ASTM INTERNATIONAL

Designation: D8299 – 19

Standard Guide for
Using Metal Ratios in Soils to Distinguish Between Anthropogenic and Natural Beryllium[1]

This standard is issued under the fixed designation D8299; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide describes a general approach to planning investigations in which the goal is to obtain background measurements of naturally occurring Beryllium (N-Be) along with one or more predictor metals in local soils, to be used in predicting the amount of N-Be expected in samples taken for evaluation using the Metal Ratio Method (MRM). Generally, systematic random sampling is recommended, after which the results are interpreted using statistical methods described in this guide.

1.2 The total Be (T-Be) measured in evaluation samples can then be compared with the predicted N-Be to estimate the amount of anthropogenic Be (A-Be) present, if any. Several scenarios are discussed in which samples taken and analyzed for T-Be for worker or public protection purposes might include both N-Be and A-Be (see 6.1). This method can allow the N-Be and A-Be components of T-Be measurements to be estimated.

1.3 Values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard guide is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appro-priate safety, health, and environmental practices and deter-mine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accor-dance with internationally recognized principles on standard-ization established in the Decision on Principles for the Development of International Standards, Guides and Recom-mendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D7458 Test Method for Determination of Beryllium in Soil, Rock, Sediment, and Fly Ash Using Ammonium Bifluo-ride Extraction and Fluorescence Detection

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of common technical terms used in this standard, refer to Terminology D653.

4. Summary of Guide

4.1 This guide describes a Metal Ratio Method (MRM) for using ratios of measurements of naturally occurring Beryllium (N-Be) with one or more predictor metals in local background soils to predict the amount of N-Be expected in samples taken in workplaces or other settings to be evaluated. The total Be (T-Be) measured in each individual sample can then be compared with the predicted N-Be to give an estimate of the amount of anthropogenic Be (A-Be) present in that sample, if any. Being based on ratios, this method can accommodate varying dilutions of the soils with other materials, including wipe matrices.

4.2 This guide describes the major steps involved in imple-menting a MRM at a site. These include:

1

4.2.1 identifying and sampling appropriate N-Be-containing site-specific background materials;

4.2.2 identifying appropriate candidate predictor metals;

4.2.3 conducting sample preparation and chemical analysis in a manner designed to promote batch-to-batch consistency (precision) of Be/Metal ratios;

4.2.4 selecting the final predictor(s) with regard to both batch-to-batch precision and predictive power;

4.2.5 using a prediction algorithm based on ratios that will be applicable to samples composed of soils diluted by other materials, including wipe matrices, and estimating its prediction error; and

4.2.6 applying the MRM at that site.

5. Significance and Use

5.1 Chronic Beryllium Disease (CBD) and Beryllium Sensitization (Be-S) caused by exposure to anthropogenic Beryllium (A-Be) are a continuing health concern in the nuclear and defense industries. The traditional worker protection strategy has been to compare total Be (T-Be) measurements taken in workplaces with appropriate limits or local background Be measurements. That strategy can be ineffective, however, where naturally occurring Be (N-Be) from local soils can be a non-negligible component of the measured T-Be. N-Be is not known to have adverse health effects. This MRM uses the ratio(s) of measurements of Be with measurements of one or more metals found in background materials, but not likely to be in facilities, to predict the amount of N-Be expected in individual samples based on the measurement(s) of the predictor metal(s) in those samples. The measured T-Be can be compared with the predictions to estimate the amount of A-Be present, if any.

5.2 In order to use the MRM it is required to identify local sources of N-Be that can be credibly transported into facilities, along with a means of sampling those sources. It would be helpful also to have insight into the metal content of the soils at those sources, to facilitate the identification of candidate predictor metals. Regarding the latter, there are published databases describing the chemistry of surface soils in many regions; however, information found in those databases should be regarded as suggestive, due to the modified sample preparation and analysis methods currently expected to be used because of the need to digest high-fired BeO, one form of A-Be of concern.

6. Procedure

6.1 *Scenarios*—This MRM can be applied in a variety of scenarios, such as the following.

