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**INTERCOMPARISON OF
ENRICHED STABLE ISOTOPE REFERENCE MATERIALS
FOR MEDICAL AND BIOLOGICAL STUDIES**

by

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1. INTRODUCTION

This report summarizes the results of an intercomparison exercise organized by IAEA during the latter part of 1988 and 1989. Data is presented for 13 different kinds of enriched stable isotope reference material containing ^2H , ^{13}C , ^{15}N and ^{18}O . Results were submitted by 40 participants in 20 countries (Annex 2).

2. DESCRIPTION OF THE MATERIALS

^2H -labelled water (IAEA-302): Two deuterium-labelled water samples (~500 and ~1000 ‰ ^2H vs VSMOW) were made from highly enriched (99.8 atom percent ^2H) deuterium oxide, which was diluted with natural-abundance distilled water. The labelling was checked by reduction over Zn to HD gas. The samples are sterile and pyrogen free.

^{13}C -labelled sodium bicarbonate (IAEA-303): Two sodium bicarbonate reference materials (~100 and ~450 ‰ ^{13}C vs VPDB) were prepared from $\text{NaH}^{13}\text{CO}_3$ (isotopic purity ~ 99 atom percent), made up in distilled water and assayed for total CO_2 by acidification in a vacuum line and manometric assay.

^{18}O -labelled water (IAEA-304): Two ^{18}O -labelled water samples (~250 and ~500 ‰ ^{18}O vs VSMOW) were made from enriched ^{18}O -water (20 atom percent ^{18}O), which was diluted with natural-abundance distilled water. The labelling was checked by CO_2 isotopic exchange. The samples are sterile and pyrogen free.

^{15}N -labelled ammonium sulphate (IAEA-305): Two materials (~40 and ~400 ‰ ^{15}N vs air) were prepared from highly enriched ^{15}N -labelled ammonium sulphate by dilution with the unlabelled compound in bidistilled water. After mixing, evaporation, freeze drying, crystallization and further mixing, the material was transferred to the IAEA in small vials containing approximately 100 mg each and stored at room temperature. (See also IAEA-311.)

^{13}C -labelled UL-glucose (IAEA-309): Two uniformly ^{13}C -labelled D-glucose standards (~100 and ~550 ‰ ^{13}C vs VPDB) were prepared by appropriate mixing of highly enriched (97 atom percent) UL-D-glucose and D-glucose at natural ^{13}C -abundance. The water soluble mixtures were recrystallized from H_2O /acetone (4 °C) and the harvested crystals dried at 45 °C for 48 hours. The materials were presented to the IAEA for distribution as approximately 100 mg UL-D-glucose in sealed glass ampoules to be stored at room temperature.

^{15}N -labelled urea (IAEA-310): Two materials (~50 and ~250 ‰ ^{15}N vs air) were prepared from highly enriched ^{15}N -labelled urea by dilution with the unlabelled compound in bidistilled water. After mixing, evaporation, freeze drying, crystallization and further mixing, the materials were transferred to the IAEA's laboratory. The final homogeneous products were dispensed into small vials containing approximately 100 mg each and stored at room temperature.

¹⁵N-labelled ammonium sulphate (IAEA-311): This material (~2 atom % ¹⁵N) was prepared from highly enriched ¹⁵N-labelled ammonium sulfate by dilution with the unlabelled compound in bidistilled water. After mixing, evaporation, freeze drying, crystallization and further mixing, the material was transferred to the IAEA in small vials containing approximately 100 mg each and stored at room temperature. (See also IAEA-305.)

3. ORGANIZATION OF THE INTERCOMPARISON

The availability of these materials was announced in the Agency's AQCS catalogue [1] for 1988 and 1989, and also in a number of scientific journals catering to scientists who are using enriched stable isotopes in medical, biological and agricultural studies. Interested scientists were offered the possibility of obtaining the materials cost free in exchange for their agreement to participate in an intercomparison exercise. Most of the participants were therefore "self selected". In addition, however, a number of scientists known by the Agency to be experts in this field were specifically invited to take part in the intercomparison.

All participants were requested to make at least three, but preferably six, separate determinations of each isotope and to report their results together with information on the method used and its limit of detection (LOD). A report form was provided for this purpose. Methods were coded according to the scheme reproduced in Annex 1.

In order to preserve anonymity in respect of the actual results reported, each participant is identified in this report only by means of a laboratory code number. There is no relation between the laboratory code number and the order in which the addresses are listed in Annex 2.

4. STATISTICAL METHODS

The results of the intercomparison were evaluated by a computer program which examines the data for each analyte and reference material in turn. This procedure [2] has been in use successfully for many years for evaluating the results of IAEA intercomparisons.

For each participant and analyte, the individual net results (up to six in number) are first reduced to a single mean value and its standard deviation. In the subsequent calculations each of these is treated as a single unweighted value. Four different outlier tests (Dixon's, Grubb's, coefficient of skewness and coefficient of kurtosis) are then applied at a significance level of 5%. Any laboratory mean thereby identified as an outlier by one or the other of these tests is subsequently excluded from further consideration. The remaining "accepted" laboratory means, say "n" in number, are then combined in the usual way, without weighting, to provide estimates of the overall mean, \bar{x} , and its associated standard deviation (SD), standard error and 95% confidence interval. The latter is defined as the range between the extreme lower and upper limits of the quantity ($\bar{x} \pm c$) where $c = SD.t/\sqrt{n}$ and t is the value of Student's t on (n - 1) degrees of freedom at the 5% probability level (2-sided).

5. RESULTS

The results presented below are largely self explanatory. For each reference material, three kinds of output are provided: (1) a table of results listing the mean value and other relevant information reported by each laboratory, (2) a statistical summary of these results, and (3) a graphical representation of the results (plots). The tables and figures are in the following order:

Table 1:	^2H in IAEA-302A (water)
Table 2:	^2H in IAEA-302B (water)
Table 3:	Statistical summary of results for ^2H samples
Figure 1:	^2H in IAEA-302A (water)
Figure 2:	^2H in IAEA-302B (water)
Table 4:	^{18}O in IAEA-304A (water)
Table 5:	^{18}O in IAEA-304B (water)
Table 6:	Statistical summary of results for ^{18}O samples
Figure 3:	^{18}O in IAEA-304A (water)
Figure 4:	^{18}O in IAEA-304B (water)
Table 7:	^{13}C in IAEA-303A (sodium bicarbonate)
Table 8:	^{13}C in IAEA-303B (sodium bicarbonate)
Table 9:	^{13}C in IAEA-309A (UL-glucose)
Table 10:	^{13}C in IAEA-309B (UL-glucose)
Table 11:	Statistical summary of results for ^{13}C samples
Figure 5:	^{13}C in IAEA-303A (sodium bicarbonate)
Figure 6:	^{13}C in IAEA-303B (sodium bicarbonate)
Figure 7:	^{13}C in IAEA-309A (UL-glucose)
Figure 8:	^{13}C in IAEA-309B (UL-glucose)
Table 12:	^{15}N in IAEA-305A (ammonium sulphate)
Table 13:	^{15}N in IAEA-305B (ammonium sulphate)
Table 14:	^{15}N in IAEA-310A (urea)
Table 15:	^{15}N in IAEA-310B (urea)
Table 16:	^{15}N in IAEA-311 (ammonium sulphate)
Table 17:	Statistical summary of results for ^{15}N samples

Figure 9:	^{15}N in IAEA-305A (ammonium sulphate)
Figure 10:	^{15}N in IAEA-305B (ammonium sulphate)
Figure 11:	^{15}N in IAEA-310A (urea)
Figure 12:	^{15}N in IAEA-310B (urea)
Figure 13:	^{15}N in IAEA-311 (ammonium sulphate)
Table 18:	Overview of all recommended values (see also section 6)

The following is some more information about the three different kinds of output:

Table of results The method codes are listed in Annex 1. In the column "mean", all values marked by an asterisk were identified as statistical outliers (see section 4). The last column (Lab LoD) refers to the Limit of Detection reported by each laboratory. (This value has not yet been used in any of the calculations.)

Statistical summary This is the output from the evaluation described in section 4. The most important values to look at are in the last line, which gives the confidence interval for the overall mean based only on the accepted results (i.e. excluding statistical outliers).

Plots Each point represents one set of results reported by one laboratory for one reference material. They have been arranged in numerical order from low to high. The error bar represents the standard deviation. The number below the error bar is the number of observations. The code on top of the error bar represents the method code (first two characters only). Results that were identified as outliers are listed but not plotted. The dashed horizontal lines indicate the 95% confidence interval (taken from the bottom line of the statistical summary table).

6. CERTIFICATION

The overall means of accepted laboratory averages (i.e. *after* removal of statistical outliers) are hereby proposed by IAEA to be the *recommended values*. These, together with their 95% confidence intervals, may be regarded as *provisional certified values*. An overview is presented in Table 18.

