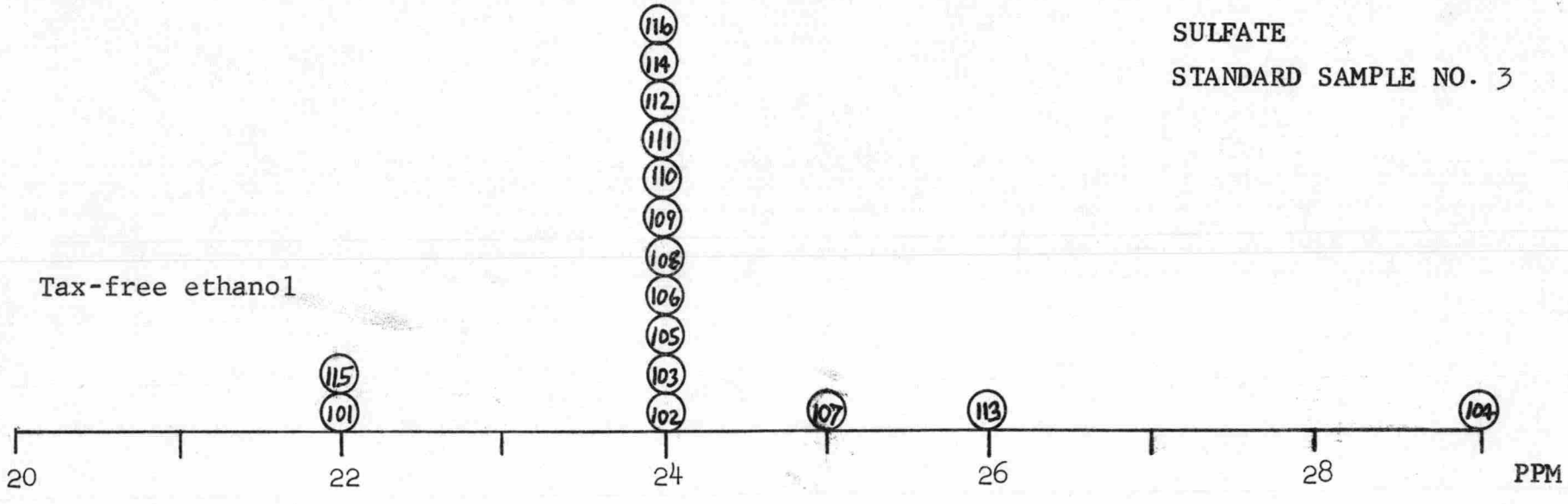
REPORTED RESULTS: SULFATE (ppm)

Standard Sample No. 4

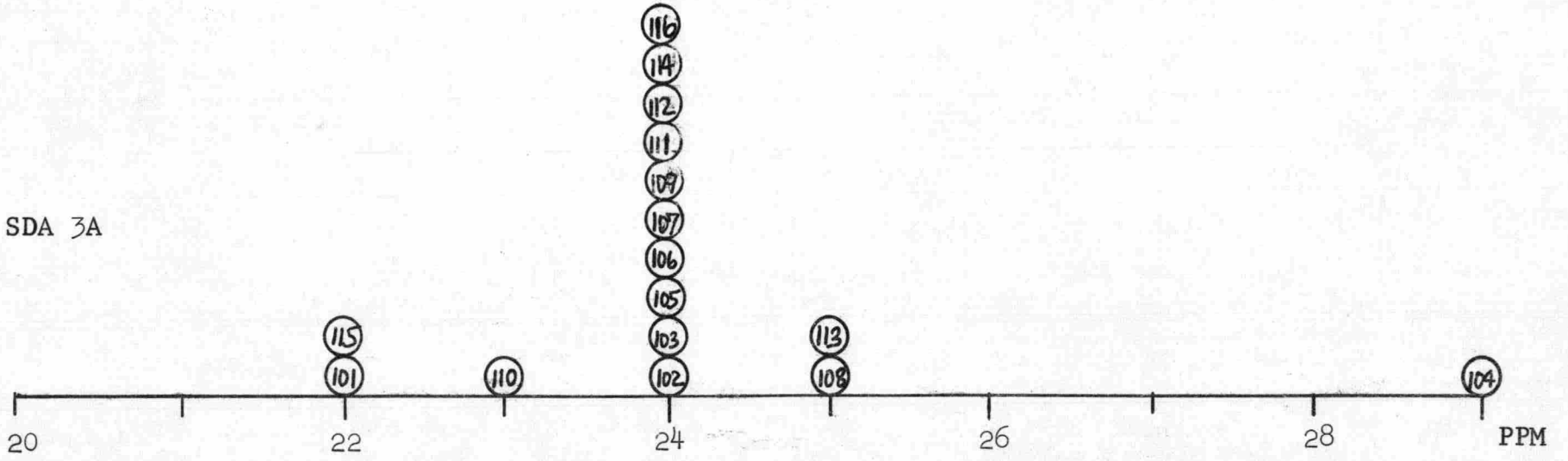
Code No.	Tax-free ethanol			SDA 3A		
	(1)	(2)	Avge.	(1)	(2)	Avge.
101	103	102	102	102	102	102
102	106	105	106	104	104	104
103	118	119	118	117	119	118
104	101	101	101	101	101	101
105	99	99	99	99	99	99
106	108	108	108	108	108	108
107	105	106	106	104	104	104
108	104	104	104	108	107	107.5
109	109	109	109	109	109	109
110	108	109	108	105	107	106
111	108	108	108	108	108	108
112	106 107	107 106	106	107 107	108 106	107
113	106	106	106	106	106	106
114	108	108	108	108	108	108
115	105	105	105	105	105	105
116	106	106	106	106	106	106

