RATE's Radiocarbon: Intrinsic or Contamination?

by <u>Kirk Bertsche</u>

The *ICR* (Institute for Creation Research) recently spent eight years on a project known as *RATE* (Radioisotopes and the Age of The Earth). The *RATE* team claims the results have yielded convincing and irrefutable scientific evidence of a young earth.

John Baumgardner, a geophysicist with expertise in tectonic modeling, presents experimental data claiming to show that all biological material contains intrinsic radiocarbon, no matter how old that material may be thought to be [1, 2]. He makes additional claims that even non-biological carbonaceous material contains intrinsic radiocarbon. He suggests that this radiocarbon is residual from the material's creation. If true, his claims would have far-reaching implications for the ages of these materials.

Baumgardner presents two classes of data. The first is a set of 90 previously published radiocarbon AMS dates of old samples (most >100k years) that he has re-analyzed. The second is a set of new samples that the *RATE* team collected and sent to a leading radiocarbon AMS laboratory to be dated. In both cases, I am convinced that the "intrinsic radiocarbon" is nothing more than contamination and instrument background.

Modern Radiocarbon Dating

New Methods Allow Smaller Samples

Willard Libby discovered radiocarbon dating in the late 1940s. He received the Nobel Prize in Chemistry for this discovery in 1960. The technique arises from radiocarbon being continually produced in the upper atmosphere by cosmic rays while it is continually decaying, so the atmospheric concentration has reached a fairly steady equilibrium. Plants are in equilibrium with atmospheric radiocarbon through respiration. This equilibrium continues through plants to herbivores and through them to carnivores. Once an organism dies, its carbon ceases exchanging with atmospheric carbon but continues decaying with a half-life of about 5730 years. Thus, measurement of the radiocarbon concentration can give the time that the organism died.

Early measurements were done by counting the beta particles (high energy electrons) liberated in radiocarbon decay. The age limit was roughly 30k years, due both to poor statistics from low decay count rates and to cosmic ray backgrounds.

Richard Muller proposed a new measurement technique, called "accelerator mass spectrometry" (AMS), in 1976 [3]. Muller suggested that particle accelerators be used to separate the atoms, allowing the radiocarbon atoms to be counted directly instead of waiting for them to decay. It was hoped that this would enable dating of much smaller and perhaps much older samples. This technique has indeed allowed use of much smaller samples and has become the dominant method of radiocarbon dating. However, the original anticipation of 100,000-year background levels has been "unrealized due to a variety of sample processing and instrument-based experimental constraints" [4].

Most radiocarbon AMS laboratories process samples using a variant of the method described by Vogel [5], with apparatus and processes typically optimized for samples containing about 1 mg of carbon. While samples containing less than 0.01 mg of carbon have been successfully dated, measurement precision begins to suffer below about 0.1 mg of carbon due to counting statistics. The maximum allowed sample size is typically about 10 mg of carbon. Larger samples produce excessive CO_2 pressure in the sealed tubes used in the process, causing tubes to explode and samples to be lost. Thus, even if larger samples like *RATE's* "on the order of 100 mg" [6] are submitted to an AMS laboratory, only about 1 mg of carbon will actually undergo analysis. Though Baumgardner calls a 1 mg sample "tiny" [6], it is generally considered "large" by AMS laboratories [e.g., 5, 7, 8], with enough carbon to provide ion source current for about a day. Most laboratories prefer to receive samples larger than 1 mg to allow some loss in cleaning and to have additional material available if needed.

Contamination and background vary significantly

Modern radiocarbon dating by AMS is a complex process with numerous potential sources of contamination requiring characterization. A typical sample must first be cleaned mechanically and chemically, then converted to CO₂ by combustion (for organic samples) or acid hydrolysis (for carbonates), then chemically reduced to graphite [9, 10]. For some samples, the process is even more complex, involving pre-separation of organic fractions from the more easily contaminated inorganic fractions (e.g., dating only cellulose from wood or only collagen from bone) [11]. Each step in this process may introduce a small amount of modern carbon contamination. More processing tends to introduce more contamination. Furthermore, the instrument itself always introduces a background, similar to most other high sensitivity analytical instruments [4]. A sample originally containing absolutely no radiocarbon will still give a nonzero measurement from such contributions.

