Draft Report on

Fourth interlaboratory comparison exercise for δ^2H and $\delta^{18}O$ analysis of water samples (WICO2011)

Prepared by

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CONTENTS

TABLE OF FIGURES

1. INTRODUCTION

The IAEA Isotope Hydrology Laboratory organized the fourth interlaboratory comparison exercise for laboratories engaged in routine analysis of hydrogen and oxygen stable isotope composition of water samples in 2011. Three similar exercises were carried out in 1995 [1], in 1999 [2] and in 2002 [3]. However, the tradition of IAEA water stable isotope inter-laboratory comparison is much older. Two interlaboratory comparison trials for isotope hydrology laboratories were carried out in the sixties and seventies, which revealed problems with use of the NBS-1 international standard; these data were used to calibrate the newly produced primary reference materials VSMOW and SLAP.

The WICO2011 exercise was announced in February 2011 on the internet, via the ISOGEOCHEM news group of Isogeochemistry [4] and by email to all participants of the former intercomparisons.

Altogether 174 laboratories showed interest to participate in the exercise. Four water samples prepared and calibrated at the IAEA Isotope Hydrology Laboratory were labelled IAEA-OH-13 to IAEA-OH-16, which are referred to in this report as OH-13 to OH-16. By the end of the reporting deadline (the end of August 2011) altogether 137 laboratories from 53 countries had submitted 172 datasets back to the IAEA on the oxygen and hydrogen isotopic composition of these water samples.

The four water samples cover the range of $\delta^{18}O$ and $\delta^{2}H$ values typical for the majority of natural waters. The samples were bottled from 30 L stainless steel storage barrels into 30 mL securely-capped brown glass bottles, serially numbered at the time of filling. Each laboratory received a set of four samples with a corresponding code. This code (assigned randomly) forms the Identification (ID) code used throughout the exercise and in the tables and graphs of this report for each laboratory. The ID code is not related to the order of the list of participating laboratories. The identity of participating laboratories will not be revealed unless each laboratory explicitly agrees to do so.

2. SAMPLE PREPARATION AND REFERENCE VALUES

Waters collected from different areas were used to prepare four water samples for this intercomparison exercise. A brief description of these waters is provided below.

OH-13: Lake water from the shoreline of the lake Neusiedl, a shallow endorheic basin near Vienna, Austria. Fifty litres were filtered with a 5 µm filter and subsequently distilled using a Barnstead MP6A laboratory distiller.

OH-14: Commercially bottled water from the Moree wells in the great artesian basin in Australia. The water was not filtered or distilled.

OH-15: Commercially bottled water from the Libyan Al-Kufra region in the Nubian Sandstone Aquifer. The water was not filtered or distilled.

OH-16: Melted alpine snow sampled in the Salzburg region of the Austrian Alps at 2000 m.a.s.l. The water was not filtered or distilled.

The physical properties of these waters can be seen in Table 1.

TABLE 1. Physical properties of water samples used for intercomparison exercise

Name	pH	Conduct. µS/cm	Visual	Odour
OH-13	4.5	2	clear	no
OH-14	7.0	900	clear	no
OH-15	5.5	450	clear	no
OH-16	4.5	24	clear	no

After preparation, all four samples were stored in airtight 30 L stainless steel drums under slight argon overpressure. The containers are equipped with a special water extraction system allowing samples to be drawn without exposition to atmospheric air, thus avoiding any risk of evaporation/contamination. A detailed description of the system is available at http://www-naweb.iaea.org/napc/ih/index.html.

The water was distributed in 30 mL brown glass bottles, using an adapted Brand® bottle-top dispenser and a concentric double-pipe nozzle, flushed with argon gas. Bottles were capped securely and labelled with a sample name and filling sequence number.

The four water samples were analyzed for $\delta^{18}O$ and $\delta^{2}H$ at the Isotope Hydrology Laboratory by a dual inlet isotope ratio mass spectrometer (Finnigan Delta plus) equipped with a 48-port equilibration unit (for $\delta^{18}O$ and $\delta^{2}H$), and four laser based liquid water isotope analyzers (LWIA), three LGR and one Picarro. The mean isotopic values determined by mass spectrometer along with associated standard uncertainties u for a single measurement at the 1σ-level (Table 2) were sent to the participating laboratories initially as reference values. The mean values and uncertainties are derived from n accepted individual measurements as stated in the columns 'number of analyses' (number of rejected values in brackets).

TABLE 2. Isotope values for the four WICO2011 water samples as determined at the IAEA Isotope Hydrology Laboratory. Standard uncertainty u for a single analysis is given at the 1σ level. The number of accepted values to derive the mean value is reported (the number of the rejected values in brackets).

Sample	$\delta^{18}O$			$\delta^{2}H$		
	mean value [‰]	standard deviation 1σ [‰]	number of values	mean value [‰]	standard deviation 1σ [‰]	number of values
OH-13	-0.96	0.04	24	-2.29	0.94	23 (1)
OH-14	-5.59	0.05	23 (1)	-37.69	0.82	20 (4)
OH-15	-9.37	0.04	23 (1)	-78.01	0.77	20 (4)
OH-16	-15.41	0.04	24	-113.81	0.94	21 (3)

Later on, it was decided not to consider our results as reference values. Instead, the reference values were calculated from the results of the 12 laboratories whose performance was the best in the last intercomparison exercise, WICO2002, having cumulative deviations for $\delta^{18}O$ and $\delta^{2}H$ less than 0.2 ‰ and 2.0 ‰, respectively. For this purpose, their results in the 2011 exercise were processed according to the statistical methods provided in the subsequent section and the reference values are shown in Table 3.

TABLE 3. Isotope reference values for the four WICO2011 water samples as determined by the twelve laboratories which had best performance in the WICO2002 exercise with cumulative deviations for $\delta^{18}O$ and $\delta^{2}H$ of less than 0.2 ‰ and 2.0 ‰, respectively.

Sample	$\delta^{18}O$			$\delta^{2}H$		
	mean value [‰]	standard deviation 1σ [‰]	number of values	mean value [‰]	standard deviation 1σ [‰]	number of values
OH-13	-0.96	0.04	11	-2.84	0.60	12
OH-14	-5.60	0.05	12	-38.30	0.36	12
OH-15	-9.41	0.04	12	-78.26	0.38	11
OH-16	-15.43	0.04	12	-114.62	0.43	12

3. EVALUATION AND PRESENTATION OF THE REPORTED DATA

All the individual results of $\delta^{18}O$ and $\delta^{2}H$ submitted to the IAEA by the participating laboratories are listed in APPENDIX I. The list of participating laboratories is presented in APPENDIX II. The WICO2011 announcement and the Laboratory Reporting Sheet used in the exercise are presented in APPENDIX III and APPENDIX IV, respectively.

3.1. Statistical methods

Statistical analysis of the submitted results was undertaken with the major objective to assess the performance of individual laboratories (precision and accuracy) with respect to the reference $\delta^{18}O$ and $\delta^{2}H$ values for the analyzed four samples.

To reach this goal, a two-stage statistical treatment, adopted in previous IAEA interlaboratory exercises of a similar nature [1-5], was applied to the entire population of submitted results. All data evaluation and outlier determination were performed with commercial EXCEL spreadsheet programme.

<u>The two-stage statistical treatment:</u>
In stage I of the statistical treatment, obvious outliers were discarded based on the frequency distribution of values: after determination of the upper (H_U) and lower (H_L) quartiles, as well as the interquartile range (H_U-H_L), values exceeding $H_U + 3.0\cdot(H_U-H_L)$ and $H_L -3.0\cdot(H_U-H_L)$ were discarded. The provisional mean compiled after stage I was further used for the stage II-outlier-rejection procedure.

In stage II of the evaluation process, the remaining results were assessed for each laboratory through examination of the difference between the reported isotope result *x* and the provisional mean *m* of stage I, divided by the standard uncertainty *s* quoted by the given laboratory. The results for which the ratio $|(x-m)|/s$ was larger than two were discarded, implying a significant deviation from the expected concordance of results. This procedure identified those results which were seriously overestimating their measurements' precision.

In the final step, the weighted average was calculated by weighing the individual results by the reciprocal of the quoted variance:

$$X_w = \frac{\sum_{i=1}^{n} \frac{x_i}{s_i^2}}{\sum_{i=1}^{n} \frac{1}{s_i^2}} \qquad (1)$$

The estimated standard uncertainty of the mean (ese) was calculated according to the following formula

$$ese(X_w) = \frac{\sigma_w}{\sqrt{\left(\sum_{i=1}^{n} \frac{1}{s_i^2}\right)}} \qquad (2)$$

where:

$$\sigma_w^2 = \frac{\sum_{i=1}^{n} \frac{(x_i - X_w)^2}{s_i^2}}{n-1} \qquad (3)$$

where *n* stands for the final number of results accepted after the second step of the data evaluation procedure.

3.2. Presentation of the results

Before rejecting outliers using statistical methods, average of $\delta^{18}O$ and $\delta^{2}H$ values for the samples, along with associated standard deviations at 1σ-level and the total number of data were determined, are provided in Table 4.

TABLE 4. Average isotope values as determined by all participating laboratories for the four WICO2011 water samples.

Sample	$\delta^{18}O$			$\delta^{2}H$		
	mean value [‰]	standard deviation 1σ [‰]	number of values	mean value [‰]	standard deviation 1σ [‰]	number of values
OH-13	-1.01	0.27	166	-2.85	1.59	159
OH-14	-5.62	0.26	167	-38.47	1.52	160
OH-15	-9.41	0.25	167	-78.49	1.39	160
OH-16	-15.46	0.28	167	-114.99	1.76	160

Long term laboratory uncertainties were provided by only 45 laboratories; therefore stage II evaluation was undertaken using: (a) standard deviations associated with present measurements, (b) maximum of the measurement and long term standard deviations. In Table 5 the average values for the four samples are provided as evaluated from all data submitted by participating laboratories using measurement standard deviations in the evaluation scheme discussed in the previous section. The results obtained by using the measurement of maximum and long term standard deviations are depicted in Table 6. Both the tables provide the average values and associated standard deviations at 1σ-level and the number of accepted laboratory values (the number of rejected laboratory values is in brackets). In the first case, some results with good accuracy were rejected in stage II due to overestimated precision, therefore Table 6 better represents the consensus values.

TABLE 5. Weighted average of accepted isotope values of all laboratories for the four WICO2011 water samples on the basis of reported measurement uncertainty. In the column 'number of values', the number of rejected values is stated in addition in brackets.

Sample	$\delta^{18}O$			$\delta^{2}H$		
	mean value [‰]	standard deviation 1σ [‰]	number of values (rejected)	mean value [‰]	standard deviation 1σ [‰]	number of values (rejected)
OH-13	-0.98	0.11	125(41)	-2.74	0.79	106(53)
OH-14	-5.59	0.10	128(39)	-38.40	0.82	117(43)
OH-15	-9.39	0.11	122(45)	-78.36	0.81	113(47)
OH-16	-15.45	0.11	123(44)	-114.94	0.70	109(51)

TABLE 6. Weighted average of accepted isotope values of all laboratories for the four WICO2011 water samples on the basis of maximum of stated and reported measurement uncertainty. In the column 'number of values', the number of rejected values is stated in addition in brackets.

Sample	$\delta^{18}O$			$\delta^{2}H$		
	mean value [‰]	standard deviation 1σ [‰]	number of values (rejected)	mean value [‰]	standard deviation 1σ [‰]	number of values (rejected)
OH-13	-0.97	0.12	140(26)	-2.73	0.91	123(36)
OH-14	-5.59	0.11	139(28)	-38.40	0.86	121(39)
OH-15	-9.39	0.13	132(35)	-78.31	0.87	126(34)
OH-16	-15.44	0.13	139(28)	-114.88	0.76	119(41)

The submitted laboratory mean values for $\delta^{18}O$ are listed in Table A1in APPENDIX I, with columns listing first the assigned laboratory identification number (ID), and equipment used (mass spectrometer, LGR laser analyzer, Picarro laser analyzer, and in the following columns several laboratory parameters: the indication of a performed VSMOW/SLAP calibration along the WICO2011 analyses, the number of laboratory water standards used for daily calibration and the amount of water and duration of the water/CO_2 equilibration. In the next columns the submitted mean values with associated standard uncertainties at a 1σ-level are reported for samples OH-13 to OH-16. The grey shaded results were rejected in the statistical evaluation and were not used to calculate the weighted averages. In the following four figures (Figs 1 to 4) these data are displayed graphically as S-shape plots versus laboratory identification numbers sorted by increasing numerical values with uncertainties provided here at the 2σ-level. Outliers having biases with respect to reference values greater than 2 times the sum of the standard deviations associated with the individual results (maximum) and the reference values at 1σ-level are marked in the figures with open symbols. The reference value and its standard uncertainty at 2σ-level are indicated in each figure as horizontal lines.

Similarly the submitted δ^2H results are listed in Table A2 in APPENDIX I, with columns listing assigned laboratory identification number (ID), equipment used (mass spectrometer, LGR laser analyzer, Picarro laser analyzer); and several laboratory parameters as the indication of a performed VSMOW/SLAP calibration, along with the WICO2011 analyses, the sample preparation method used, the number of laboratory water standards used for daily calibration and the amount of water used per measurement. In the following columns the submitted mean values with associated standard uncertainties at a 1σ-level are reported for samples OH-13 to OH-16. In Figs 5 to 8, these data are displayed graphically as S-shape plots versus laboratory identification numbers sorted by increasing numerical values. Outliers as defined above are marked in the figures with open symbols.

A large spread of uncertainties is visible in the Figs 1 to 8; for a considerable number of laboratories the uncertainties are of such an order of magnitude, that measured data could not be used in a meaningful way in hydrological applications. This is recognized, but was not used as acceptance criterion in the evaluation, since the exercise was intentionally open to all laboratories measuring stable isotopes in water samples and was not limited to isotope hydrology laboratories.

A different display of the same data is provided by δ^2H/δ^{18}O plots. These plots are quite useful to visualize the spread of hydrogen and oxygen data provided for any given sample. In Figs. 9 to 12, the reported laboratory mean data are plotted for each water sample. In the same plots, the respective average of the reported values calculated from all participating laboratories (except rejected values) is marked as a circle with associated uncertainty (1σ-level). The squared symbol marks the reference value and its uncertainty (see Table 2). Several values could not be displayed in the plot at the chosen scales, but all the four plots cover a similar range of δ-values (with a range of 1.2 ‰ and 12 ‰ each). It is worthwhile to note that the average values for all laboratories are not much different from the reference values. For OH-13 sample, only δ^{18}O is slightly biased, while for OH-16 sample, both δ^{18}O and δ^2H are slightly biased towards more negative δ-values versus the reference values.

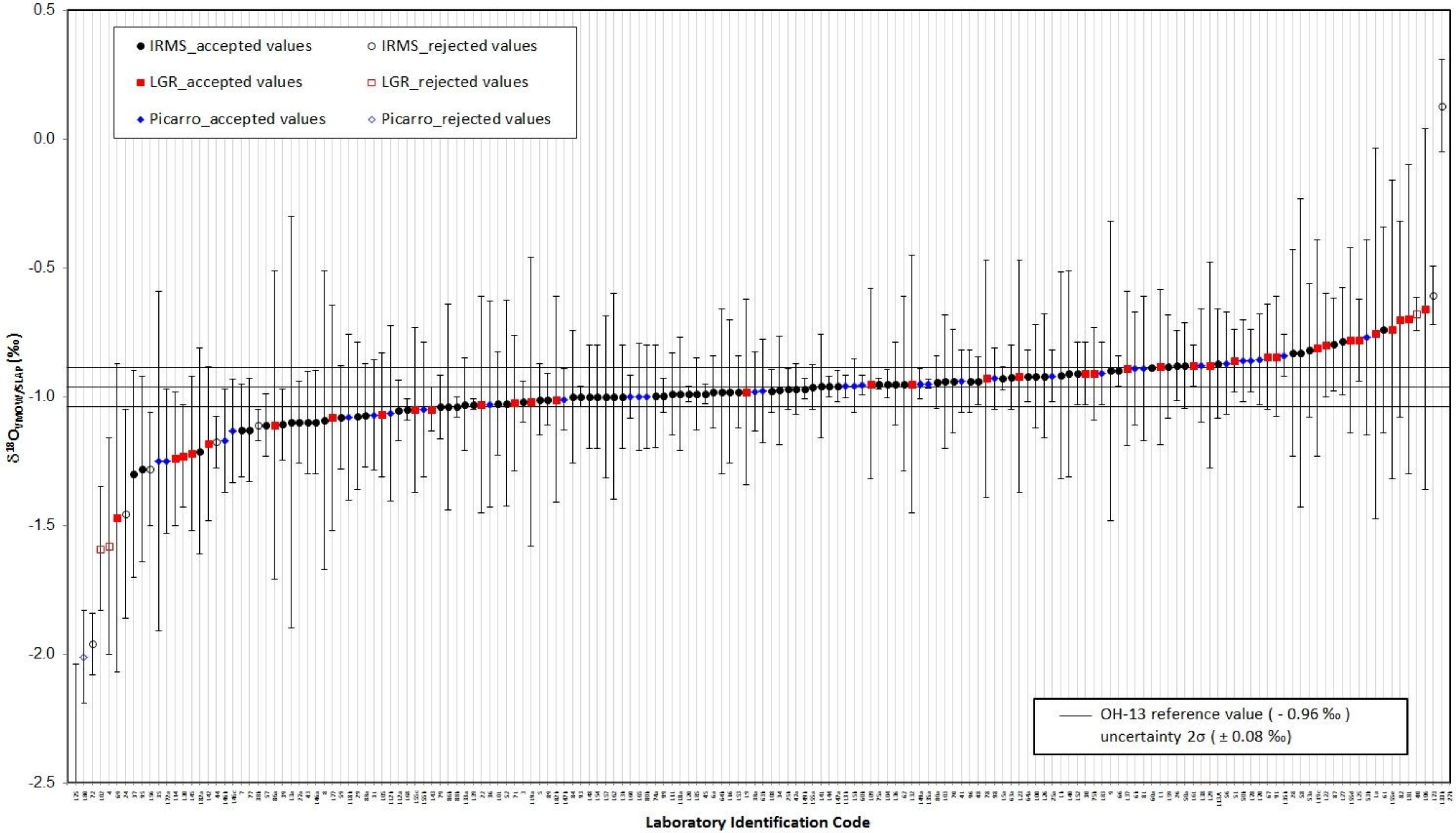

FIG. 1: S-shape plot for $\delta^{18}O$ values of sample IAEA-OH-13. Outliers as recognized during the statistical evaluation are marked by open symbols.