SULFATE

STANDARD SAMPLE NO. 3

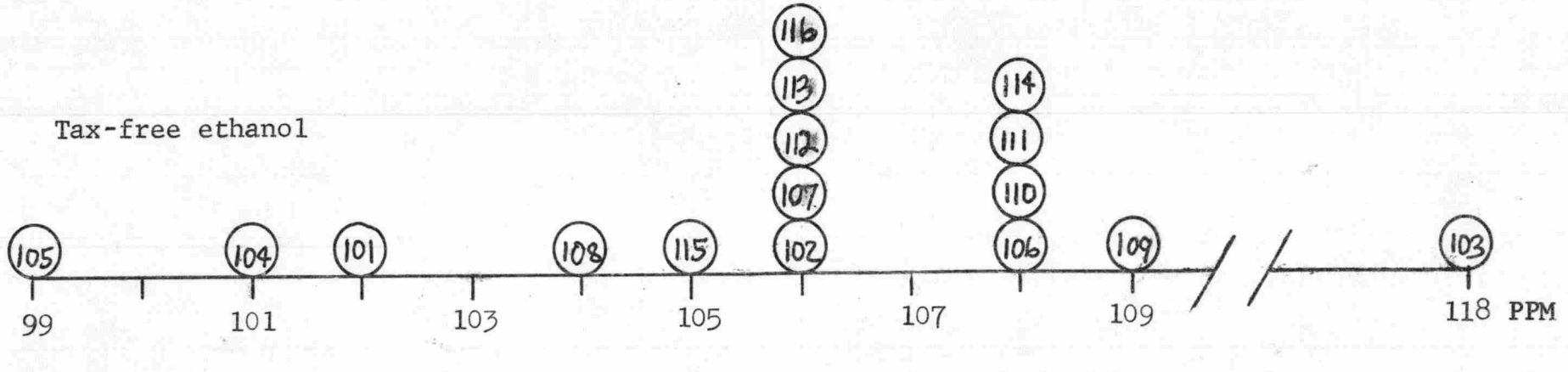


perfect result

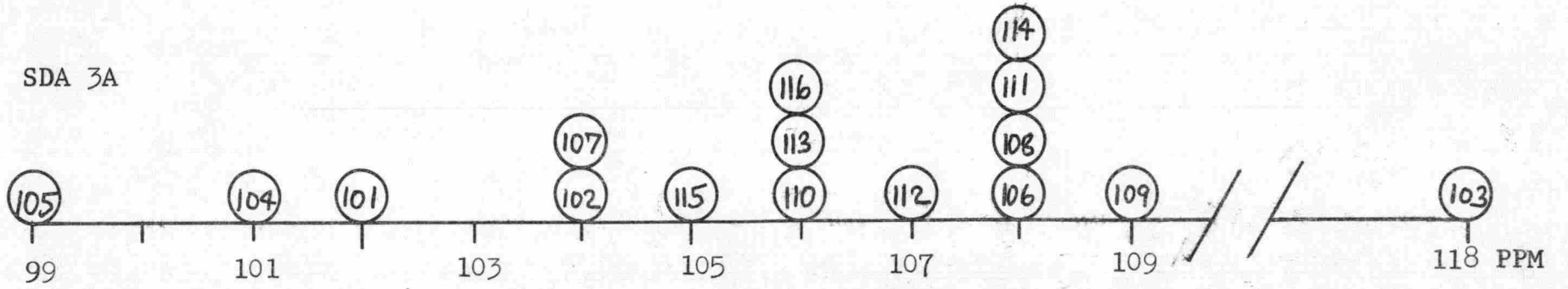


SULFATE

STANDARD SAMPLE NO. 4



perfect result



Methods used: Sulfate (SO_4)

