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^{129}I Interlaboratory Comparison: Phase I and Phase II Results

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Abstract

An interlaboratory comparison exercise for ^{129}I was organized and conducted. Nine laboratories participated in the exercise to either a full or limited extent. In Phase I of the comparison, 11 samples were measured. The suite of samples contained both synthetic ‘standard type’ materials (i.e., AgI) and environmental materials. The isotopic ^{129}I/^{127}I ratios of the samples varied from 10^{-8} to 10^{-14}. In this phase, each laboratory was responsible for its own chemical preparation of the samples. In Phase I, the ^{129}I AMS measurements for prepared AgI were in good agreement. However, large discrepancies were seen in ^{129}I AMS measurements of environmental samples.

Because of the large discrepancies seen in the Phase I ^{129}I intercomparison, a subsequent study was conducted. In Phase II of the ^{129}I intercomparison, three separate laboratories prepared AgI from two environmental samples (IAEA 375 soil and maples leaves). Each laboratory used its own chemical preparation method with each of the methods being distinctly different. The resulting six samples (two sets of three) were then re-distributed to the participating ^{129}I AMS facilities and ^{129}I/^{127}I ratios measured. Results and discussion of both the Phase I and Phase II interlaboratory comparison are presented.

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Introduction

In April 1993, at an International Atomic Energy Agency (IAEA) consultant's meeting in Vienna, several technical issues relating to environmental monitoring, sampling, and analyses for the detection of undeclared nuclear activities were raised and discussed. Accelerator Mass Spectrometry (AMS) was identified as an important high sensitivity analysis technique for the detection of ^{14}C and ^{129}I. Detection of ^{14}C using AMS is a well established technique used in a wide range of scientific applications. About thirty laboratories worldwide have an established ^{14}C AMS analysis capability. Through the efforts of the radiocarbon dating community, appropriate standards, blanks, and analysis protocols have been determined and numerous interlaboratory comparisons have been performed. Given established sample handling and preparation procedures, the precision and accuracy of a ^{14}C AMS analysis is seldom in doubt. However, this level of analytical robustness has not yet been achieved for ^{129}I AMS analysis. A very limited number of laboratories have established analysis techniques for ^{129}I AMS, and typically, each laboratory has its own independent standards and blanks by which abundance concentrations are determined.

Because of these unresolved questions about ^{129}I AMS measurements, Lawrence Livermore National Laboratory (LLNL) sponsored an ^{129}I intercomparison exercise. The purpose of the ^{129}I intercomparison exercise was to assess the suitability and effectiveness of ^{129}I AMS measurements for possible safeguards use (since ^{129}I is a potential tracer of reactor or reprocessing operations). To maximize the utility of the intercomparison exercise, the sample suite necessarily included materials expected in a typical field trial or inspection. Accordingly the sample suite included swipes, filters, soils, grasses, lichens or moss, deciduous leaves, tree bark, pine needles, sediments, water, and water biota (e.g., algae, mussels, plants).

Nine laboratories participated in the exercise to either a full or limited extent. Laboratories that participated in the exercise are listed in the acknowledgments.

Phase I Samples

In Phase I of the ^{129}I intercomparison exercise, the specific samples were:

Sample #1: Prepared AgI. ^{129}I/^{127}I ratio calculated to be 90308 x 10^{-15}.
Sample #2: Prepared AgI. ^{129}I/^{127}I ratio calculated to be 45474 x 10^{-15}.
Sample #3: Prepared AgI. ^{129}I/^{127}I ratio calculated to be 21729 x 10^{-15}.
Sample #4: Prepared AgI. ^{129}I/^{127}I ratio calculated to be 4922 x 10^{-15}.
Sample #5: Water sample. ^{129}I/^{127}I ratio unknown but less than 10^{-10}.
Sample #6: Spiked swipe. A Whatman filter paper spiked with ^{129}I.
Sample #7: Pine needles. ^{129}I/^{127}I ratio unknown but less than 10^{-10}.
Sample #8: Maple leaves. ^{129}I/^{127}I ratio unknown but less than 10^{-8}.
Sample #9: Dried seaweed. ^{129}I/^{127}I ratio unknown but less than 10^{-8}.
Sample #10: Soil. ^{129}I/^{127}I ratio unknown but less than 10^{-10}.
Sample #11: Woodward Iodine. ^{129}I/^{127}I ratio approximately 50 x 10^{-15}.

The first three AgI samples had ^{129}I/^{127}I ratios readily measured by the AMS technique. These three samples provided some statistically meaningful intercomparison of the AMS technique at the various participating laboratories. The fourth AgI sample had an ^{129}I/^{127}I ratio much lower than the first three AgI samples and provided a ‘low-level’ intercomparison sample. The Woodward iodine sample was included to help in the determination of backgrounds. All prepared AgI samples were precipitated from a bulk solution that was derived by successive dilutions of a National Institute of Standards and Technology (NIST) standard material. The $^{129}I/^{127}I$ ratio of the original NIST standard material was 0.4091.

The water sample was created using de-ionized purified water. Iodine was added to the water in the form of potassium iodide and, to prevent loss of iodide, both NaOH and sodium bisulfite were added. The iodine concentration for the sample was approximately 250 μg I /g of H_2O.

The remaining five samples on the list were included to represent environmental samples of the type that the IAEA would typically collect. The swipe sample was a Whatman filter paper spiked with a known amount of ^{129}I. The pine needle and soil samples were collected in the vicinity of LLNL and were believed to have ^{129}I concentrations such that the high sensitivity of the AMS technique is truly required to obtain isotopic abundances. The seaweed and maple leaf samples were not collected locally to the LLNL area and possessed sufficiently high ^{129}I concentrations such that measurement by both thermal emission mass spectrometry and AMS was possible. The seaweed, maple leaf, pine needle, and soil samples provided to the participating laboratories were representative aliquots from a larger supply of sample material. To ensure homogeneity of the samples, each sample was extensively ground and mixed. All samples were prepared identically, at the same time, and under the same conditions.

