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DEVELOPMENT OF THE NBS BERYLLIUM ISOTOPIC STANDARD REFERENCE MATERIAL[†]

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ABSTRACT

The National Bureau of Standards, in conjunction with the Oak Ridge National Laboratory and the Accelerator Mass Spectrometry community, is in the process of developing a beryllium isotopic solution Standard Reference Material. The master $^{10}\text{Be}/^9\text{Be}$ solution was characterized isotopically by resonance-ionization and secondary-ion mass-spectrometric-based techniques, and radioactivity measurements were by liquid scintillation counting. The master solution was gravimetrically diluted with ^9Be to a final $^{10}\text{Be}/^9\text{Be}$ atomic ratio of 3×10^{-11} . The preliminary data indicate a half life for ^{10}Be of 1.3 million years, and AMS measurements are within ten percent of the known beryllium isotopic ratio.

MASTER

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There have been recent developments in analytical technology which resulted in significant improvements for atom-counting sensitivity and selectivity. At hand are capabilities of measuring picogram quantities of actinides [1] and isotopic ratios of selected radionuclides on the order of 10^{-13} [2]. Also, increased analytical selectivity through the application of laser ionization have made it possible to significantly reduce isobaric interferences which have limited traditional mass spectrometry in the past, eg. ^{10}B interference in ^{10}Be analysis [3].

However, as ultrasensitive analytical methodologies are extended, the need for high-quality reference materials become increasingly critical for two reasons: a) measurement coherence and b) continued refinement of technology. For example, the Accelerator Mass Spectrometry (AMS) community have been making $^{10}\text{Be}/^9\text{Be}$ isotopic ratio measurements of 10^{-10} to 10^{-15} for the past ten years [4, 5]. However, there are no less than six $^{10}\text{Be}/^9\text{Be}$ in-house reference materials produced by the various AMS laboratories, and intercomparison of these reference materials have indicated discrepancies in excess of ten percent [6]. Additionally, some of the in-house reference materials are dependent on knowing the half life of ^{10}Be , which has an "accepted" uncertainty of twelve percent ($\pm 1\text{s}$) [6]. The AMS community has been in need of an accepted world-wide standard which will bring coherence within and between laboratories. Since relatively few AMS measurements can be made each year, it is imperative that interlaboratory data be reliable so that resources can be managed wisely.

The National Bureau of Standards (NBS), in conjunction with the Oak Ridge National Laboratory (ORNL) and the AMS community, is in the process of developing a beryllium-isotopic solution Standard Reference Material (SRM). The beryllium-isotopic SRM will serve as the primary measurement standard for AMS studies of important geoscience fields such as plate tectonics, terrestrial

accumulation of cosmic dust, meteorite irradiation histories, soil erosion, terrestrial magnetic reversals, solar radiation cycle, and sediment and ice accumulation rates [7].

Since there was no existing beryllium isotopic SRM nor any high purity ^{10}Be , several mass spectrometric methods were employed for the isotopic concentration determinations in an attempt to evaluate the isotopic discrimination effects in the measurements. The atom counting techniques employed were resonance ionization (RIMS) [3], secondary ionization (SIMS) [8, 9], inductively coupled plasma, and thermal mass spectrometry. The RIMS and SIMS results were the most reliable and were used to certify the beryllium isotopic material (± 7 percent). The certified beryllium material was then measured by radioactivity liquid-scintillation counting [10, 11] and finally diluted to a $^{10}\text{Be}/^9\text{Be}$ atomic ratio of 3×10^{-11} (Fig 1). The diluted material is currently being measured in an interlaboratory AMS comparison exercise and will be issued as a Standard Reference Material in the near future.

Preliminary results indicate: a) a half life value for ^{10}Be of 1.3 ± 0.1 million years, and b) AMS $^{10}\text{Be}/^9\text{Be}$ atomic ratio of 3.3×10^{-11} . The causes for the discrepancies between these values and the "accepted" half life and the nominal isotopic ratio of the SRM material should be resolved in the near future.