Lab.	Method	Modifications
101	Spectrophotometric thorin. WSP 1454, D:38a-2	Lumetron colorimeter; K=490 mμ. E.P. at 0.19 absorbance.
102	" "	None
103	" "	"
104	" "	"
105	" "	Titration from absorbance of 0.100 to 0.300; 100-ml beakers used instead of 50-mm cells.
106	" "	None
107	" "	Titration from absorbance of 0.100 to 0.300.
108	" "	None
109	" "	"
110	" "	"
111	Visual thorin. WSP 1454, D:38a-1	ETOH used for thorin reagent; exchange columns are 18"x1"-diameter tubes containing a 10"-column of Amberlite resin; flow rate of sample is approx. 20 ml/min.
112	" "	--
113	(not designated)	--
114	Spectrophotometric thorin. WSP 1454, D:38a-2	Solvent-indicator solution unstable, therefore separate solutions of each prepared; indicator 0.5 g thorin and 10 g NaOAc per 500 ml; solvent 12 ml HOAc per 1,000 ml alcohol; 1 ml of indicator and 40 ml of solvent added to each sample.
115	" "	None
116	" "	pH of sample adjusted to 2.5 before indicator added.

SULFATE DETERMINATION

Standard Water Sample No. 3, 24 ppm

Errors

Error (absolute)	Number of laboratories reporting		Percentage of 16 laboratories reporting	
	Tax-free ethanol	SDA 3A	Tax-free ethanol	SDA 3A
0 ppm	11	10	69 percent	62 percent
±1 "	12	13	75 "	81 "
±2 "	15	15	94 "	94 "
⋮	⋮	⋮	⋮	⋮
±5 "	16	16	100 "	100 "

Comparison of results obtained using SDA 3A with results obtained using tax-free ethanol

Laboratories reporting -1 ppm difference - 3
" " 0 " " 12 (75 percent)
" " +1 " " 1

Standard Water Sample No. 4, 107 ppm

Errors

Error (absolute)	Number of laboratories reporting		Percentage of 16 laboratories reporting	
	Tax-free ethanol	SDA 3A	Tax-free ethanol	SDA 3A
0 ppm	0	1	0 percent	6 percent
±1 "	9	8	56 "	50 "
±2 "	11	10	69 "	62 "
±3 "	12	12	75 "	75 "
⋮	⋮	⋮	⋮	⋮
±5 "	13	13	81 "	81 "
±6 "	14	14	87 "	87 "
⋮	⋮	⋮	⋮	⋮
±8 "	15	15	94 "	94 "
⋮	⋮	⋮	⋮	⋮
±11 "	16	16	100 "	100 "

Comparison of results obtained using SDA 3A
with results obtained using tax-free ethanol

Laboratories	reporting	-2	ppm	difference	- 3
"	"	-1	"	"	0
"	"	0	"	"	11 (69 percent)
"	"	+1	"	"	1
"	"	+4	"	"	1

The calculated values for sulfate were 24 ppm (Sample No. 3) and 107 ppm (Sample No. 4). The bias in favor of reporting even-numbered values can easily be seen from this data. Eleven of the 16 laboratories reported 24 ppm for Sample No. 3 and, of these, 9 also reported perfect results using denatured ethanol. For Sample No. 4 only one laboratory reported a perfect result; more than half the laboratories reported either 106 ppm or 108 ppm. There was considerable spread in the results for Sample No. 4 with a tendency to report values lower than the calculated value. The results were good for Sample No. 3 with 94 percent of the laboratories reporting within ±2 ppm of the calculated value.

The use of denatured alcohol presents no problem in the sulfate determination. More than 75 percent of the participating laboratories obtained identical values using both alcohols and for either sample. The other results were either positive or negative with a slight trend in the negative direction.