Contamination and instrument background are sometimes collectively referred to as "total background." Taylor and Southon have characterized six general types of total background, each of which has multiple specific sources [4]. For our purposes, we will group these contributions into three general classes of contamination and background:

- 1. contamination of the sample before reaching the testing laboratory (primarily contamination *in situ* but also during collection or storage)
- 2. laboratory contamination before placement in the accelerator (handling, sample chemistry, etc.)
- 3. instrument background, including sample contamination in the AMS accelerator system

The first contribution often results in sample-position-dependent variations in radiocarbon content, thus is often detectable by measuring multiple pieces of the same sample. But the amount of contamination is generally impossible to quantify. An old sample with *in situ* contamination cannot generally provide an accurate date.

The second contribution, laboratory contamination, is largely due to sample chemistry (pretreatment, hydrolysis or combustion to CO₂, and reduction to graphite), which generally introduces a small amount of modern carbon, typically at least 1 microgram [8, 12, 13, 14]. Thus a 1 mg sample of infinitely old carbon would measure at least 0.1 pMC (percent modern carbon) before background subtraction. At least one laboratory reports sample chemistry contamination as low as 0.08 pMC (excluding chemical pretreatment, which can be a significant contribution), but this value does "not necessarily apply to other laboratories" [15]. Different sample chemistry techniques and processing equipment and variations in chemical batches can result in significantly more sample chemistry contamination.

The third contribution, instrument background, has a number of sources. The main sources are generally the following:

- a. ion source "memory" of previous samples, due to radiocarbon sticking to the walls of the ion source, thermally desorbing, and then sticking to another sample
- b. mass spectrometer background, non-radiocarbon ions that are misidentified as radiocarbon, sometimes through unexpected mechanisms [16]
- c. detector background, including cosmic rays and electronics noise

Baumgardner claims that instrument background "is routinely and reliably tested by running the system with no sample in the aluminum sample holder," [6] but this technique underestimates the true instrument background contribution and provides only a lower limit. Specifically, this technique underestimates both the ion source memory and mass spectrometer backgrounds. Even with this underestimation, Baumgardner's claim of "about 0.0005 pMC" [6] using this technique seems unreasonably low, since IsoTrace Laboratory has measured "approximately 0.0025 pMC" [15].

Surface-dependent "sticking coefficients" are a well-known issue in ion source design. Recent tests suggest, not surprisingly, that this effect also applies to the sample surface itself, causing ion source memory to be sample-surface-dependent due to different sticking coefficients for carbon-containing molecules in the ion source. For tests on the UCI AMS system, graphite gave instrument backgrounds of 0.020 to 0.035 pMC, while natural diamond gave 0.005 to 0.02 pMC [4]. Differences in ion sources, beamline components, mass separation techniques, and detectors will cause the instrument background to vary significantly from laboratory to laboratory.

Laboratory contamination and instrument background can also vary with time at a single laboratory. Many unexpected sources can introduce additional contamination. Thus frequent characterizations of the measurement background are necessary. The most straightforward approach is to obtain "radiocarbon-free" material similar to the unknown, process it identically to the unknown and treat its radiocarbon content as a measurement background to subtract from the unknown. Most laboratories include such "process blanks" with each set of samples to get an accurate and contemporaneous reflection of the true background. A few laboratories *may* apply a "standard background" rather than such parallel processing of "radiocarbon-free" samples, but this approach is somewhat risky and thus not common.

AMS laboratories have been able to identify and reduce many sources of contamination through years of care and attention. The remaining amounts of laboratory contamination and instrument background vary between laboratories but are well characterized by frequent use of process blanks. Frequent intercomparisons between AMS laboratories demonstrate the effectiveness of this approach. For example, the Fourth International Radiocarbon Intercomparison (FIRI) included identical samples of very old wood with a radiocarbon content of about 0.2 pMC, corresponding to an age of about 50,000 years BP (before present). The mean value measured by over 30 AMS laboratories was 0.27 +/- 0.05 pMC for Kauri wood sample A and 0.24 +/- 0.04 pMC for Kauri wood sample B [17]. These low variations demonstrate very good consistency between laboratories, in spite of the presence of laboratory contamination and instrument background.