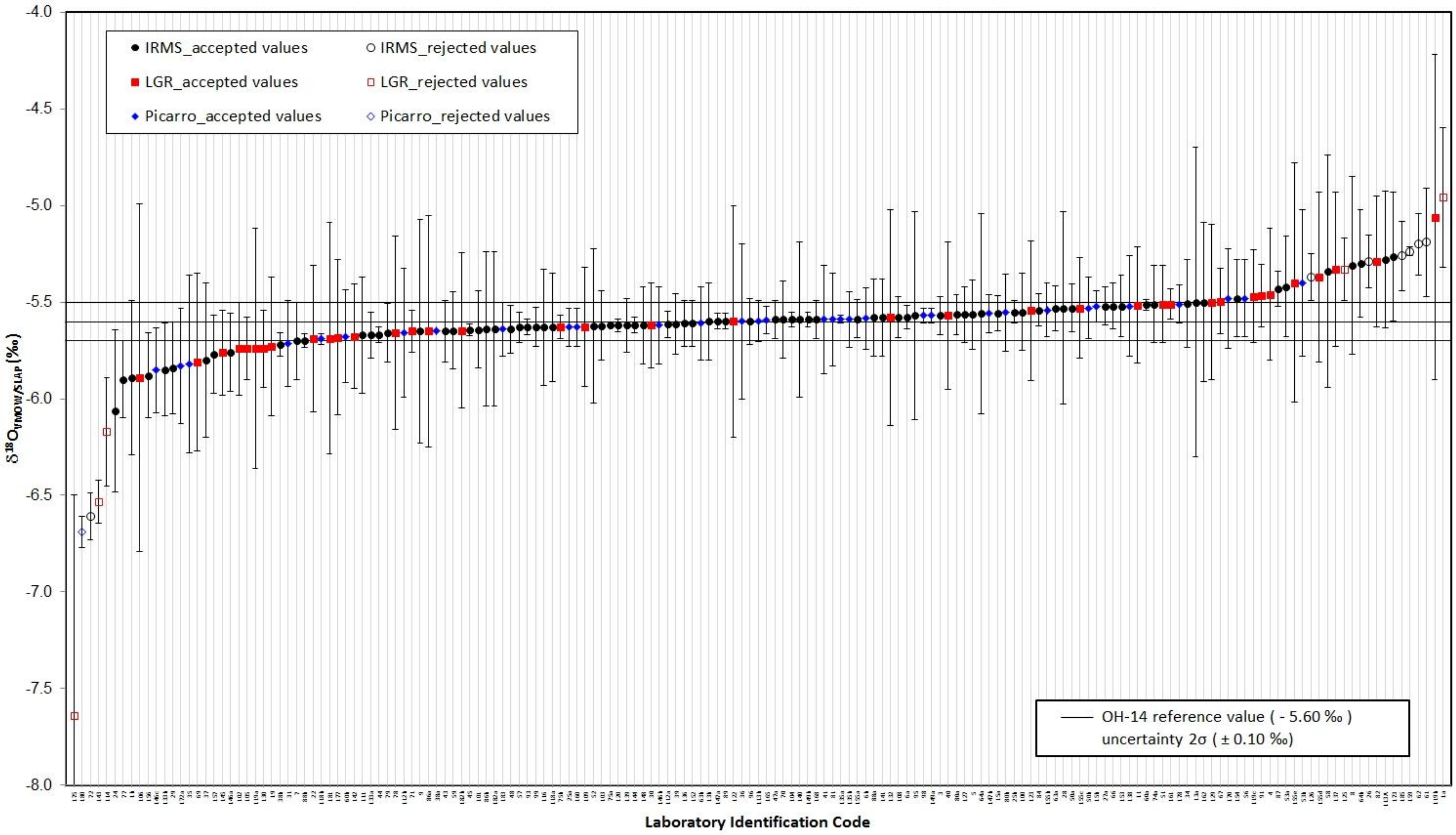

FIG. 2. S-shape plot for $\delta^{18}O$ values of sample IAEA-OH-14. Outliers as recognized during the statistical evaluation are marked by open symbols.

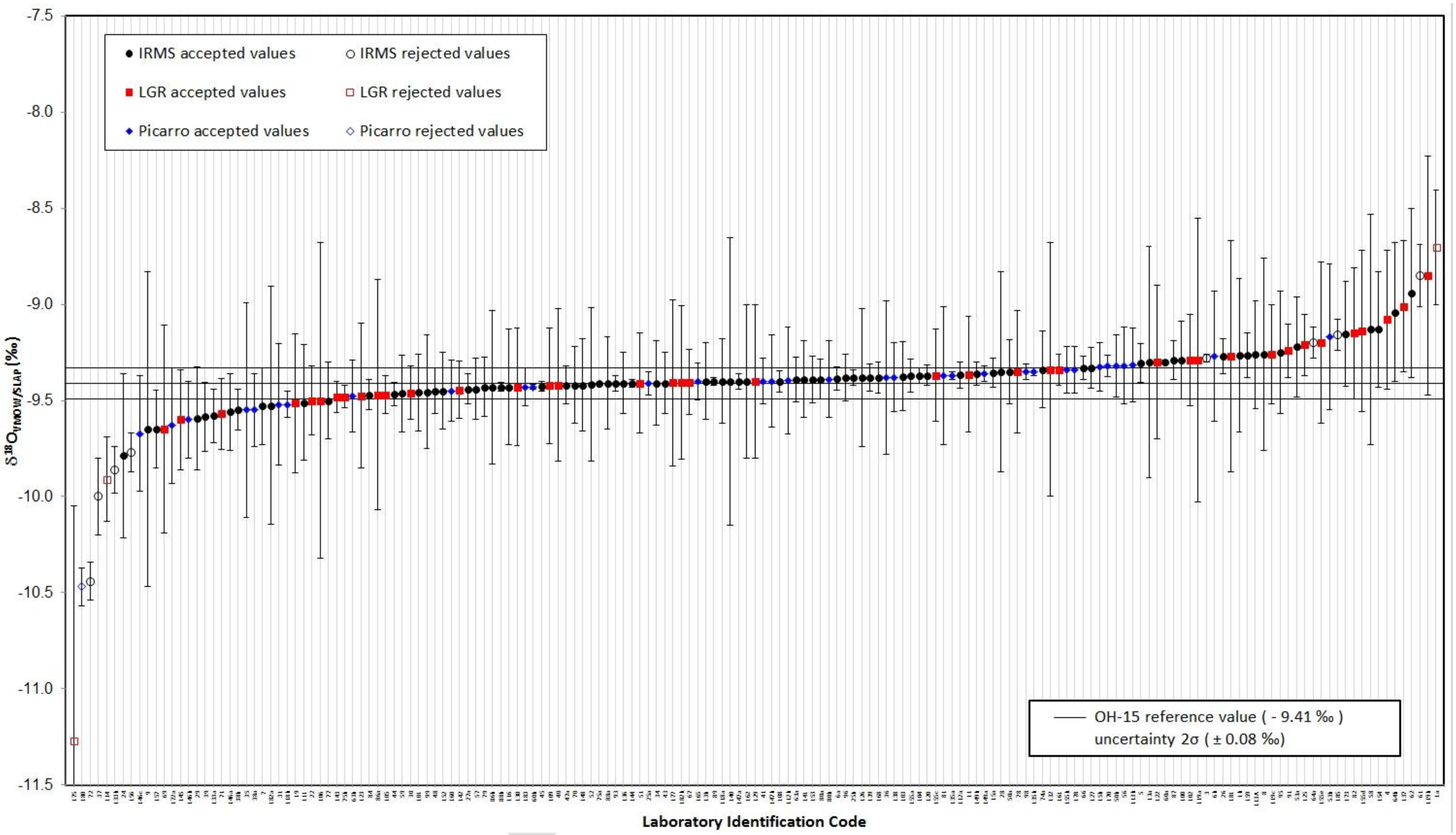

FIG. 3. S-shape plot for $\delta^{18}O$ values of sample IAEA-OH-15. Outliers as recognized during the statistical evaluation are marked by open symbols.

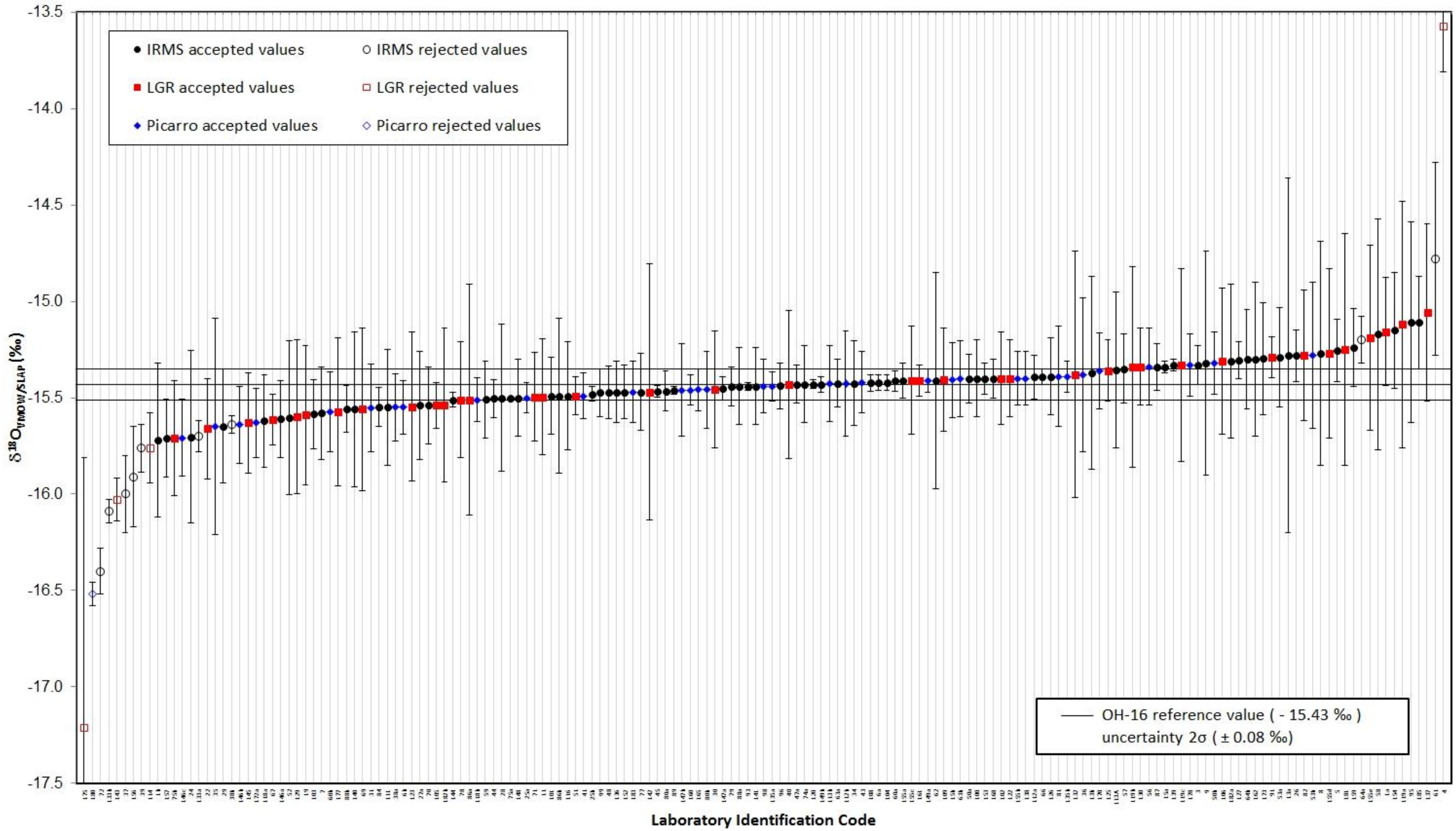

FIG. 4. S-shape plot for $\delta^{18}O$ values on sample IAEA-OH-16. Outliers as recognized during the statistical evaluation are marked by open symbols.

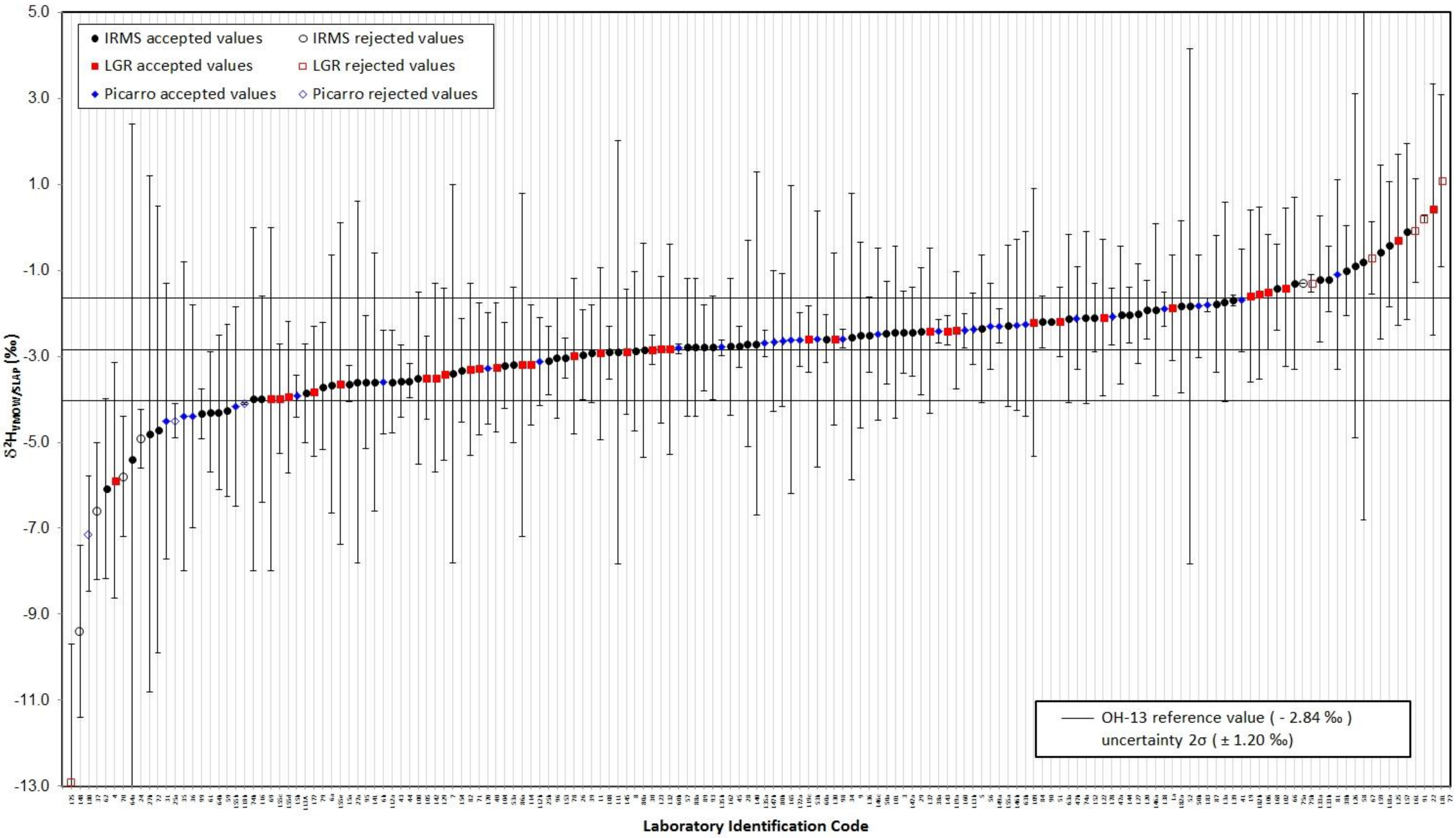

FIG. 5. S-shape plot for δ^2H values on sample IAEA-OH-13. Outliers as recognized during the statistical evaluation are marked by open symbols.

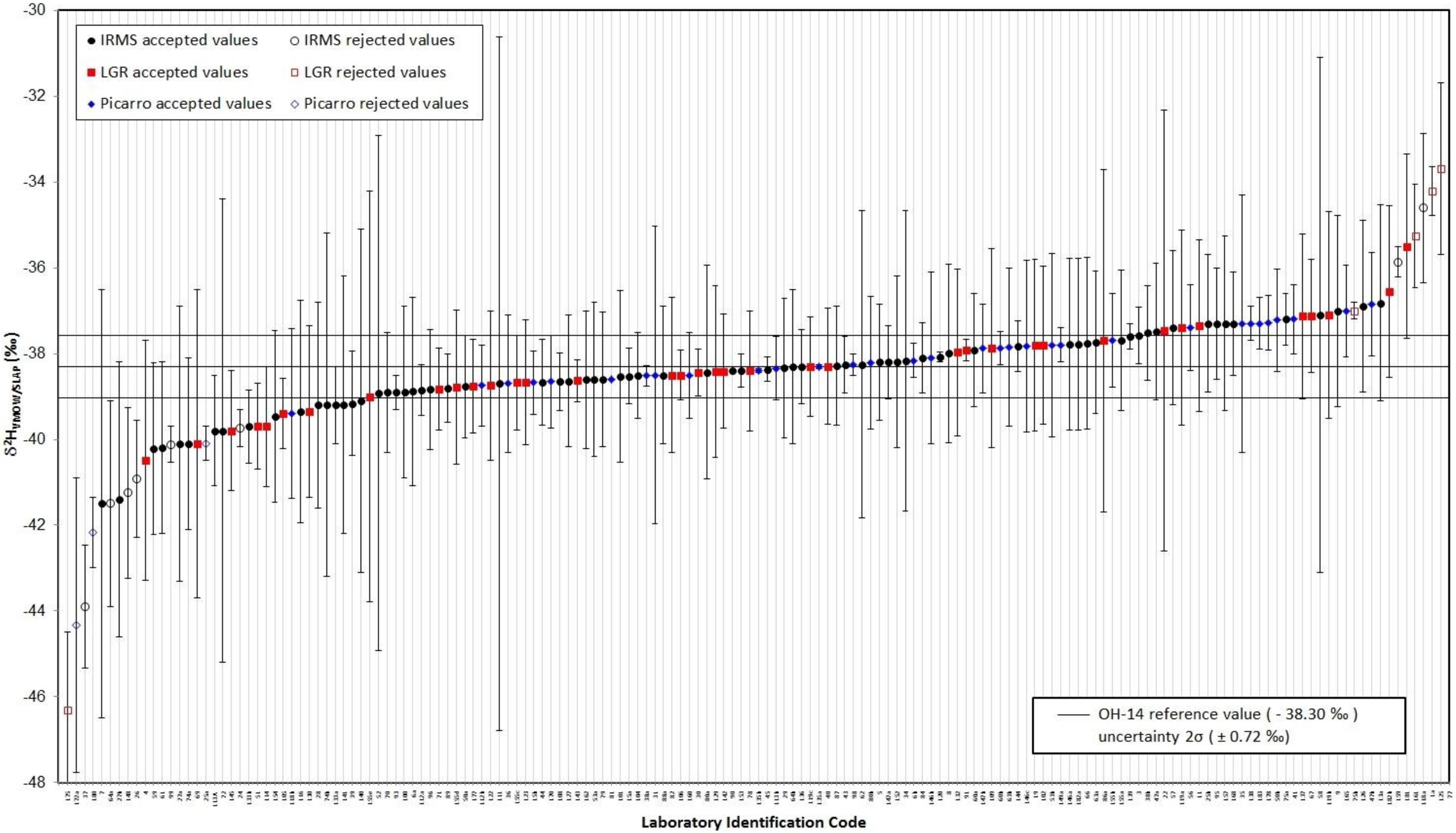

FIG. 6. S-shape plot for δ2H values on sample IAEA-OH-14. Outliers as recognized during the statistical evaluation are marked by open symbols.

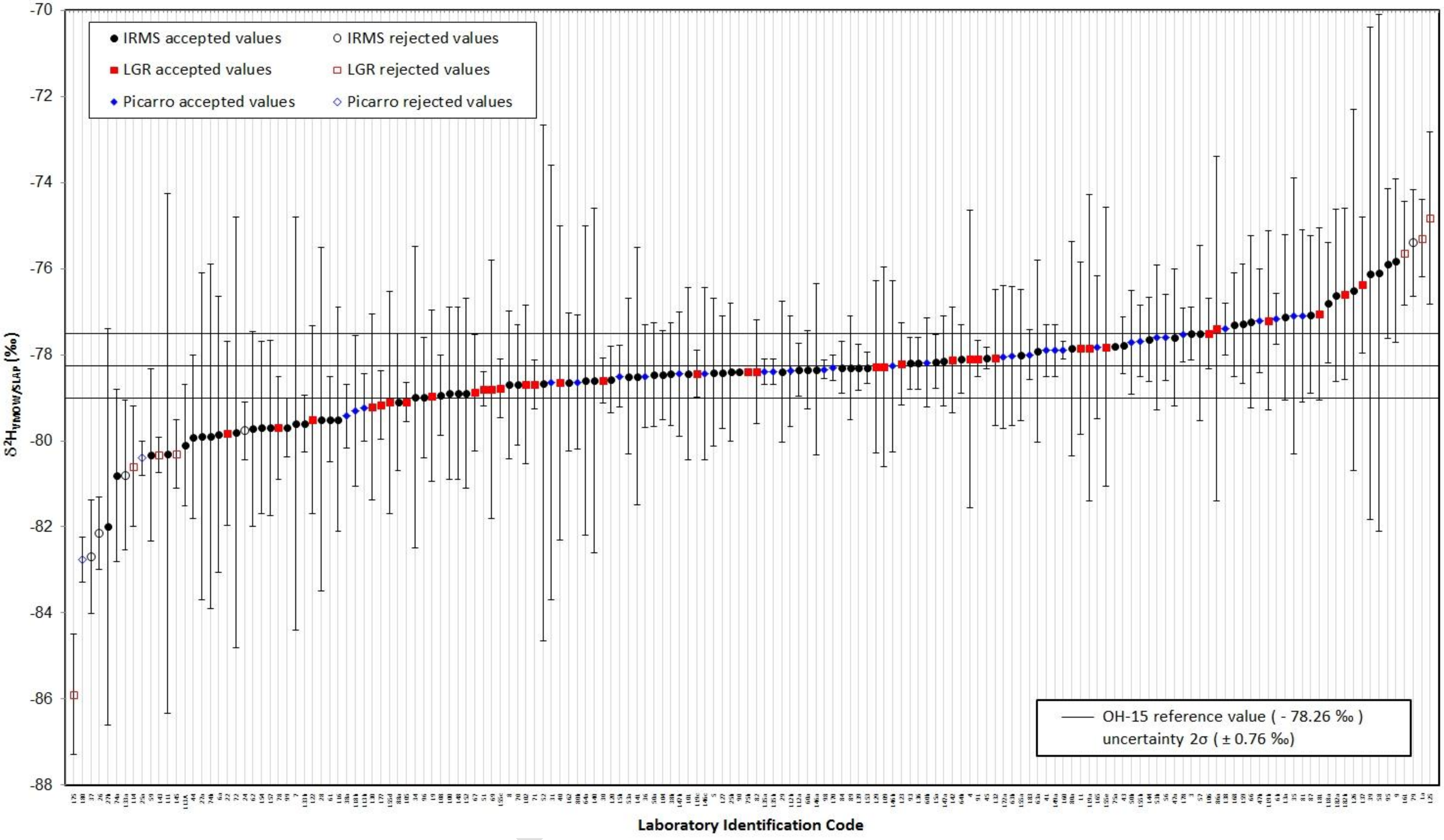

FIG. 7. S-shape plot for δ^2H values on sample IAEA-OH-15. Outliers as recognized during the statistical evaluation are marked by open symbols.

