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The Critical Role of Analytical Science in the Study of Anomalies

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Abstract — Controversy surrounding the validity of anomalous events can often be resolved by the proper use of analytical science. Similarly, improper use of analytical science can lead to the appearance of anomalies when none exist. This paper examines these issues, points out errors that have been made in previous investigations of anomalies, and makes recommendations for the correct use of analytical science in anomalies research.

Introduction

It seems that disagreement between skeptics and believers over the validity of anomalous events often comes down to an understanding (or lack thereof) of the fundamental characteristics and requirements of analytical science (a.k.a. analytical chemistry, the science of materials characterization for chemical composition and physical properties). Claims of biological transmutations have been based on differences in element concentrations before and after the growth of a plant. Claims of cold fusion have been based on the detection of tritium, neutrons, gamma rays, or He^3. Claims of polymeric water were based on physical and spectral properties without an examination of chemical composition. Claims of homeopathy are based on the effectiveness of preparations in which no active agent can be detected. Claims for miraculous religious relics have been based on physical and spectral properties obtained using antiquated or inappropriate instrumentation. The list is extensive and perhaps it is time to examine how analytical science works.

The Role of Analytical Science

Analytical science is often described as the science of materials characterization for chemical composition and physical properties, but that doesn't adequately portray the role that it plays in the verification of anomalous events. Chemical composition and physical properties are more than "what something is made of' or "how it behaves." They are also "where it came from" and "how old it is." In an recent article, Hieftje (1993) provided an overview and appraised the current status and future prospects of analytical science, as well as defining what analytical scientists do. Typical research areas include method development, chemometrics, instrument development, measurement science (principles, signal-to-noise enhancement, and noise analysis), and fundamen-

tal characterization of instruments and measurements. Applied activities include sampling, routine analysis, statistical data treatment, and automation. The range of activities is enormous, and overlaps almost every other scientific field. And although analytical science has sometimes been relegated to second-class status in academic institutions, the fact is that good analytical scientists understand better than anyone else the capabilities and limitations of measurement instrumentation. I find that many people underestimate the complexity of analytical science. It seems so simple. You stick a sample in an analytical instrument and out comes the composition. In reality it is an extremely complex process that is made up of sampling, sample preparation, analysis, and data interpretation. Ralph E. Oesper, Emeritus Professor at the University of Cincinnati, said it best: *"It is a process in which you can do a hundred things right and only one thing wrong, and still come out with the wrong answer."*

Since the verification of an anomalous event usually relies on the characterization of materials remaining after the event, analytical science often plays the vital role in providing conclusive evidence. Obviously, the more information that can be obtained, the better. The amount of information that can be obtained from an analytical method is called the *informing power* of the method. Most common analytical methods only provide information about the bulk composition of a material. These methods generally require several milligrams of sample and need some preparation or dissolution. Method sensitivities vary, but often extend down to parts-per-billion of analyte in the bulk material. Specialized techniques, such as laser, electron or x-ray microprobe techniques, can characterize the composition of microscopic particles, and some methods are useful for determining the composition of surfaces or the distribution of analyte as a function of material depth. Perhaps the most valuable information for the anomalist comes from techniques that can provide information about the form or species of the analyte. Is it a pure element or a compound? What isotopes are present? And, of course, isotopic analysis can sometimes indicate the age of an object as well.

There is obviously a wealth of information that is available by expert application of analytical science. But who is an expert? An expert is someone who has had years of intimate experience with the technique. *This is critical!* An expert must know the theory, instrumentation, and practical applications of an analytical technique. Someone who teaches an academic course may know the theory and instrumentation, but may not be able to deal with all the variables involved in a practical analysis. Conversely, a technician who has years of practical experience may not know the theory and instrumentation well-enough to deal with the complex data interpretation that analysis of an anomalous material may present. There is, for example, no such thing as an "expert in spectrometric analysis," a term I have heard used before to justify the validity of analytical results. Why? Because, to name a few methods of spectrometric analysis, there is atomic spectrometry, mass spectrometry, x-ray spec-

trometry, UV-visible spectrometry, infrared spectrometry, and so on. And each of these areas has a large number of permutations, not to mention hyphenated methods that cross area boundaries. Suffice it to say that a real expert is not as easy to find as one might think.