These recommended values will remain in force pending a more detailed evaluation of the results so far submitted, and of any new results that may be provided in the meantime. This new evaluation will take account of comments, if any, submitted by individual participants and will also place greater weight on an appraisal of the sample preparation procedures and analytical methods actually employed. It will be conducted by experts appointed by IAEA, probably during 1991.

7. ACKNOWLEDGEMENTS

This intercomparison would not have been possible without the assistance of many persons and institutes. IAEA would like to thank all persons who submitted analytical results, and is particularly grateful to the following institutes and persons who provided the labelled materials used in these intercomparisons:

The USDA/ARS Children's Nutrition Center, Baylor College of Medicine, Houston, USA (Dr. P. Klein), and MSD Canada, Ltd. (Mr. I.I. Lesk) for the ^2H - and ^{18}O -labelled water samples and for the ^{13}C -labelled sodium bicarbonate reference materials;

The Nestlé Research Centre, Vevey, Switzerland (Dr. E. Fern) for the ^{15}N -labelled ammonium sulphate reference materials;

The Clinical Research Centre, Harrow, UK (Dr. D. Halliday) for the ^{13}C -labelled UL-glucose reference materials;

The Central Institute of Isotope and Radiation Research, Leipzig, Germany (Prof. H. Faust) for the ^{15}N -labelled urea reference materials;

8. REFERENCES

1. AQCS, Analytical Quality Control Services, IAEA Vienna (issued annually in January).
2. Dybczynski, R., Comparison of the effectiveness of various procedures for the rejection of outlying results and assigning consensus values in interlaboratory programmes involving determination of trace elements or radionuclides. *Anal. Chim. Acta*, **117** (1980) 53-70.

Table 1: ²H in IAEA-302A (Water)

Recommended value: 508.4 per mille (vs VSMOW)
95% Confidence interval: 505.5 to 511.3 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	10	A30	3	508.73	0.90	0.20	1.50
2	14	A30	6	511.73	0.70	0.10	1.00
3	18	A20	3	506.33	3.51	0.70	1.40
4	21	A28	4	501.25	1.50	0.30	6.00
5	24	A20	6	505.57	1.77	0.40	2.25
6	32	A38	6	506.28	2.71	0.50	0.97
7	33	A30	5	515.50	3.65	0.70	
8	35	A38	2	509.35	0.35	0.10	4.20
9	36	A30	4	506.77	2.36	0.50	1.00
10	38	A2	1	527.54*			
11	40	A30	6	512.53	0.57	0.10	1.20

Table 2: ²H in IAEA-302B (Water)

Recommended value: 996 per mille (vs VSMOW)
95% Confidence interval 987 to 1004 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	10	A30	3	997.40	1.31	0.10	1.50
2	14	A30	6	1007.63	1.17	0.10	1.00
3	18	A20	3	993.00	7.94	0.80	1.40
4	21	A28	4	984.25	3.30	0.30	6.00
5	24	A20	6	981.39	5.03	0.50	2.25
6	32	A38	3	1005.73	4.32	0.40	0.97
7	33	A30	5	1015.60	8.59	0.80	
8	36	A30	4	1000.10	1.74	0.20	1.00
9	38	A2	1	978.26			
10	40	A30	6	994.42	4.26	0.40	1.20

Table 3: Statistical summary of results for ^2H samples

Isotope determined:		^2H	^2H
Material:		IAEA-302A	IAEA-302B
Unit:		per mille	per mille
Number of reported results	Laboratory averages:	11	10
	Individual determinations:	46	41
Number of accepted results	Laboratory averages:	10	10
	Individual determinations:	45	41
Total range of Laboratory averages:		501.25 - 527.54	978.26 - 1015.60
Range of accepted Laboratory averages:		501.25 - 515.50	978.26 - 1015.60
Percentage of outlying Laboratories:		9	0
Overall mean of accepted Laboratory averages:		508.41	995.78
Standard deviation (S.D.)	Abs:	4.08	12.07
	Rel %:	0.8	1.2
Standard error (S.E.)	Abs:	1.29	3.82
	Rel %:	0.3	0.4
Confidence limits for the mean of population for probability level .95		505.48 - 511.33	987.15 - 1004.41

Figure 1: ^2H in IAEA-302A (Water)

Recommended value: 508.4 per mille (vs VSMOW)
95% Confidence interval: 505.5 to 511.3 per mille

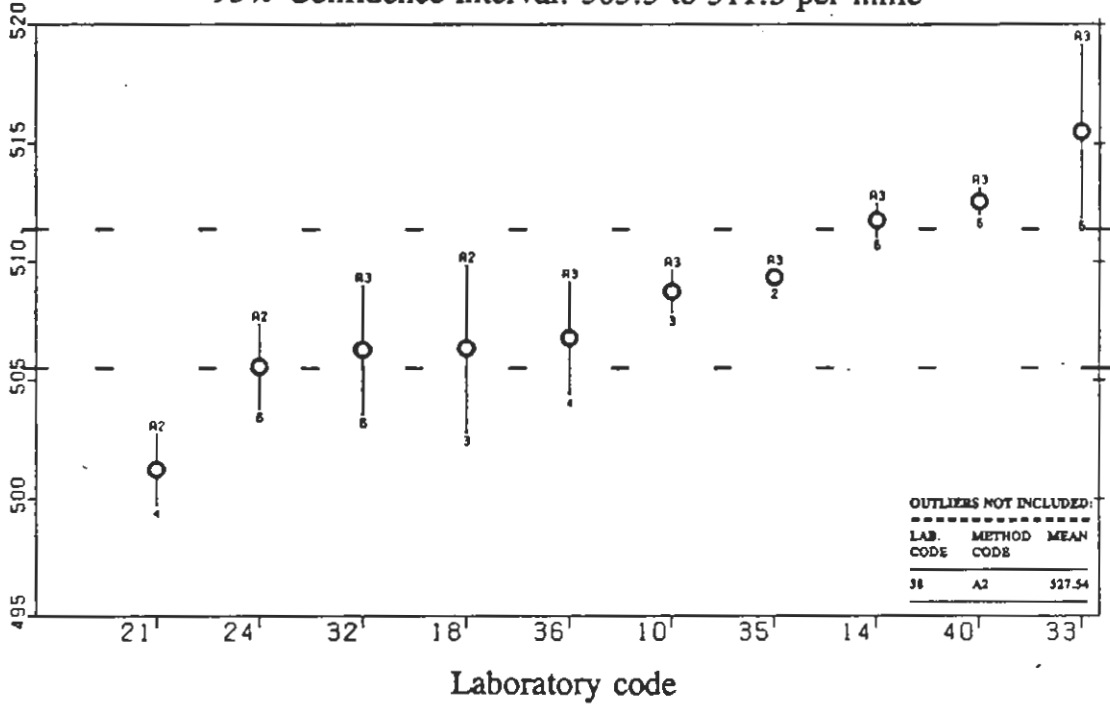


Figure 2: ^2H in IAEA-302B (Water)

Recommended value: 996 per mille (vs VSMOW)
95% Confidence interval 987 to 1004 per mille

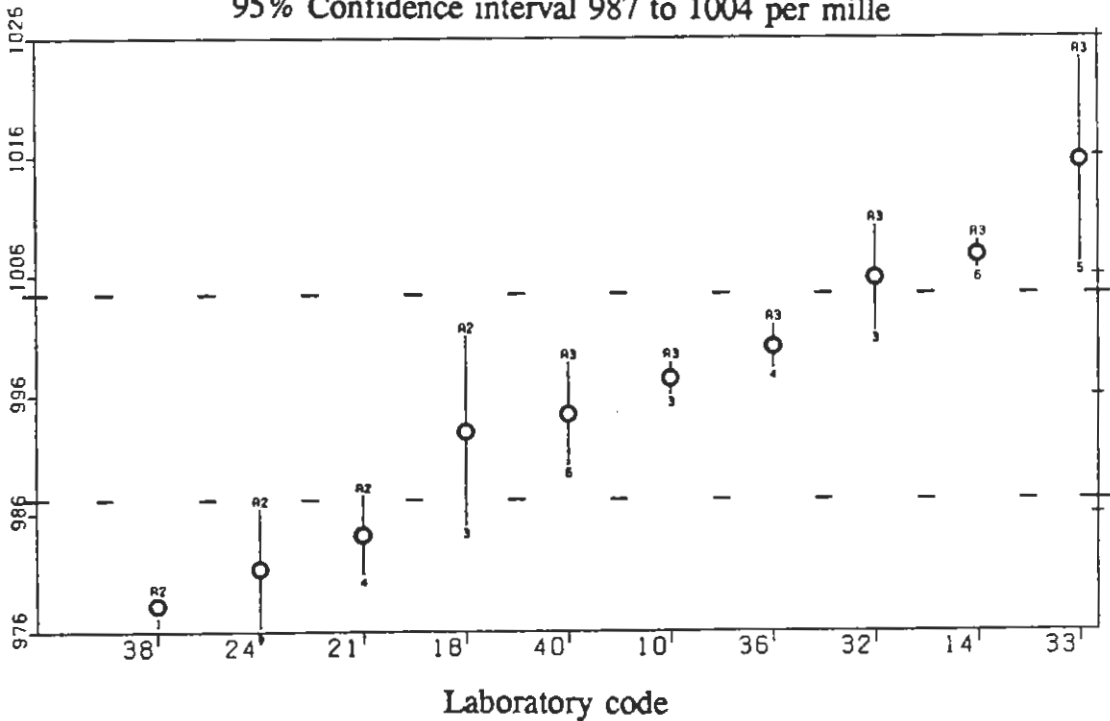


Table 4: ¹⁸O in IAEA-304A (Water)