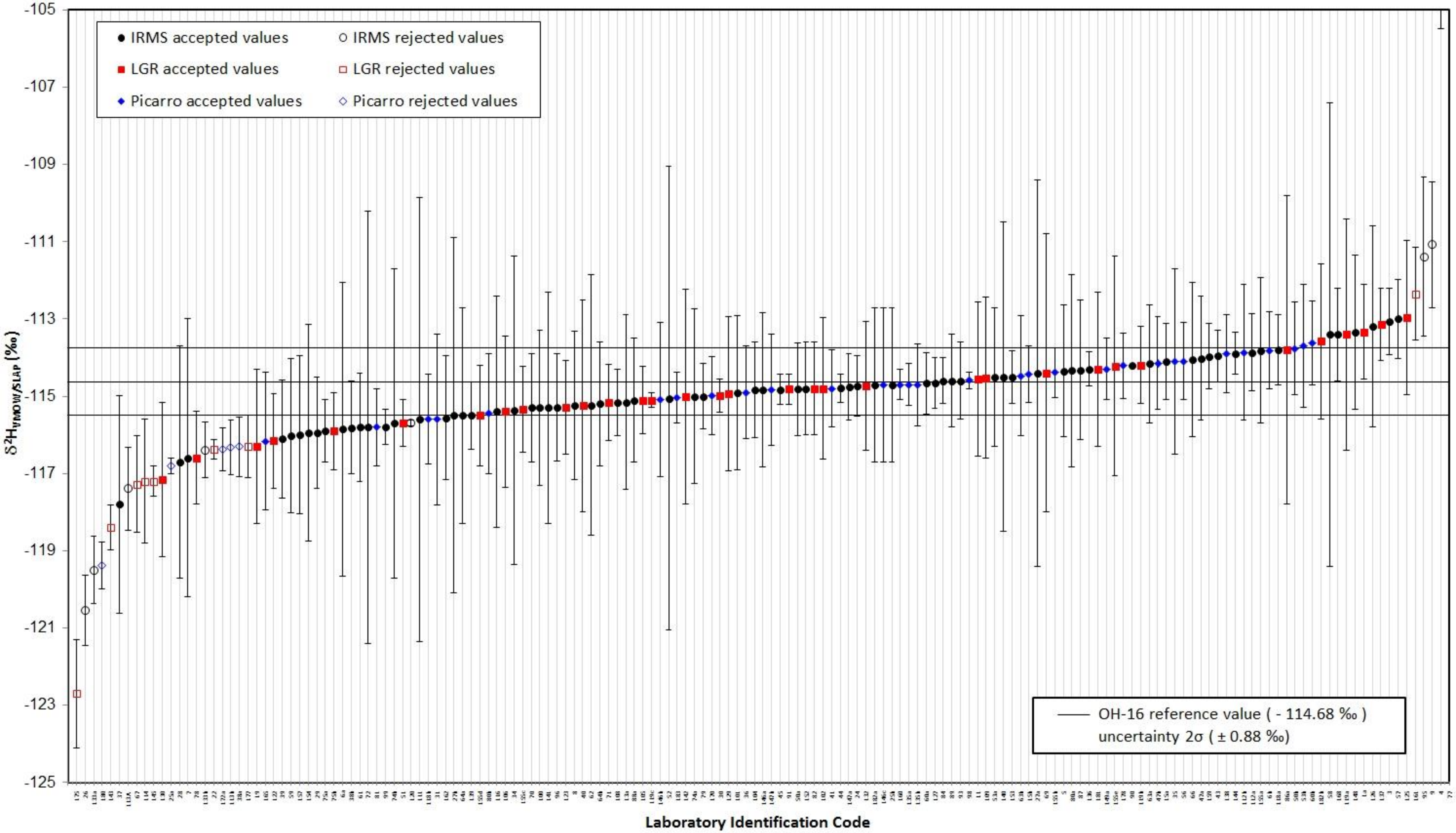

FIG. 8. S-shape plot for δ^2*H values on sample IAEA-OH-16. Outliers as recognized during the statistical evaluation are marked by open symbols.*

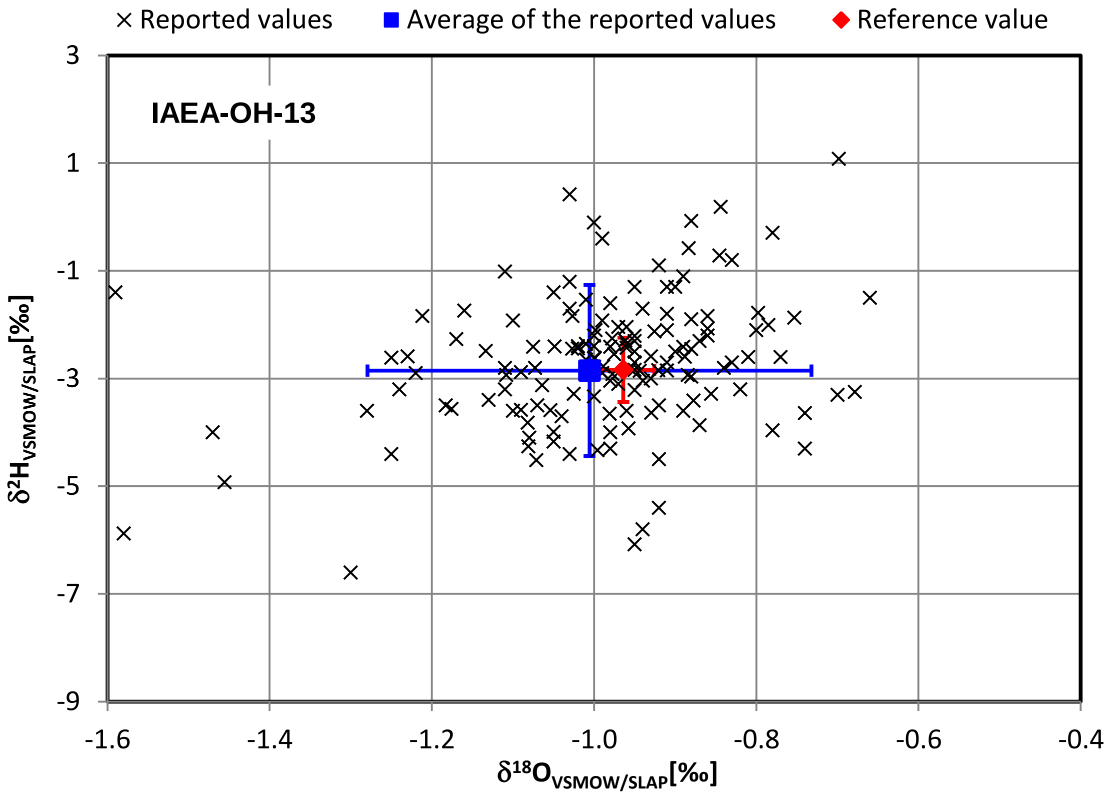

FIG. 9. $\delta^2H/\delta^{18}O$ plot of all reported laboratory mean values for the sample IAEA-OH-13. Not all values could be displayed using the selected scale.

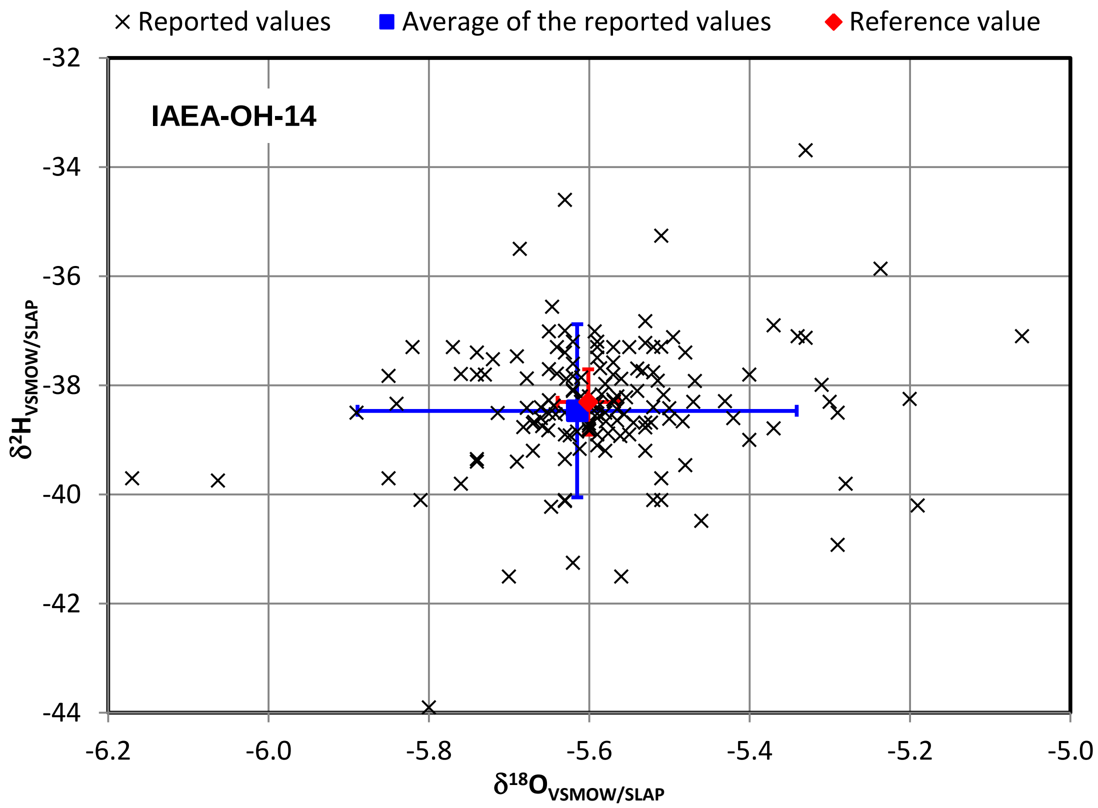

FIG. 10. $\delta^2H/\delta^{18}O$ plot of all reported laboratory mean values for the sample IAEA-OH-14. Not all values could be displayed using the selected scale.

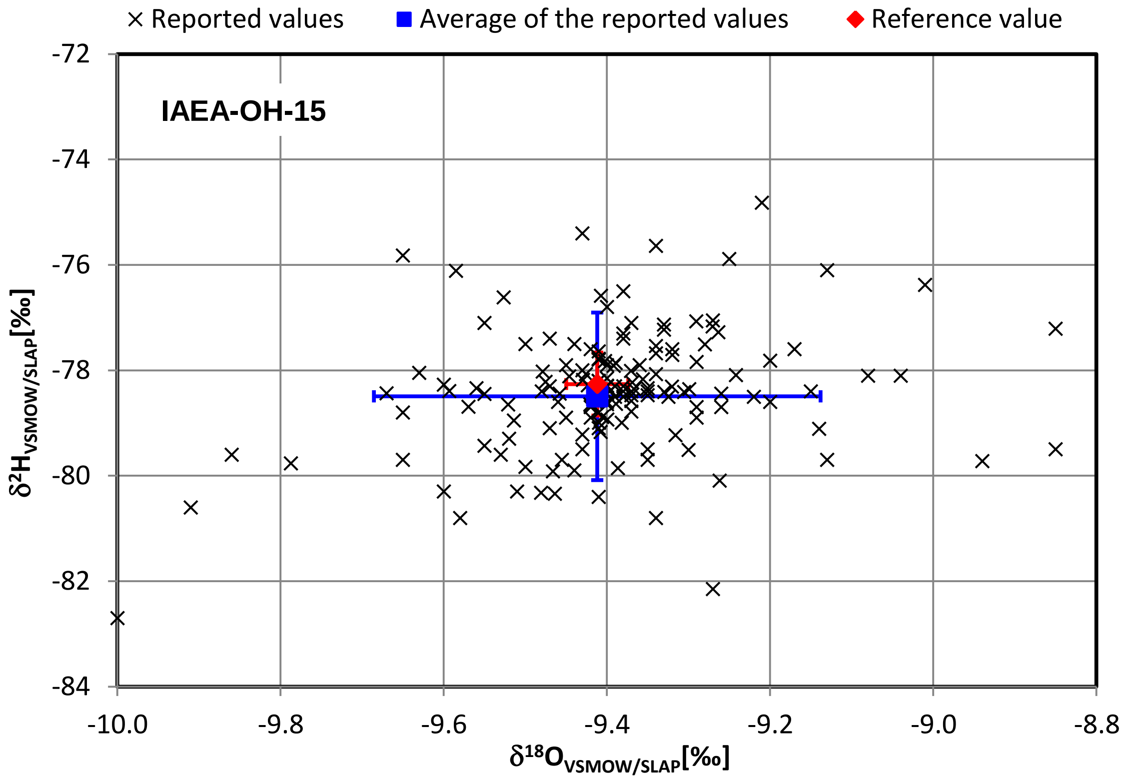

FIG. 11. $\delta^2H/\delta^{18}O$ plot of all reported laboratory mean values for the sample IAEA-OH-15. Not all values could be displayed using the selected scale.

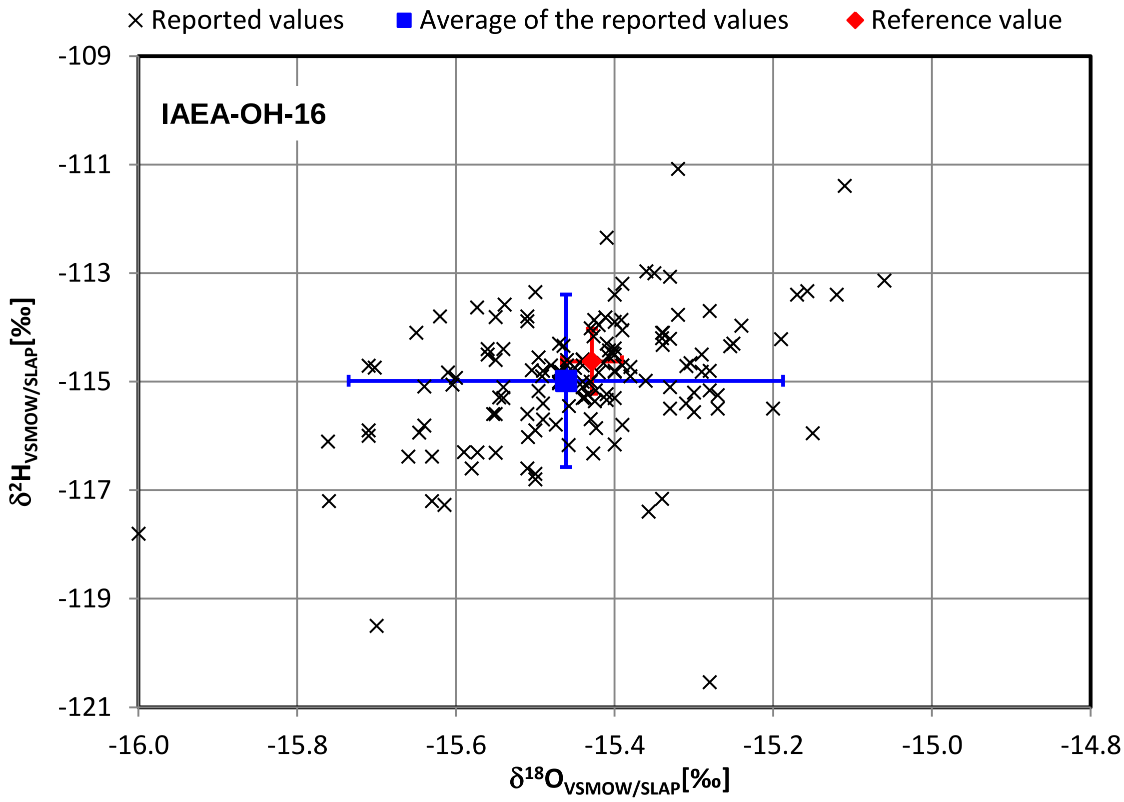

FIG. 12. $\delta^2H/\delta^{18}O$ plot of all reported laboratory mean values for the sample IAEA-OH-16. Not all values could be displayed using the selected scale.

3.3. Performance rating

In order to obtain information on relative performance of the participating laboratories, the results were processed as they were in the last IAEA exercises [2, 3]. An obvious measure for the accuracy of a result is the deviation from the respective reference value. Since the measurement process for all four samples is the same and the precision of measurements should theoretically not be strongly dependent on this range of δ-values, the four sample results can be treated as providing independent tests for the accuracy of measurements in each laboratory. In Fig. 13 the cumulative deviation from the reference value for all four $\delta^{18}O$ results is displayed as a stack plot for each laboratory. A value equal to the reference value therefore has a value of zero on a $\Delta\delta^{18}O$ scale ($\Delta\delta^{18}O=\delta^{18}O_{lab}-\delta^{18}O_{reference}$). The laboratories are sorted in order of increasing absolute cumulative deviation. A similar approach was used for cumulative deviation from the reference value for all four $\delta^{2}H$ measurements and is shown in Fig. 14.

Deviations of the reported values from the reference values were also compared with the uncertainties to check the performance of a laboratory. As indicated above, most laboratories did not provide long term uncertainties, therefore reported measurement uncertainties were used in such cases. The cumulated deviation of $\delta^{18}O$ values from the reference values for all four values is plotted in Fig. 15. The thick black line indicates the laboratory uncertainty estimate (four times the standard uncertainty or sum of the measurement uncertainties for four samples). Columns much higher than the black line indicate an underestimation of uncertainty for those laboratories (deviations that are much larger than expected). On the other hand, columns being lower than the black line indicate an excessively conservative approach in uncertainty evaluation at a particular laboratory (real precision and accuracy better than assumed). A similar plot for $\delta^{2}H$ measurements is provided in Fig. 16.

For overall of laboratory performance assessment of both $\delta^{18}O$ and for $\delta^{2}H$, laboratory deviations as displayed in Fig. 15 and Fig. 16 were combined on the basis of their natural correlation by the mean meteoric water line [3]. The absolute value of all sample deviations for $\delta^{2}H$ was combined with similar data for $\delta^{18}O$, the latter data being multiplied by a factor of eight to weigh the deviations at a similar level. The resulting numerical value for each laboratory provides an indicator for laboratory deviation from reference values for the four distributed samples. A smaller value can be interpreted as better corresponding to the reference values. Those data are displayed in Fig. 17. Laboratories which only analyzed $\delta^{18}O$ or $\delta^{2}H$ are displayed separately at the right side of the plot. Such a performance indicator is somewhat arbitrary, as it combines different and independent measurements performed outside of the routine modus and provides only a snapshot of laboratory performance limited to the time period when the measurements were actually performed. No extrapolation is possible to the past and future based on the performance indicators presented here.