It is important to note that the environmental samples included in the ^{129}I intercomparison exercise were not intended to become 'NIST type' environmental standards. Our main intent with the chosen set of environmental samples was to determine to what level one could expect agreement between results obtained from different ^{129}I AMS laboratories.

Phase I Intercomparison Procedure

The only information given to the participating laboratories regarding the $^{129}I/^{127}I$ ratios of the individual samples was an approximate guide to the upper limit of the expected $^{129}I/^{127}I$ ratio. Sufficient sample material was given to each laboratory to allow several repeat measurements. Laboratories were asked to report the results for the AgI samples (samples 1, 2, 3, and 4) and the Woodward Iodine sample (sample 11) as ratios (i.e., number of ^{129}I atoms per number of ^{127}I atoms). For the water, swipe, and other environmental samples (samples 5, 6, 7, 8, 9, and 10), laboratories were asked to report results as concentrations (i.e., the number of ^{129}I atoms per gram of sample).

Phase I Results

The results of the Phase I ^{129}I intercomparison are shown in Table 1 and in Figures 1 and 2. To preserve the anonymity promised to the participating laboratories, individual laboratories are identified only by code. Because some of the participants in the exercise did not have the requisite chemical preparation lines and procedures necessary to prepare and measure the environmental samples, some of the laboratories were only able to report results for the AgI samples.

As can be seen from Figure 1 and Table 1, excellent agreement between $^{129}I/^{127}I$ ratios is obtained for the AgI samples (samples 1, 2, 3, and 4). For the AgI samples, the differences of the un-weighted means from the expected $^{129}I/^{127}I$ ratios were always less than 5%, while the standard deviations of the means were generally around 5%. This agreement of $^{129}I/^{127}I$ ratios for AgI samples between different laboratories is remarkable considering the difficulty of the analytical technique as well as the fact that different laboratories tend to use their own independent standards and blanks.

Looking at Figure 2 and Table 1, however, one can see that, except for sample 5, there is large disagreement between measured ^{129}I concentrations from the various environmental samples. For example, differences in ^{129}I concentrations of two to three orders of magnitude were reported for samples 8 and 9. These large differences between reported ^{129}I concentrations are particularly noticeable considering the excellent agreement obtained between the AgI samples. One possible cause of this discordance is the different chemical preparation methods used by each laboratory. Another possible explanation for the widely differing results is ^{129}I contamination of the chemical preparation laboratories. While contamination was not believed to be a problem at all the participating laboratories there is substantial anecdotal history of ^{129}I contamination at some laboratories.

^{129}I Workshop

On May 16, 1996, in conjunction with the 7th International Conference on Accelerator Mass Spectrometry, LLNL hosted a one day pre-conference workshop that concentrated on the application of the ^{129}I AMS technique. Most of the participants in the ^{129}I intercomparison exercise were present at the workshop. A major part of this workshop was the discussion of results from the present ^{129}I intercomparison exercise and possible tests needed to understand the large discrepancies in ^{129}I results obtained for the environmental samples. As was expressed at both formal and informal discussions, widely differing ^{129}I environmental results could possibly be explained by differences in ^{129}I extraction chemistry. To better characterize this extraction chemistry theory, it was agreed to conduct Phase II of the ^{129}I intercomparison exercise.

In Phase II of the ^{129}I intercomparison exercise, it was agreed that AgI was to be prepared from two different environmental samples by three separate laboratories. The resulting six samples (two sets of three) were then to be re-distributed to all the ^{129}I AMS facilities and ^{129}I/^{127}I ratios measured. It was believed that results from this Phase II of the exercise would help determine whether the large discrepancies seen in results obtained for the Phase I environmental samples were due to chemical extraction procedures or due to some problem with the analytical method.

Phase II Samples

Three laboratories volunteered to chemically prepare samples for Phase II of the ^{129}I intercomparison. The three laboratories were 1) Texas A&M University, College Station, Texas, 2) PSI/ETH, Zürich, Switzerland, and 3) LLNL, Livermore, California. Each laboratory uses its own distinctly different ^{129}I chemical preparation method. Texas A&M prepares ^{129}I samples using an alkali leach and fusion method[1,2]. PSI/ETH works in conjunction with ZSR (Center of Radiation Protection and Radioecology) at the University of Hannover, Germany and uses a dry-ash combustion procedure to prepare environmental ^{129}I samples[3]. LLNL uses a wet-ash distillation method to prepare environmental ^{129}I samples.

In June of 1996, LLNL shipped soil, maple leaves, and potassium iodide carrier solution to three laboratories listed above. The soil was IAEA #375 reference material (IAEA reference sheet, reference material IAEA 375-soil, reference number G4.12), reported to have an ^{129}I concentration of 1.7(±0.4) x 10^{-3} Bq/kg or 1.2(±0.3) x 10^{9} ^{129}I atoms/gram material. Approximately 10 grams of soil was provided to each of the three chemical preparation laboratories. The maple leaves were collected near the town of Sequim, Olympic Peninsula, Washington State, USA. These leaves were also used as sample 8 in Phase I of the intercomparison. The precise ^{129}I concentration of the maple leaves is unknown but was tentatively measured in Phase I of the intercomparison exercise to be between 3.1 x 10^{7} and 5.2 x 10^{9} ^{129}I atoms/gram material. Approximately 9 grams of leaves were provided to each chemical preparation laboratory.

In conjunction with the soil and maple leaves, LLNL also provided KI carrier solution to each of the chemical preparation laboratories. The KI Carrier Solution was a 1 Normal or 12.3 mg I/gram solution. The ^{129}I/^{127}I ratio of KI precipitated from the carrier solution has been measured to be less than 2 x 10^{-14}.