6.1.1 *Scenario WF*[3]—When dealing with N-Be blown into facilities from local surface soils, samples to be evaluated are bulk and wipe samples taken inside the facilities. This situation is found at sites in arid locations.

6.1.2 *Scenario WA*—Similar considerations could apply where samples to be evaluated are air sampling filters, but the N-Be source is still windblown soils.

6.1.3 *Scenario WD*—Another scenario is where the samples to be evaluated are from soil areas, either near facilities containing A-Be or having been remediated, where the concern is still with possible N-Be being blown from nearby uncontaminated soils.

6.1.4 *Scenario IF*—In some settings there may not be a clear and accessible local background source of N-Be, but other facilities can be identified that are known quite credibly to have NOT had operations involving A-Be, in which the bulk or wipe sample chemistry ought to be similar to that in facilities of concern, except for the operations involving A-Be.

6.2 *Background Data*—The fundamental idea is that background samples should chemically resemble those that would be found in facilities of concern (allowing for dilutions) so long as operations at those facilities have not involved A-Be.

6.2.1 *Identifying Background Locations*—In scenarios **WF**, **WA**, and **WD** background sources would be local surface soils. There may be a possibility that A-Be had been transported from a facility (operating or not) to surrounding soils. If this is a concern, test samples can be obtained that extend outward from that facility to determine whether a gradient in Be concentrations can be discerned. If such a gradient is found, it can be estimated how far away samples must be taken to obtain acceptably uncontaminated background samples. On the other hand, natural soil chemistry does vary spatially, so it should be considered how far away credibly comparable background samples can or should still be obtained. For example, at the site for which this MRM was developed, there is clear variation in the soil chemistry, but also notable winds, so a large background area was used. Issues arising in identifying background facilities for scenario **IF** were mentioned in 6.1.4.

6.2.2 *Sampling at the Background Locations*—Systematic random sampling is recommended for obtaining background samples in scenarios **WF**, **WA**, and **WD**. A grid of approximately equally sized cells is superimposed on a chart of the background area identified in 6.2.1. One location is chosen at random in each grid cell, at which "blowable" surface soils are collected using standard methods. For example, equal amounts of soil could be collected at four points within a meter of each selected location. These would then be dried, sieved to less than 1000 μm, and homogenized. Sufficient amounts of these soils, termed "source materials", should be obtained to allow for aliquots to be provided to the laboratory in multiple analytical batches, as discussed in 6.4.2 and 6.6.3. The number of locations from which to obtain source materials will be based on the anticipated variation in natural soil chemistry. If available survey data suggest that the soil chemistry is quite homogeneous, the survey might start with as few as 20 or so locations, but in a situation with more complex spatial patterns in soil chemistry more background sampling locations would be desired.[4]

6.2.2.1 *Advantages of the Systematic Grid Sampling Approach*—A grid sampling approach is recommended, even though subsequent data analysis uses techniques designed for simple random sampling (SRS). There are two possibilities.

---

[3] This is the scenario for which the MRM was originally developed at the US Department of Energy Hanford Site.

[4] At the Hanford Site for which the MRS was developed, 65 locations were used.

One is that there really are no spatial patterns or autocorrelations present in the population being sampled. In that case no statistical differences are expected between samples obtained regardless of the sampling plan used; grid sampling is essentially equivalent to SRS. The other is that there really are spatial patterns or autocorrelations. In that case it has been shown that statistical methods designed for SRS actually perform better in terms of statistical precision and accuracy than they would in an actual SRS situation.[5]

6.2.2.2 *Stratified Sampling*—This is an extension of systematic sampling, in which available information will allow areas expected to have differences in soil chemistry to be identified. In this situation such areas should be identified and appropriate proportions of the overall background sampling should be obtained from each such stratum. A challenge here, though, is determining what those appropriate proportions ought to be; in principle, they should be related to the proportions of soils to be found in facilities expected to have come from each stratum.