REPORTED RESULTS: CHROMIUM (ppm)

Standard Sample No. 3

Code No.	Tax-free ethanol			SDA 3A		
	(1)	(2)	Avge.	(1)	(2)	Avge.
101	--	--	--	--	--	--
102	--	--	--	--	--	--
103	0.16	0.16	0.16	0.16	0.15	0.16
104	0.15	0.13	0.14	0.13	0.14	0.14
105	0.14	0.14	0.14	0.14	0.14	0.14
106	--	--	--	--	--	--
107	--	--	--	--	--	--
108	0.15	0.15	0.15[a/]	--	--	--
109	--	--	--	--	--	--
110	--	--	--	--	--	--
111	0.13	0.13	0.13	0.13	0.13	0.13
112	--	--	--	--	--	--
113	--	--	--	--	--	--
114	--	--	--	--	--	--
115	--	--	--	--	--	--
116	0.07	0.07	0.07[b/]	0.08	0.08	0.08[b/]

a/ 1:1 (acetone:water) substituted for ethanol.

b/ Calculation error; later corrected by participating laboratory to 0.14 ppm, tax-free ethanol, and 0.16 ppm, SDA 3A.

REPORTED RESULTS: CHROMIUM (ppm)

Standard Sample No. 4

Code No.	Tax-free ethanol			SDA 3A		
	(1)	(2)	Avge.	(1)	(2)	Avge.
101	--	--	--	--	--	--
102	--	--	--	--	--	--
103	0.80	0.80	0.80	0.80	0.81	0.80
104	0.84	0.82	0.83	0.83	0.83	0.83
105	0.78	0.78	0.78	0.78	0.78	0.78
106	--	--	--	--	--	--
107	--	--	--	--	--	--
108	0.87	0.87	0.87[a/]	--	--	--
109	--	--	--	--	--	--
110	--	--	--	--	--	--
111	0.79	0.79	0.79	0.79	0.78	0.78
112	--	--	--	--	--	--
113	--	--	--	--	--	--
114	--	--	--	--	--	--
115	--	--	--	--	--	--
116	0.37	0.36	0.36[b/]	0.36	0.39	0.37[b/]

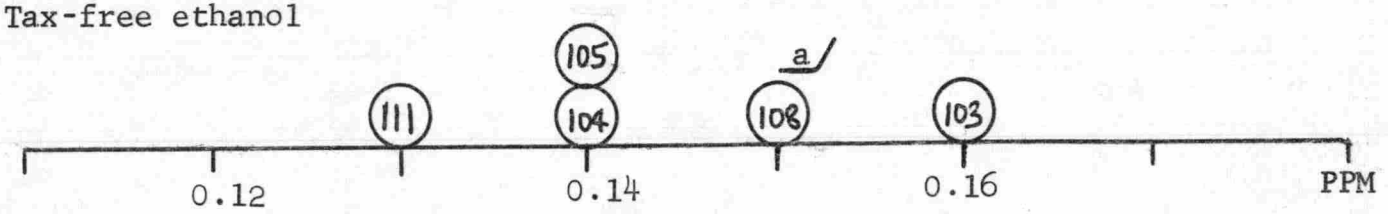
a/ 1:1 (acetone:water) substituted for ethanol.

b/ Calculation error; later corrected by participating laboratory to 0.72 ppm, tax-free ethanol, and 0.79 ppm, SDA 3A.