Recommended value: 251.7 per mille (vs VSMOW)

95% Confidence interval: 249.2 to 254.2 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	10	A37	2	258.24	1.15	0.40	0.09
2	14	A37	5	253.21	0.87	0.30	0.10
3	15	A37	5	249.74	0.49	0.20	0.30
4	18	A27	2	256.05	0.07	0.00	0.10
5	21	A27	3	248.83	3.15	1.30	0.60
6	24	A27	2	251.20	0.86	0.30	0.45
7	30	A27	5	258.77	5.62	2.20	
8	32	A30	3	252.36	1.16	0.50	1.91
9	33	A37	2	252.20	0.14	0.10	
10	35A	A47	3	252.53	0.38	0.10	1.20
11	35B	A30	2	251.10	0.68	0.30	1.20
12	36	A37	3	252.95	0.06	0.00	0.15
13	38	A2	1	243.34			
14	39	A27	2	242.45	0.10	0.00	0.30
15	40	A37	6	251.90	1.10	0.40	0.06

Table 5: ¹⁸O in IAEA-304B (Water)

Recommended value: 502.5 per mille (vs VSMOW)

95% Confidence interval: 498.9 to 506.1 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	10	A37	2	512.33	0.47	0.10	0.09
2	14	A37	5	502.53	0.98	0.20	0.10
3	15	A37	3	496.05	0.39	0.10	0.30
4	18	A27	2	510.60	0.14	0.00	0.10
5	21	A27	3	494.70	6.09	1.20	0.60
6	24	A27	2	502.13	0.43	0.10	0.45
7	30	A27	5	504.48	6.41	1.30	
8	32	A30	3	505.72	0.31	0.10	1.91
9	33	A37	2	502.40	0.28	0.10	
10	35A	A47	3	503.03	0.15	0.00	1.20
11	35B	A30	2	502.18	1.58	0.30	1.20
12	36	A37	2	505.35	0.40	0.10	0.15
13	38	A2	1	488.14			
14	39	A27	2	381.39*	0.87	0.20	0.30
15	40	A37	6	505.68	0.46	0.10	0.06

Table 6: Statistical summary of results for ^{18}O samples

Isotope determined:		^{18}O	^{18}O
Material:		IAEA-304A	IAEA-304B
Unit:		per mille	per mille
Number of reported results	Laboratory averages:	15	15
	Individual determinations:	46	43
Number of accepted results	Laboratory averages:	15	14
	Individual determinations:	46	41
Total range of Laboratory averages:		242.45 - 258.77	381.39 - 512.33
Range of accepted Laboratory averages:		242.45 - 258.77	488.14 - 512.33
Percentage of outlying Laboratories:		0	7
Overall mean of accepted Laboratory averages:		251.66	502.52
Standard deviation (S.D.)	Abs:	4.51	6.23
	Rel %:	1.8	1.2
Standard error (S.E.)	Abs:	1.16	1.66
	Rel %:	0.5	0.3
Confidence limits for the mean of population for probability level .95		249.16 - 254.16	498.93 - 506.12

Figure 3: ^{18}O in IAEA-304A (Water)

Recommended value: 251.7 per mille (vs VSMOW)
 95% Confidence interval: 249.2 to 254.2 per mille

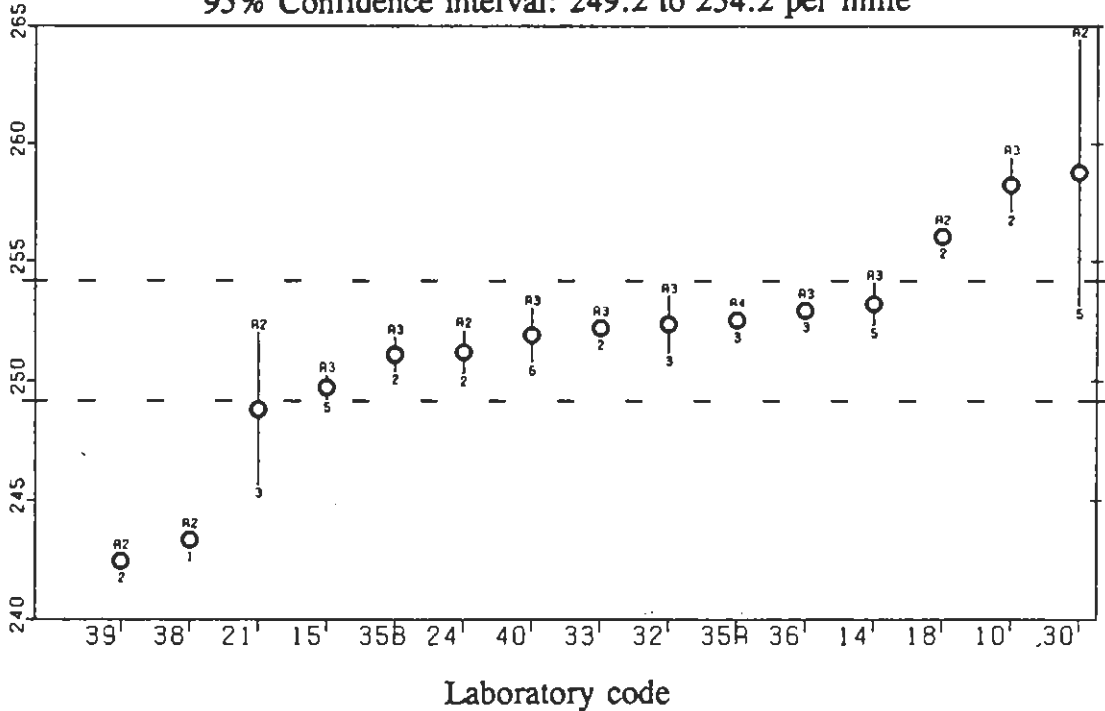


Figure 4: ^{18}O in IAEA-304B (Water)

Recommended value: 502.5 per mille (vs VSMOW)
 95% Confidence interval: 498.9 to 506.1 per mille

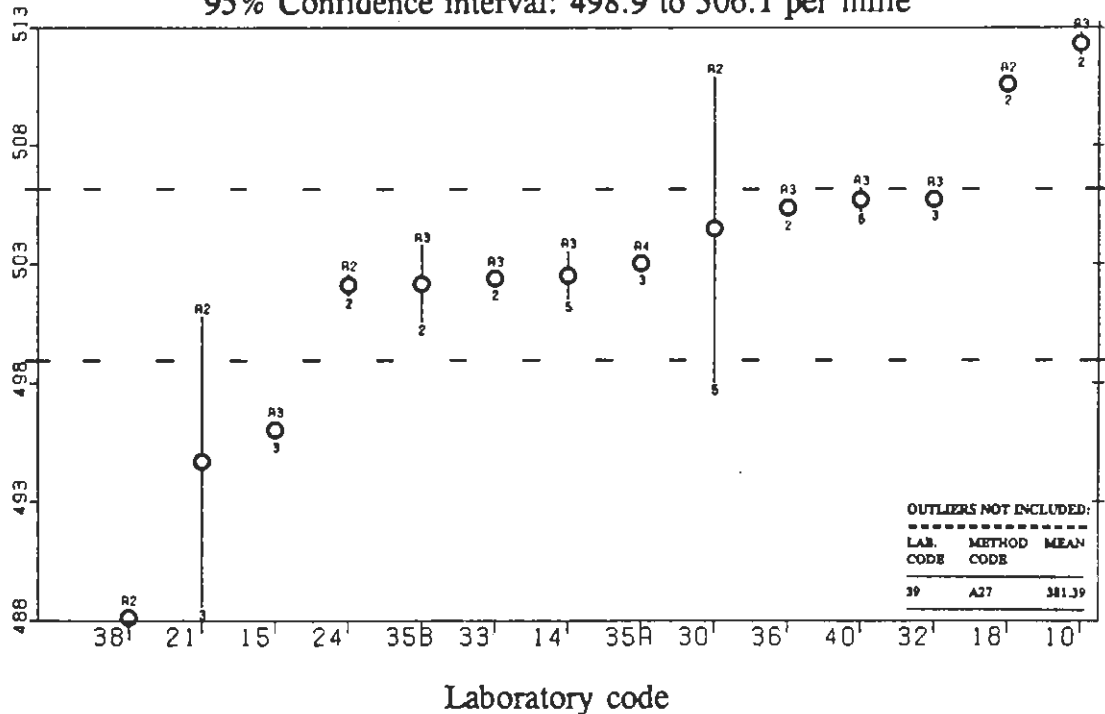


Table 7: ^{13}C in IAEA-303A (Sodium bicarbonate)