Each chemical preparation laboratory was asked to make approximately 50 to 60 mg of AgI from both the soil and leaf samples. This amount of AgI was enough to provide each of the ^{129}I AMS measurement laboratories with approximately 5 to 6 mg of AgI. Each laboratory was asked to add enough carrier to each sample so that one could make the assumption that all the ^{127}I comes from the carrier and all the ^{129}I comes from the sample.

In addition to the six environmental samples, LLNL prepared an AgI sample that was precipitated from a bulk solution that had been derived by successive dilutions of a NIST standard material. This sample was identical to sample 2 used in Phase I of the intercomparison. This sample is useful to check for normalization errors and to provide a statistically meaningful comparison of the AMS technique at the various participating laboratories using a well constrained sample.

Details of the seven samples are as follows:

Sample 31. AgI prepared from NIST standard material
Same as sample 2 used in Phase I of the intercomparison
^{129}I/^{127}I ratio calculated to be 45474 x 10^{-15}
Sample 32. AgI prepared from IAEA #375 reference material soil
Amount of sample used: 2.84 g
Amount of KI carrier added: 69.7 mg
Sample 33. AgI prepared from maple leaves
Amount of sample used: 5.30 g
Amount of KI carrier added: 100 mg
Sample 34. AgI prepared from IAEA #375 reference material soil
Amount of sample used: 9.98 g
Amount of KI carrier added: 29.89 mg
Sample 35. AgI prepared from maple leaves
Amount of sample used: 5.87 g
Amount of KI carrier added: 22.76 mg
Sample 36. AgI prepared from IAEA #375 reference material soil
Amount of sample used: 1.20 g
Amount of KI carrier added: 61.5 mg
Sample 37. AgI prepared from maple leaves
Amount of sample used: 3.60 g
Amount of KI carrier added: 61.5 mg

Phase II Intercomparison Procedure

After LLNL received bulk aliquots of AgI from the three chemical preparation laboratories, the bulk AgI was subdivided at LLNL and distributed to all participating laboratories. Each participating laboratory was given an approximate guide to the upper limit of the expected $^{129}I/^{127}I$ ratios. All laboratories were asked to report results as ratios (i.e., number of ^{129}I atoms per number of ^{127}I atoms).

Phase II Results

Results of the Phase II ^{129}I intercomparison are shown in Figures 3, 4, 5, 6, and 7 and in Tables 2 and 3. Individual laboratories are identified only by code.

With the exception of one measurement, the agreement between $^{129}I/^{127}I$ ratios from different laboratories is good for sample 31 (Figure 3 and Table 2). Including laboratory B, the difference of the un-weighted mean from the expected $^{129}I/^{127}I$ ratio is about 10% with a standard deviation of the means of about 15%. Excluding laboratory B, the difference of the un-weighted mean from the expected $^{129}I/^{127}I$ ratio is approximately 5% with a standard deviation of the means of about 4%. As was the case in Phase I of the ^{129}I intercomparison, the agreement of $^{129}I/^{127}I$ ratios for the AgI 'standard type' material is excellent.

Results from the IAEA #375 reference material soil are shown in Figures 4 and 5 and in Tables 2 and 3. Measured $^{129}I/^{127}I$ ratios for samples 32, 34, and 36 are shown in Figure 4 and in Table 2 while ^{129}I concentrations calculated from the measured $^{129}I/^{127}I$ ratios are shown in Figure 5 and in Table 3. The agreement of $^{129}I/^{127}I$ ratios amongst the various AgI aliquots is good (Figure 4). After $^{129}I/^{127}I$ ratios have been converted to ^{129}I concentrations, as is shown in Figure 5 and Table 3, the three differing chemical preparation methods give consistent results for the ^{129}I concentration of the IAEA #375 soil. Examining the un-weighted means or 'average' ^{129}I concentration, however, it appears that all three differing chemical preparation methods yield an average ^{129}I concentration that is slightly lower than the reported IAEA ^{129}I concentration value of $1.7(\pm 0.4) \times 10^{-3}$ Bq/kg or $1.2(\pm 0.3) \times 10^{9}$ ^{129}I atoms/gram material. It may be premature, however, to draw major conclusions from this slight systematic shift considering the 25% error in the reported IAEA concentration and the 10-20% standard deviations associated with the AMS results.

Results from the maple leaf sample are shown in Figures 6 and 7 and in Tables 2 and 3. Measured $^{129}I/^{127}I$ ratios for samples 33, 35, and 37 are shown in Figure 6 and in Table 2 while ^{129}I concentrations calculated from the measured $^{129}I/^{127}I$ ratios are shown in Figure 7 and in Table 3. As one can see from Figure 6 the agreement of $^{129}I/^{127}I$ ratios between various AgI aliquots is poor relative to the agreement obtained for the soil samples. Because of the poor agreement of $^{129}I/^{127}I$ ratios, ^{129}I concentrations are also in poor agreement as is shown in Figure 7 and Table 3. Although there is a factor of four difference between the un-weighted mean ^{129}I concentration of samples 33 and sample 35 (with sample 37 lying somewhere in between), it is hard to draw any conclusions about the three differing chemical preparations due to the large standard deviations.

Discussion of Phase II Results

Results of Phase II of the ^{129}I AMS intercomparison show good agreement of $^{129}I/^{127}I$ ratios for AgI 'standard type' material. This good agreement was also seen in Phase I of the intercomparison.

The phase II ^{129}I AMS intercomparison results show agreement with ^{129}I concentrations in IAEA #375 soil measured by other techniques. This agreement is independent of the chemical preparation method.