CHROMIUM

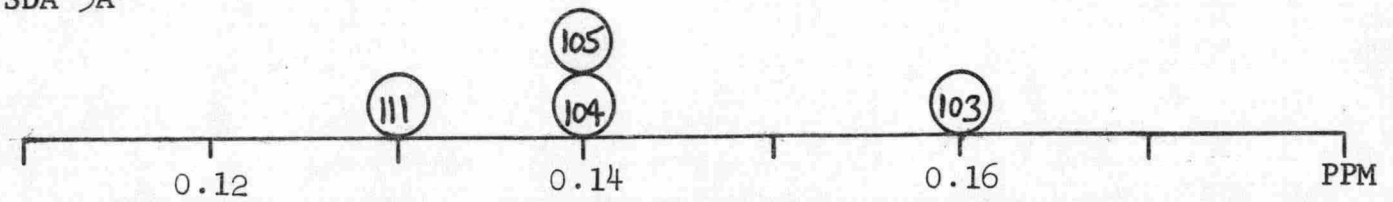
STANDARD SAMPLE NO.3

Tax-free ethanol



perfect result

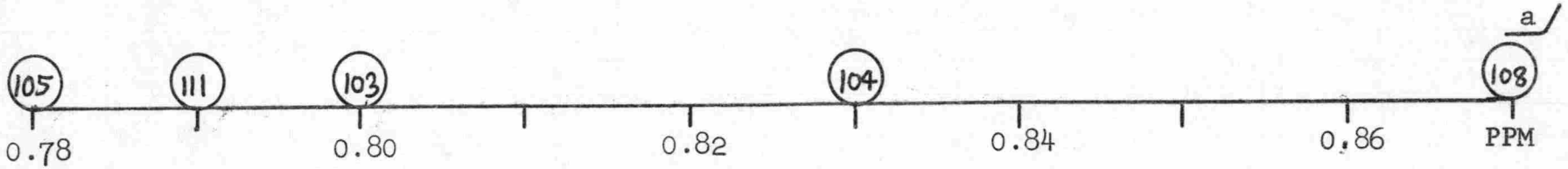
SDA 3A



a/ 1:1 (acetone:water) substituted for ethanol

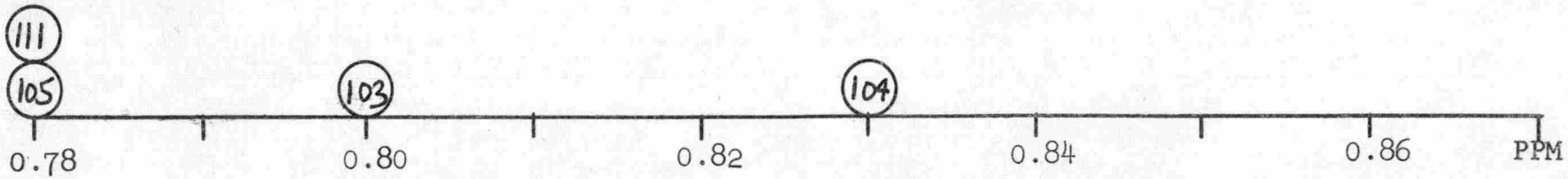
CHROMIUM
STANDARD SAMPLE NO.4

Tax-free ethanol



perfect result

SDA 3A



a/ 1:1 (acetone:water) substituted for ethanol

Methods used: Chromium (Cr)

Lab.	Method	Modifications
101	(not determined)	--
102	" "	--
103	WSP 1454, D-12a-1	None
104	" "	"
105	" "	"
106	(not determined)	--
107	" "	--
108	Diphenylcarbazide	Diphenylcarbazide reagent prepared in 1:1 acetone:water.
109	(not determined)	--
110	" "	--
111	WSP 1454, D:12a-1	None
112	(not determined)	--
113	" "	--
114	" "	--
115	" "	--
116	WSP 1454, D:12b-1	None

CHROMIUM DETERMINATION

Standard Water Sample No. 3, 0.14 ppm

Errors

Error (absolute)	Number of laboratories reporting		Percentage of 4 laboratories reporting[a/]	
	Tax-free ethanol	SDA 3A	Tax-free ethanol	SDA 3A
0.00 ppm	2	2	50 percent	50 percent
±0.01 "	3	3	75 "	75 "
±0.02 "	4	4	100 "	100 "

a/ One laboratory reported 0.15 ppm; they substituted 1:1 (acetone:water) for ethanol. Another laboratory reported 0.07 ppm (using tax-free ethanol) and 0.08 ppm (using SDA 3A), later changed to 0.14 ppm and 0.16 ppm, respectively, after correcting for a calculation error. These results are not included in the evaluation.

Comparison of results obtained using SDA 3A with results obtained using tax-free ethanol

Laboratories reporting 0.00 ppm difference - 4 (100 percent)

Standard Water Sample No. 4, 0.82 ppm

Errors

Error (absolute)	Number of laboratories reporting		Percentage of 4 laboratories reporting[a/]	
	Tax-free ethanol	SDA 3A	Tax-free ethanol	SDA 3A
0.00 ppm	0	0	0 percent	0 percent
±0.01 "	1	1	25 "	25 "
±0.02 "	2	2	50 "	50 "
±0.03 "	3	2	75 "	50 "
±0.04 "	4	4	100 "	100 "

a/ One laboratory reported 0.87 ppm; they substituted 1:1 (acetone:water) for ethanol. Another laboratory reported 0.36 ppm (using tax-free ethanol) and 0.37 ppm (using SDA 3A), later changed to 0.72 ppm and 0.74 ppm, respectively, after correcting for a calculation error. These results are not included in the evaluation.

Comparison of results obtained using SDA 3A
with results obtained using tax-free ethanol

Laboratories reporting -0.01 ppm difference - 1
" " 0.00 " " 3 (75 percent)

Only four laboratories participated in the chromium determination. The results were excellent. For Sample No. 3, the four participating laboratories were within ±0.02 ppm of the calculated value (0.14 ppm), and for Sample No. 4 (0.82 ppm) were within ±0.04 ppm.

The choice of solvent for the diphenylcarbazide reagent does not affect the results. One laboratory used neither ethanol nor denatured alcohol, but used a 1:1 ratio of acetone and water, and their results were as good as the other data submitted using denatured or tax-free ethanol.