3.4. Comparison of performance with previous exercises

In order to compare the performance of the laboratories which participated in WICO2002 and/or WICO1999, cumulative deviations from the reference values for all four $\delta^{18}O$ and $\delta^{2}H$ results are plotted in the form of bar charts (Figs. 18 and 19). Most of the laboratories have improved their performance compared to the previous exercises.

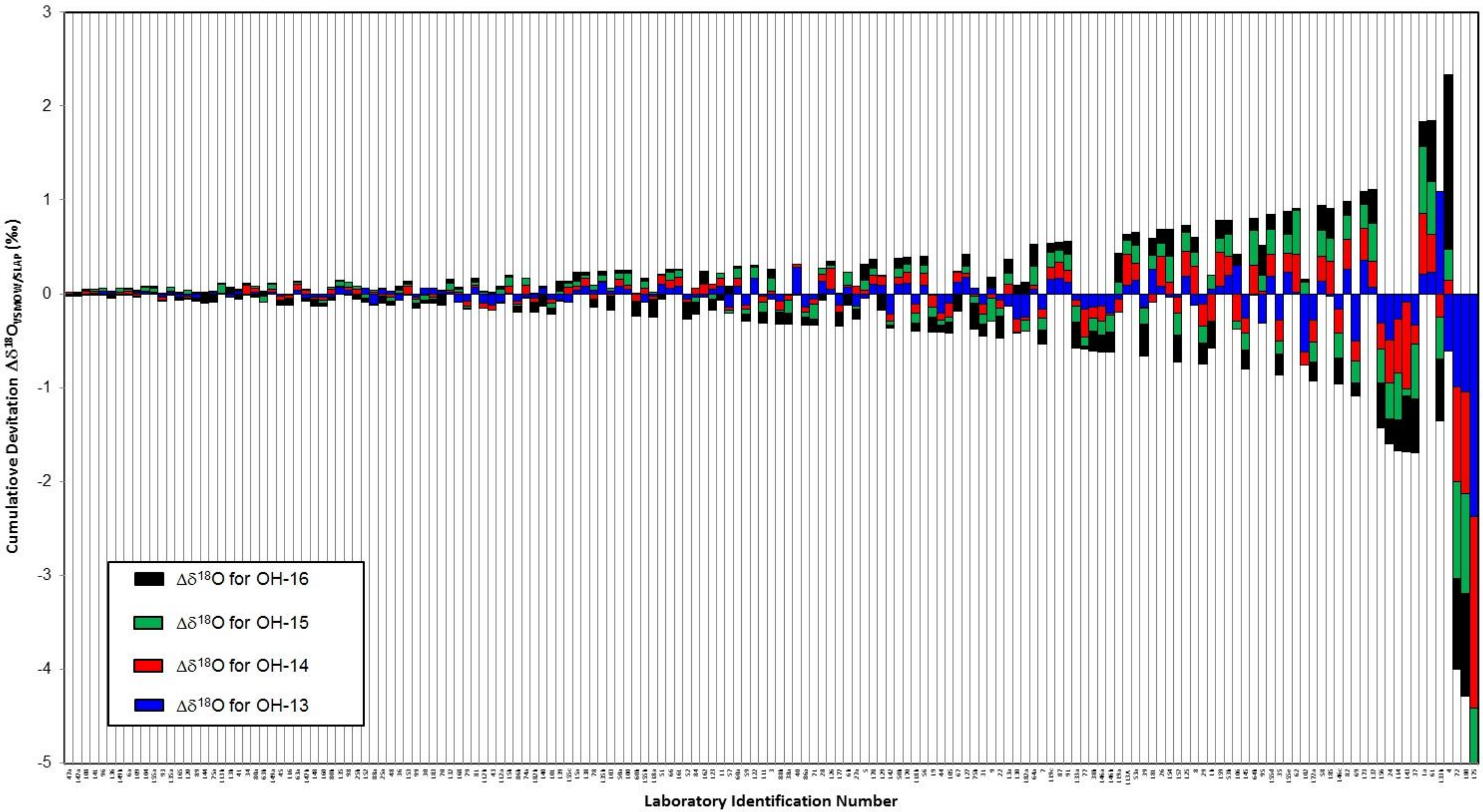

FIG. 13. Deviation of $\delta^{18}O$ results versus reference values for all four samples analyzed by each laboratory and sorted by increasing deviation.

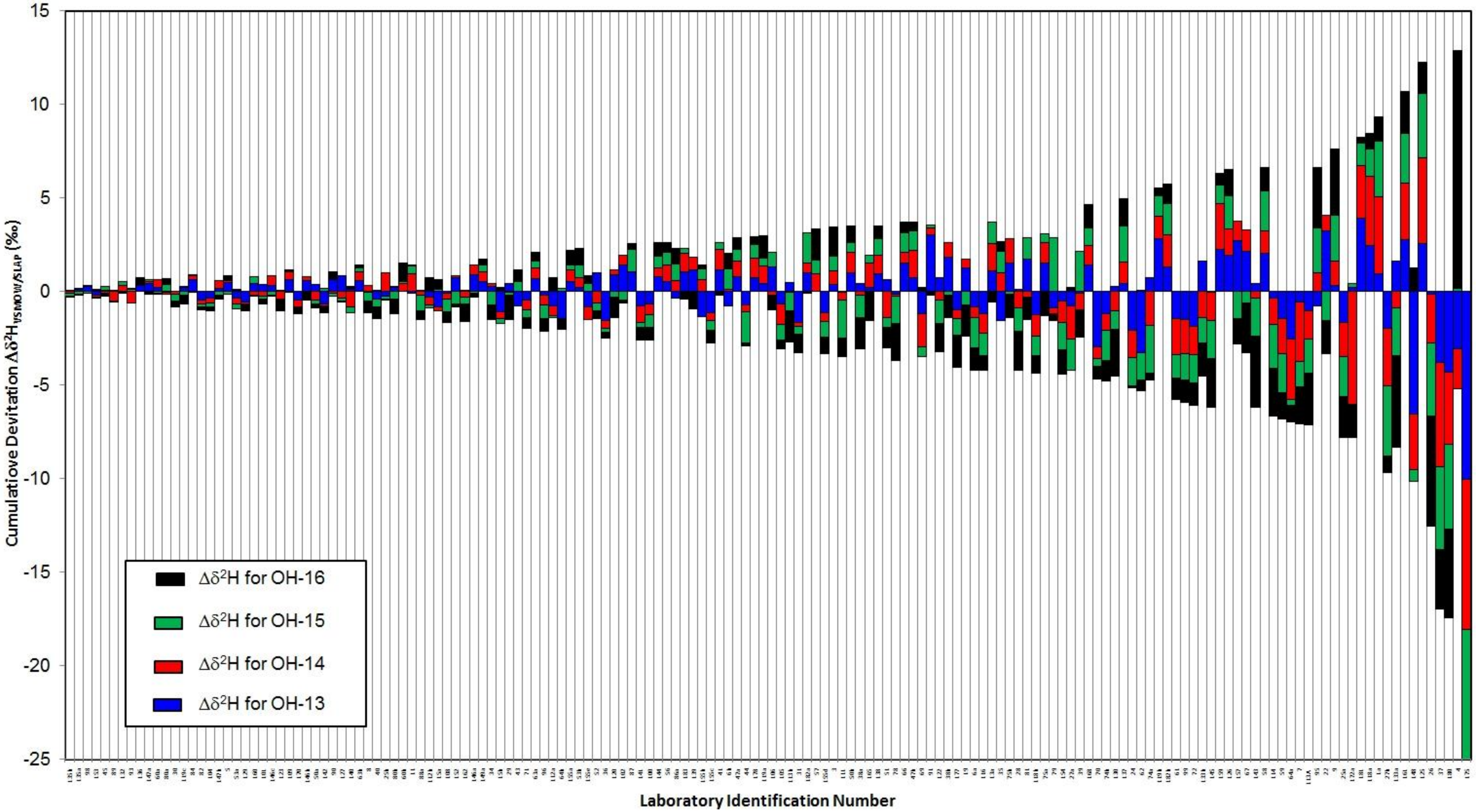

FIG. 14. Deviation of δ^2H results versus reference values for all four samples analyzed by each laboratory and sorted by increasing deviation.

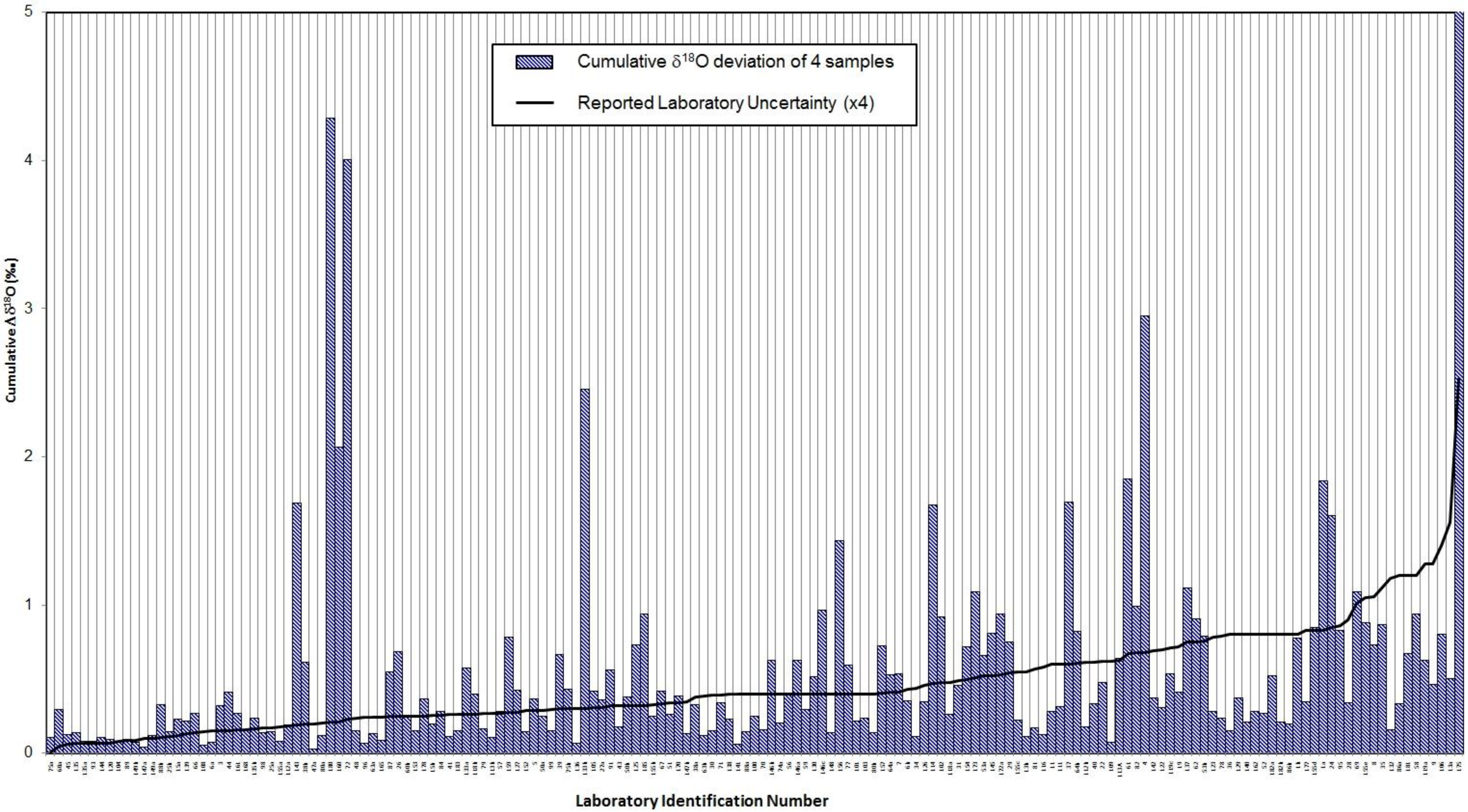

FIG. 15. Comparison between reported $\delta^{18}O$ uncertainty and deviation from the reference value for laboratories. Columns much higher than the black line indicate an underestimation of uncertainty (deviations much greater than expected). On the other hand, columns much lower than the black line indicate an excessively conservative approach in uncertainty evaluation (real precision and accuracy better than assumed).

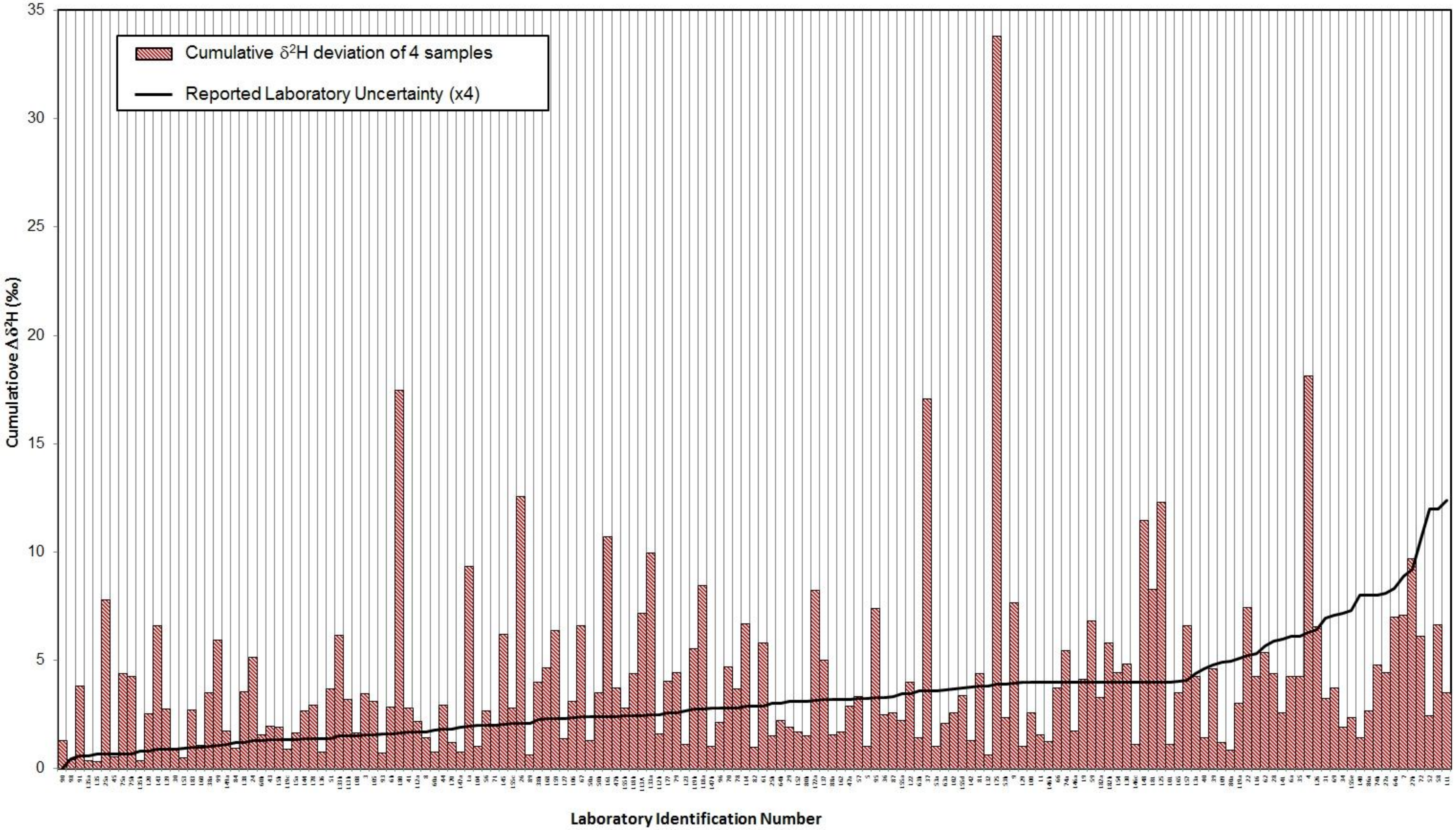

FIG. 16. Comparison between reported δ^2H uncertainty and deviation from the reference value for laboratories. Columns much higher than the black line indicate an underestimation of uncertainty (deviations much greater than expected). On the other hand, columns much lower than the black line indicate an excessively conservative approach in uncertainty evaluation (real precision and accuracy better than assumed).

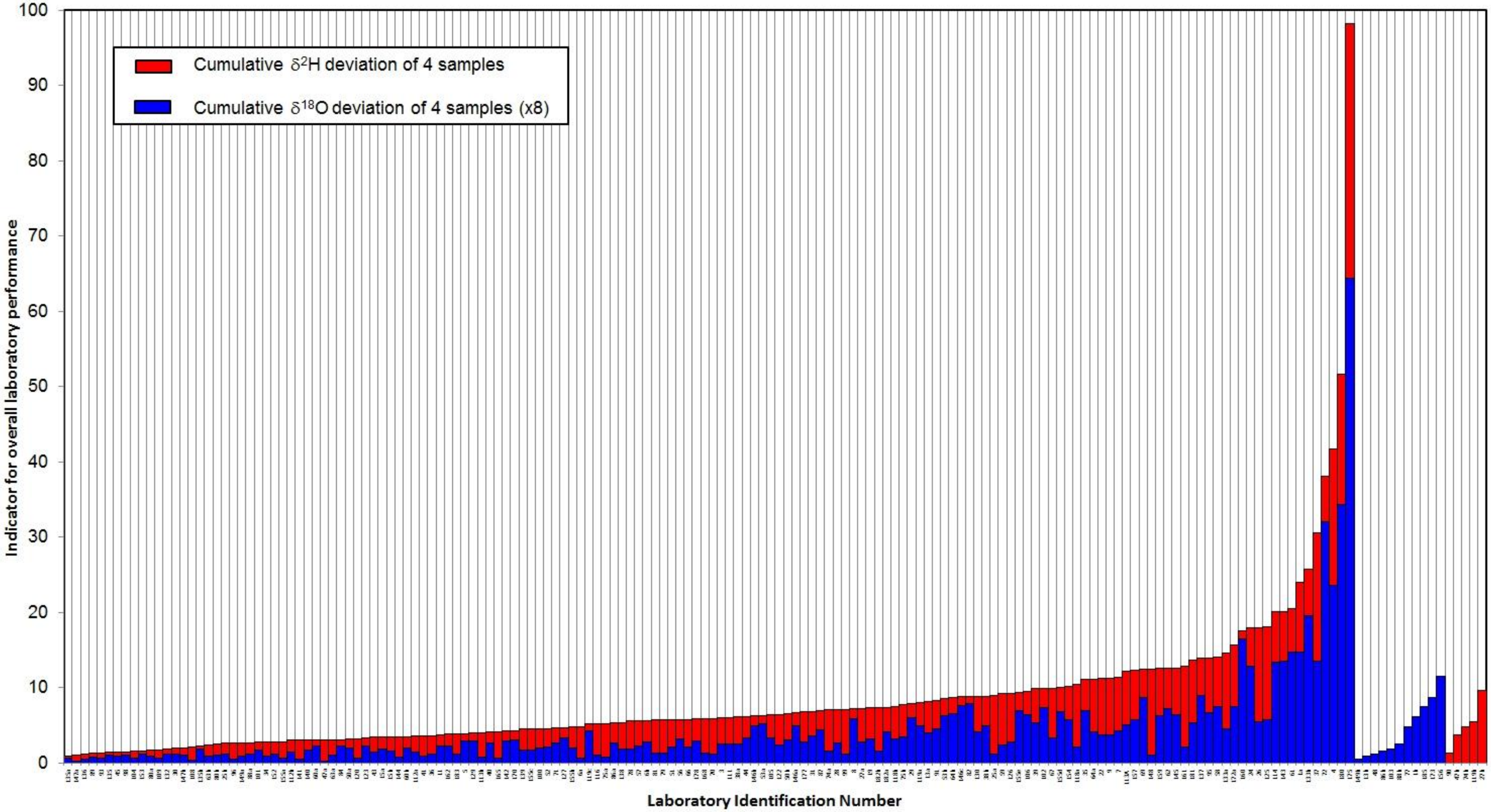

FIG. 17. Indicator for overall laboratory performance derived from both δ^2H and $\delta^{18}O$ data.