The phase II ^{129}I AMS intercomparison results show relatively poor agreement of ^{129}I concentrations in the maple leaves. This relatively poor agreement of ^{129}I concentrations appears largely to be due to the fact that different AMS laboratories do not obtain the same ratio from aliquots of the same AgI. While we are unable to unambiguously explain the cause of these disagreements, we note the following:

1) The analytical difficulties for the environmental samples may imply the presence of trace contaminating constituents within the original sample and that this trace contaminate is not removed completely by any of the chemistries. To test this hypothesis, we plan on performing Proton Induced X-Ray Emission (PIXE) measurements of the various samples to see if gross elemental differences between AgI made from the soil and AgI made from maple leaves can be detected.

2) Another possibility is that some AMS laboratories are more susceptible to certain contaminates than others. For example, the charge state of the ^{129}I may play a role. The laboratories participating in this exercise measure ^{129}I in various charge states starting at 3^+ going up. Other systematic differences include stages of magnetic or electrostatic analysis and particle detection techniques.

Conclusions

Because of unresolved questions about ^{129}I AMS measurements, an interlaboratory comparison exercise for ^{129}I has been organized and conducted. The primary purpose of this ^{129}I intercomparison exercise was to assess the suitability and effectiveness of ^{129}I AMS measurements for possible IAEA safeguards use. From the data collected so for, it appears that:

1) Good agreement of ^{129}I/^{127}I ratios can be obtained for AgI ‘standard type’ material using AMS.

2) Good agreement of ^{129}I concentrations in soils (in this case IAEA #375 soil) can be obtained using AMS. This agreement can be obtained largely independent of the chemical preparation method.

3) Relatively poor agreement of ^{129}I concentrations in low activity organic material (in this case maple leaves) is obtained using AMS. The cause of this poor agreement is unknown. For this class of samples, more effort is needed to understand the cause of this large discrepancy.

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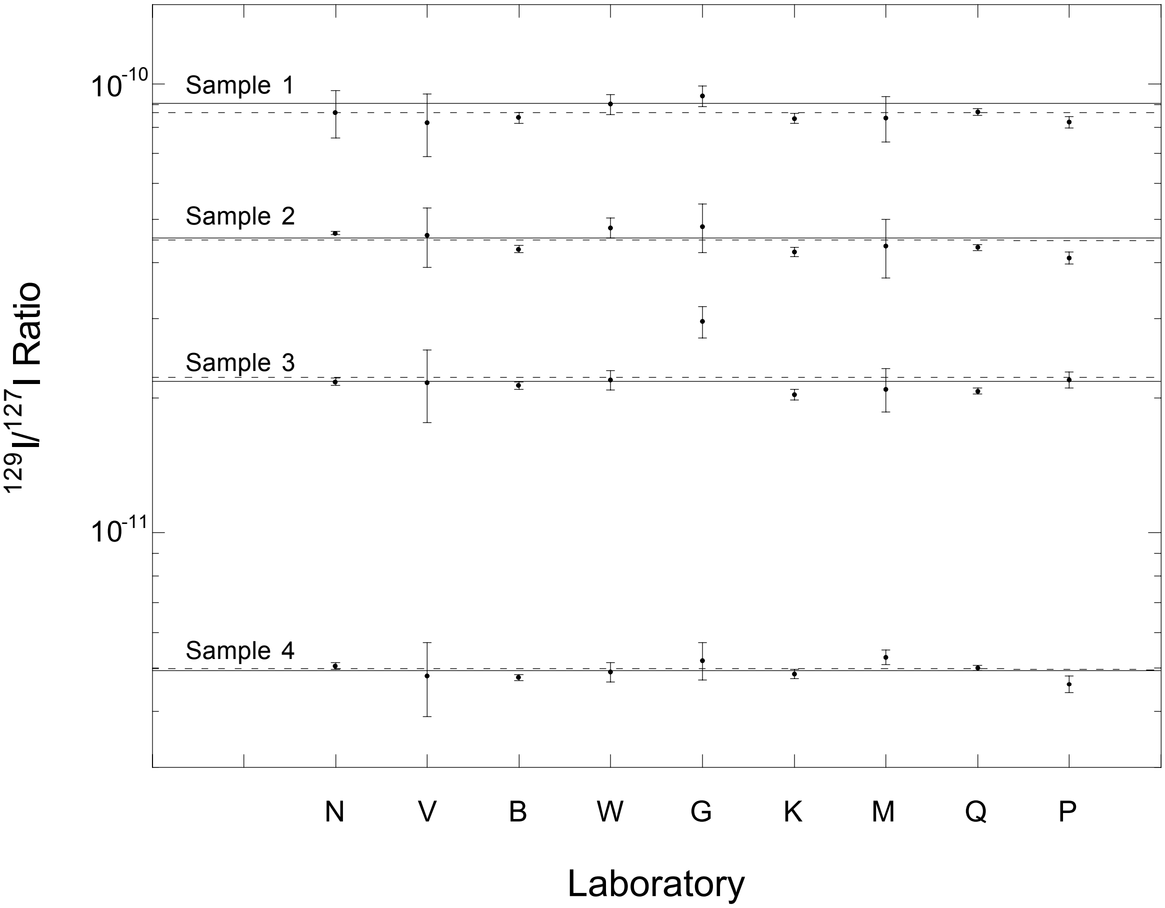

Figure 1. Results of the Phase I ^{129}I intercomparison exercise for AgI samples 1, 2, 3, and 4. The dashed line is an un-weighted mean of the results. The solid line is the expected value based upon successive dilutions of a NIST standard material. As one can see, the agreement between different laboratories for AgI is quite good.