REPORTED RESULTS: COPPER (ppm)

Standard Sample No. 3

Code No.	Tax-free ethanol			SDA 3A		
	(1)	(2)	Avge.	(1)	(2)	Avge.
101	0.20	0.20	0.20	0.20	0.20	0.20
102	--	--	--	--	--	--
103	0.15	0.15	0.15	0.15	0.15	0.15
104	0.18	0.18	0.18	0.19	0.19	0.19
105	0.17	0.17	0.17	0.15	0.15	0.15
106	--	--	--	--	--	--
107	0.20	0.20	0.20	0.20	0.20	0.20
108	0.19	0.19	0.19[a/]	--	--	--
109	--	--	--	--	--	--
110	--	--	--	--	--	--
111	0.16	0.16	0.16	0.15	0.15	0.15
112	0.15	--	0.15	0.15	--	0.15
113	0.08	0.07	0.08[b/]	0.07	0.07	0.07[b/]
114	0.17	0.17	0.17	0.18	0.18	0.18
115	--	--	--	--	--	--
116	0.17	0.17	0.17	0.17	0.17	0.17

a/ Methanol substituted for ethanol.

b/ Calculation error; later reported by participating laboratory as 0.19 ppm (tax-free ethanol) and 0.18 ppm (SDA 3A).

REPORTED RESULTS: COPPER (ppm)

Standard Sample No. 4

Code No.	Tax-free ethanol			SDA 3A		
	(1)	(2)	Avge.	(1)	(2)	Avge.
101	0.75	0.75	0.75	0.75	0.75	0.75
102	--	--	--	--	--	--
103	0.75	0.75	0.75	0.75	0.75	0.75
104	0.86	0.85	0.86	0.87	0.88	0.88
105	0.75	0.75	0.75	0.75	0.74	0.75[a/]
106	--	--	--	--	--	--
107	0.80	0.84	0.82	0.80	0.84	0.82
108	0.76	0.76	0.76[b/]	--	--	--
109	--	--	--	--	--	--
110	--	--	--	--	--	--
111	0.78	0.78	0.78	0.78	0.78	0.78
112	0.77	--	0.77	0.75	--	0.75
113	0.31	0.31	0.31[c/]	0.31	0.31	0.31[c/]
114	0.77	0.77	0.77	0.77	0.78	0.78
115	--	--	--	--	--	--
116	0.77	0.77	0.77	0.81	0.80	0.80

a/ Should have been reported as 0.74 ppm.

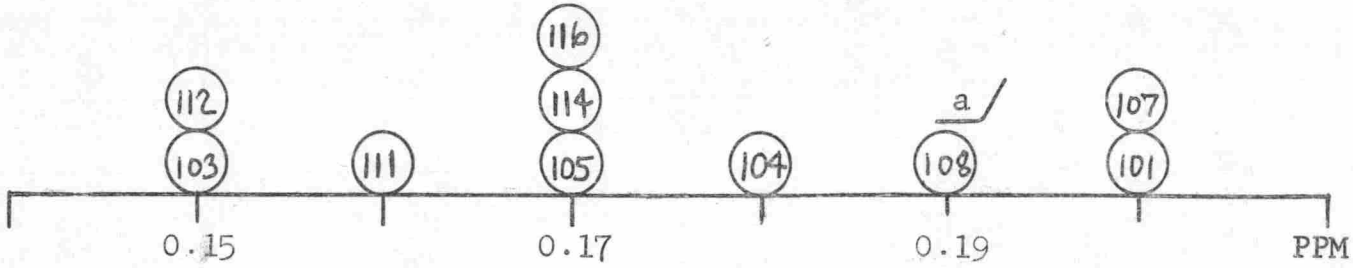
b/ Methanol substituted for ethanol.

c/ Calculation error; later reported by participating laboratory as 0.77 ppm (tax-free ethanol) and 0.77 ppm (SDA 3A).