6.2.3 *The IF Scenario*—In the **IF** scenario, samples from the facility of concern may be bulk samples or wipe samples or both. In the background comparison facility sub-facilities (clusters of offices, shop, etc.) can be identified that would play the role of grid cells in systematic sampling. One (or a few adjacent) location(s) would be selected in each sub-facility, to be sampled in the same manner as the facility of concern would be sampled; that is, using the same methods, same selection of specific locations within the sub-facility, etc. Multiple side-by-samples (for wipes) or splits (for bulks) should be obtained from each location, in order to allow for the multiple analytical batches discussed in 6.4.2 and 6.6.3. If the background comparison facility has areas of different types, such as machine shops, labs, and offices, sampling separately from each type (stratum) can be considered. Depending on available numbers of locations, sampling 15-20 locations from each such stratum could be suggested.

6.3 *Identifying Candidate Predictor Metals*—This will necessarily be site-specific. At some sites there may be databases based on prior geological surveys that can suggest initial candidates. Minerals prominent in local soils and select metals characteristic of those could be considered.

6.3.1 Three caveats are offered. One is that some metals, such as aluminum, chromium, iron, lead, and perhaps even lithium, may be present inside facilities from non-natural sources either from operations of interest/concern or even from routine maintenance. These would clearly not be suitable as predictors of N-Be to be found in facilities.

6.3.2 Another is that although it may be tempted to select candidate predictor metals based on their apparent correlations with N-Be, there may be advantages in including predictors which can aid in differentiating heterogeneous mineral sources. The idea is this: suppose that N-Be has a ratio around 1:5 with metal M_1 in one mineral form but around 1:10 in another mineral form. If there is a metal M_2 with different concentra-

tions in the two mineral forms, including M_2 may allow the combination to provide superior overall predictions of N-Be, even though M_2 by itself might not be such a good predictor of N-Be.

6.3.3 The third caveat is that even though soil chemical databases may be available, they may not include measurements of all potential candidate predictor metals that are desired, and will be unlikely to have measurements made using aggressive sample preparation procedures currently desired for making Be measurements because of the desire to digest high-fired BeO.

6.4 *Producing a "Short List" of Candidate Predictors*—Once background source soils (or in-facility samples in scenario IF) have been obtained and the candidate predictors identified, several (at least four) analytical batches should be prepared and analyzed (for both soil and wipe samples if appropriate). It will be useful to first obtain some idea of the N-Be concentrations present in the background soils in scenarios WF and WA as well as the (diluted) concentrations anticipated in future in-facility analyses in application. (In scenarios WD and IF it is more likely that N-Be concentrations would be similar for the background and comparison samples.)

6.4.1 *Batching Considerations*—If information regarding anticipated in-facility N-Be concentrations can be obtained, it can be used to guide the assignment of soils to different analytical batches. If both bulk and wipe samples (or just wipes) are to be analyzed, it can be used to guide the amounts of source soils added to the wipes in an attempt to have N-Be concentrations blanketing the anticipated range of measurements. If only bulk samples are to be obtained in facilities, it might be considered to provide the lab with some samples which contain mixtures of background soil with field blank materials, using soil amounts designed to cover the range of N-Be concentrations anticipated based on the nature of the dilutions anticipated in facilities. If field blank materials are added, their chemical composition should be taken into consideration in evaluating the results. In any situation, where possible it should be attempted to create analytical batches for submission to the lab such that each batch includes a relevant variety of concentrations.

6.4.2 *Batch-to-Batch Consistency (Precision)*—Quality control provisions in standard analytical methods address precision and accuracy of measurements of individual analytes, but do not address precision of ratios of those measurements. The MRM relies on such precision, though, so one part of identifying superior predictor metals is evaluating the precisions of their Be/Metal ratios. The idea is to be confident that for the selected predictor(s) such ratios will be reliable not only in the background data but also in future measurements yet to be obtained.

6.4.2.1 Toward that end it is recommended that sample aliquots from each source location be included in at least four background analytical batches (four batches for soils plus four batches for wipes, if applicable). These batches would ideally be analyzed at somewhat separated times.

6.4.2.2 After the measurements from these analyses are available, the Be/Mi ratios for each candidate predictor metal (Mi) can be calculated. A useful way of evaluating these is to

---

[5] This use of systematic grid sampling ideas at the design phase of a study, followed by treating data as if they had been obtained from a SRS design, is described in US EPA US EPA's Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G-4, 2006, and Guidance for Data Quality Analysis, EPA QA/G-9, 2000.