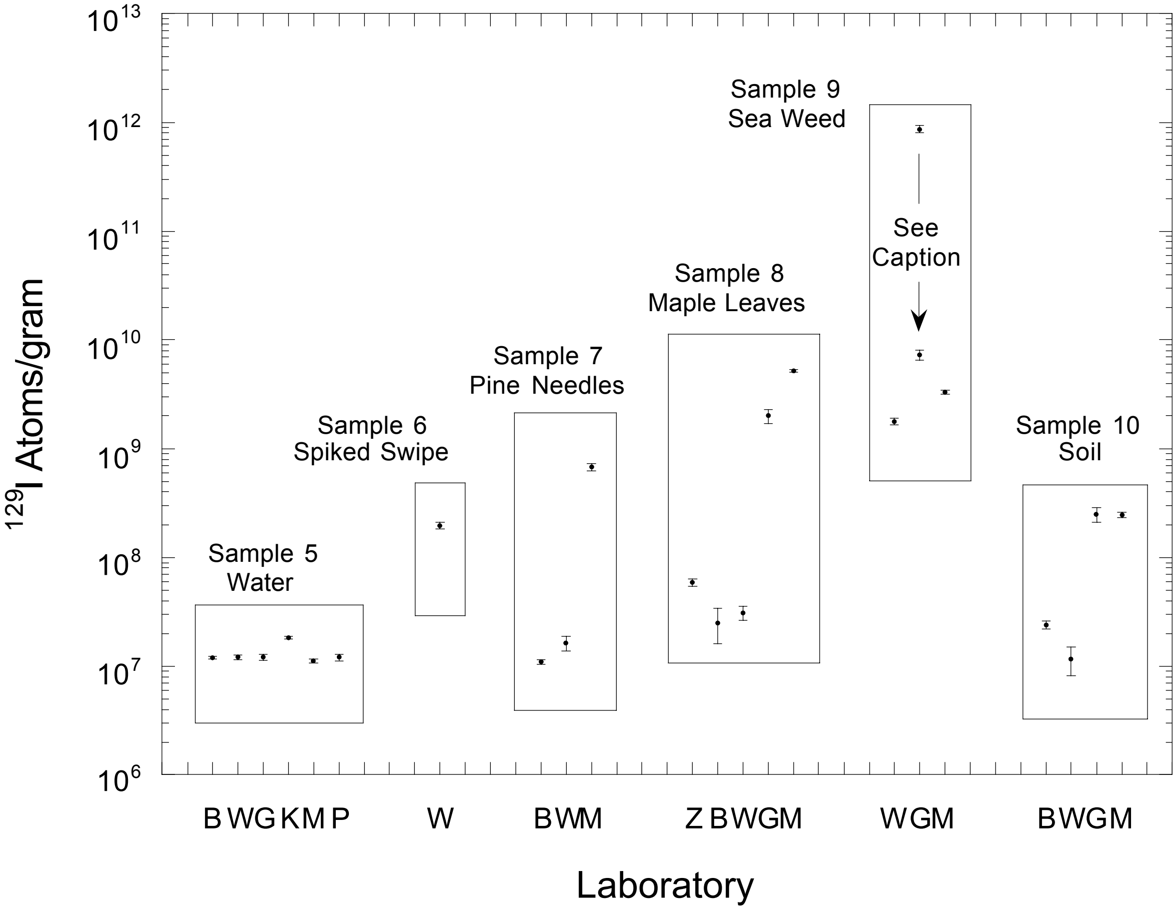

Figure 2. Results of the Phase I ^{129}I intercomparison exercise for samples 5, 6, 7, 8, 9, and 10. Except for sample 5, the agreements between measured ^{129}I concentrations are poor. After initial results were generally available, laboratory G repeated sample 9 and obtained the new value shown.