COPPER

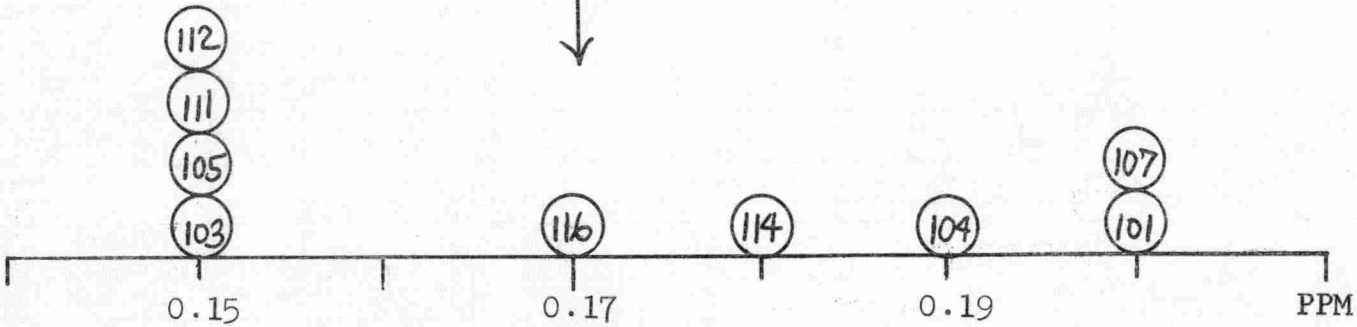
STANDARD SAMPLE NO. 3

Tax-free ethanol



perfect result

SDA 3A

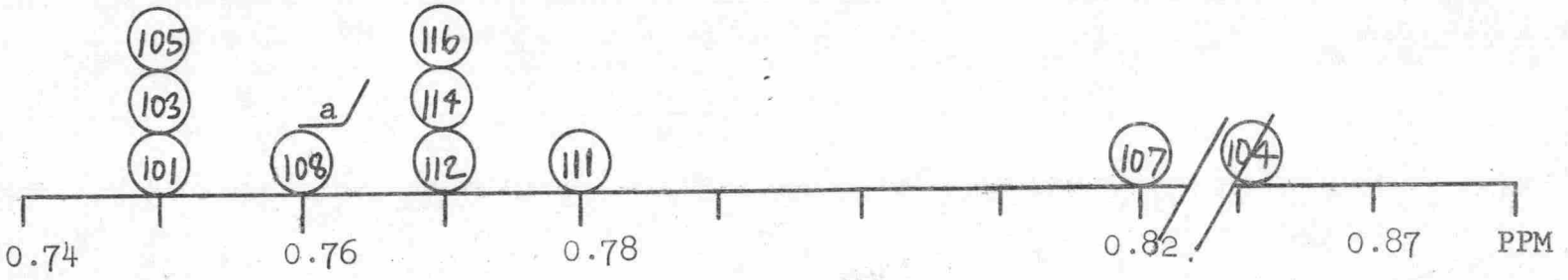


a / Methanol substituted for ethanol

COPPER

STANDARD SAMPLE NO. 4

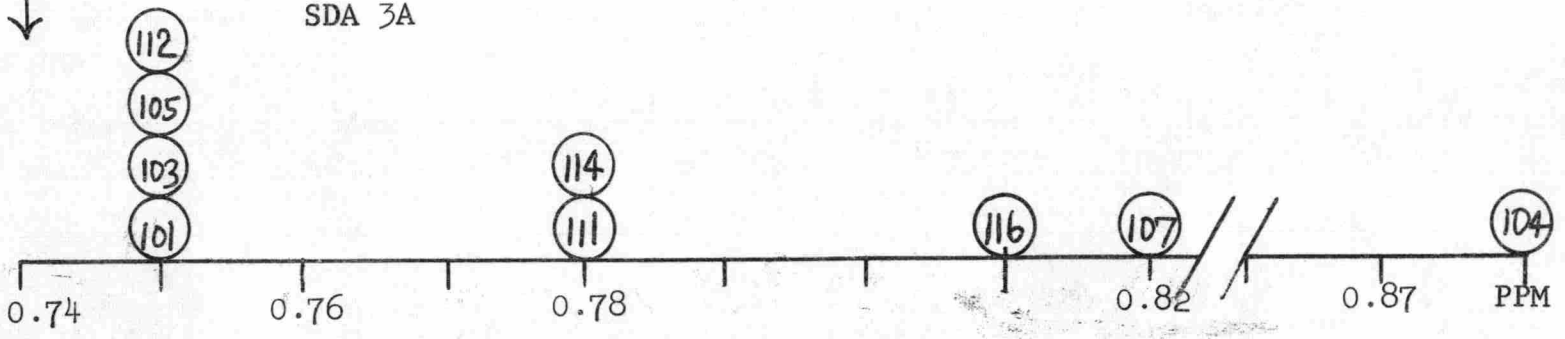
Tax-free ethanol



31

perfect result

SDA 3A



a/ Methanol substituted for ethanol

Methods used: Copper (Cu)