Interestingly, *none* of these laboratories found either of the Kauri wood samples to be at or below measurement background levels. This fact is at odds with Baumgardner's accusation that "most commercial labs" apply a "high 'standard background' to the samples," potentially as high as 0.8 pMC, allowing them to report "an 'infinite' radiocarbon age" and so to avoid "the awkward difficulty of explaining" non-zero levels of radiocarbon for presumably radiocarbon-free samples [6].

Analysis of RATE's claims

Previously published data: sample processing affects results

Baumgardner's first class of data is a set of 90 previously published radiocarbon AMS dates. He has selectively divided these into two groups for re-analysis: 34 Precambrian geological samples and 40 Phanerozoic biological samples. The remaining samples, including marbles of uncertain origin and a few reprocessed samples, were not re-analyzed.

The Precambrian geological subset Baumgardner analyzed has a mean radiocarbon content of 0.06 pMC. The Phanerozoic biological samples have a mean radiocarbon content of 0.29 pMC, about five times that of the geological samples. Baumgardner concludes from this "unambiguously higher mean" that "organic samples from every level in the Phanerozoic portion of the geological record ... display significant and reproducible amounts of ¹⁴C" [1].

Baumgardner fails to note that nearly all of these geological samples are actually of geological *graphite*, so did not undergo the sample chemistry required for the biological samples. (Geological graphite typically requires only a mechanical surface cleaning with no chemical processing.) This omission is crucial, because Baumgardner asserts evidence for increased intrinsic radiocarbon in the biological samples on the basis of these lower results from the geological samples.

Baumgardner also omits two important geological graphite samples from his analysis, namely entries 21 and 40 in his *Table 1* [1]. These samples were identical to two natural graphite samples, entries 62 and 79 respectively, but were combusted and re-graphitized in the laboratory using identical chemistry to biological samples. This procedure provided controlled characterizations of contamination from sample chemistry, which added 0.25 and 0.14 pMC respectively [18, 19]. Entry #10 in Baumgardner's *Table 1* compares radiocarbon AMS with radiocarbon decay counting, showing roughly a 0.4 pMC contamination level for AMS due to sample chemistry [20]. These tests used identical materials with and without sample chemistry, not relying on assumptions that any of the materials were "radiocarbon-free," showing that sample chemistry produces values in the range seen in the Phanerozoic biological samples.

Many of Baumgardner's references report characterizations of various contamination sources, with sample chemistry adding from about 0.1 to 0.7 pMC (highly dependent on sample size and procedure). This range is essentially the same as that of Baumgardner's biological samples. The highest value of 0.7 pMC comes from an older sample chemistry procedure and may have been somewhat overestimated [20]. Baumgardner's biological sample #10 mentioned above is from this reference and is well within this sample chemistry background. Jull *et al* characterize a total process background of 0.58 pMC, with about 0.5 pMC attributed to sample chemistry, and Baumgardner's biological sample #8 from the same reference is consistent with this contamination [21]. Thus the main difference Baumgardner sees between geological and biological samples is contamination introduced by sample chemistry.

While this conclusion explains the higher values for the biological samples in general, it does not account for all the details. Some biological samples *do* have radiocarbon levels not explainable by sample chemistry. These samples are mostly coals and biological carbonates, both of which are prone to *in situ* contamination.

Coal is notorious for contamination [22]. Uranium is often found in or near coal, releasing neutrons that generate radiocarbon in the coal from nitrogen. Mobile humic acids are almost always present and can transport more recent carbon to the coal. Microbial growth can incorporate modern carbon from

groundwater while *in situ* and from air after sample collection. Coal can easily adsorb atmospheric CO₂ after collection.

Carbonates often exhibit anomalous radiocarbon values, potentially becoming contaminated by adsorption of atmospheric CO₂ [23]. Nadeau *et al* detail anomalies with marine carbonates, i.e., shells and foraminifera, suggesting that "the carbonate crystal structure of the shells ... may incorporate atoms, at some later stage, from its surrounding [sic] for the curing process" [24]. A similar contamination mechanism occurs in bone, where carbonates can be "transported into the bone matrix from the groundwater and soil environment by chemical exchange and/or through dissolution and reprecipitation processes" and bone collagen has been found to give much more reliable dates than bone carbonates [25]. But these anomalies are specific to carbonates and do not apply to other materials, e.g., wood. Most of the wood samples and some of the coal and carbonate samples in Baumgardner's *Table 1* show radiocarbon values consistent with sample chemistry, thus showing no evidence of intrinsic radiocarbon.