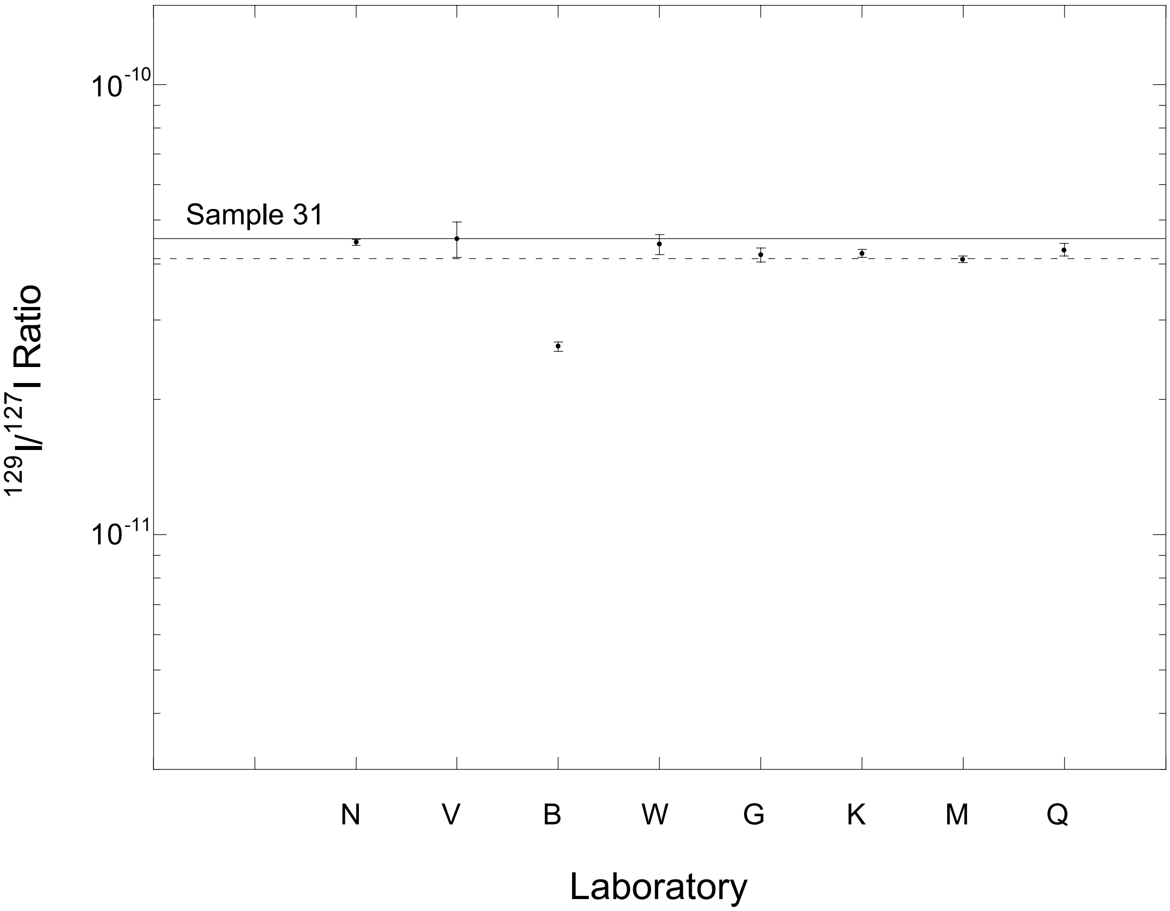

Figure 3. Results of the Phase II ^{129}I intercomparison exercise for AgI sample 31. Sample 31 is a repeat of sample 2 used in Phase I of the exercise. The dashed line is the un-weighted mean of the results. The solid line is the expected value based upon successive dilutions of a NIST standard material. With the exception of one point, the agreement between different laboratories for AgI is quite good.

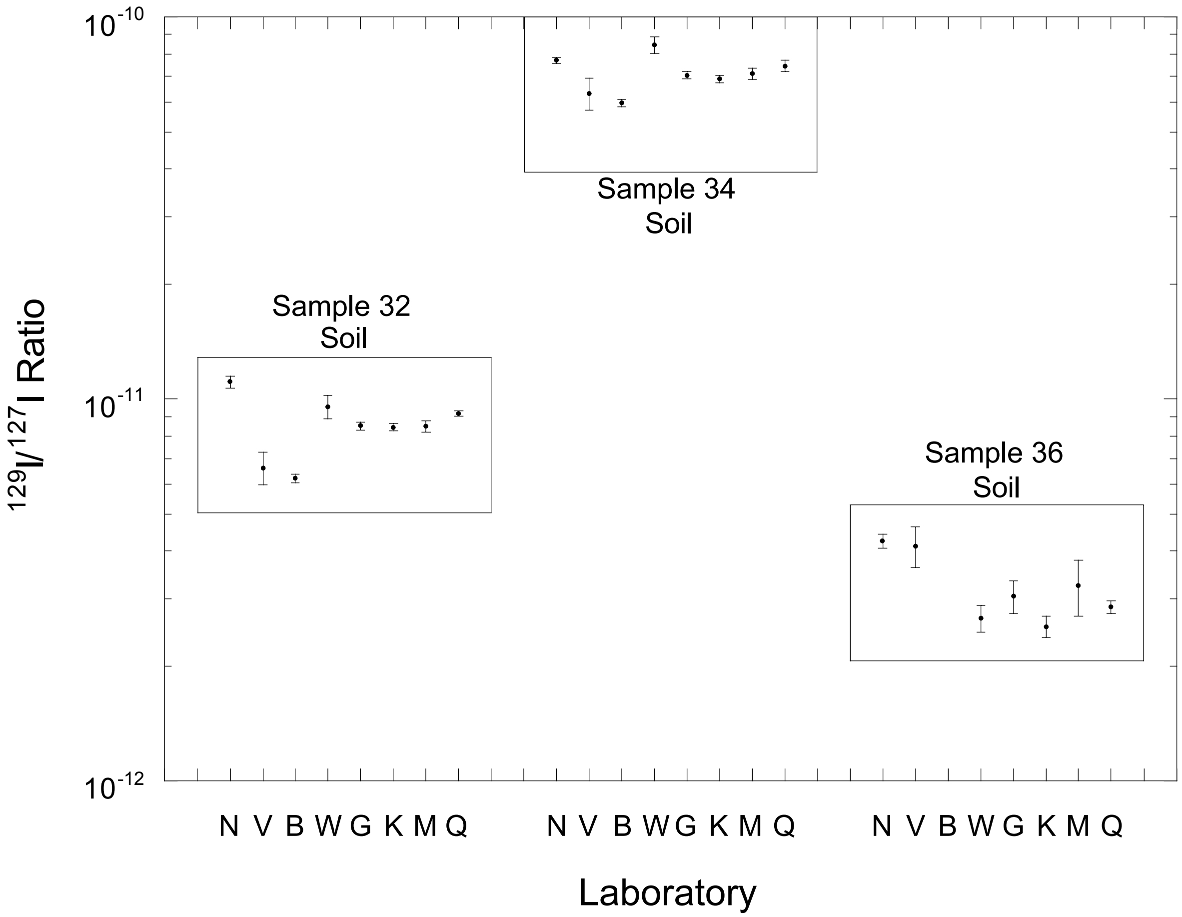

Figure 4. Measured $^{129}I/^{127}I$ ratios for samples 32, 34, and 36 of Phase II of the ^{129}I intercomparison exercise. Samples 32, 34, and 36 are AgI prepared from IAEA 375 soil using three differing chemical preparation methods. $^{129}I/^{127}I$ ratios for samples 32, 34, and 36 are not expected to agree because of the differing amount of carrier material used in the differing sample preparation methods.