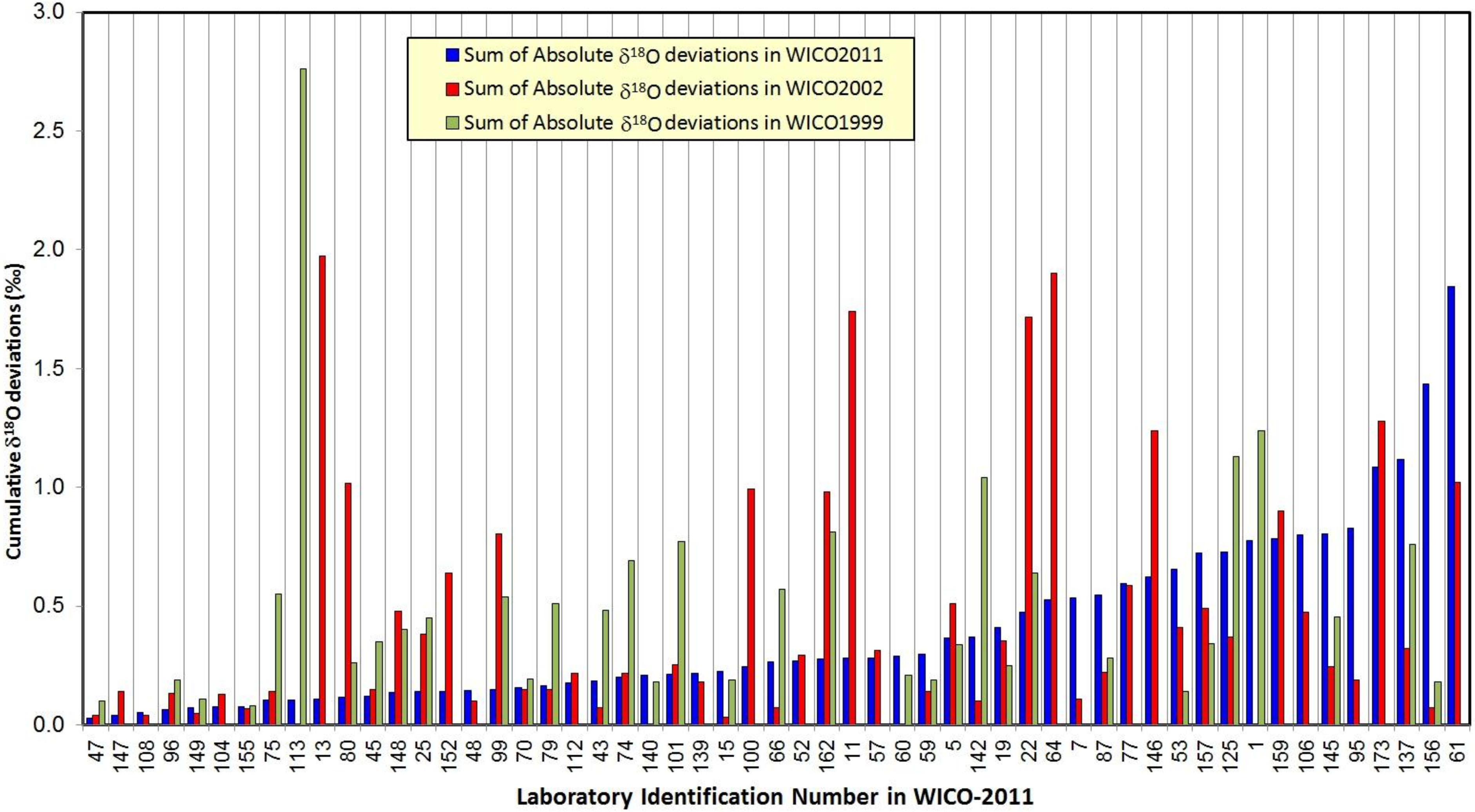

FIG. 18. Comparison of laboratory performance in WICO2011 with that of previous exercises for analysis of $\delta^{18}O$ in water samples.

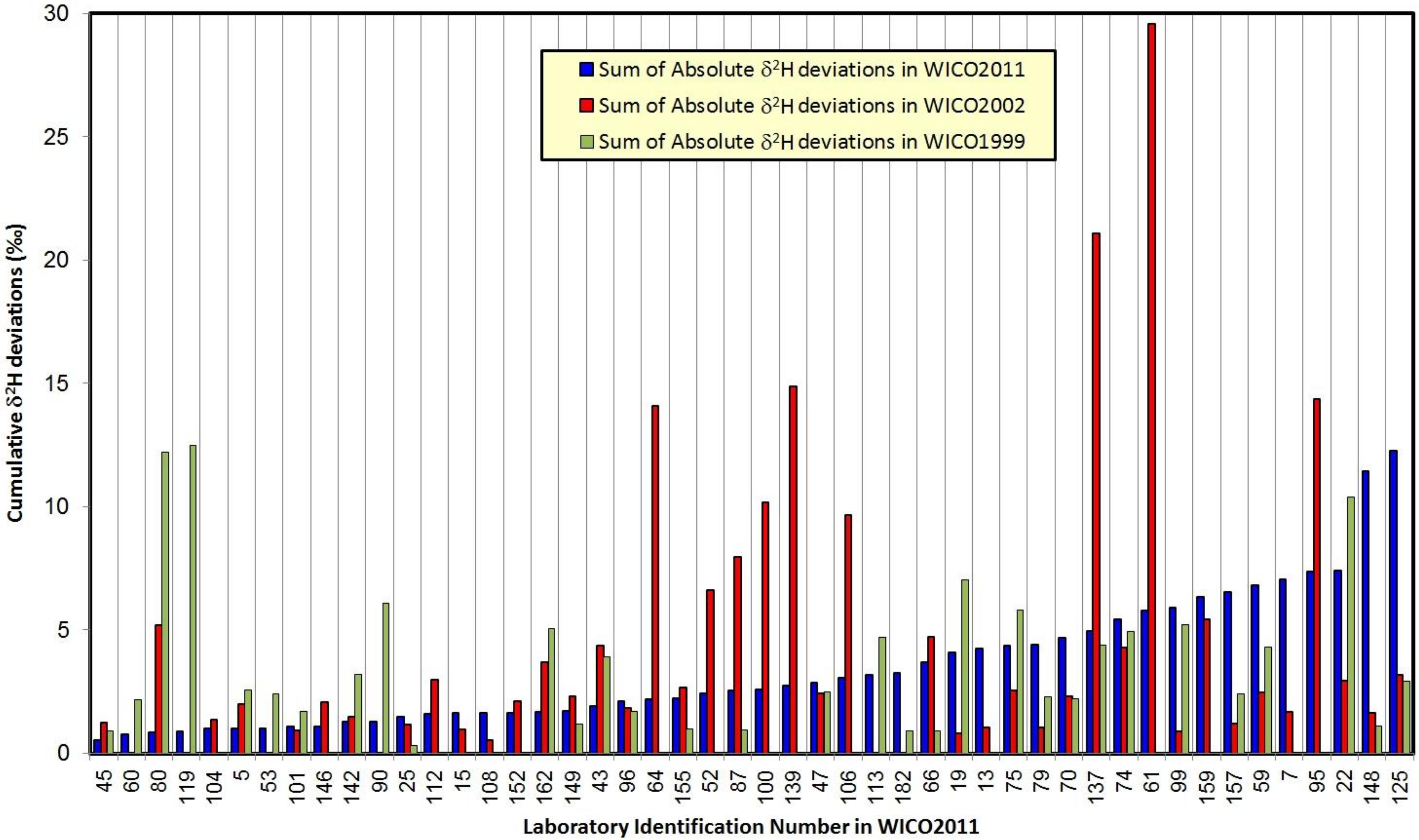

FIG. 19. Comparison of laboratory performance in WICO2011 with that of previous exercises for analysis of δ^2H in water samples.

4. COMPARISON OF RESULTS OF ANALYTICAL INSTRUMENTS

Results of three analytical instruments i.e. mass spectrometers, LGR laser-based water isotope analyzers and Picarro laser-based water isotope analyzers were compared. Average values of accepted results from WICO2011 samples determined by these instruments are provided in Tables 7 to Table 9. Graphic comparisons of average $\delta^{18}O$ and $\delta^{2}H$ values of the four samples determined from the results of these analyzers are shown in Fig. 20 and Fig. 21, respectively. It is noted that the average values for the three instruments are in very good agreement, being well within 1σ-uncertainties. The average values are so close that their maximum standard deviations at 1σ-level for $\delta^{18}O$ and $\delta^{2}H$ are 0.01‰ and 0.1‰, respectively.

TABLE 7. Weighted average of accepted isotope values analyzed by mass spectrometers of the four WICO2011 water samples on the basis of maximum stated and measurement uncertainty. In the column 'number of laboratories', the number of rejected laboratories is additionally stated in brackets.

Sample	$\delta^{18}O$			$\delta^{2}H$		
	Mean value [‰]	standard deviation 1σ [‰]	number of laboratories (rejected)	mean value [‰]	standard deviation 1σ [‰]	number of laboratories (rejected)
OH-13	-0.98	0.10	75(15)	-2.77	1.00	64(18)
OH-14	-5.60	0.10	72(18)	-38.37	0.94	67(15)
OH-15	-9.39	0.13	72(18)	-78.32	1.02	68(14)
OH-16	-15.44	0.13	71(19)	-114.83	0.81	66(16)

TABLE 8. Weighted average of accepted isotope values analyzed by LGR laser spectroscopic analyzers of the four WICO2011 water samples on the basis of maximum of stated and measurement uncertainty. In the column 'number of laboratories', the number of rejected laboratories is additionally stated in brackets.

Sample	$\delta^{18}O$			$\delta^{2}H$		
	mean value [‰]	standard deviation 1σ [‰]	number of laboratories (rejected)	mean value [‰]	standard deviation 1σ [‰]	number of laboratories (rejected)
OH-13	-0.97	0.18	34(7)	-2.68	0.75	32(9)
OH-14	-5.61	0.14	35(7)	-38.50	0.89	33(9)
OH-15	-9.41	0.13	33(9)	-78.46	0.71	30(12)
OH-16	-15.46	0.15	35(7)	-115.05	0.74	28(14)

TABLE 9. Weighted average of accepted isotope values analyzed by Picarro laser spectroscopic analyzers of the four WICO2011 water samples on the basis of maximum stated and measurement uncertainty. In the column 'number of laboratories', the number of rejected laboratories is additionally stated in brackets.

Sample	$\delta^{18}O$			$\delta^{2}H$		
	mean value [‰]	standard deviation 1σ [‰]	number of laboratories (rejected)	mean value [‰]	standard deviation 1σ [‰]	number of laboratories (rejected)
OH-13	-0.96	0.11	31(4)	-2.73	0.84	27(9)
OH-14	-5.58	0.09	32(3)	-38.40	0.47	21(15)
OH-15	-9.38	0.11	27(8)	-78.31	0.50	28(8)
OH-16	-15.45	0.09	33(2)	-114.89	0.62	25(11)

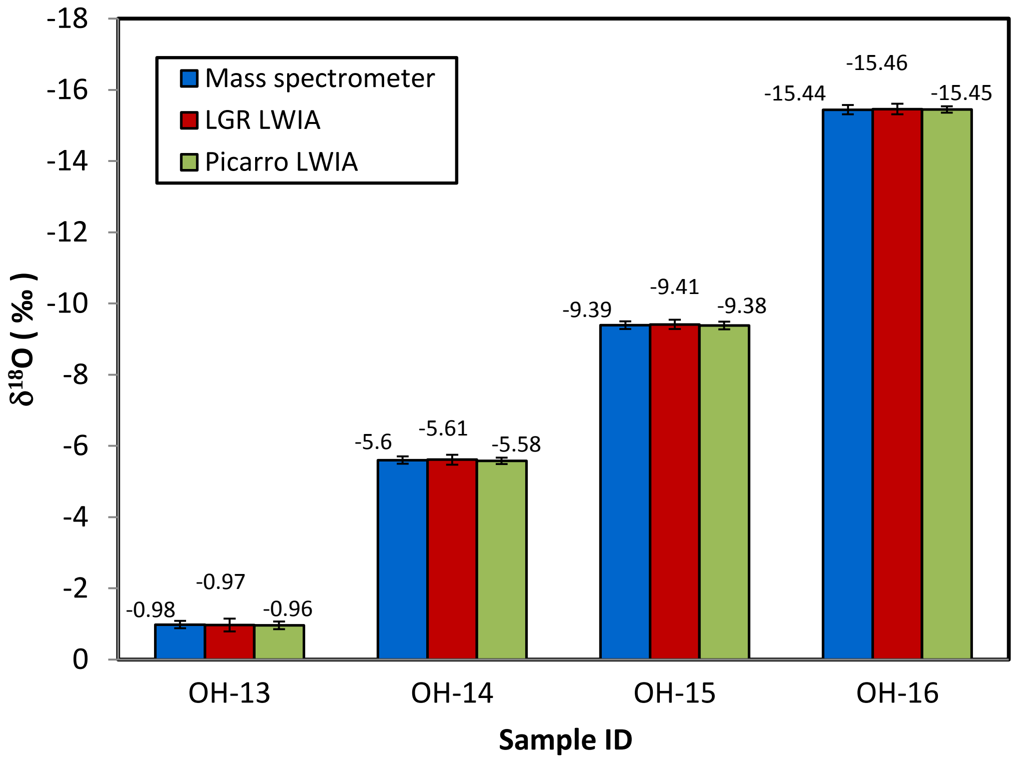

FIG. 20. Comparison of average $\delta^{18}O$ values of four samples determined from the results of mass spectrometers and laser-based water isotope analyzers.

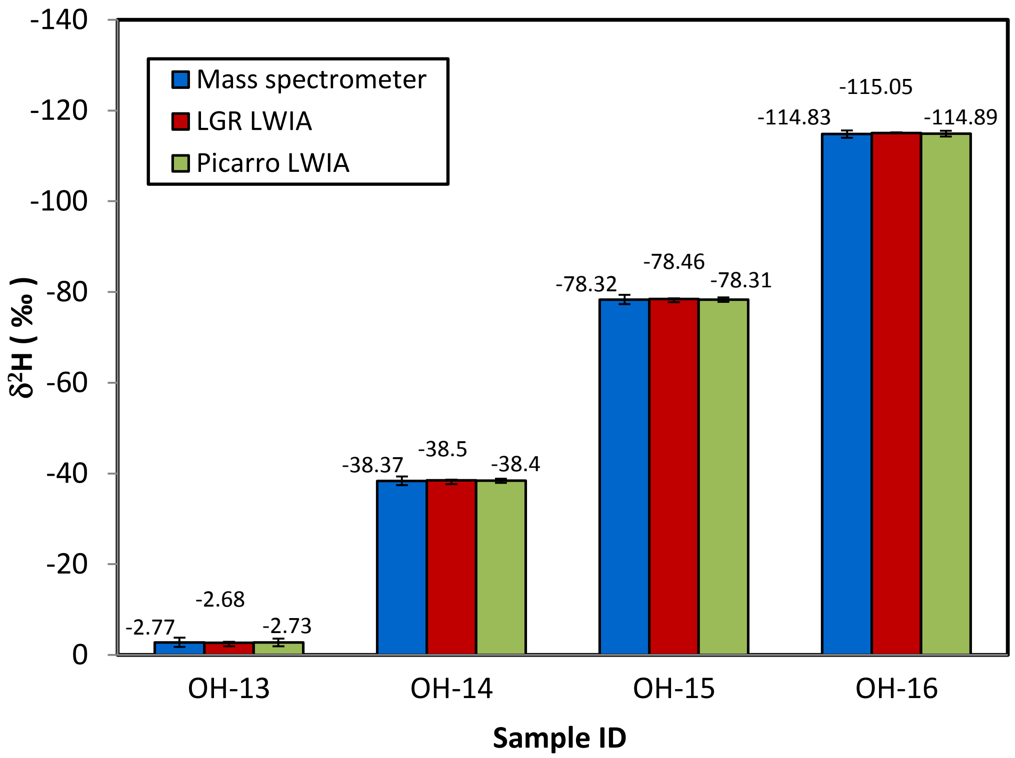

FIG. 21. Comparison of average δ^2H values of four samples determined from the results of mass spectrometers and laser-based water isotope analyzers.

5. BASIC QUALITY ASSURANCE ISSUES AND RECOMMENDATIONS

The following are the main quality assurance issues:

Unacceptably high errors: Of all the participating laboratories, 35% for δ^{18}O and 39% for δ^2H, have considerable problems in achieving the precision and accuracy targets required for most hydrological applications. Their cumulative biases for δ^{18}O and δ^2H are greater than 0.4‰ and 4‰, respectively.

Systematic errors: A general bias of reported results with respect to reference values exists towards both positive and negative values for the set of all participating laboratories. Inconsistencies in the results arise depending on the sample measurements carried out with aliquots of VSMOW and SLAP (for direct calibration) or using internal laboratory water standards calibrated earlier. Evaporated or contaminated standards result in systematic off-sets. Most of the laboratories have either positive or negative deviations for all four samples which cannot be attributed to random offsets in ^{2}H or ^{18}O measurements alone. One possible explanation for the shift towards more negative values could be slight evaporative enrichment of the used internal laboratory standards with time after calibration against VSMOW. Laboratory standards must be stored in proper containers with dispensing systems and slightly positive inert gas pressure to avoid evaporation and contamination by atmospheric moisture. Positive shifts could be due to evaporative enrichment of the primary standard (VSMOW), used multiple times for calibration of internal laboratory standards after the first opening of ampoules and improper storage.

Reporting of uncertainties: Stated long term and individual measurement uncertainties from some laboratories are too high or too low. The underestimation of uncertainty is especially critical. Other uncertainties stated are so huge that the results themselves become very uncertain and are of limited usefulness for most hydrological applications. Some laboratories quoted extremely low uncertainties averaging a few analytical measurements which are very close, especially regarding laser spectroscopic analysis. Overestimated uncertainties lead to the rejection of such results. Proper procedure should be used to reject outlying measurements for the calculation of average values and estimation of realistic uncertainties. Proper procedure should be used to reject outlying measurements for the calculation of average values and the estimation of realistic uncertainties.

Routine calibration with internal standards: It is obvious that for stable isotope measurements, daily routine calibration using only one internal laboratory standard cannot be used to correct for any scale normalization effect. Two internal laboratory standards would allow, for scale normalization. In the present exercise, seven laboratories used only one standard for calibration. The results of two to four samples of six of those laboratories are not within an acceptable range.

6. CONCLUSIONS

The number of participating laboratories in WICO2011 is twice the number of participants in WICO2002, mainly because of a revolution brought on by the expansion in the number of laser water isotope analyzers. It also shows increased interest in the application of environmental isotopes in hydrology and other disciplines.

No significant dependence of obtained δ-values on the analyzer type could be found. The results of laser water isotope analyzers and mass spectrometers are within the acceptable range of uncertainties.

Data from 52 sets out of 172 sets showed considerable deviation from the reference values for at least three sample values. On the other hand, a majority of laboratories have proven themselves capable of providing high quality data which can be used confidently for hydrological/ hydrogeological studies.

Precise calibration possible creates a bottleneck for those laboratories which are still subject to considerable offsets from the reference values. The basic requirement remains to suitably calibrate and store internal laboratory water standards.

The WICO2011 interlaboratory comparison has been helpful in detecting potential problem areas for laboratories. There is still a considerable number of laboratories with basic problems in accuracy, precision, and in stating justifiable measurement uncertainty. Further efforts are needed to improve this situation. Some improvements will be suggested to individual laboratories having large deviations in results and uncertainty problems.

Most of the laboratories which participated in WICO2002 and/or WICO1999 have improved their performance as compared to the previous exercises.

7. ACKNOWLEDGEMENTS

The efforts of all laboratories which participated in the 4th IAEA interlaboratory comparison exercise and contributed their results to the success of this test are highly appreciated.