Baumgardner observes that "the variation in ¹⁴C content for the Phanerozoic samples is large" at +/- 0.16 pMC [1]. He suggests that this is caused by variations of *in situ* radiocarbon contamination due to "accelerated nuclear decay" of nearby material. I agree that these large variations suggest contamination, but the main contributor seems to be sample chemistry contamination, not *in situ* contamination.

Baumgardner also concludes that the geological samples show evidence of intrinsic radiocarbon with values above instrument background. But their radiocarbon content of 0.06 +/- 0.03 pMC is in good agreement with the instrument backgrounds characterized in many of Baumgardner's references. One may perhaps charge circular reasoning since instrument backgrounds are often found by measuring geological graphite assumed to be "radiocarbon-free." However, applying a very low value from one laboratory to data from all other laboratories, as Baumgardner does, is improper. Each AMS instrument's background will be different and must be determined individually.

More evidence against intrinsic radiocarbon appears in multi-laboratory intercomparisons. In *FIRI*, mentioned earlier, the mean value of old wood measured by over 30 AMS laboratories was 0.27 ± 0.05 pMC for Kauri wood sample A and 0.24 ± 0.04 pMC for Kauri wood sample B [17]. These low variations show very good consistency between laboratories. Each laboratory used separate process blanks to characterize and subtract total background. If the blank values really contained intrinsic radiocarbon, the subtraction would have biased their results to varying degrees and would have produced large variations in their reported results, similar to the ± 0.16 pMC seen in the Phanerozoic biological samples.

RATE's new data

Baumgardner's second class of data consists of samples that the *RATE* team collected and sent to a leading radiocarbon AMS laboratory for analysis, including a set of ten coal samples and a number of diamond samples [1]. Measurements of both materials show large variations, suggesting contamination.

Coal

Baumgardner claims that his coal results of 0.25 ± 0.11 pMC "fall nicely within the range for similar analyses reported in the radiocarbon literature." This claim is misleading. Baumgardner's coal results already include background subtraction, whereas the literature results are generally raw values and are treated *as* the measurement backgrounds [1]. Unlike the literature values, Baumgardner's coal samples *do*

show significant radiocarbon above background, inviting explanation. The measurements also show relatively large variations, suggesting contamination.

The expert who prepared and measured the *RATE* samples suspects that the coal samples had been contaminated before reaching his laboratory, probably *in situ*. As mentioned earlier, coal is easily contaminated both *in situ* and after collection. Though precautions were taken, the coal samples may have also been contaminated while stored in a DOE geology laboratory refrigerator [1]. Geology laboratories often have elevated levels of radiocarbon due to tracer studies, neutron activation studies, and dust from uranium-bearing rocks. Carbon is highly mobile and contamination can spread through an entire laboratory and persist for decades [26].

With extreme care and isotopic enrichment techniques, anthracite coal has been measured with an apparent age of more than 75,000 years (<0.01 pMC), below the detection limit of the procedure [27]. Thus coal exists that shows no evidence of intrinsic radiocarbon.

Diamond

Diamond is difficult to combust. The *RATE* samples apparently required modifications to the normal procedure [1], presumably higher combustion temperatures and longer combustion times, likely increasing the sample chemistry contamination. The samples were reportedly pitted and may have been subjected to previous analyses and to unknown contamination. Nevertheless, *RATE's* five deep-mine diamond samples had radiocarbon levels only slightly above background (0.01 to 0.07 pMC after background subtraction), while the seven alluvial samples ranged from 0.03 to 0.31 pMC after background subtraction.

Subsequently, the *RATE* team inserted diamond directly into an ion source, eliminating the sample chemistry, and measured much lower radiocarbon values, "between 0.008 and 0.022 pMC, with a mean value of 0.014 pMC," apparently with no background subtraction [6]. This much lower value for unprocessed diamond provides strong evidence that their processed diamond samples had been contaminated, most likely by the modified sample chemistry.