Recommended value: 93.3 per mille (vs VPDB)
95% Confidence interval: 91.1 to 95.4 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	3	A40	6	90.26	1.08	1.20	1.10
2	7	A4	6	96.79	0.33	0.30	0.33
3	11	A20	6	96.96	0.34	0.30	0.03
4	12	A20	6	92.41	1.21	1.30	0.02
5	15	A20	3	93.32	0.20	0.20	0.35
6	16	A30	5	94.87	0.63	0.70	0.15
7	17	A42	6	88.11	0.48	0.50	0.06
8	24	A20	6	93.06	1.17	1.30	0.60
9	35	A42	3	107.50*	1.17	1.30	1.65
10	38	A2	1	109.38*			
11	40	A30	2	93.47	0.22	0.20	0.03

Table 8: ^{13}C in IAEA-303B (Sodium bicarbonate)

Recommended value: 466 per mille (vs VPDB)
95% Confidence interval: 460 to 472 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	3	A40	6	459.58	3.55	0.80	1.10
2	7	A4	6	477.17	1.17	0.20	0.33
3	11	A20	6	467.24	9.85	2.10	0.03
4	12	A20	6	466.60	5.11	1.10	0.02
5	15	A20	5	469.45	0.44	0.10	0.35
6	17	A42	4	439.32*	0.29	0.10	0.06
7	24	A20	3	463.03	6.18	1.30	-0.60
8	35	A42	3	453.58	0.97	0.20	1.65
9	38	A2	1	503.68*			
10	40	A30	2	470.29	2.74	0.60	0.03

Table 9: ¹³C in IAEA-309A (UL-glucose)

Recommended value: 93.9 per mille (vs VPDB)
95% Confidence interval: 92.9 to 94.9 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	3	A40	6	95.11	0.47	0.50	1.10
2	7	A4	6	94.97	0.13	0.10	0.16
3	12	A23	6	94.28	0.24	0.30	0.02
4	15	A22	4	82.22*	1.28	1.60	
5	16	A30	6	92.06	0.45	0.50	0.60
6	17	A42	6	94.75	0.11	0.10	0.60
7	24	A20	6	92.04	0.53	0.60	0.60
8	26	A80	1	38.70*			
9	33	A33	2	92.95	0.21	0.20	
10	35	A42	6	133.60*	2.67	2.00	1.65
11	38	A2	1	79.43*			
12	40A	A33	5	93.55	0.50	0.50	0.05
13	40B	A33	3	95.40	0.38	0.40	0.04

Table 10: ¹³C in IAEA-309B (UL-glucose)

Recommended value: 535.3 per mille (vs VPDB)
95% Confidence interval: 530.6 to 540.1 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	3	A40	6	544.85	0.55	0.10	1.10
2	7	A4	6	540.55	0.18	0.00	0.16
3	12	A23	4	536.69	3.50	0.70	0.02
4	15	A22	3	529.67	1.31	0.20	
5	17	A42	6	539.19	0.90	0.20	0.60
6	24	A20	6	524.42	1.05	0.20	0.60
7	26	A80	1	81.90*			
8	33	A33	2	530.15	0.78	0.10	
9	35	A42	6	528.81	6.48	1.20	1.65
10	38	A2	1	471.04*			
11	40A	A33	5	537.02	3.45	0.60	0.05
12	40B	A33	4	541.83	1.06	0.20	0.04

Table 11: Statistical summary of results for ¹³C samples

Isotope determined:		¹³ C	¹³ C	¹³ C	¹³ C
Material:		IAEA-303A	IAEA-303B	IAEA-309A	IAEA-309B
Unit:		per mille	per mille	per mille	per mille
Number of reported results	Laboratory averages:	11	10	13	12
	Individual determinations:	50	42	58	50
Number of accepted results	Laboratory averages:	9	8	9	10
	Individual determinations:	46	37	46	48
Total range of Laboratory averages:		88.11 - 109.38	439.32 - 503.68	38.70 - 133.60	81.90 - 544.85
Range of accepted Laboratory averages:		88.11 - 96.96	453.58 - 477.17	92.04 - 95.40	524.42 - 544.85
Percentage of outlying Laboratories:		18	20	31	17
Overall mean of accepted Laboratory averages:		93.25	465.87	93.90	535.32
Standard deviation (S.D.)	Abs:	2.85	7.18	1.3	6.67
	Rel %:	3.1	1.5	1.4	1.2
Standard error (S.E.)	Abs:	0.95	2.54	0.43	2.11
	Rel %:	1	0.5	0.5	0.4
Confidence limits for the mean of population for probability level .95		91.06 - 95.44	459.86 - 471.88	92.90 - 94.90	530.55 - 540.09

Figure 5: ^{13}C in IAEA-303A (Sodium bicarbonate)

Recommended value: 93.3 per mille (vs VPDB)
 95% Confidence interval: 91.1 to 95.4 per mille

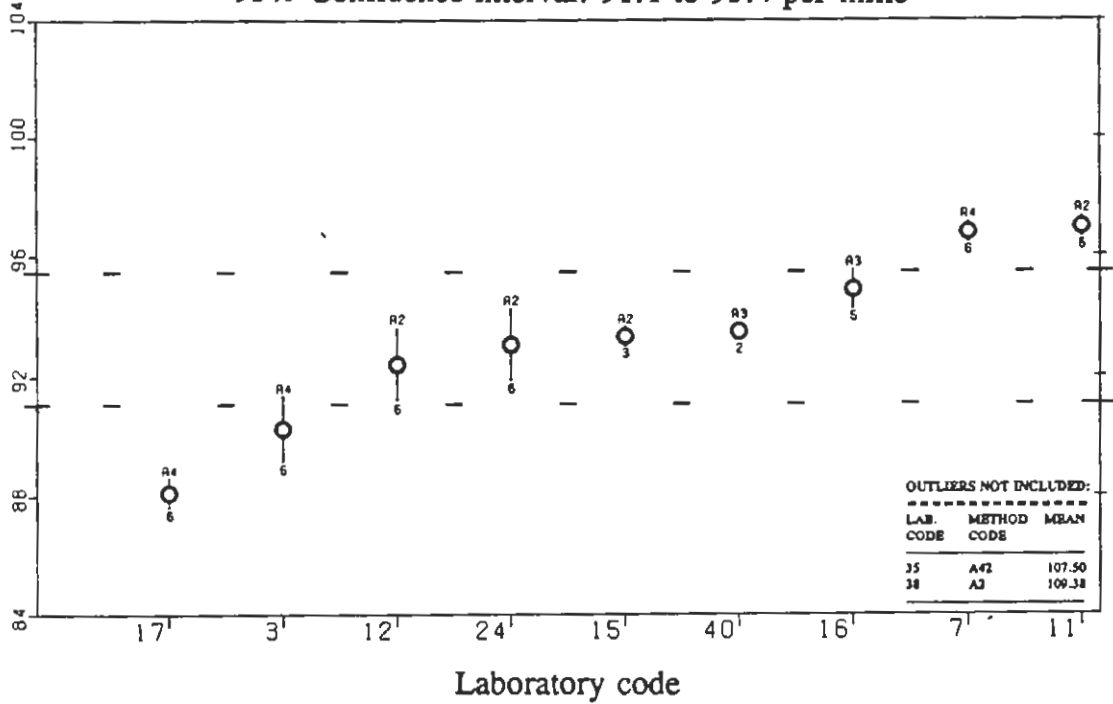


Figure 6: ^{13}C in IAEA-303B (Sodium bicarbonate)

Recommended value: 466 per mille (vs VPDB)
 95% Confidence interval: 460 to 472 per mille