8. REFERENCES

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APPENDIX I. LISTING OF RESULTS FOR ALL FOUR SAMPLES

Table A1: Submitted $\delta^{18}O$ results from all participating laboratories for samples OH-13 to OH-16 with measurement uncertainties at the 1σ-level. Gray-shaded values are regarded as outliers according to the statistical criteria applied. Column 'ID' gives the assigned laboratory identification number; column 'Equip' indicates equipment (mass spectrometer, LGR laser analyzer, Picarro laser analyzer) used; column 'CA' states in Yes/No modus, whether a calibration with VSMOW was performed along with WICO2011 measurements; 'NS' provides the number of laboratory water standards used for daily calibration; 'AW' shows the amount of water used for a single measurement; 'ET' provides the used equilibration time for the H_2O/CO_2 exchange reaction as used where applicable; 'Stated Unc.' indicates the long term laboratory uncertainties. Other parameters provided by the laboratories are not listed in this report. The abbreviations used in the columns:
NA: not applicable, NM: not mentioned, IRMS: isotope ratio mass spectrometer, LGR: Los Gatos Research laser analyzer, Pic: Picarro laser analyzer.

Lab ID	Instrument	CA	NS	AW (μL)	ET (hr)	IAEA- OH-13 $\delta^{18}O$	IAEA- OH-13 1σ[‰]	IAEA- OH-14 $\delta^{18}O$	IAEA- OH-14 1σ[‰]	IAEA- OH-15 $\delta^{18}O$	IAEA- OH-15 1σ[‰]	IAEA- OH-16 $\delta^{18}O$	IAEA- OH-16 1σ[‰]	Stated Unc.	Remarks
Reference values						-0.96	0.04	-5.60	0.05	-9.41	0.04	-15.43	0.04		
1A	LGR	N	5	0.70	NA	-0.75	0.36	-4.96	0.18	-8.70	0.15	-15.16	0.14		
1B	IRMS	N	1	1000	25	-0.92	0.05	-5.89	0.11	-9.27	0.14	-15.72	0.14	0.20	
3	IRMS	NM	2	0.10	NM	-1.02	0.04	-5.57	0.05	-9.28	0.01	-15.33	0.05	0.15	
4	LGR	NM	1	NM	NA	-1.58	0.21	-5.46	0.17	-9.08	0.18	-13.57	0.12		
5	IRMS	Y	1	1000	NM	-1.01	0.07	-5.56	0.09	-9.31	0.05	-15.25	0.08		
6A	IRMS	Y	3	5000	10	-0.98	0.07	-5.58	0.03	-9.39	0.03	-15.42	0.02		
6B	PIC	Y	NM	1.75	NA	-0.89	0.11	-5.58	0.08	-9.27	0.17	-15.55	0.07		
7	IRMS	Y	2	3000	4	-1.13	0.09	-5.70	0.10	-9.53	0.10	-15.58	0.12		
8	IRMS	N	3	400	4	-1.09	0.29	-5.31	0.23	-9.26	0.25	-15.27	0.29	0.15	
9	IRMS	Y	3	0.70	NM	-0.90	0.29	-5.65	0.29	-9.65	0.41	-15.32	0.29		
11	LGR	NM	6	1000	NA	-0.88	0.06	-5.56	0.05	-9.37	0.05	-15.50	0.04	0.15	
13A	IRMS	Y	3	0.50	NM	-1.16	0.40	-5.53	0.34	-9.33	0.31	-15.28	0.46		
13B	IRMS	Y	3	0.50	18	-1.04	0.15	-5.61	0.13	-9.36	0.13	-15.37	0.25		

Lab ID	Instrument	CA	NS	AW (µL)	ET (hr)	IAEA- OH-13		IAEA- OH-14		IAEA- OH-15		IAEA- OH-16		Stated Unc.	Remarks
						$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]		
Reference values						-0.96	0.04	-5.60	0.05	-9.41	0.04	-15.43	0.04		
15A	IRMS	NM	4	5000	5	-0.93	0.02	-5.56	0.05	-9.36	0.04	-15.34	0.02		
15B	PIC	NM	NM	NM	NA	-0.96	0.05	-5.52	0.04	-9.32	0.06	-15.41	0.10		
19	LGR	N	3	0.97	NA	-0.98	0.06	-5.73	0.10	-9.51	0.08	-15.59	0.14	0.18	
22	LGR	N	2	NM	NA	-1.03	0.21	-5.69	0.19	-9.50	0.09	-15.66	0.13		
24	IRMS	Y	3	1.00	NA	-1.46	0.20	-6.06	0.21	-9.79	0.21	-15.70	0.22		
25A	PIC	Y	3	2.00	NA	-0.92	0.05	-5.63	0.05	-9.41	0.03	-15.50	0.04		
25B	IRMS	Y	3	NM	18	-0.97	0.04	-5.55	0.03	-9.38	0.02	-15.48	0.02		
26	IRMS	NM	3	2000	18	-0.88	0.07	-5.29	0.07	-9.27	0.05	-15.28	0.07		
27A	IRMS	N	3	200	18	-1.10	0.08	-5.52	0.05	-9.44	0.04	-15.54	0.14		
28	IRMS	Y	2	NM	NA	-0.83	0.20	-5.53	0.25	-9.35	0.26	-15.50	0.19		
29	IRMS	NM	NM	1.00	NA	-1.08	0.14	-5.84	0.12	-9.59	0.13	-15.65	0.15		
30	LGR	NM	3	NM	NA	-0.91	0.06	-5.62	0.11	-9.46	0.07	-15.46	0.15		
31	PIC	Y	NM	NM	NA	-1.07	0.11	-5.71	0.11	-9.52	0.16	-15.55	0.11		
34	IRMS	Y	3	300	20	-0.98	0.10	-5.51	0.11	-9.41	0.11	-15.42	0.11		
35	PIC	NM	2	10	NA	-1.25	0.33	-5.82	0.23	-9.55	0.28	-15.65	0.28	0.39	
36	PIC	Y	3	1.00	NA	-1.03	0.02	-5.60	0.02	-9.38	0.04	-15.38	0.02	0.20	
37	IRMS	Y	2	0.15	NA	-1.30	0.15	-5.80	0.21	-10.00	0.13	-16.00	0.14	0.33	
38A	PIC	N	3	1.80	NA	-0.98	0.08	-5.65	0.15	-9.55	0.09	-15.55	0.09		
38B	IRMS	N	3	0.50	18	-1.11	0.03	-5.72	0.09	-9.55	0.05	-15.64	0.02		
39	IRMS	Y	3	200	34	-1.11	0.07	-5.61	0.08	-9.58	0.09	-15.76	0.06		
40	LGR	Y	2	1.00	NA	-0.68	0.03	-5.57	0.19	-9.42	0.20	-15.43	0.19		
41	PIC	Y	2	1.77	NA	-0.94	0.02	-5.59	0.14	-9.40	0.04	-15.49	0.06	0.06	
43	PIC	N	2	1.80	NA	-1.09	0.10	-5.65	0.07	-9.41	0.04	-15.42	0.06	0.08	
44	IRMS	Y	3	200	7	-1.176	0.05	-5.67	0.02	-9.47	0.03	-15.50	0.05		
45	IRMS	N	7	2500	8	-0.989	0.02	-5.64	0.01	-9.43	0.01	-15.47	0.02		

Lab ID	Instrument	CA	NS	AW (μL)	ET (hr)	IAEA- OH-13 $\delta^{18}O$	1σ[‰]	IAEA- OH-14 $\delta^{18}O$	1σ[‰]	IAEA- OH-15 $\delta^{18}O$	1σ[‰]	IAEA- OH-16 $\delta^{18}O$	1σ[‰]	Stated Unc.	Remarks
Reference values						-0.96	0.04	-5.60	0.05	-9.41	0.04	-15.43	0.04		
47A	IRMS	NM	4	4000	4	-0.970	0.04	-5.59	0.04	-9.42	0.05	-15.43	0.03	0.05	
48	IRMS	N	6	600	36	-0.94	0.05	-5.64	0.06	-9.45	0.06	-15.47	0.07		
50A	IRMS	Y	3	4000	4	-0.88	0.08	-5.53	0.06	-9.35	0.08	-15.40	0.06	0.06	
50B	PIC	NM	NM	1.70	NA	-0.86	0.03	-5.53	0.03	-9.32	0.04	-15.32	0.07	0.08	
51	LGR	N	3	1	NA	-0.86	0.06	-5.51	0.10	-9.41	0.13	-15.49	0.05		
52	IRMS	N	3	200	24	-1.03	0.02	-5.62	0.01	-9.42	0.02	-15.60	0.02	0.20	
53A	IRMS	NM	3	500	20	-0.82	0.05	-5.42	0.06	-9.22	0.08	-15.29	0.07	0.13	
53B	PIC	NM	3	10	NA	-0.77	0.08	-5.40	0.14	-9.17	0.11	-15.28	0.06	0.19	
56	PIC	NM	3	NM	NA	-0.87	0.07	-5.48	0.07	-9.32	0.06	-15.34	0.09	0.10	
57	IRMS	NM	2	500	20	-1.11	0.06	-5.63	0.04	-9.44	0.08	-15.35	0.09		
58	IRMS	Y	3	NM	NA	-0.83	0.05	-5.34	0.16	-9.13	0.16	-15.17	0.00	0.30	
59	IRMS	Y	3	200	12	-1.08	0.10	-5.65	0.09	-9.46	0.09	-15.51	0.06	0.10	
60A	IRMS	NM	2	1.00	NA	-0.89	0.00	-5.51	0.01	-9.30	0.01	-15.41	0.03		
60B	PIC	NM	2	NM	NA	-0.96	0.02	-5.68	0.12	-9.43	0.01	-15.57	0.10		
61	IRMS	Y	2	200	4	-0.74	0.20	-5.19	0.14	-8.85	0.08	-14.78	0.25		
62	IRMS	NM	1	5000	8	-0.95	0.17	-5.2	0.08	-8.94	0.22	-15.41	0.28		
63A	IRMS	N	4	500	NM	-0.93	0.06	-5.53	0.06	-9.39	0.06	-15.43	0.06		
63B	PIC	N	4	1.85	NA	-0.98	0.10	-5.61	0.09	-9.48	0.09	-15.40	0.10		
64A	IRMS	NM	2	500	24	-0.92	0.05	-5.56	0.26	-9.20	0.04	-15.20	0.06		
64B	IRMS	NM	2	0.70	NA	-0.980	0.16	-5.30	0.14	-9.04	0.18	-15.30	0.13		
66	IRMS	N	3	2000	2	-0.90	0.03	-5.52	0.06	-9.33	0.03	-15.39	0.02		
67	LGR	NM	3	1.00	NA	-0.85	0.10	-5.50	0.08	-9.41	0.08	-15.61	0.07		
69	LGR	NM	3	0.75	NA	-1.47	0.30	-5.81	0.23	-9.65	0.27	-15.56	0.21		
70	IRMS	Y	3	1.00	NA	-0.94	0.10	-5.59	0.10	-9.42	0.10	-15.54	0.10	0.10	
71	LGR	NM	NM	NM	NA	-1.02	0.13	-5.65	0.06	-9.57	0.09	-15.50	0.12		

Lab ID	Instrument	CA	NS	AW (µL)	ET (hr)	IAEA- OH-13		IAEA- OH-14		IAEA- OH-15		IAEA- OH-16		Stated Unc.	Remarks
						$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]		
Reference values						-0.96	0.04	-5.60	0.05	-9.41	0.04	-15.43	0.04		
72	IRMS	Y	3	200	4	-1.96	0.06	-6.61	0.06	-10.44	0.05	-16.40	0.06		
74A	IRMS	NM	3	NM	6	-1.00	0.03	-5.51	0.03	-9.34	0.03	-15.43	0.03	0.10	
75A	IRMS	N	3	400	24	-0.95	0.01	-5.62		-9.41		-15.50			
75B	LGR	N	3	0.92	NA	-0.91	0.09	-5.63	0.03	-9.48	0.03	-15.71	0.15		
77		NM	NM	NM	NM	-1.13	0.10	-5.90	0.10	-9.50	0.10	-15.47	0.10		
78	LGR	NM	3	NM	NA	-0.93	0.23	-5.66	0.25	-9.35	0.16	-15.51	0.15		
79	IRMS	N	2	500	18	-1.04	0.06	-5.66	0.08	-9.43	0.08	-15.44	0.05		
80A	IRMS	Y	NM	5000	12	-0.94	0.05	-5.57	0.05	-9.39	0.05	-15.46	0.05		
80B	PIC	Y	NM	1.80	NA	-1.00	0.10	-5.55	0.10	-9.39	0.10	-15.46	0.10		
81	PIC	Y	2	NM	NA	-0.89	0.14	-5.59	0.12	-9.37	0.18	-15.39	0.13		
82	LGR	N	5	0.75	NA	-0.70	0.19	-5.29	0.04	-9.15	0.15	-15.28	0.08	0.17	
84	IRMS	N	2	500	18	-1.00	0.13	-5.54	0.04	-9.47	0.04	-15.55	0.05	0.04	
86A	LGR	Y	2	NM	NA	-1.11	0.30	-5.65	0.30	-9.47	0.30	-15.51	0.30		
86B	IRMS	Y	2	500	55	-1.04	0.20	-5.64	0.20	-9.43	0.20	-15.49	0.20		
87	IRMS	Y	2	NM	24	-0.80	0.09	-5.90	0.05	-9.29	0.05	-15.34	0.06		
88A	IRMS	N	5	400	6	-1.0726	0.07	-5.58	0.07	-9.41	0.12	-15.44	0.06	0.10	
88B	IRMS	N	5	200	6	1.04	0.02	-5.70	0.02	-9.43	0.01	-15.56	0.06		
89	IRMS	N	3	200	7	-1.01	0.05	-5.60	0.02	-9.40	0.01	-15.46	0.01	0.04	
91	LGR	N	3	NM	NA	-0.84	0.12	-5.47	0.08	-9.24	0.07	-15.29	0.05		
93	IRMS	Y	3	1000	9	-1.00	0.02	-5.63	0.02	-9.41	0.02	-15.44	0.01		
95	IRMS	NM	1	NM	NA	-1.28	0.18	-5.57	0.27	-9.25	0.15	-15.11	0.26	0.16	
96	IRMS	N	2	1500	6	-0.94	0.03	-5.60	0.03	-9.38	0.04	-15.44	0.04	0.06	
98	PIC	NM	4	2.00	NA	-0.93	0.06	-5.57	0.02	-9.35	0.02	-15.44	0.07		
99	IRMS	N	2	2000	10	-1.00	0.03	-5.63	0.05	-9.46	0.15	-15.47	0.06		
100	IRMS	N	5	3000	21	-0.92	0.09	-5.55	0.07	-9.29	0.09	-15.40	0.03	0.10	

Lab ID	Instrument	CA	NS	AW (µL)	ET (hr)	IAEA- OH-13 $\delta^{18}O$	IAEA- OH-13 1σ[‰]	IAEA- OH-14 $\delta^{18}O$	IAEA- OH-14 1σ[‰]	IAEA- OH-15 $\delta^{18}O$	IAEA- OH-15 1σ[‰]	IAEA- OH-16 $\delta^{18}O$	IAEA- OH-16 1σ[‰]	Stated Unc.	Remarks
Reference values						-0.96	0.04	-5.60	0.05	-9.41	0.04	-15.43	0.04		
101	IRMS	Y	2	200	NM	-1.03	0.06	-5.64	0.04	-9.46	0.04	-15.49	0.06	0.10	
102	LGR	N	3	NM	NA	-1.59	0.09	-5.74	0.10	-9.29	0.05	-15.40	0.04	0.12	
103	IRMS	NM	5	1.00	24	-0.94	0.13	-5.62	0.09	-9.37	0.09	-15.59	0.11		
104	IRMS	Y	3	4000	9	-0.95	0.03	-5.59	0.01	-9.37	0.01	-15.42	0.02	0.02	
105	LGR	Y	3	NM	NA	-1.07	0.12	-5.74	0.08	-9.47	0.05	-15.54	0.06		
106	LGR	Y	2	NM	NA	-0.66	0.35	-5.89	0.45	-9.50	0.41	-15.31	0.19		
108	IRMS	NM	3	NM	4	-0.98	0.04	-5.58	0.05	-9.40	0.03	-15.42	0.02		
109	LGR	NM	3	NM	NA	-0.95	0.18	-5.63	0.15	-9.42	0.15	-15.41	0.13		
111	IRMS	Y	2	200	24	-0.99	0.08	-5.67	0.06	-9.51	0.03	-15.55	0.08	0.15	
112A	IRMS	N	3	3000	6	-1.05	0.06	-5.61	0.03	-9.37	0.03	-15.39	0.06		
112B	LGR	N	3	NM	NA	-1.06	0.17	-5.66	0.17	-9.40	0.14	-15.43	0.14		
113A	IRMS	Y	4	NM	18	-0.87	0.11	-5.28	0.18	-9.26	0.14	-15.36	0.20		
113B	PIC	Y	4	NM	NA	-0.96	0.02	-5.60	0.05	-9.32	0.10	-15.43	0.10		
114	LGR	Y	3	NM	NA	-1.24	0.13	-6.17	0.14	-9.91	0.11	-15.76	0.09		
116	IRMS	Y	3	4000	4	-0.98	0.14	-5.63	0.15	-9.43	0.15	-15.49	0.14	0.10	
118A	IRMS	Y	2	2000	12	-0.99	0.11	-5.63	0.14	-9.40	0.11	-15.62	0.12		
118B	PIC	NM	2	NM	NA	-1.08	0.16	-5.69	0.01	-9.52	0.03	-15.51	0.06		
119A	LGR	N	2	NM	NA	-1.02	0.28	-5.74	0.31	-9.29	0.37	-15.12	0.32		
119B	LGR	NM	NM	NM	NA			-5.06	0.42	-8.85	0.31	-15.34	0.26		
119C	LGR	NM	NM	NM	NA	-0.81	0.21	-5.47	0.12	-9.26	0.13	-15.33	0.25		
120	IRMS	NM	2	NM	12	-0.99	0.02	-5.62	0.02	-9.37	0.03	-15.43	0.01		
122	LGR	N	3	0.75	NA	-0.80	0.10	-5.60	0.30	-9.30	0.20	-15.40	0.10		
123	LGR	Y	3	1.20	NA	-0.92	0.22	-5.54	0.18	-9.48	0.19	-15.55	0.19		
125	LGR	N	3	0.75	NA	-0.78	0.06	-5.33	0.04	-9.21	0.05	-15.36	0.08	0.08	
126	IRMS	N	3	200	5	-0.92	0.12	5.37	0.06	-9.38	0.18	-15.39	0.10		