Lab.	Method	Modifications
101	WSP 1454, D:14a-1	None
102	(not determined)	--
103	WSP 1454, D:14a-1	None
104	" "	"
105	" "	50-mm optical depth cells used.
106	(not determined)	--
107	WSP 1454, D:14a-1	None
108	Carbamate method	MeOH substituted for ETOH.
109	(not determined)	--
110	" "	--
111	WSP 1454, D:14a-1	None
112	" "	"
113	" "	"
114	" "	"
115	(not determined)	--
116	WSP 1454, D:14a-1	None

COPPER DETERMINATION

Standard Water Sample No. 3, 0.17 ppm

Errors

Error (absolute)	Number of laboratories reporting		Percentage of 9 laboratories reporting[a/]	
	Tax-free ethanol	SDA 3A	Tax-free ethanol	SDA 3A
0.00 ppm	3	1	33 percent	11 percent
±0.01 "	5	2	55 "	22 "
±0.02 "	7	7	78 "	78 "
±0.03 "	9	9	100 "	100 "

a/ One laboratory reported 0.19 ppm; they substituted methanol for ethanol. Another laboratory reported 0.08 ppm (using tax-free ethanol) and 0.07 ppm (using SDA 3A), later changed to 0.19 ppm and 0.18 ppm, respectively, after correcting for a calculation error. These results are not included in the evaluation.

Comparison of results obtained using SDA 3A with results obtained using tax-free ethanol

Laboratories reporting	-0.02 ppm	difference	-	1
"	-0.01 "	"		1
"	0.00 "	"		5 (55 percent)
"	+0.01 "	"		2

Standard Water Sample No. 4, 0.74 ppm

Errors

Error (absolute)	Number of laboratories reporting: Tax-free ethanol	Number of laboratories reporting: SDA 3A	Percentage of 9 laboratories reporting[a]: Tax-free ethanol	Percentage of 9 laboratories reporting[a]: SDA 3A
0.00 ppm	0	0	0 percent	0 percent
±0.01 "	3	4	33 "	44 "
±0.03 "	6		67 "	
±0.04 "	7	6	78 "	67 "
±0.06 "		7		78 "
±0.08 "	8	8	89 "	89 "
±0.12 "	9		100 "	
±0.14 "		9		100 "

a/ One laboratory reported 0.76 ppm; they substituted methanol for ethanol. Another laboratory reported 0.31 ppm (for both alcohols), later changed to 0.77 ppm (for both alcohols) after correcting for a calculation error. These results are not included in the evaluation.

Comparison of results obtained using SDA 3A with results obtained using tax-free ethanol

Laboratories reporting	-0.02 ppm	difference	- 1	
"	"	-0.01 "	"	0
"	"	0.00 "	"	5 (55 percent)
"	"	+0.01 "	"	1
"	"	+0.02 "	"	1
"	"	+0.03 "	"	1

Eleven laboratories determined copper. However, only 9 sets of data were used in the evaluation since one of the two laboratories not included used methanol in place of ethanol, while the other had calculation errors. The copper method appears to be satisfactory, with 78 percent of the data falling within ±0.02 ppm of the calculated value for Sample No 3, and between ±0.04 and ±0.06 ppm for Sample No. 4.

SDA 3A presents no problem in the copper determination; more than half of the laboratories reported identical results using either alcohol. The remaining laboratories reported results on both sides of their tax-free alcohol value. These differences were within the limits of the method.

CONCLUSIONS AND RECOMMENDATIONS

From the data for each method tested, denatured alcohol (SDA 3A) can be substituted for tax-free ethanol with no adverse effect. The data from the sulfate determination were superior to the data from the other methods, and the sulfate method gives us the best test for this study since 80 percent of the sample solution is alcohol and 5 percent of this is methanol. In the other methods, only minute amounts of methanol from the SDA 3A formulation are present.

Good results were obtained by one laboratory using methanol in the copper procedure and a 1:1 ratio of acetone to water in the chromium procedure. It is possible that these solvents may be used as substitutes for ethanol in these two determinations.

Hardness

1. Hardness concentrations of the order of 25 ppm can be determined to within ±1 ppm.

2. Hardness concentrations above 100 ppm cannot be determined to within ±1 ppm by the present method. The reliability of the present method for determining hardness above 100 ppm is approximately ±3 ppm, and results should be reported with this notation.

Sulfate

1. Concentrations of the order of 24 ppm can be reported accurately to within ±1 ppm.

2. Sulfate concentrations greater than 100 ppm cannot be determined to within ±1 ppm by the present method. The accuracy of the method at this concentration level is probably ±5 ppm, and results should be reported with this notation.