Taylor and Southon have also measured unprocessed diamond, finding a similar range of 0.005 to 0.03 pMC without background subtraction. They interpret this result as their instrument background, primarily due to ion source memory. Their ion source current varied, unintentionally, over about a factor of two, perhaps due to crystal face orientation or to conductivity differences between samples. "The oldest ¹⁴C age equivalents were measured on natural diamonds which exhibited the highest current yields" [4]. This important observation provides evidence about the source of the radiocarbon.

If the radiocarbon were intrinsic to the sample, there would be no change in the radiocarbon ratio with sample current. The ¹⁴C, ¹³C, and ¹²C would change in unison. However, if the radiocarbon were coming from ion source memory or elsewhere in the accelerator, it should give a count rate independent of ion source current. Normalizing the radiocarbon count rate to the ion source current, which is predominantly ¹²C, would result in higher radiocarbon content for lower source currents, as observed. This data provides clear evidence that at least a significant fraction of the radiocarbon detected by Taylor and Southon in diamond measurements did *not* come from the diamonds themselves and thus could not be "intrinsic radiocarbon."

The lower values for unprocessed diamond and the current-dependent behavior find no explanation in Baumgardner's "intrinsic radiocarbon" model. But these results fit well with the Taylor and Southon evidence that instrument background (specifically ion source memory) is material-dependent, with

diamond exhibiting significantly less ion source memory than graphite. The radiocarbon detected in natural, unprocessed diamond measurements seems to be nothing more than instrument background.

Summary

Radioisotope evidence presents significant problems for the young earth position. Baumgardner and the *RATE* team are to be commended for tackling the subject, but their "intrinsic radiocarbon" explanation does not work. The previously published radiocarbon AMS measurements can generally be explained by contamination, mostly due to sample chemistry. The *RATE* coal samples were probably contaminated *in situ. RATE*'s processed diamond samples were probably contaminated in the sample chemistry. The unprocessed diamond samples probably reflect instrument background. Coal and diamond samples have been measured by others down to instrument background levels, giving no evidence for intrinsic radiocarbon.

While some materials, e.g., coals and carbonates, often *do* show radiocarbon contamination that cannot be fully accounted for, resorting to "intrinsic radiocarbon" raises more questions than it answers. Why do only *some* materials show evidence of this intrinsic radiocarbon? Why does some anthracite and diamond exist with *no* measurable intrinsic radiocarbon? Why is its presence in carbonates so much more variable than in other materials, e.g., wood and graphite? Why is it often found in bone carbonates but not in collagen from the same bone? Since intrinsic radiocarbon would be mistakenly interpreted as AMS process background, why do multi-laboratory intercomparisons not show a much larger variation than is observed? Why does unprocessed diamond seem to have less intrinsic radiocarbon than processed diamond?

These and many other considerations are inconsistent with the *RATE* hypothesis of "intrinsic radiocarbon" but are consistent with contamination and background. "Intrinsic radiocarbon" is essentially a "radiocarbon-of-the-gaps" theory. As contamination becomes better understood, the opportunities to invoke "intrinsic radiocarbon" will diminish. Most radiocarbon measurements of old materials, including many of shells and coal, can be accounted for by known contamination mechanisms, leaving absolutely no evidence for intrinsic radiocarbon. The evidence falsifies the *RATE* claim that "all carbon in the earth contains a detectable and reproducible ... level of ¹⁴C" [1].

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About the author

Dr. Bertsche received a PhD in Physics from the University of California, Berkeley in 1989 under the direction of Prof. Richard A. Muller, the inventor of radiocarbon AMS. Dr. Bertsche's thesis involved the design and testing of a small cyclotron for radiocarbon AMS. He subsequently received a postdoctoral appointment in the AMS laboratory of Lawrence Livermore National Laboratory, where he was involved with accelerator design and operation and also with sample preparation and analysis. In 2005, he received an MA in Exegetical Theology from Western Seminary, Portland, Oregon. He is the author of 25 publications and 13 patents, primarily dealing with particle accelerator and electron microscope design.

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