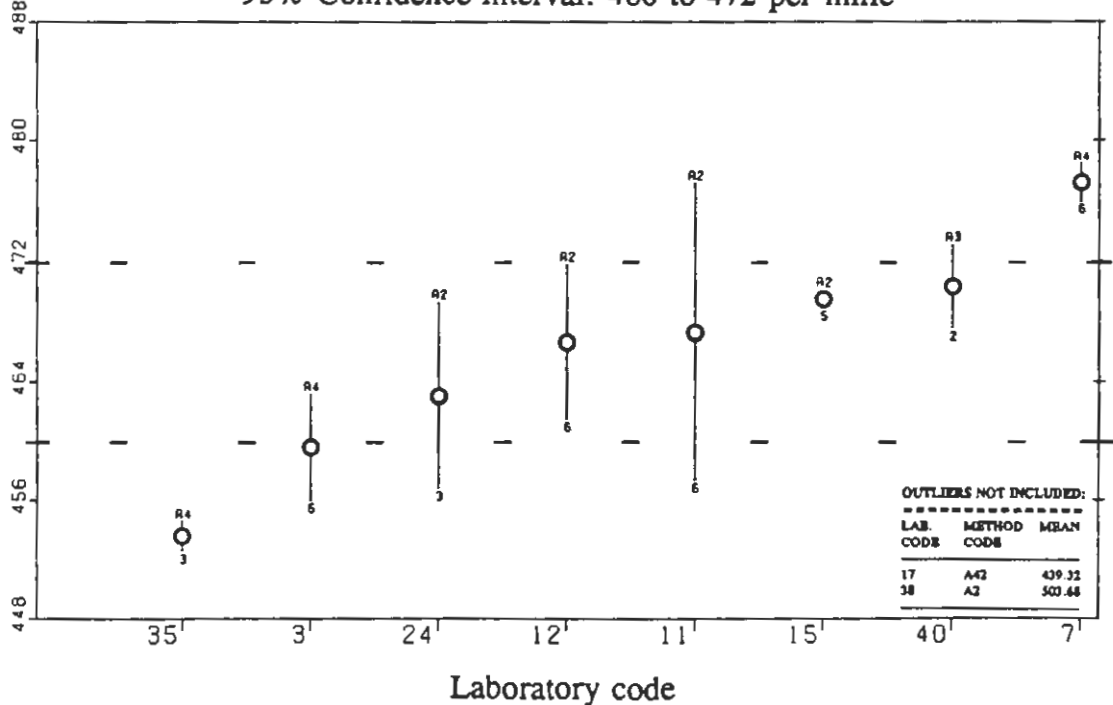


Figure 7: ^{13}C in IAEA-309A (UL-glucose)

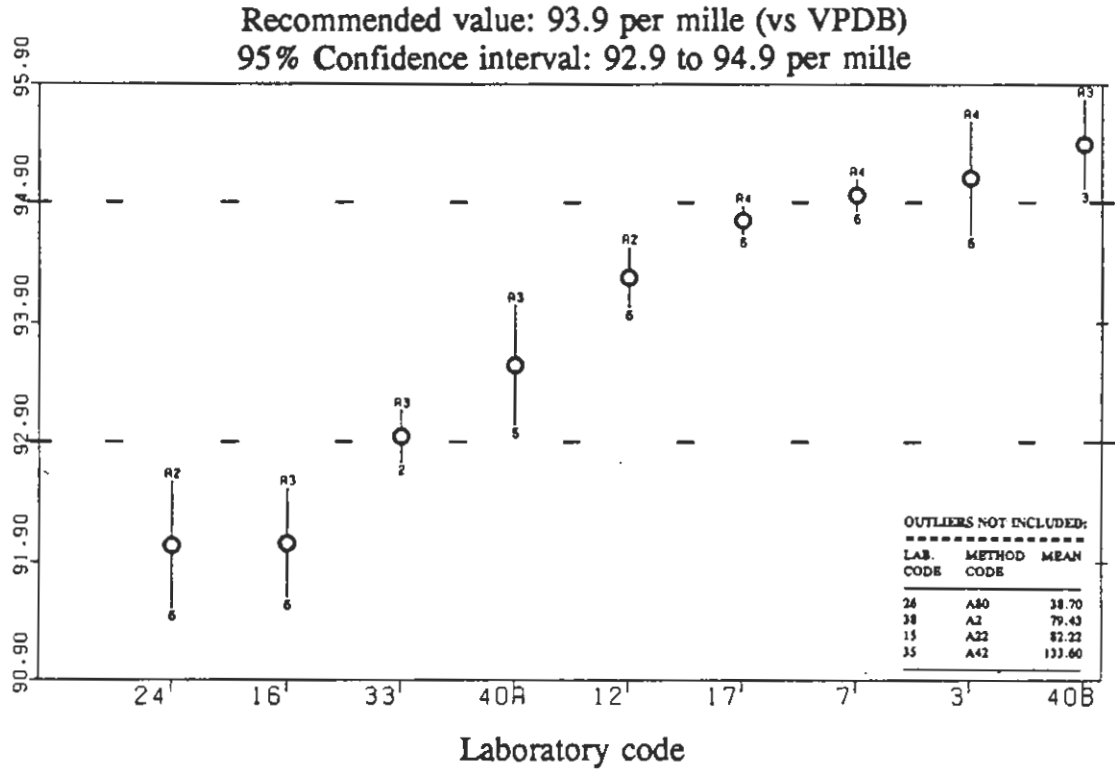


Figure 8: ^{13}C in IAEA-309B (UL-glucose)

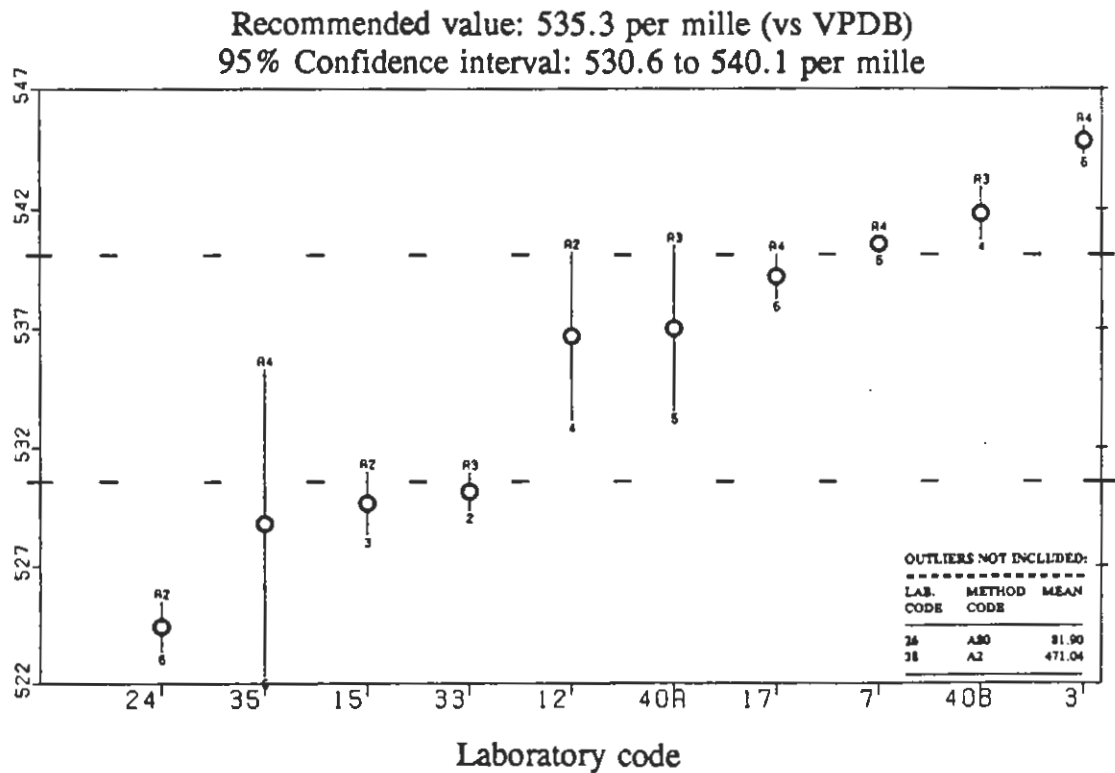


Table 12: ¹⁵N in IAEA-305A (ammonium sulphate)

Recommended value: 39.8 per mille (vs air)
95% Confidence interval: 39.3 to 40.3 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	1	A42	6	46.82*	3.70	7.90	0.0015
2	2	A42	6	39.05	0.41	1.10	0.5200
3	4	A42	6	41.24	0.23	0.60	0.0010
4	5A	A21	5	38.26	0.25	0.60	0.0020
5	5B	A21	5	39.52	0.41	1.00	0.0020
6	6	A31	5	39.90	0.57	1.40	0.0006
7	7	A4	6	40.23	0.19	0.50	0.3000
8	8A	A23	3	40.83	0.00	0.00	0.0001
9	8B	A42	3	43.94*	1.58	3.60	0.0020
10	9	A42	6	39.55	0.09	0.20	0.3300
11	11	A31	6	39.39	0.45	1.20	0.0001
12	14	A31	4	39.84	0.09	0.20	0.2000
13	15	A21	5	39.41	0.12	0.30	0.3000
14	16A	A4	5	41.16	0.39	1.00	0.6000
15	16B	A31	5	39.36	1.34	3.40	0.1500
16	17	A42	5	37.84	0.42	1.10	1.0000
17	19	A11	4	40.90	3.79	9.30	0.0030
18	23	A11	6	38.00	2.06	5.40	0.0010
19	24	A21	3	41.04	1.14	2.80	0.6000
20	25	A33	6	38.41	0.36	0.90	0.0003
21	27	A34	3	41.03	0.90	2.20	0.3000
22	28	A73	5	39.92	2.90	7.30	0.3000
23	29A	A23	6	40.72	0.02	0.00	
24	29B	A23	6	24.30*	13.36	55.00	0.0200
25	29C	B20	5	30.97*	7.15	23.10	0.0200
26	29D	B21	6	24.30*	13.58	55.90	0.0200
27	31	B22	5	38.58	1.58	4.10	0.0003
28	34	B1	6	47.14*	24.25	51.50	0.0061
29	35	A42	6	41.06	2.39	5.80	0.0008