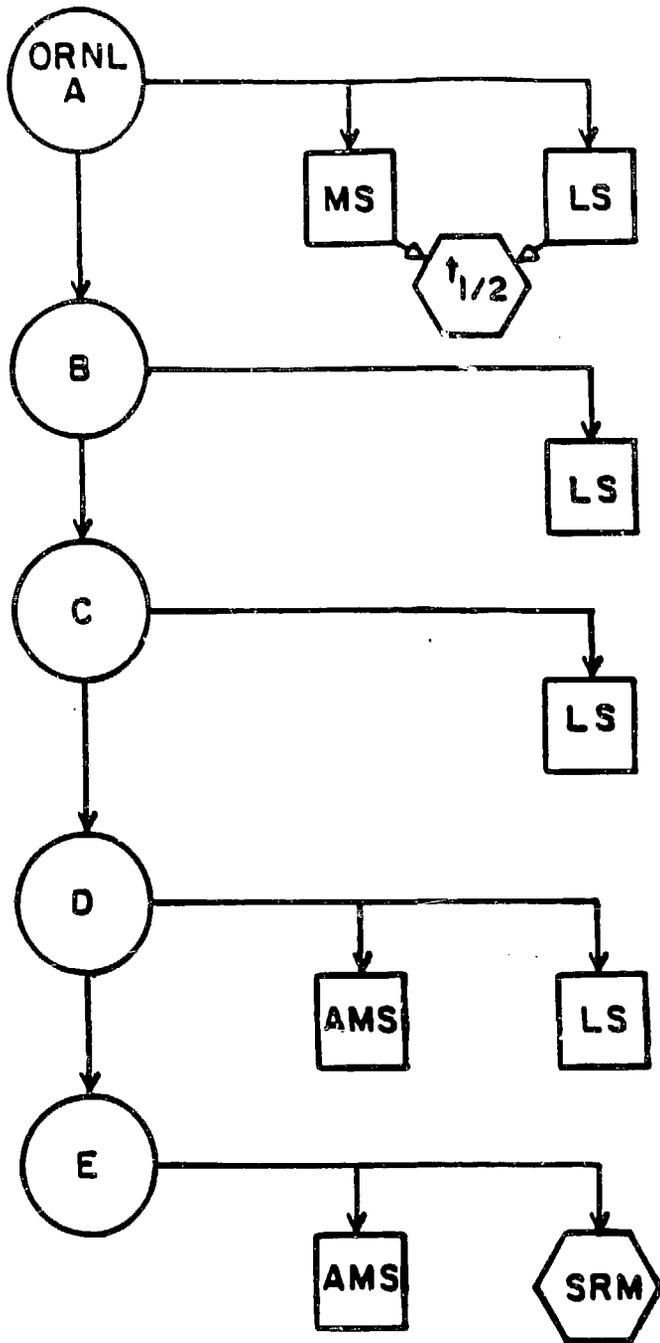
Following the issue of the beryllium isotopic SRM, the production of additional radioactivity standards for the ultrasensitive atom counting community such as ^{36}Cl , ^{46}Ca , ^{90}Sr , and ^{129}I will be considered. Since measurements of these nuclides are still in their infancy, it is suggested that appropriate standards be developed early on instead of allowing years of uncorrelated data to be collected, as was the case for ^{10}Be measurements.

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REFERENCES

- [1] W.R. Kelly and J.D. Fassett, *Anal. Chem.*, 55, 1040 (1983).
- [2] Forth International Symposium on Accelerator Mass Spectrometry, April 27-30, 1987, Niagra-on-the-Lake, Ontario, Canada.
- [3] J.D. Fassett, J.C. Travis and T.B. Lucatorto, 32nd Annual Conference on Bioassay, Analytical and Environmental Radiochemistry, National Bureau of Standards, Gaithersburg, MD, October 21-23, 1986.
- [4] D. Elmore, ed., Proceedings of an Interagency Briefing and Workshop to Explore Needs and Opportunities for AMS Application, NSF, DOE, USGS, Washington D.C., March 28-29, 1985.
- [5] D. Elmore and F.M. Phillips, *Science*, 236, 543 (1986).
- [6] H.J. Hofmann, J. Beer, G. Bonani, H.R. von Gunten, S. Raman, M. Suter, R.L. Walker, W. Woelfli, and D. Zimmermann, Forth International Symposium on Accelerator Mass Spectrometry, April 27-30, 1987, Niagra-on-the-Lake, Ontario, Canada.
- [7] B.R. Doe, *Geochimica et Cosmochimica Acta*, 47, 1341 (1983).
- [8] W.H. Christie, R.E. Eby, R.J. Warmack and L. Landau, *Anal. Chem.*, 53, 13 (1981).
- [9] R.L. Walker, J.L. Botts, J.H. Cooper, H.L. Adair, J.E. Bigelow and S. Raman, ORNL-5986, October, 1983.
- [10] A. Grau Malonda, E. Garcia-Torano, J.E.N. Report 488, Appendix III p.2, Madrid (1981).
- [11] B.M. Coursey, W.E. Mann, A. Grau Malonda, E. Garcia-Torano, J.M. Los Arcos, J.A.B. Gibson and D. Reher, *Int. J. Radiat. Appl. and Instrum.*, Part A 37, 403 (1986).



NBS dilution and measurement scheme to produce NBS Solution E as an SRM. Mass spectrometry (MS), liquid scintillation (LS), and accelerator mass spectrometry (AMS) were used to calibrate the solutions and confirm the dilutions. The MS and LS measurements on solution B were made to evaluate the half life of ^{10}Be .

Figure 1