Lab ID	Instrument	CA	NS	AW (µL)	ET (hr)	IAEA- OH-13		IAEA- OH-14		IAEA- OH-15		IAEA- OH-16		Stated Unc.	Remarks
						$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]	$\delta^{18}O$	1σ[‰]		
Reference values						-0.96	0.04	-5.60	0.05	-9.41	0.04	-15.43	0.04		
127	IRMS	Y	2	3000	5	-0.78	0.10	-5.56	0.07	-9.33	0.05	-15.30	0.05		
129	LGR	NM	3	NM	NA	-0.88	0.10	-5.50	0.10	-9.40	0.08	-15.60	0.13	0.20	
130	LGR	NM	3	NM	NA	-1.23	0.09	-5.74	0.08	-9.43	0.15	-15.34	0.07	0.30	
132	LGR	N	3	1.20	NA	-0.95	0.25	-5.58	0.28	-9.34	0.33	-15.38	0.32		
133A	IRMS	N	3	0.10	NM	-1.03	0.09	-5.67	0.06	-9.58	0.07	-15.70	0.04		
133B	IRMS	NM	NM	NM	NA	0.13	0.09	-5.85	0.12	-9.86	0.06	-16.09	0.03		
135A	PIC	Y	3	2.00	NA	-0.95	0.01	-5.59	0.01	-9.37	0.01	-15.44	0.04		
135B	PIC	Y	3	2.00	NA	-0.84	0.041	-5.59	0.07	-9.35	0.01	-15.39	0.04		
136	IRMS	N	2	2000	11	-0.95	0.08	-5.61	0.06	-9.41	0.08	-15.47	0.08		
137	LGR	N	2	1.20	NA	-0.89	0.15	-5.33	0.20	-9.01	0.17	-15.06	0.23		
138	PIC	NM	NM	NM	NA	-0.88	0.11	-5.52	0.13	-9.38	0.09	-15.40	0.07		
139	IRMS	NM	2	NM	2	-1.03	0.01	-5.62	0.07	-9.38	0.04	-15.33	0.02		
140A	IRMS	NM	2	500	24	-0.91	0.14	-5.59	0.12	-9.40	0.37	-15.56	0.12	0.20	
141	IRMS	N	2	200	24	-0.96	0.04	-5.58	0.05	-9.39	0.08	-15.44	0.06	0.10	
142	LGR	N	2	NM	NA	-1.18	0.15	-5.68	0.13	-9.45	0.07	-15.47	0.33		
143	LGR	Y	3	NM	NA	-1.05	0.04	-6.53	0.05	-9.48	0.04	-16.03	0.06		
144	IRMS	N	3	1000	9	-0.96	0.02	-5.62	0.02	-9.41	0.01	-15.51	0.02		
145	LGR	NM	7	1.00	NA	-1.22	0.15	-5.76	0.11	-9.60	0.13	-15.63	0.13		
146A	IRMS	N	1	3000	24	-1.10	0.05	-5.76	0.07	-9.56	0.05	-15.61	0.02	0.10	
146B	PIC	N	3	1.00	NA	-1.17	0.02	-5.62	0.10	-9.60	0.07	-15.64	0.06	0.10	
146C	PIC	N	3	1.00	NA	-1.13	0.04	-5.85	0.11	-9.67	0.15	-15.71	0.05	0.10	
147A	IRMS	N	3	5000	10	-0.96	0.03	-5.60	0.02	-9.40	0.02	-15.45	0.03		
147B	PIC	N	3	2.00	NA	-1.01	0.06	-5.56	0.05	-9.40	0.12	-15.46	0.12		
148	IRMS	N	2	5000	10	-1.00	0.03	-5.62	0.08	-9.42	0.12	-15.50	0.04	0.10	
149A	PIC	Y	3	1.80	NA	-0.95	0.03	-5.57	0.02	-9.36	0.02	-15.41	0.03		
149B	IRMS	Y	3	3000	4	-0.97	0.02	-5.59	0.02	-9.36	0.03	-15.43	0.02		

Lab ID	Instrument	CA	NS	AW (µL)	ET (hr)	IAEA- OH-13 $\delta^{18}O$	1σ[‰]	IAEA- OH-14 $\delta^{18}O$	1σ[‰]	IAEA- OH-15 $\delta^{18}O$	1σ[‰]	IAEA- OH-16 $\delta^{18}O$	1σ[‰]	Stated Unc.	Remarks
Reference values						-0.96	0.04	-5.60	0.05	-9.41	0.04	-15.43	0.04		
152	IRMS	NM	3	1000	8	-0.91	0.06	-5.61	0.06	-9.45	0.10	-15.47	0.07		
153	IRMS	Y	3	400	7	-0.98	0.07	-5.52	0.08	-9.39	0.06	-15.40	0.04		
154	IRMS	NM	3	500	20	-1.00	0.10	-5.48	0.10	-9.13	0.15	-15.15	0.15		
155A	IRMS	Y	3	4000	NM	-0.96	0.04	-5.59	0.05	-9.37	0.04	-15.41	0.04	0.01	
155B	PIC	Y	3	1.00	NA	-1.05	0.13	-5.54	0.07	-9.34	0.06	-15.40	0.07		
155C	LGR	Y	3	0.75	NA	-1.05	0.16	-5.53	0.13	-9.37	0.12	-15.41	0.14		
155D	LGR	Y	3	0.75	NA	-0.78	0.18	-5.37	0.22	-9.14	0.21	-15.27	0.22		
155E	LGR	Y	3	0.75	NA	-0.74	0.29	-5.40	0.31	-9.20	0.21	-15.19	0.24		
156	IRMS	NM	2	1.00	NM	-1.28	0.11	-5.88	0.11	-9.77	0.05	-15.91	0.13		
157	IRMS	Y	1	NM	15	-1.00	0.16	-5.77	0.09	-9.65	0.09	-15.71	0.05	0.10	
159	IRMS	Y	2	NM	18	-0.88	0.10	-5.24	0.01	-9.26	0.06	-15.24	0.10		
160	PIC	Y	10	1.00	NA	-1.00	0.04	-5.63	0.05	-9.45	0.08	-15.46	0.04		
161	LGR	Y	3	NM	NA	-0.88	0.01	-5.51	0.02	-9.34	0.03	-15.41	0.01	0.04	
162	IRMS	N	4	200	5	-1.00	0.17	-5.50	0.21	-9.40	0.13	-15.30	0.10	0.20	
165	PIC	NM	3	NM	NA	-1.00	0.10	-5.59	0.03	-9.40	0.05	-15.46	0.05		
168	IRMS	Y	4	5000	4	-1.05	0.02	-5.59	0.05	-9.38	0.04	-15.40	0.05		
170	PIC	N	3	1.70	NA	-0.86	0.09	-5.48	0.13	-9.32	0.03	-15.36	0.10		
172	PIC	Y	3	1.70	NA	-1.25	0.14	-5.83	0.15	-9.63	0.15	-15.63	0.09		
173	IRMS	N	3	200	10	-0.61	0.06	-5.26	0.17	-9.15	0.14	-15.30	0.15		
175	LGR	NM	3	1.20	NA	-3.34	0.65	-7.64	0.57	-11.27	0.61	-17.21	0.70		
177	LGR	NM	3	NM	NA	-1.08	0.22	-5.68	0.20	-9.41	0.22	-15.57	0.19		
178	PIC	NM	3	NM	NA	-0.86	0.06	-5.51	0.05	-9.34	0.06	-15.33	0.08		
180	PIC	NM	NM	1.10	NA	-2.01	0.09	-6.69	0.04	-10.47	0.05	-16.52	0.03		Manual Injection
181	LGR	NM	3	NM	NA	-0.70	0.09	-5.69	0.25	-9.27	0.20	-15.25	0.12	0.30	
182A	IRMS	Y	2	5000	5	-1.21	0.04	-5.64	0.04	-9.53	0.31	-15.31	0.01	0.20	

Lab ID	Instrument	CA	NS	AW (µL)	ET (hr)	IAEA- OH-13 δ^{18}O	1σ[‰]	IAEA- OH-14 δ^{18}O	1σ[‰]	IAEA- OH-15 δ^{18}O	1σ[‰]	IAEA- OH-16 δ^{18}O	1σ[‰]	Stated Unc.	Remarks
Reference values						-0.96	0.04	-5.60	0.05	-9.41	0.04	-15.43	0.04		
182B	LGR	Y	2	0.75	NA	-1.01	0.03	-5.65	0.09	-9.41	0.07	-15.54	0.11	0.20	
183	PIC	Y	NM	1.80	NA	-0.91	0.06	-5.64	0.07	-9.43	0.05	-15.47	0.08		
185	IRMS	NM	2	500	16	-0.99	0.07	-5.26	0.09	-9.16	0.04	-15.11	0.12		

Table A2: Submitted δ^{2}H results from all participating laboratories for samples OH-13 to OH-16 with measurement uncertainties at the 1σ-level. Gray-shaded values are regarded as outliers according to the statistical criteria applied. Column 'ID' gives the assigned laboratory identification number; column 'Equip' indicates equipment (mass spectrometer, LGR laser analyzer, Picarro laser analyzer) used; column 'CA' states in Yes/No modus, whether a calibration with VSMOW was performed along with WICO2011 measurements; 'Method' provides the indication of the sample preparation: e.g. reduction methods (Zn for Zinc, Cr for Chromium), pyrolysis (Pyro), equilibration of H2 gas with Pt-catalyst; the column 'NS' provides the number of laboratory water standards used for daily calibration; 'AW' shows the amount of water used for a single measurement; ET' provides the used equilibration time for the H_2O/H_2 exchange reaction where applicable; 'Stated Unc' indicates the long term uncertainties. Other parameters provided by the laboratories are not listed in this report. The abbreviations used in the columns are: NA: not applicable, NM: not mentioned, IRMS: isotope ratio mass spectrometer, LGR: Los Gatos Research laser analyzer, Pic: Picarro laser analyzer.

Lab ID	Equip.	CA	Method	NS	AW (µL)	ET (hr)	IAEA-OH-13 δ^{2}H	1σ[‰]	IAEA-OH-14 δ^{2}H	1σ[‰]	IAEA-OH-15 δ^{2}H	1σ[‰]	IAEA-OH-16 δ^{2}H	1σ[‰]	Stated Unc.	
Reference values							-2.84	0.60	-38.30	0.36	-78.26	0.38	-114.62	0.43		
1a	LGR	N	NA	5	0.70	NA	-1.87	0.61	-34.21	0.28	-75.29	0.45	-113.33	0.61		
3	IRMS	NM		2	0.10	NM	-2.44	0.48	-37.58	0.33	-77.51	0.31	-113.07	0.43	0.60	
4	LGR	NM	NA	3	NM	NA	-5.88	1.37	-40.48	1.40	-78.10	1.73	-101.90	1.79		
5	IRMS	Y	Pt	1	1000	NM	-2.35	0.86	-38.20	0.68	-78.41	0.86	-114.34	0.85		
6a	IRMS	Y		3	5000	2.00	-3.66	1.50	-38.88	1.10	-79.86	1.60	-115.86	1.90		
6b	PIC	Y	NA		1.75	NA	-3.60	0.6	-38.16	0.20	-77.17	0.30	-101.90	0.50		

Lab ID	Equip.	CA	Method	NS	AW (μL)	ET (hr)	IAEA-OH-13 δ²H	IAEA-OH-13 1σ[‰]	IAEA-OH-14 δ²H	IAEA-OH-14 1σ[‰]	IAEA-OH-15 δ²H	IAEA-OH-15 1σ[‰]	IAEA-OH-16 δ²H	IAEA-OH-16 1σ[‰]	Stated Unc.	
Reference values							-2.84	0.60	-38.30	0.36	-78.26	0.38	-114.62	0.43		
7	IRMS	Y		2	3000	1.45	-3.40	2.2	-41.50	2.50	-79.60	2.40	-116.6	1.8		
8	IRMS	N		3	400	1.50	-2.88	0.93	-37.99	-1.04	-78.70	0.86	-115.25	0.96	0.50	
9	IRMS	Y			0.70	NM	-2.50	1.08	-37.01	1.11	-75.82	0.95	-113.81	0.82		
11	LGR	NM	NA	6	1000	NA	-2.93	0.35	-38.93	0.48	-78.23	0.35	-114.56	0.15	1.00	
13a	IRMS	Y		3	0.50	NM	-1.74	1.16	-36.82	1.14	-77.13	0.96	-115.16	1.13		
15a	IRMS	NM	Cr	3	5000	0.05	-3.63	0.21	-38.53	0.33	-78.17	0.31	-114.10	0.50		
15b	PIC	NM	NA		NM	NA	-3.93	0.243	-38.68	0.37	-78.50	0.36	-114.43	0.37		
19	LGR	N	NA	3	0.97	NA	-1.60	0.34	-37.80	0.46	-78.95	0.60	-116.30	0.53	1.00	
22	LGR	N	NA		NM	NA	0.42	1.46	-37.47	2.57	-79.83	1.07	-116.38	0.13		
24	IRMS	Y		3	1.00	NM	4.92	0.34	-39.74	0.22	-79.77	0.33	-114.74	0.39		
25a	PIC	Y	NA	3	2.00	NA	-4.50	0.20	-40.10	0.20	-80.40	0.20	-116.80	0.10		
25b	IRMS	Y		3	NM	18.00	-3.10	0.40	-37.30	0.80	-78.40	0.80	-114.70	1.00		
26	IRMS	NM	Cr	3	2000	0.02	-2.96	0.52	-40.92	0.68	-82.15	0.42	-120.54	0.46		
27a	IRMS	N		3	200	4.00	-3.60	2.10	-40.10	1.60	-79.90	1.90	-114.40	2.50		
27b	IRMS	NM		3	200	4.00	-4.80	3.00	-41.40	1.60	-82.00	2.30	-115.50	2.30		
28	IRMS	Y		2	NM	NM	-2.70	1.20	-39.20	1.20	-79.50	2.00	-116.70	1.50		
29	IRMS	NM			1.00	NM	-2.41	0.74	-38.34	0.81	-78.40	0.82	-115.94	0.72		
30	LGR	NM	NA		NM	NA	-2.85	0.17	-38.44	0.27	-78.60	0.26	-114.98	0.21		
31	PIC	Y	NA		NM	NA	-4.51	1.60	-38.51	1.74	-78.65	2.53	-115.60	1.11		
34	IRMS	Y		3	300	6.00	-2.55	1.67	-38.17	1.75	-78.99	1.75	-115.36	1.99		
35	PIC	NM	NA	2	10.00	NA	-4.40	1.80	-37.30	1.50	-77.10	1.60	-114.10	1.20	1.46	
36	PIC	Y	NA		1.00	NA	-4.40	0.67	-38.70	0.40	-78.50	0.25	-114.90	0.27	0.60	
37	IRMS	Y	Pyro	2	0.15	NA	-6.60	0.80	-43.90	0.72	-82.70	0.66	-117.80	1.41	2.00	
38a	PIC	N	NA	3	1.80	NA	-2.41	0.14	-38.52	0.12	-79.43	0.37	-116.31	0.39		
38b	IRMS	N		3	0.10	NM	-1.01	0.52	-37.52	0.55	-78.45	0.59	-115.81	0.58		
39	IRMS	Y		3	200	34.00	-2.93	0.57	-39.16	0.61	-76.11	2.86	-116.11	0.77		

Lab ID	Equip.	CA	Method	NS	AW (µL)	ET (hr)	IAEA-OH-13 δ^2H	IAEA-OH-13 1σ[‰]	IAEA-OH-14 δ^2H	IAEA-OH-14 1σ[‰]	IAEA-OH-15 δ^2H	IAEA-OH-15 1σ[‰]	IAEA-OH-16 δ^2H	IAEA-OH-16 1σ[‰]	Stated Unc.	
Reference values							-2.84	0.60	-38.30	0.36	-78.26	0.38	-114.62	0.43		
40	LGR	Y	NA	2	1.00	NA	-3.25	0.75	-38.29	0.68	-78.65	1.82	-115.25	1.37		
41	PIC	Y	NA	2	1.77	NA	-1.70	0.60	-37.20	0.40	-77.90	0.20	-114.80	0.50	0.30	
43	PIC	N		2	1.80	NM	-3.58	0.42	-38.27	0.10	-77.78	0.28	-113.96	0.21	0.33	
44	IRMS	Y	Cr		0.30	NM	-3.56	0.20	-38.67	0.50	-79.92	0.95	-114.79	0.18		
45	IRMS	N	Cr	7	0.50	NM	-2.77	0.24	-38.37	0.14	-78.07	0.12	-114.82	0.19		
47a	IRMS	NM	Cr	4		NM	-2.04	0.74	-37.49	0.76	-77.60	0.63	-114.02	0.74	0.80	
47b	PIC	NM	NA			NA	-2.11	0.30	-36.85	0.40	-77.22	0.39	-114.15	0.43	0.60	
50a	IRMS	Y	Pt	3	4000	0.75	-2.45	0.16	-38.77	0.22	-78.47	0.20	-114.82	0.18	0.60	
50b	PIC	NM	NA	3	1.70	NA	-1.84	0.27	-37.22	0.25	-77.71	0.20	-113.77	0.12	0.60	
51	LGR	N	NA	3	1.00	NA	-2.20	0.40	-39.70	0.50	-78.80	0.20	-115.70	0.30		
52	IRMS	N		3	0.20	48.00	-1.84	0.65	-38.92	0.52	-78.66	0.73	-115.06	0.72	3.00	
53a	IRMS	NM	Cr	3	500	NM	-3.20	0.25	-38.60	0.26	-78.50	0.32	-114.50	0.38	0.90	
53b	PIC	NM	NA	3	10.00	NA	-2.60	1.49	-37.80	1.07	-77.60	0.84	-113.70	0.52	0.80	
56	PIC	NM	NA	3		NA	-2.30	0.30	-37.40	0.30	-77.60	0.50	-114.10	0.50	0.50	
57	IRMS	NM	Pyro	2	0.40	20.00	-2.80	0.80	-37.40	0.90	-77.50	1.02	-113.00	0.51		
58	IRMS	Y	Pyro	3		NM	-0.80	0.65	-37.10	1.06	-76.10	0.78	-113.40	0.02	3.00	
59	IRMS	Y	Zn	3	8.00	30.00	-4.26	0.57	-40.22	0.50	-80.34	0.48	-116.02	1.00	1.00	
60a	IRMS	NM		2	1.00	NM	-2.59	0.28	-37.92	0.66	-78.35	0.45	-114.66	0.40		
60b	PIC	NM	NA	2		NA	-2.82	0.06	-37.87	0.19	-78.18	0.51	-113.63	0.54		
61	IRMS	Y	Cr	2	0.90	NM	-4.30	0.70	-40.20	1.00	-79.50	0.50	-115.80	0.70		
62	IRMS	NM	Pt	1	5000	1.00	-6.08	1.05	-38.25	1.79	-79.72	1.13	-115.23	1.69		
63a	IRMS	N	Cr	4	1.20	NM	-2.13	0.98	-37.74	0.83	-77.91	1.06	-114.16	0.77		
63b	PIC	N	NA	4	1.85	NA	-2.25	1.07	-37.86	0.92	-78.02	0.81	-114.48	0.78		
64a	IRMS	NM	Pyro	2	500	24.00	-5.40	3.90	-41.50	1.20	-78.60	1.80	-115.50	1.40		
64b	IRMS	NM		2	0.70	NM	-4.30	0.90	-38.30	0.90	-78.10	0.40	-115.20	0.80		
66	IRMS	Y	Zn	3	10	NA	-1.30	0.46	-37.76	0.53	-77.23	0.78	-114.05	0.34		

Lab ID	Equip.	CA	Method	NS	AW (µL)	ET (hr)	IAEA-OH-13 δ^2H	IAEA-OH-13 1σ[‰]	IAEA-OH-14 δ^2H	IAEA-OH-14 1σ[‰]	IAEA-OH-15 δ^2H	IAEA-OH-15 1σ[‰]	IAEA-OH-16 δ^2H	IAEA-OH-16 1σ[‰]	Stated Unc.	
Reference values							-2.84	0.60	-38.30	0.36	-78.26	0.38	-114.62	0.43		
67	LGR	NM	NA	3	1.00	NA	-0.71	0.42	-37.11	0.66	-78.88	0.68	-117.27	0.63		
69	LGR	NM	NA	3	0.75	NA	-4.00	2.00	-40.10	1.80	-78.80	1.50	-114.40	1.80		
70	IRMS	Y	Pyro	3	1.00	NM	-5.80	0.70	-38.90	0.70	-78.70	0.70	-115.30	0.70	0.70	
71	LGR	NM	NA			NA	-3.28	0.77	-38.82	0.48	-78.69	0.28	-115.17	0.49		
72	IRMS	Y	Cr	3		NM	-4.70	2.60	-39.80	2.70	-79.80	2.50	-115.80	2.80		
74a	IRMS	NM		3		4.00	-2.10	0.39	-40.10	0.35	-80.80	0.64	-115.00	1.13	1.00	
74b	IRMS	NM		3	2.00	NM	-4.00	0.10	-39.20	1.22	-79.90	1.16	-115.70	0.90	2.00	
75a	IRMS	N	Cr	3	0.50	NM	-1.30		-37.20	0.30	-77.80	0.00	-115.90	0.40		
75b	LGR	N	NA	3		NA	-1.30	0.10	-37.00	0.10	-78.40	0.00	-115.90	0.50		
78	LGR	NM	NA	3		NA	-3.00	0.90	-38.40	0.70	-79.70	0.60	-116.60	0.60		
79	IRMS	N	EA	2	0.20	NM	-3.70	0.74	-38.60	0.79	-75.40	0.62	-115.00	0.42		
80a	IRMS	Y		2		6.00	-2.86	1.25	-38.44	1.25	-77.86	1.25	-114.34	1.25		
80b	PIC	Y	NA	2	1.80	NA	-2.63	0.78	-38.22	0.78	-78.64	0.78	-115.45	0.78		
81	PIC	Y	NA	2		NA	-1.10	1.10	-38.60	1.20	-77.10	1.00	-115.80	0.50		
82	LGR	N	NA	5	0.75	NA	-3.30	1.00	-38.50	0.90	-78.40	0.40	-114.80	0.60	0.60	
84	IRMS	N	Cr	2	1.00	NM	-2.20	0.22	-38.10	0.41	-78.30	0.24	-114.60	0.23	0.30	
86a	LGR	Y	NA	2		NA	-3.20	2.00	-37.70	2.00	-77.40	2.00	-113.80	2.00		
87	IRMS	Y	Pt	2		1.00	-1.78	0.79	-38.29	0.69	-77.07	0.92	-114.32	0.91		
88a	IRMS	N	Cr	5		NM	-2.80	0.36	-38.50	0.18	-79.10	0.39	-115.10	0.10	0.80	
89	IRMS	N	Pt	3		4.00	-2.80	0.50	-38.80	0.40	-78.30	0.60	-114.60	0.60	1.05	
90	IRMS	NM	Cr	2	1.00	NM	-2.20		-38.40		-78.40		-114.20			
91	LGR	N	NA	3		NA	0.19	0.05	-37.92	0.13	-78.09	0.21	-114.82	0.20		
93	IRMS	Y		3	1000	1.00	-2.80	0.60	-38.90	0.20	-78.20	0.30	-114.60	0.50		
95	IRMS	NM	Pyro	1		NM	-3.60	0.77	-37.30	0.60	-75.89	0.87	-111.39	1.03	0.65	
96	IRMS	N	Cr	2	0.50	NM	-3.04	0.60	-38.83	0.40	-78.99	0.20	-115.29	0.30	0.70	
98	PIC	NM	NA	4	2.00	NA	-2.59	0.11	-38.26	0.13	-78.34	0.11	-114.59	0.11		