Table 13: ¹⁵N in IAEA-305B (ammonium sulphate)

Recommended value: 375.3 per mille (vs air)
95% Confidence interval: 373.0 to 377.6 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard	Deviation	Lab. LoD
					Abs %	Rel %	
1	1	A42	5	381.73	3.47	0.90	0.0015
2	2	A42	6	375.30	0.37	0.10	0.5200
3	4	A42	6	377.41	0.34	0.10	0.0010
4	5A	A21	5	373.70	0.67	0.20	0.0020
5	5B	A21	5	370.90	6.12	1.60	0.0020
6	6	A31	5	388.93	2.22	0.60	0.0006
7	7	A4	6	379.48	0.11	0.00	0.3000
8	8A	A23	3	377.92	0.16	0.00	0.0001
9	8B	A42	3	377.83	0.00	0.00	0.0020
10	9	A42	6	368.43	1.05	0.30	0.3300
11	11	A31	6	372.83	0.74	0.20	0.0001
12	14	A31	4	374.48	0.19	0.10	0.2000
13	15	A21	5	376.53	0.28	0.10	0.3000
14	17	A42	5	374.70	0.45	0.10	1.0000
15	19	A11	5	378.43	5.91	1.60	0.0030
16	23	A11	6	369.58	0.00	0.00	0.0010
17	24	A21	3	373.37	1.36	0.40	0.6000
18	25	A33	6	362.35	0.73	0.20	0.0003
19	27	A34	3	380.00	1.35	0.40	0.3000
20	28	A73	5	374.80	0.84	0.20	0.5000
21	29A	A23	6	378.04	0.49	0.10	
22	29B	B20	6	340.28*	20.75	6.10	0.0200
23	29C	B21	5	383.32	27.68	7.20	0.0200
24	29D	B24	6	337.99*	12.38	3.70	0.0200
25	31	A31	6	373.98	4.24	1.10	0.0003
26	34	B1	6	365.46	29.53	8.10	0.0061
27	35	A42	6	372.92	2.04	0.50	0.0008

Table 14: ¹⁵N in IAEA-310A (urea)

Recommended value: 47.2 per mille (vs air)
95% Confidence interval: 46.0 to 48.5 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	1	A42	6	53.58	3.97	7.40	0.0015
2	2	A42	6	47.68	0.17	0.40	0.9600
3	3	A40	6	46.86	1.03	2.20	0.0010
4	4	A42	6	49.10	0.11	0.20	0.0005
5	5A	A24	5	46.81	0.36	0.80	0.0020
6	5B	A24	5	47.74	0.74	1.50	0.0020
7	7	A4	6	48.70	0.15	0.30	0.3500
8	8A	A23	3	48.96	0.16	0.30	0.0001
9	8B	A42	3	45.77	0.00	0.00	0.0020
10	9	A42	6	47.71	0.17	0.40	0.3300
11	11	A34	6	66.77*	0.40	0.60	0.0001
12	15	A24	3	48.04	0.30	0.60	0.3000
13	16A	A4	5	49.68	0.54	1.10	0.6000
14	16B	A34	5	49.30	0.29	0.60	0.1500
15	17	A42	5	43.34	0.23	0.50	1.0000
16	19	A11	6	39.92	6.85	17.20	0.0030
17	23	A11	6	55.82*	2.83	5.10	0.0010
18	24	A24	3	48.02	1.14	2.40	0.6000
19	25	A33	6	46.09	0.36	0.80	0.0002
20	27	A34	3	48.63	0.67	1.40	0.3000
21	28	A73	5	43.44	1.47	3.40	0.5000
22	29A	A23	5	48.94	0.11	0.20	
23	29B	B21	5	44.67	9.61	21.50	0.0200
24	29C	B24	6	48.96	13.70	28.00	0.0200
25	34	B1	5	41.38	24.25	58.60	0.0061
26	35	A42	6	50.43	0.77	1.50	0.0008
27	37	B15	5	185.63*	56.90	30.60	

Table 15: ¹⁵N in IAEA-310B (urea)

Recommended value: 244.6 per mille (vs air)
95% Confidence interval: 243.9 to 245.4 per mille

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	1	A42	6	243.03	3.88	1.60	0.0015
2	2	A42	6	242.75	0.53	0.20	0.9600
3	3	A40	6	247.42	0.66	0.30	0.0010
4	4	A42	6	244.63	0.17	0.10	0.0005
5	5A	A24	5	244.08	0.67	0.30	0.0020
6	5B	A24	5	245.56	0.66	0.30	0.0020
7	7	A4	6	247.45	0.10	0.00	0.3500
8	8A	A23	3	246.28	0.00	0.00	0.0001
9	8B	A42	3	243.26	0.00	0.00	0.0020
10	9	A42	6	240.84	0.86	0.40	0.3300
11	11	A34	6	243.93	0.26	0.10	0.0001
12	15	A24	3	244.83	0.22	0.10	0.3000
13	16A	A4	5	245.08	0.55	0.20	0.6000
14	16B	A34	5	243.76	0.87	0.40	0.1500
15	17	A42	5	243.22	0.37	0.20	1.0000
16	19	A11	6	232.87*	10.09	4.30	0.0030
17	23	A14	6	246.00	0.00	0.00	0.0010
18	24	A24	3	242.53	0.64	0.30	0.6000
19	25	A33	6	235.07*	0.94	0.40	0.0003
20	27	A34	4	242.45	1.88	0.80	0.3000
21	28	A73	5	245.60	1.95	0.80	0.5000
22	29A	A23	3	246.31	0.59	0.20	
23	29B	B21	5	244.90	16.76	6.80	0.0200
24	29C	B24	6	228.62*	18.34	8.00	0.0200
25	34	B1	6	221.31*	44.69	20.20	0.0061
26	35	A42	6	246.46	3.04	1.20	0.0008
27	37	B15	5	246.03 ⁺	100.11	40.70	

* not included in plot because of large standard deviation

Table 16: ¹⁵N in IAEA-311 (ammonium sulphate)

Recommended value: 2.05 atom %
95% Confidence interval: 2.03 to 2.06 atom %

No.	Lab. Code No.	Method Code	No. of Results	Mean	Standard Deviation		Lab. LoD
					Abs %	Rel %	
1	1	A42	6	2.06	0.01	0.40	0.0015
2	2	A42	6	2.05	0.00	0.10	0.0002
3	3	A40	6	2.09	0.00	0.10	0.0010
4	4	A42	6	2.06	0.00	0.00	0.0020
5	5A	A21	5	2.05	0.00	0.10	0.0020
6	5B	A21	5	2.06	0.00	0.00	0.0020
7	6	A31	4	2.12	0.01	0.40	0.0240
8	7	A4	6	2.07	0.00	0.00	0.3500
9	8A	A23	3	2.06	0.00	0.00	0.0001
10	8B	A42	3	2.06	0.00	0.00	0.0020
11	11	B21	6	2.04	0.01	0.50	0.0360
12	13	B10	5	1.64*	0.01	0.60	0.1381
13	14	A31	4	2.04	0.00	0.20	0.0040
14	15	A21	6	2.08	0.00	0.10	0.1000
15	17	A42	5	2.06	0.00	0.00	0.0004
16	19	A11	6	2.05	0.02	1.20	0.0030
17	20	B14	2	2.01	0.02	0.90	0.0020
18	22A	B11	1	2.08			0.0300
19	22B	B14	6	2.08	0.01	0.50	0.0120
20	23	A11	6	2.07	0.01	0.60	0.0010
21	24	A21	3	1.99	0.01	0.40	0.0003
22	25	A33	6	1.99	0.00	0.10	0.0001
23	28	A73	5	2.09	0.01	0.70	0.0003
24	29A	B20	6	2.00	0.01	0.70	0.0200
25	29B	B21	5	2.07	0.01	0.30	0.0200
26	29C	B24	6	1.96	0.01	0.40	0.0200
27	31	A31	5	2.02	0.01	0.50	0.0003
28	34	B1	6	2.00	0.01	0.70	0.0003
29	35	A42	6	2.05	0.01	0.70	0.0008