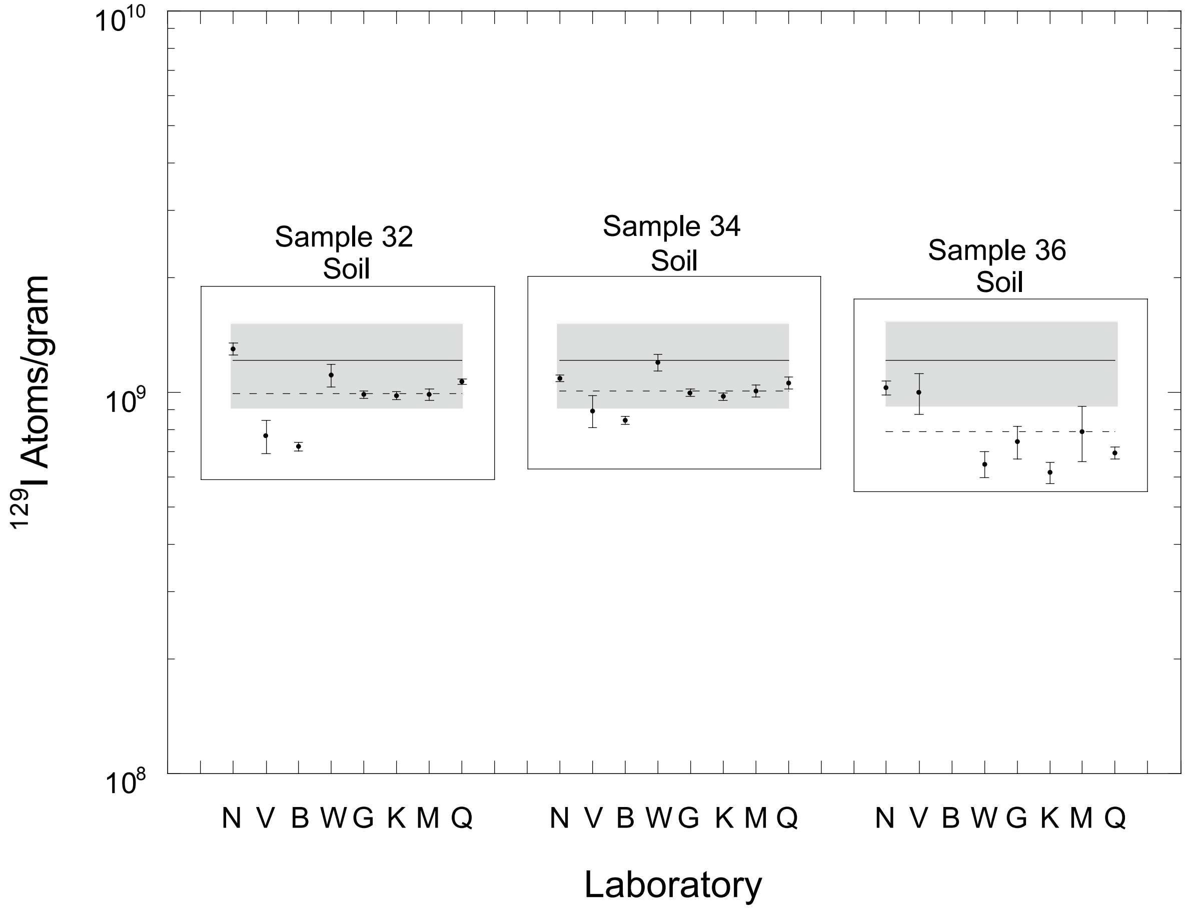

Figure 5. Measured ^{129}I concentrations for samples 32, 34, and 36 of Phase II of the ^{129}I intercomparison exercise. Samples 32, 34, and 36 were prepared from IAEA 375 soil using three differing chemical preparation methods. The dashed line is an un-weighted mean of the various results. The solid line is the reported IAEA ^{129}I concentration value of 1.7(±0.4) x 10^{-3} Bq/kg or 1.2(±0.3) x 10^{9} ^{129}I atoms/gram material. The grayed area is the bounds the upper and lower limits of the IAEA reported value. As one can see, the agreement between differing measurements and differing chemical preparation methods is quite good.