Lab ID	Equip.	CA	Method	NS	AW (µL)	ET (hr)	IAEA-OH-13 δ²H	1σ[‰]	IAEA-OH-14 δ²H	1σ[‰]	IAEA-OH-15 δ²H	1σ[‰]	IAEA-OH-16 δ²H	1σ[‰]	Stated Unc.	
Reference values							-2.84	0.60	-38.30	0.36	-78.26	0.38	-114.62	0.43		
99	IRMS	N	Pt	2	2000	1.40	-4.33	0.29	-40.12	0.21	-79.70	0.34	-115.79	0.23		
100	IRMS	N		5	3000	21.00	-3.50	0.35	-38.90	0.17	-78.90	0.45	-115.30	0.22	1.00	
101	IRMS	Y		2	200	NM	-2.44	0.88	-38.53	0.58	-78.44	0.60	-114.90	0.80	1.00	
102	LGR	N	NA	3		NA	-1.40	0.41	-37.80	0.62	-78.70	0.71	-114.80	0.74	0.92	
104	IRMS	Y	Pt	3	4000	2.00	-3.21	0.34	-38.52	0.47	-78.47	0.52	-114.83	0.62	0.50	
105	LGR	Y	NA	3		NA	-3.50	0.48	-39.40	0.41	-79.10	0.23	-115.10	0.44		
106	LGR	Y	NA	2		NA	-1.50	0.67	-38.50	0.29	-77.50	0.41	-115.40	0.98		
108	IRMS	NM		3		3.00	-2.91	0.31	-38.66	0.33	-78.94	0.47	-115.16	0.43		
109	LGR	NM	NA	3		NA	-2.21	1.56	-37.88	1.16	-78.29	1.16	-114.53	1.04		
111	IRMS	Y		2	200	24.00	-2.90	2.46	-38.70	4.04	-80.30	3.02	-115.60	2.88	2.00	
112a	IRMS	N		3	3000	3.00	-3.59	0.60	-38.85	0.30	-78.36	0.31	-113.86	0.50		
112b	LGR	N	NA	3		NA	-3.12	0.51	-38.75	0.47	-78.38	0.64	-113.87	0.88		
113A	IRMS	Y	Pt	4		1.00	-3.87	0.57	-39.80	0.64	-80.09	0.71	-117.40	0.54		
113b	PIC	Y	NA	4		NA	-2.36	0.42	-38.34	0.37	-79.23	0.39	-116.32	0.35		
114	LGR	Y	NA	3		NA	-3.20	0.70	-39.70	0.70	-80.60	0.70	-117.20	0.80		
116	IRMS	Y		3	4000	1.50	-4.00	1.20	-39.35	1.30	-79.50	1.30	-115.40	1.50	1.00	
118a	IRMS	Y	Zn	2	2.00	NM	-0.40	0.73	-34.60	0.87	-76.80	0.70	-113.80	0.46		
118b	PIC	NM	NA	2	NM	NA	-4.10	0.01	-39.40	0.99	-79.30	0.88	-115.60	0.58		
119a	LGR	N	NA	2	NM	NA	-2.40	0.68	-37.40	1.14	-77.84	1.78	-113.40	1.50		
119b	LGR	NM	NA		NM	NA			-37.10	1.20	-77.21	1.04	-114.20	0.50		
119c	LGR	NM	NA		NM	NA	-2.60	0.39	-38.30	0.58	-78.44	0.27	-115.10	0.10		
120	IRMS	NM			NM	NM	-1.92	0.34	-38.08	0.06	-78.58	0.38	-115.69	0.04		
122	LGR	N	NA	3	0.75	NA	-2.10	0.91	-38.74	0.87	-79.51	1.09	-116.16	0.61		
123	LGR	Y	NA	3	1.20	NA	-2.84	0.86	-38.68	0.73	-78.22	0.48	-115.29	0.61		
125	LGR	N	NA	3	NM	NA	-0.29	0.32	-33.69	0.29	-74.82	0.16	-112.97	0.48	1.00	
126	IRMS	N		3	200	1.50	-0.90	2.00	-36.90	1.00	-76.50	2.10	-113.20	1.30		

Lab ID	Equip.	CA	Method	NS	AW (µL)	ET (hr)	IAEA-OH-13 δ²H	IAEA-OH-13 1σ[‰]	IAEA-OH-14 δ²H	IAEA-OH-14 1σ[‰]	IAEA-OH-15 δ²H	IAEA-OH-15 1σ[‰]	IAEA-OH-16 δ²H	IAEA-OH-16 1σ[‰]	Stated Unc.	
Reference values							-2.84	0.60	-38.30	0.36	-78.26	0.38	-114.62	0.43		
127	IRMS	Y		2	3000	3.00	-2.01	0.58	-38.64	0.77	-78.41	0.65	-114.66	0.33		
129	LGR	NM	NA	3	NM	NA	-3.41	0.31	-38.42	0.32	-78.29	0.40	-114.93	0.64	1.00	
130	LGR	NM	NA	3	NM	NA	-2.59	0.39	-39.35	0.74	-79.22	1.08	-117.16	0.49	1.00	
132	LGR	N	NA	3	1.20	NA	-2.84	1.22	-37.97	0.97	-78.07	0.79	-114.73	0.83		
133a	IRMS	N	Pyro	3	0.10	NM	-1.20	0.73	-39.20	0.45	-80.80	0.87	-119.50	0.44		
133b	IRMS	NM			NM	NM	-1.20	0.38	-39.70	0.43	-79.60	0.33	-116.40	0.36		
135a	PIC	Y	NA	3	2.00	NA	-2.7	0.15	-38.3	0.02	-78.4	0.15	-114.7	0.27		
135b	PIC	Y	NA	3	2.00	NA	-2.8	0.09	-38.4	0.02	-78.4	0.15	-114.7	0.53		
136	IRMS	N		2	2000	3.00	-2.50	0.44	-38.30	0.43	-78.20	0.30	-114.30	0.22		
137	LGR	N	NA	2	1.20	NA	-2.41	0.96	-37.13	0.96	-76.38	0.79	-113.14	0.47		
138	PIC	NM	NA		NM	NA	-1.90	0.20	-37.30	0.20	-77.40	0.30	-113.90	0.50		
139	IRMS	NM		2	NM	1.00	-1.70	0.06	37.60	0.15	-78.30	0.26	-115.50	0.44		
140b	PIC	NM	NA	2	1.00	NA	-2.70	0.08	-39.10	0.43	-78.60	0.22	-114.50	0.39	2.00	
141	IRMS	N	Pt	2	200	12.00	-3.60	1.43	-39.20	1.22	-78.50	0.73	-115.30	0.98	1.50	
142	LGR	N	NA	2	NM	NA	-3.50	1.10	-38.41	0.67	-78.12	0.61	-115.00	1.39		
143	LGR	Y	NA	3	NM	NA	-2.40	0.17	-38.63	0.24	-80.32	0.21	-118.40	0.29		
144	IRMS	N		3	1000	9.00	-2.04	0.32	-37.83	0.30	-77.64	0.49	-113.89	0.27		
145	LGR	NM	NA	7	1.00	NA	-2.90	0.73	-39.80	0.70	-80.30	0.40	-117.20	0.20		
146a	IRMS	N		1	3000	1.00	-1.92	0.61	-37.79	0.48	-78.34	0.45	-114.83	0.66	1.00	
146b	PIC	N	NA	3	1.00	NA	-2.27	0.11	-38.10	0.17	-78.27	0.16	-115.09	0.20	1.00	
146c	PIC	N	NA	3	1.00	NA	-2.49	0.24	-37.83	0.38	-78.44	0.51	-114.71	0.51	1.00	
147a	IRMS	N	Pt	3	5000	2.00	-2.43	0.52	-38.20	0.43	-78.15	0.52	-114.75	0.43		
147b	PIC	N	NA	3	2000	NA	-2.65	0.82	-37.88	0.52	-78.45	0.72	-114.83	0.72		
148	IRMS	N	Cr	2	1.50	NM	-9.40	0.85	-41.25	0.49	-78.90	0.14	-113.35	0.78	1.00	
149a	PIC	Y	NA	3	1.80	NA	-2.30	0.20	-37.80	0.20	-77.90	0.30	-114.30	0.40		
152	IRMS	NM	Pt	3	1000	1.00	-2.10	0.40	-38.20	1.00	-78.90	1.10	-114.80	0.60		

Lab ID	Equip.	CA	Method	NS	AW (µL)	ET (hr)	IAEA-OH-13 δ^2H	IAEA-OH-13 1σ[‰]	IAEA-OH-14 δ^2H	IAEA-OH-14 1σ[‰]	IAEA-OH-15 δ^2H	IAEA-OH-15 1σ[‰]	IAEA-OH-16 δ^2H	IAEA-OH-16 1σ[‰]	Stated Unc.	
Reference values							-2.84	0.60	-38.30	0.36	-78.26	0.38	-114.62	0.43		
153	IRMS	Y		3	400	2.50	-3.04	0.23	-38.40	0.19	-78.30	0.18	-114.50	0.34		
154	IRMS	NM	Pt	3	200	1.00	-3.33	0.60	-39.46	1.00	-79.70	1.00	-115.95	1.40		
155a	IRMS	Y	Pt	3	4000	NM	-2.3	0.9	-37.7	0.8	-78.0	0.8	-113.8	0.9	0.21	
155b	PIC	Y	NA	3	1.00	NA	-4.17	1.16	-37.69	0.55	-77.68	0.42	-114.39	0.32		
155c	LGR	Y	NA	3	0.75	NA	-3.99	0.64	-38.68	0.55	-78.78	0.34	-115.34	0.55		
155d	LGR	Y	NA	3	0.75	NA	-3.96	0.88	-38.79	0.90	-79.11	1.29	-115.50	0.65		
155e	LGR	Y	NA	3	0.75	NA	-3.64	1.87	-39.00	2.39	-77.82	1.62	-114.22	1.42		
157	IRMS	Y	Zn	1	NM	1.00	-0.10	1.02	-37.30	0.45	-79.70	0.38	-116.00	0.25	1.02	
159	IRMS	Y	Cr	2	NM	0.10	-0.58	1.02	-35.86	0.17	-77.28	0.69	-113.97	0.42		
160	PIC	Y	NA	10	1.00	NA	-2.40	0.20	-38.50	0.50	-77.90	0.10	-114.70	0.20		
161	LGR	Y	NA	3	NM	NA	-0.07	0.02	-35.26	0.10	-75.64	0.12	-112.35	0.09	0.60	
162	IRMS	N	Cr	3	0.20	NM	-2.78	0.44	-38.61	0.66	-78.64	0.31	-115.56	0.44	0.80	
165	PIC	NM	NA	3	NM	NA	-2.61	1.79	-37.01	0.53	-77.82	0.83	-116.17	0.89		
168	IRMS	Y	Pyro	4	NM	NM	-1.40	0.50	-37.30	0.60	-77.30	0.60	-113.40	0.60		
170	PIC	N	NA	3	NM	NA	-3.28	0.65	-38.66	0.54	-78.31	0.15	-114.98	0.50		
172a	PIC	Y	NA	3	1.70	NA	-2.61	0.31	-44.33	1.72	-78.05	0.83	-116.38	0.28		
175	LGR	NM	NA	3	1.20	NA	-12.90	1.60	-46.30	0.90	-85.90	0.70	-122.70	0.70		
177	LGR	NM	NA	3	NM	NA	-3.82	0.75	-38.76	0.55	-79.17	0.40	-116.30	0.86		
178	PIC	NM	NA	3	NM	NA	-2.07	0.33	-37.29	0.32	-77.54	0.31	-114.21	0.42		
180	PIC	NM	NA	no!	1.10	NA	-7.13	0.67	-42.18	0.41	-82.77	0.26	-119.38	0.30		Manual injection
181	LGR	NM	NA	3	NM	NA	1.08	0.42	-35.50	1.08	-77.05	0.19	-114.30	0.56	1.00	
182a	IRMS	Y		2	5.00	35.00	-1.84	0.50	-37.79	0.60	-76.62	0.50	-114.71	0.40	1.00	
182b	LGR	Y	NA	2	0.75	NA	-1.54	0.40	-36.56	0.30	-76.59	0.30	-113.58	0.60	1.00	
183	PIC	Y	NA		1.80	NA	-1.80	0.08	-37.30	0.30	-78.00	0.29	-115.03	0.33		

APPENDIX II. LIST OF PARTICIPANTS

Note: This list is sorted alphabetically by country and is not related to the assigned Laboratory ID number used throughout the report.

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APPENDIX III. WICO2011 ANNOUNCEMENTS

The following announcements were made through IAEA Website www.iaea.org/water, ISOGEOCHEM News Group on Isogeochemistry and emails to all participants of the former intercomparisons.

Announcement of WICO 2011, IAEA Water Isotope Interlaboratory Comparison

The Isotope Hydrology Laboratory of the International Atomic Energy Agency is organising the fourth interlaboratory comparison for laboratories engaged in stable isotope analyses (^{18}O & ^{2}H) of water samples. The exercise is part of the IAEA Water Resources Programme to promote quality assurance and to improve the reliability and comparability of stable isotope measurements. Those willing to join the exercise should confirm their participation by sending the complete postal address of the participating institution with name of the investigator, e-mail, fax number and homepage (if available) to the Acting Unit Head of Isotope Hydrology Laboratory with the subject field "WICO 2011": *Manzoor Choudhry, Isotope Hydrology Laboratory, International Atomic Energy Agency, P.O. Box 100, Wagramerstrasse 5, A-1400 Vienna, Austria; fax:43-1-26007; e-mail: isotope.hydrology.lab@iaea.org.* Detailed plan will be provided later.

Progress in WICO 2011, IAEA Water Isotope Interlaboratory Comparison organized by the Isotope Hydrology Laboratory of Water Resources Programme

In response to our announcement, 173 laboratories have been registered to participate in the fourth interlaboratory comparison exercise on stable isotope analyses (^{18}O & ^{2}H) of water samples. The samples along with covering letter and data reporting sheet have been dispatched to all the laboratories, which are expected to arrive soon. Electronic version of the data reporting sheet has been sent to each laboratory and it can also be downloaded from our website www.iaea.org/water. Please use this data reporting sheet to submit the results for each method used for analysis. The participants are requested to send the report electronically and a hard copy to the address given below before 31st August, 2011.

Manzoor Choudhry
Isotope Hydrology Laboratory,
International Atomic Energy Agency,
P.O. Box 100, Wagramerstrasse 5,
A-1400 Vienna, Austria;
Phone: +43 1 2600 21758
Fax: +43 1 26007;
Email: isotope.hydrology.lab@iaea.org

APPENDIX IV. LABORATORY REPORTING SHEET

FOURTH IAEA INTERLABORATORY COMPARISON FOR STABLE ISOTOPE ANALYSES OF WATER

4th IAEA INTERLABORATORY COMPARISON FOR STABLE ISOTOPE ANALYSES OF WATER	
Laboratory ID	
Laboratory name	
Person in charge	
Reporting date	

Methods:

Analyte	Method	Sample preparation/equilibration	Analyzer
$\delta^{18}O$	1		
	2		
	3		
$\delta^{2}H$	1		
	2		
	3		

Remarks

Results

Sample	Method	$\delta^{18}O$			$\delta^{2}H$		
		‰	unc.	n	‰	unc.	n
OH-13	1						
	2						
	3						
OH-14	1						
	2						
	3						
OH-15	1						
	2						
	3						
OH-16	1						
	2						
	3						

Individual results

Sample	Analysis	$\delta^{18}O$			$\delta^{2}H$		
		Method 1	Method 2	Method 3	Method 1	Method 2	Method 3
OH13	1						
	2						
	3						
	4						
	5						
	6						
	7						
	8						
	9						
	10						
OH14	1						
	2						
	3						
	4						
	5						
	6						
	7						
	8						
	9						
	10						
OH15	1						
	2						
	3						
	4						
	5						
	6						
	7						
	8						
	9						
	10						
OH16	1						
	2						
	3						
	4						
	5						
	6						
	7						
	8						
	9						
	10						

Laboratory Manager................................
Date:...............................

Please send this reporting sheet as electronic version to:

Manzoor Choudhry
Team Leader, Isotope Hydrology Laboratory
International Atomic Energy Agency
P.O.Box 100 A-1400 Vienna AUSTRIA
Phone: +43-1-2600-21758/21766 Fax: +43-1-2600-7
e-mail (SMTP) : isotope.hydrology.lab@iaea.org

Explanations

Field	Information required	Example
Analyzer	Equilibration system	Brand, custom made
	Mass Spectrometer	Brand, type (dual inlet / continuous flow)
	Laser Spectroscopic Analyzer	Brand, settings (No. of injections)
Detail of sample preparation for each method	Volume of water sample used	4 mL, 10 µL
	Transfer	direct, capsule
	Reaction (when applicable)	Zinc, 2 hours at 500°C, Glassy Carbon at 1050°C
	Equilibration	4 hours at 25°C
	Separation (when applicable)	GC at 80°C
Normalization	Type of lab standards	How are the lab standards calibrated? Using IAEA VSMOW and SLAP or any other standards
	Number of Standards	1-standard or 2-standard normalisation
	Values of lab. standards	….‰ vs VSMOW normalized on VSMOW-SLAP scale
	Sequence of samples and standards	e.g. calibration standard1, 2, control, sample1,2 etc.
	Calculations method	formulas / templates
	Date of last calibration	Date the lab standards were last calibrated using VSMOW SLAP GISP
Final results	Average of n measurements	….‰ vs VSMOW (2 decimal place for $\delta^{18}O$ and 1 decimal place for $\delta^{2}H$)
Uncertainty	Long term sd of laboratory standards	….‰ (2 decimal place for $\delta^{18}O$ and 1 decimal place for $\delta^{2}H$)
	Standard deviation of individual measurements	….‰ (2 decimal place for $\delta^{18}O$ and 1 decimal place for $\delta^{2}H$)
Individual results	Results of all single measurements.	Ratio ($^{18}O/^{2}H$) vs working standard in case of IRMS
		Please attach machine raw data electronically, eg. LGR: H_2O_yyyymmdd.xxx.txt, Picarro: hbdsxx_isowater_yyyymmdd_xxxxx.csv