Table 17: Statistical summary of results for ¹⁵N samples

Isotope determined:		¹⁵ N	¹⁵ N	¹⁵ N	¹⁵ N	¹⁵ N
Material:		IAEA-305A	IAEA-305B	IAEA-310A	IAEA-310B	IAEA-311
Unit:		per mille	per mille	per mille	per mille	atom %
Number of reported results	Laboratory averages:	29	27	27	27	29
	Individual determinations:	148	139	137	137	145
Number of accepted results	Laboratory averages:	23	25	24	23	28
	Individual determinations:	116	127	120	113	140
Total range of Laboratory averages:		24.30 - 47.14	337.99 - 388.93	39.92 - 185.63	221.31 - 247.45	1.64 - 2.12
Range of accepted Laboratory averages:		37.84 - 41.24	362.35 - 388.93	39.92 - 53.58	240.84 - 247.45	1.96 - 2.12
Percentage of outlying Laboratories:		21	7	11	15	3
Overall mean of accepted Laboratory averages:		39.79	375.3	47.24	244.63	2.05
Standard deviation (S.D.)	Abs:	1.09	5.60	3.00	1.73	0.04
	Rel %:	2.7	1.5	6.3	0.7	1.8
Standard error (S.E.)	Abs:	0.23	1.12	0.61	0.36	0.01
	Rel %:	0.6	0.3	1.3	0.1	0.3
Confidence limits for the mean of population for probability level .95		39.32 - 40.26	372.99 - 377.61	45.97 - 48.51	243.88 - 245.37	2.03 - 2.06

Figure 9: ^{15}N in IAEA-305A (ammonium sulphate)

Recommended value: 39.8 per mille (vs air)
 95% Confidence interval: 39.3 to 40.3 per mille

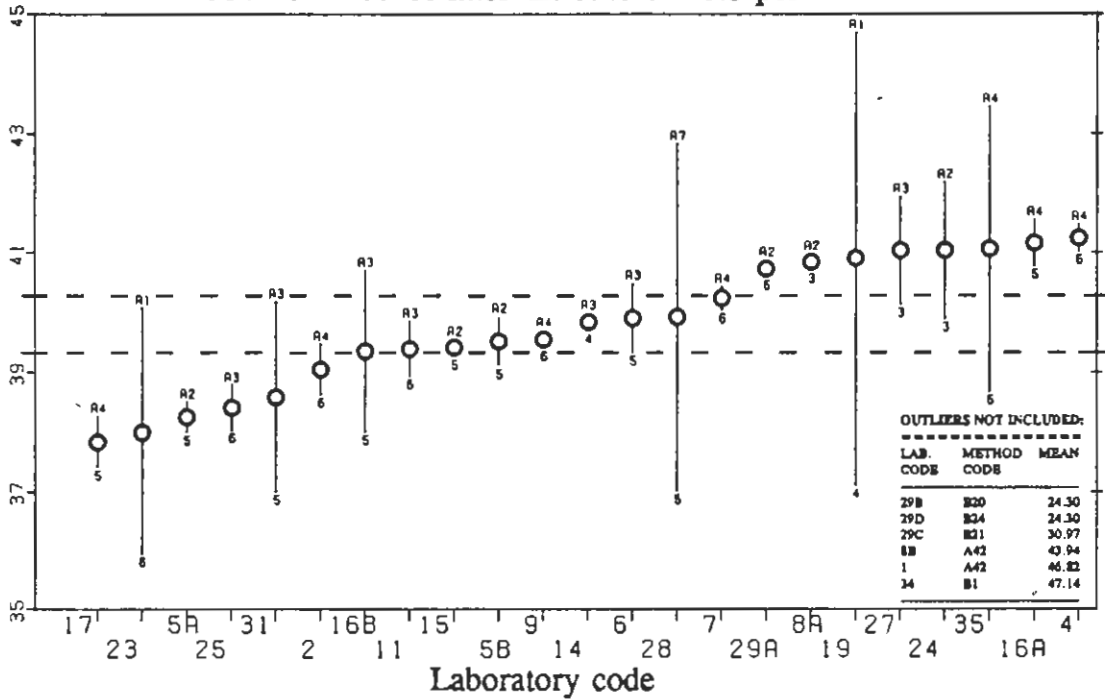


Figure 10: ^{15}N in IAEA-305B (ammonium sulphate)

Recommended value: 375.3 per mille (vs air)
 95% Confidence interval: 373.0 to 377.6 per mille

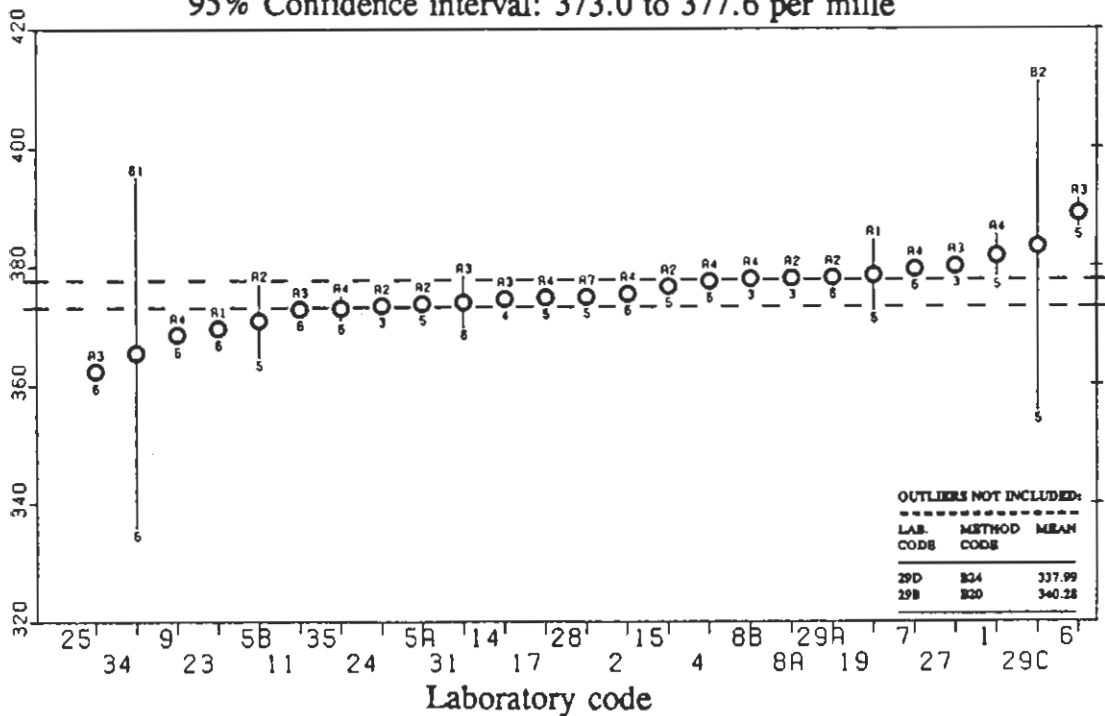


Figure 11: ¹⁵N in IAEA-310A (urea)

Recommended value: 47.2 per mille (vs air)
 95% Confidence interval: 46.0 to 48.5 per mille

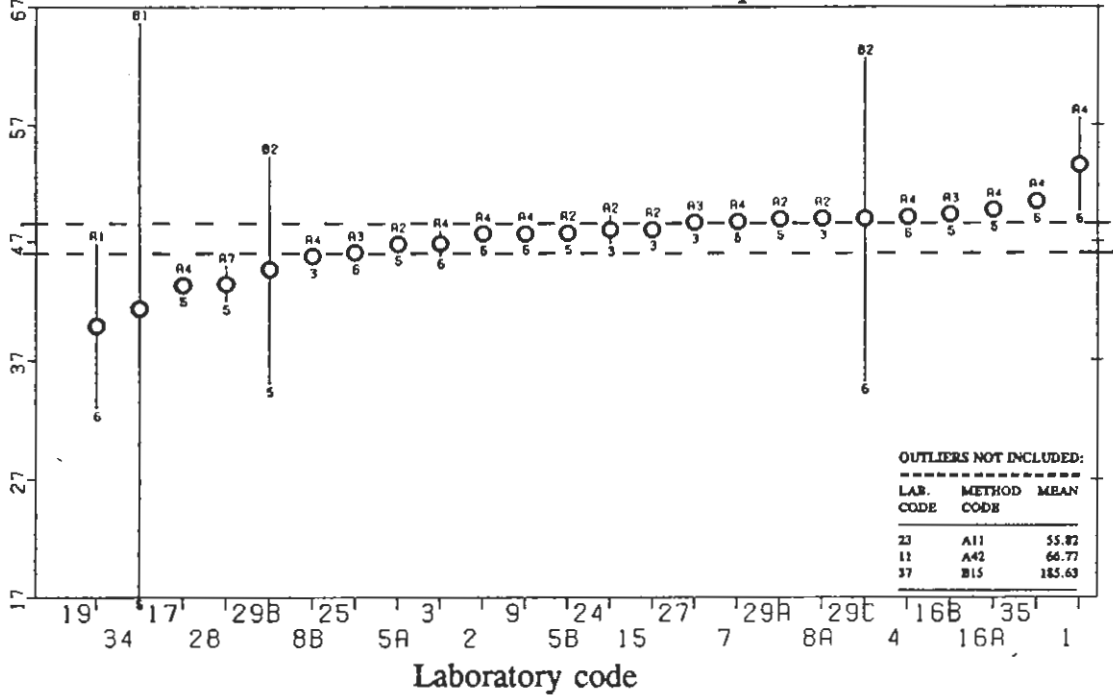


Figure 12: ¹⁵N in IAEA-310B (urea)