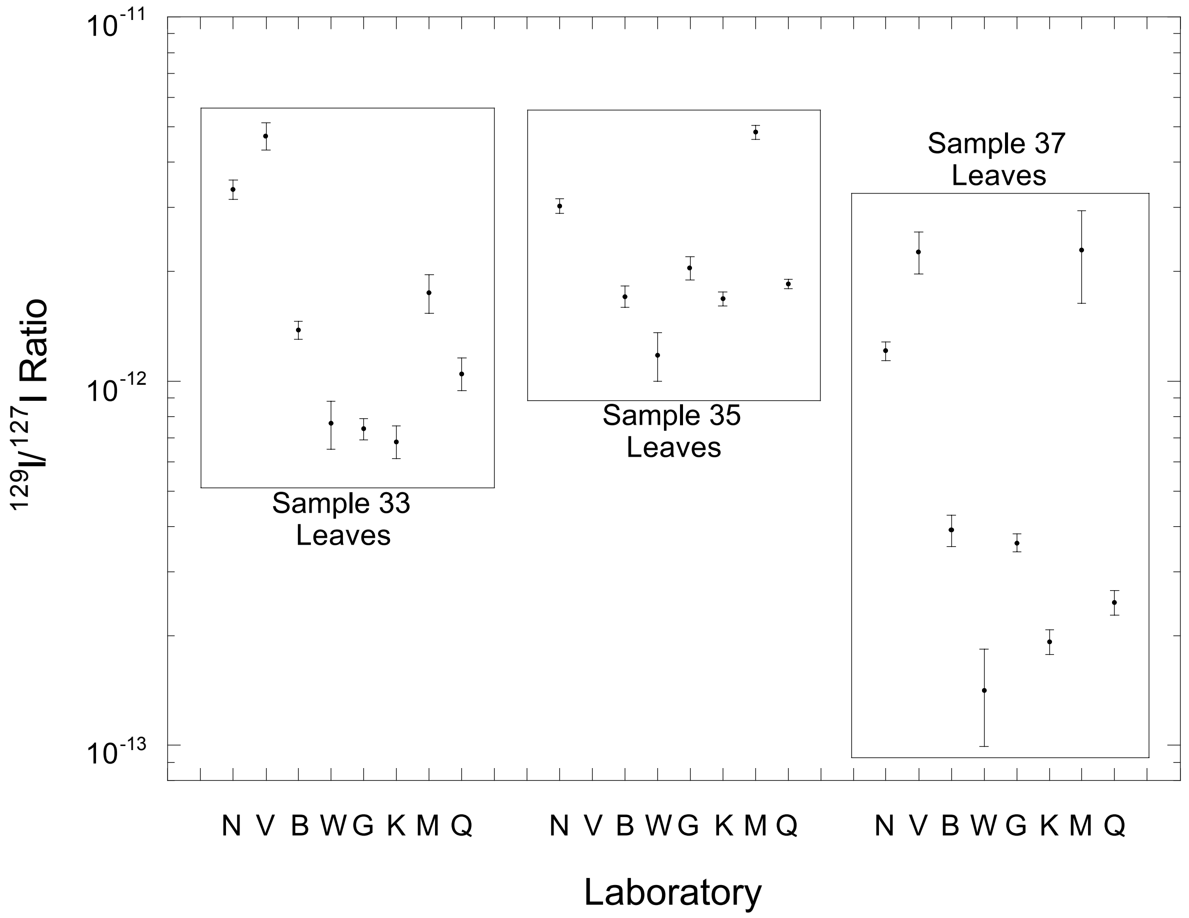

Figure 6. Measured $^{129}I/^{127}I$ ratios for samples 33, 35, and 37 of Phase II of the ^{129}I intercomparison exercise. Samples 33, 35, and 37 are AgI prepared from maple leaves using three differing chemical preparation methods. $^{129}I/^{127}I$ ratios for samples 33, 35, and 37 are not expected to agree because of the differing amount of carrier material used in the differing sample preparation methods.

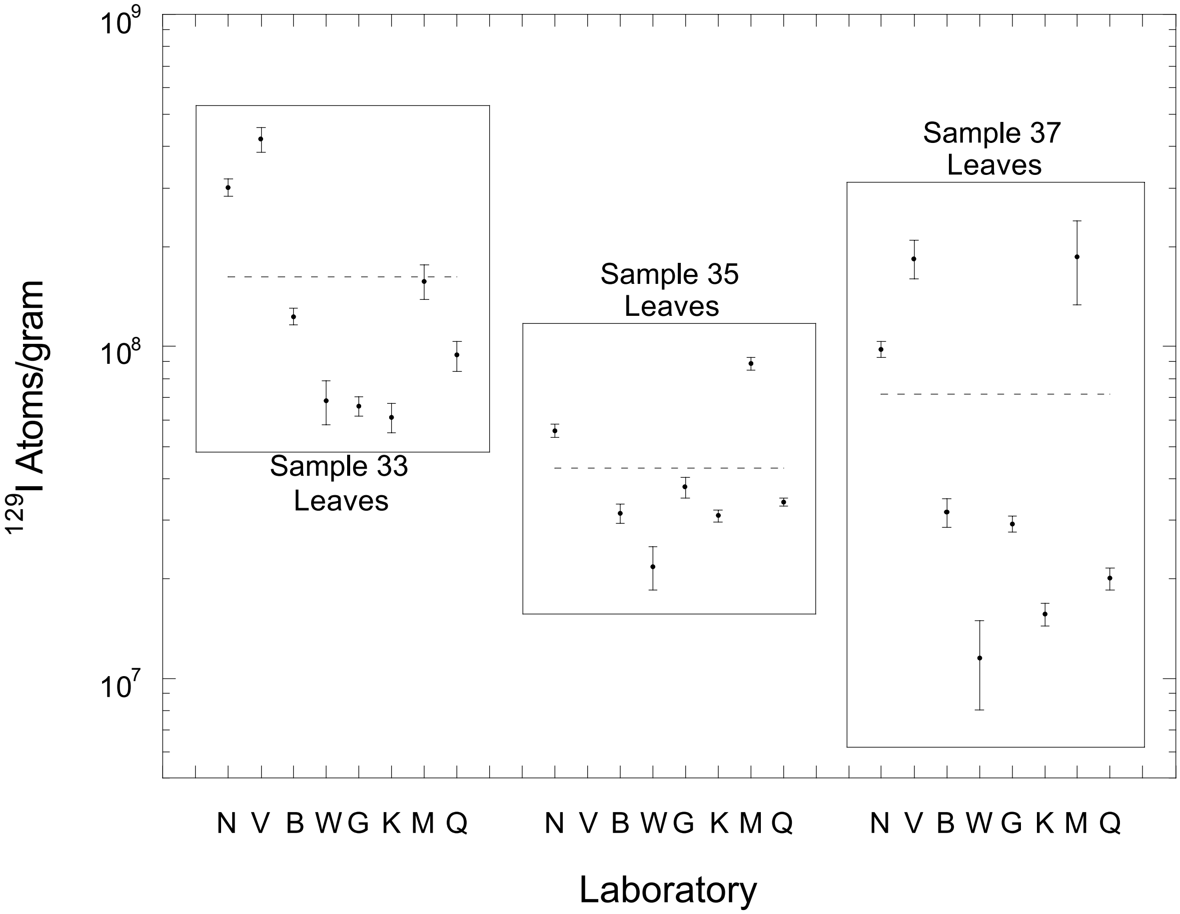

Figure 7. Measured ^{129}I concentrations for samples 33, 35, and 37 of Phase II of the ^{129}I intercomparison exercise. Samples 33, 35, and 37 were prepared from maple leaves using three differing chemical preparation methods. The dashed line is an un-weighted mean of the various results. As one can see, the agreement between differing measurements and differing chemical preparation methods is not very good.