Common Errors

Following are a few examples of errors which might be expected in the application of analytical science to verification of anomalies:

- *The measured subsample is not representative* of the original sample because of inhomogeneity or poor sampling technique.
- *The sample is contaminated* before it gets to the analytical instrument. Contamination control for ubiquitous elements such as sodium, calcium and iron is critical to successful analytical science.
- *The appropriate method is used incorrectly.* Interferences are not understood and corrected for, or calibration is performed improperly.
- *An inappropriate method is used,* leading to information that neither proves nor disproves the existence of the anomaly. A subset of this is the *use of equipment of less than optimum sensitivity,* which brings results down to the random noise level, and leaves them open to imaginative interpretation.
- *A new method is invented* to validate an anomaly, even though there is little theoretical basis or practical experience in the use of the method. Analytical methods should be thoroughly tested before they are applied to complex samples.
- *Accurate analytical data is misinterpreted,* either accidentally or deliberately, so as to steer conclusions in the wrong direction. This includes disregarding evidence that doesn't fit within the theoretical constraints of the experimenter.
- *The uncertainty of an analytical result is underestimated by ignoring the significance of non-random bias* in reporting the uncertainty and thus the significance of the results. The random precision of a measurement is meaningless unless all sources of non-random bias are under control.

Of all these errors, poor control of non-random bias is perhaps the most pervasive. It is extremely difficult in most experimental situations to characterize every possible source of bias, so it is necessary for the scientist to run numerous and proper controls and to conservatively estimate the total uncertainty of his measurements. The Vernon Hughes law of low-level statistics applies here. "Despite the fact that a three-sigma effect appears to have a 99.73 percent chance of being right, it will be wrong half the time." What Hughes is saying is that while the probability based on the random error of measurement may indicate significance, there is a equal chance that some source of non-random bias will have been ignored in the experimental design, and the anomalous result is just a chance occurrence.

Past Mistakes

If we examine the history of anomalies research, we see numerous examples of these errors. The verification of religious relics is almost always complicated by restrictions placed on investigators by the religious authorities, who wish to avoid defiling the relic. This can force investigators into analytical errors by limiting measurement options. *The* blood of *Saint Januarius* is a case in point (Epstein, 1992). This miraculous blood relic undergoes a transformation from solid to liquid when displayed by the Bishop of Naples, Italy on the saint's feast days, three times a year. Since the Church will not allow the ampoule holding the relic to be opened for direct sampling of the relic, to see if it is really blood, any testing has to be done by external optical probing. In 1902, scientists were permitted to perform a spectroscopic analysis of the relic by shining a light beam through the glass case and visually studying the absorption spectrum. By early 20th century standards, this equipment was state-of-the-art, and they saw an absorption band that looked similar to that of blood. However, the data interpretation was colored by poor wavelength resolution and coverage, and the nature of the detection system (i.e., their eyes), which was certainly subject to bias. When the Church, in 1989, again permitted a spectroscopic analysis, were scientists permitted to use modern laser-based fluorescence or high-resolution absorption detection systems? No, they used a spectroscope, similar to that used in 1902, but at least augmented with photographic detection. As might be expected, the results were no more conclusive than in 1902. The instrumentation used had insufficient informing power to provide conclusive evidence. The results were hidden in the noise or uncertainty of the measurement, allowing observers to interpret the data as they wished: believers continued to believe and skeptics continued to scoff. Nothing was accomplished.