Recommended value: 244.6 per mille (vs air)
 95% Confidence interval: 243.9 to 245.4 per mille

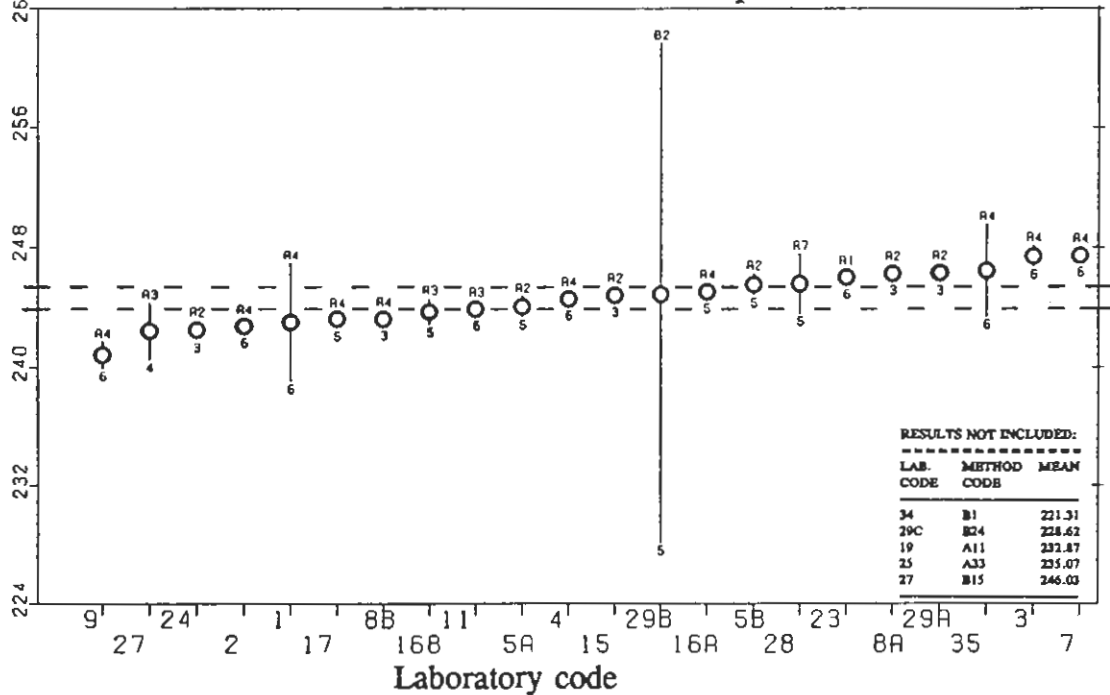


Figure 13: ^{15}N in IAEA-311 (ammonium sulphate)

Recommended value: 2.05 atom %
 95% Confidence interval: 2.03 to 2.06 atom %

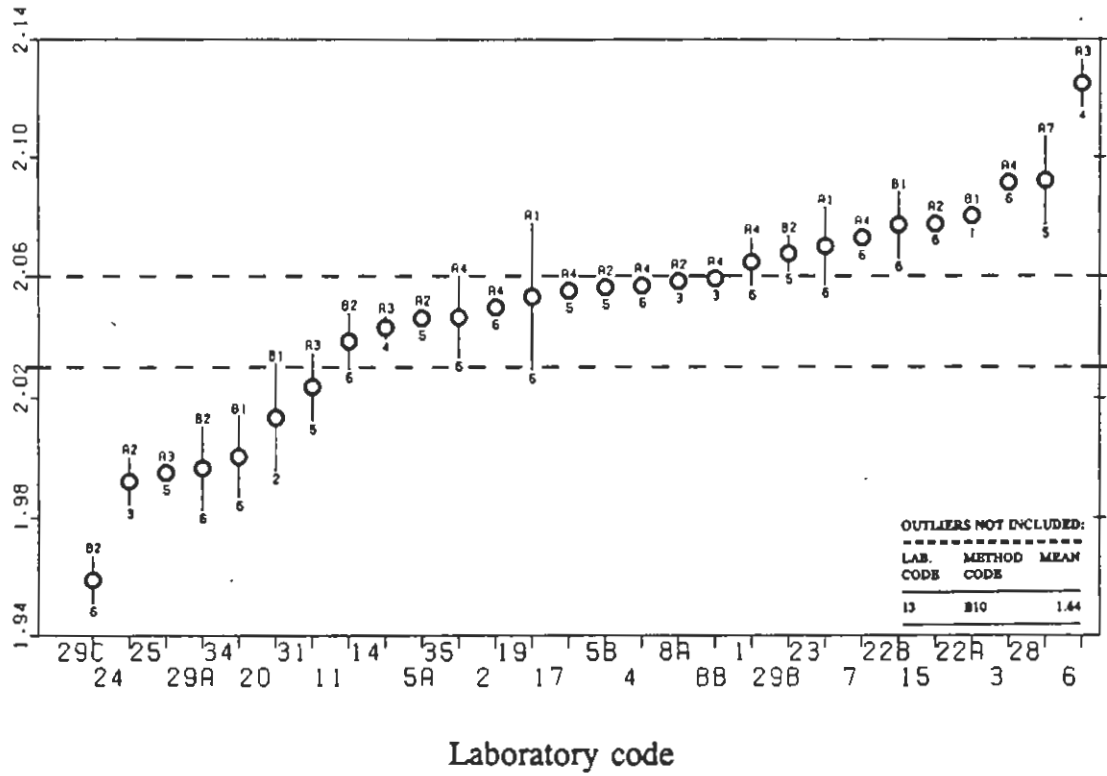


Table 18: Overview of all recommended values

Sample	Isotope	Enrichment* per mille	95% Confidence Interval	vs
302A	² H	508.4	505.5 - 511.3	VSMOW
302B	² H	996	987 - 1004	VSMOW
303A	¹³ C	93.3	91.1 - 95.4	VPDB
303B	¹³ C	466	460 - 472	VPDB
304A	¹⁸ O	251.7	249.2 - 254.2	VSMOW
304B	¹⁸ O	502.5	498.9 - 506.1	VSMOW
305A	¹⁵ N	39.8	39.3 - 40.3	air
305B	¹⁵ N	375.3	373.0 - 377.6	air
309A	¹³ C	93.9	92.9 - 94.9	VPDB
309B	¹³ C	535.3	530.6 - 540.1	VPDB
310A	¹⁵ N	47.2	46.0 - 48.5	air
310B	¹⁵ N	244.6	243.9 - 245.4	air
311	¹⁵ N	2.05	2.03 - 2.06	

* All values as per mille, except for IAEA-311, which is as atom percent

METHOD CODES

- △ → GROUP OF ANALYTICAL INSTRUMENTS
- △ → INDIVIDUAL SYSTEM FOR ISOTOPIC ANALYSIS
- △ → SAMPLE PREPARATION METHOD IDENTIFICATION *
- △△△

A mass spectrometry

- A 1 conventional routine mass spectrometry (MS)
- A 2 conventional high precision MS (dual inlet, dual (triple) collector)
- A 3 modern isotope ratio MS (IRMS)
- A 4 on line EA/MS (EA: elemental analyser)
- A 5 quadrupole MS
- A 6 chemical ionization/MS
- A 7 IRM-GCMS (gas chromatographic effluent is catalytically combusted to N₂, CO₂ or H₂O prior to entering a modified IRMS)
- A 8 SIM-GCMS (isotope enrichment is determined from the ion produced by the entire derivatized molecule or from a suitable fragment ion)

B emission spectrometry

- B 1 optical emission spectrometry (ES), conventional procedure by NOI-5 type instruments
- B 2 automated ES
- B 3 on line EA/ES (EA: elemental analyser)

C other or unspecified methods

- C 1 NMR
- C 2 ESR
- C 3 infrared absorption spectroscopy

D special micromethods

* CODES FOR SAMPLE PREPARATION METHODS

(includes codes for ²H, ¹³C, ¹⁵N and ¹⁸O)

- 1 RITTENBERG preparation (hypobromite)
- 2 DUMAS preparation I (combustion, cyclic method)
- 3 DUMAS preparation II (combustion, ampoule method)
- 4 KJELDAHL digestion plus RITTENBERG
- 5 KJELDAHL digestion plus DUMAS I
- 6 KJELDAHL digestion plus DUMAS II
- 7 CO₂ - water exchange
- 8 reduction of water to H₂ over uranium metal
- 9 reduction of water to H₂ over chromium
- 0 other

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