In stark contrast to this was the extensive analytical work performed on the Shroud of Turin, claimed by some to be the burial shroud of Christ. Perhaps because the Church has never taken an official stand on the Shroud's validity, scientists were allowed to extensively study the relic, including the removal of samples for carbon-14 dating, which provided an origination date between 1260 and 1390 A.D. Techniques used prior to the carbon-14 dating included x-ray and ultraviolet fluorescence, mass spectrometry, and visible and electron microscopy, and the work, in general, was of good quality, although some incorrect conclusions were drawn because of inappropriate sampling methods (Craig, 1994; Wilson 1986). An interesting sidelight to the main investigation was a study of the claim that button-like images in the eye areas of the Shroud were lepton coins of Pontius Pilate, placed over the eyelids. This claim was made by Chicago theology professor Francis Filas, who noted that under high-magnification, the image on the right eye appeared to show the letters UCAI and a shepherd's crook, which were characteristics of a coin in existence at the time of the crucifixion. This inspired Alan and Mary Whanger (1985) to invent a technique called "Polarized Image Overlay," in which two projectors were

fitted with oppositely polarized filters. One projected the image from the eye of the Shroud, and the other the image of a lepton coin. A third polarizer was used by the observer to switch from one image to the other so as to note congruencies. Was this really any more than a complex Rorschach test that misinterpreted scorch marks on the cloth? The Whangers attempted to alleviate that concern by doing the image analysis of other coins and with other observers, although there is no evidence that the tests were done blind. In any event, the question of Shroud authenticity was settled by the carbon-14 dating results.

The existence of *polywater,* also known as anomalous water, water II, orthowater, and superwater, was brought to the scientific forefront by Soviet scientists Fedyakin and Derjaguin in the 1960s. They discovered that water condensed in a capillary tube that was kept in an atmosphere of saturated water vapor for several days behaved with physical characteristics completely different from normal water. It froze at -50 degrees C and boiled at about 300 degrees C, and was thought to be a polymer of water molecules. Infrared spectra of the material, which was available only in microliter volumes, provided the strongest evidence that polywater was a new kind of water. But while infrared spectroscopy was the correct method to elucidate molecular structure, it was not the correct method to determine what some skeptics were claiming... that polywater was simply contaminated water. Further examination using analytical techniques that could characterize inorganic and organic contamination proved conclusively that polywater was not polymeric water, but only water contaminated by inorganic cations like sodium and silicon, or bio-organic matter. Depending on whom one believes, this final conclusion was reached as a result of work by Rousseau (1992), who correlated spectra of biological substances with polywater, or by Derjaguin and others (Reese, 1993; Franks, 1981), who claimed polywater to result from dissolution of quartz by condensing water. In any event, polywater was no longer anomalous.

During the first 40 years of the 20th century, scientists were eagerly trying to find real evidence for the existence of elements 85 and 87, which had been predicted by Mendeleyev in the late 19th century. It was during this time that Fred Allison, an American physicist, devised an analytical method that he called the *magneto-optic method of chemical analysis.* The method was based on the comparison of the difference in response of the Faraday effect induced in different liquids (Allison, 1927). The Faraday effect involves the rotation of polarized light passing through a liquid, induced by a magnetic field applied to the liquid. He constructed an apparatus in which light from a high-voltage spark was directed through crossed polarizers and two tubes containing the liquids to be analyzed, and surrounded by coils of opposite winding. The time at which the magnetic field was applied to the different liquids could be varied by adjusting the distance the electric current had to travel to reach the cells. The observer looked for a minimum in the light from the spark, which indicated the delay in the appearance of the Faraday effect (relative to the reference liquid) after the magnetic field had been applied. Allison first applied this pro-

cedure to simple solutions of carbon bisulfide and hydrochloric acid and later to mixtures. He became convinced that compounds would retain their individual minima in a mixture, regardless of the other components. He claimed a sensitivity down to less than part-per-billion levels, and he began to use his method to search for the elusive elements 85 and 87. He rapidly found them, and published studies on spectra and compounds of virginium and alabamine. His results were replicated in part by several other laboratories, but detailed examination by others (MacPherson, 1934) proved conclusively that the minima observed by Allison and others "had no objective reality" and "were not a function of the chemical solutions used." The Allison magneto-optic method disappeared from the pages of scientific journals. Looking back in retrospect, the modem analytical spectroscopist can easily find errors in Allison's reasoning. He never offered a substantial theory to explain his observations, but more importantly, he ignored several experimental factors, such as the variability and temporal length of the pulse of light from the spark, that limited his approach from the start. This was a classic example of the investigator finding what he believed in. But it certainly didn't hurt his career, since his biography lists him as head of the Auburn physics department and dean of the graduate school in 1953, with the physics building at Auburn named in his honor. Furthermore, he is still listed as the discover of astatine (i.e., element 85, alabamine) in the 1991 Concise Columbia Encyclopedia and similarly credited in the 1994 edition of Microsoft Encarta. Being a distinguished scientist does not make one immune to mistakes, particularly when working outside of one's area of expertise.

The analytical scientist has no control over how his data are used once they leave the laboratory. He can suggest how the data should be interpreted, but the suggestions may not be favored by the recipient. A classic example is a recent report on the letterhead of the Chief of Police of Fyffe, Alabama, reporting on a series of suspected *UFO-related cattle mutilations* in 1992 and 1993. In this report, it is noted that a flaky white material was found on and near the mutilated animal (Garmony, 1993). The material was placed in a cigarette wrapper and then transferred to a glass jar. During the transfer, material that came in contact with the brass tip of a ball-point pen was reported to melt into an almost clear liquid. The material was sent to a molecular biologist for analysis. According to the report, after two tests, the scientist determined that the substance was composed of aluminum, titanium, oxygen and silicon in significant amounts, and that the amount of titanium was larger than he would ever expect to see in any substance and that there was no way this combination of elements could ever occur in nature. Attached to the report were two spectra and an information sheet provided by the analytical scientist. The white powder is reported to be insoluble in water and not radioactive. Scanning electron microscopy with energy dispersive detection of x-ray fluorescence was used to determine the four elements noted. The attachment says that the spectra do not necessarily indicate relative amounts of each element accurately, but should

provide a reasonably good fingerprint for future analysis. That sounds like good analytical science to me. Nothing is said about the amount of titanium or the combination of elements being unusual. Shortly afterwards, it was reported in a UFO journal (Ecker, 1993) that further investigation by James Deveroux, a wildlife biologist, indicated that there is a filler and opacifier used in paper production that is similar in element concentration to the mystery white substance found on the animal. It is insoluble in water, and there is a paper manufacturer a short distance away from where the dead animal was found. This appears to be a likely solution, and one that came directly from the proper interpretation of the information provided by the analytical scientist.

The concept of biological transmutation of elements was popularized by Louis Kervran (1972) in his book Biological Transmutations and their Applications, in which he claimed transmutations of elements such as sodium, potassium, magnesium, calcium, silicon, phosphorus, manganese and iron by living systems, primarily plants. According to Kervran, these transmutations occurred by simple addition or subtraction of elements, such as iron minus hydrogen equals manganese. P. M. Baranger and others (Bounias, 1993) have also reported on the study of anomalous changes in calcium, phosphorus, manganese and iron during the growth of plants. Unfortunately, these results were likely (shall we say 3 sigma) to be the result of contamination, inexpert use of analytical methods, or unidentified non-random bias. But it did get me thinking how one might really verify or invalidate the concept of these purported anomalous changes in elemental concentrations. Baranger's experiments involved placing seeds in a clean growing atmosphere of a Pyrex glass or polyethylene dish with double-distilled water to which was added a known amount of nutrient solution. Assuming that Baranger had adequate control over contamination and concentration of nutrients, which I am not willing to believe without substantial proof, any errors would lie in the analytical measurements. Still, arguing about work done 30 years ago is useless. Is there a better way to investigate this? Yes, modern inductively-coupled plasma mass spectrometry (ICP-MS) instrumentation allows the simultaneous determination of a large number of elemental isotopes as well as accurate measurement of isotope ratios. By using stable isotope spiking of nutrient solutions for plant growth, it would rapidly become evident if biological transmutations occurred, since isotope ratios would be changed if transmutations to different elements occurred.

Conclusion

Anomalies can be investigated and resolved, and paradigms shifted as necessary, if the analytical science is done correctly. This means proper sampling and contamination control, appropriate instrumentation, valid controls and standards, expert analysts, and careful and conservative data interpretation. In the past, that has been the exception rather than the rule. Let us hope that the lessons of the past will guide us in future attempts to resolve the many mysteries of nature.

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