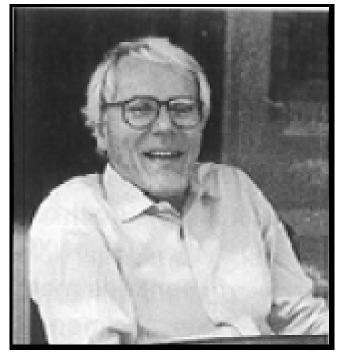
PREFACE

Specimen preparation



It is well known that improper specimen preparation can cause errors greater than 100% during an analysis. However if one examines the literature one often finds that details on how a specimen was prepared are often omitted not only in papers dealing with XRD or XRF, but also in papers dealing with other analytical methods of analysis. Why do authors do this? Why do editors allow this to happen? I do not know. Is it because they feel this information is not important? I hope not. Evidence of this neglect can however be found by an examination of the literature. Select any ten papers and find out how many do or do not include this information. I believe many papers on XRD and XRF usually include incomplete information, or none at all, about the procedure used to prepare the specimen. I do not know for sure how many papers dealing with methods other than X-rays lack this information, but I believe there are many. Do editors and authors assume all readers can duplicate the results in a publication without a detailed explanation of how the specimen was prepared? If they do, I disagree with them. Of course there are times when the analyst has to analyze the sample without any treatment (as submitted) because the request is to analyze it this way. There may be other cases when the sample can not be treated because it is in a form which prevents any treatment. When these situations exist, it would be much better if authors would state this in their discussion. I do not believe this scarcity of information in publications is because there were no problems preparing the specimen. Even if this were true, the author should state it in the paper. I believe a paper should include all the information necessary to permit one to duplicate an experiment. Is this asking too much? I think not.

There are more than 100,000 XRD and XRF units in operation in teaching institutions, industrial, and research laboratories all over the world. They are used to study a gamut of materials and are a vital tool for industrial quality assurance... probably the major application. The materials are an eclectic mixture of powders, solids, metals, liquids, plastics, environmental, forensic, etc. Do you believe that every one of these users has ready access to literature containing the proper method of preparing his or her specimens? I do not think so. Would these analysts be able to do better work if they had this information and did not have to find it out by trial and error over many months of work?

Definitions of the terms bulk, sample, and specimen are given at this point because they appear below. Although it is often untrue, it is assumed the material submitted for analysis (referred to as the sample) has been sampled (taken) correctly from a source (the bulk). When the specimen is properly prepared from the sample it is ready for analysis.

Most analysts are aware that several errors occur during an XRD or XRF analysis. These errors include standard counting errors, instrument variation errors, instrument operation errors, and specimen preparation errors. Much to the chagrin of the analyst, he is faced with the reality that specimen preparation errors can be, and usually are very large and are the only ones which can not be corrected by the use of software and computers. These errors can be as large as several hundred percent. Admittedly, such large errors are rare, however, they can occur if the specimen is not prepared properly. This statement may shock readers; however, it comes as a bigger shock to a person just starting to use XRD or XRF. The real shock is when the beginning analyst tries to find the procedure in the literature.

It may not be comforting for an analyst to know other users of XRD or XRF equipment have the same problem; namely, the one of preparing a meaningful specimen which will yield accurate data. The goal of every XRF analyst is to produce accurate results quickly and inexpensively. In the case of XRF this requires that the small volume of the specimen exposed to the X-ray beam contains material which has the desired physical form and chemical composition. This implies the specimen is homogeneous, free of contamination, in the desired physical condition, and represents the sample from which it was prepared. In many XRD specimens, preferred orientation can be a serious problem.

100,000 users is an impressive number! It is an army. It is all the more impressive when one realizes how the X-ray work done in these laboratories affects our daily lives. Just imagine what a nightmare it would be if all XRF and XRD units stopped working at one time.

What about training in the science of XRD, XRF, and in specimen preparation? Where can one get this training? Is it readily available? Unfortunately, there are not many places one can get this training. The ICDD (International Centre for Diffraction Data) presents clinics on XRF and XRD and these are probably the most comprehensive ones given in the United States. Workshops at the Denver Conference contain one half or full day instruction on a variety of XRD and XRF subjects and on specimen preparation. These workshops are not as thorough as the ICDD clinics but instructors do an excellent job in the time allowed. Instructors have extensive experience... they are either college instructors or scientists working in industry and using the techniques on a daily basis. Are there other places to receive this kind of instruction in XRD or XRF? Courses in X-ray diffraction and single crystal analysis do exist. However, I do not believe there are courses dedicated entirely to XRF at any American college or university. XRF may get one lecture during the semester in a course on instrumental analysis, but that is about the extent of it. Students taking these instrumental analysis courses may receive some basic information about the technique of XRF analysis but I do not believe they receive extensive training on specimen preparation. Students entering industry are not totally prepared to us XRF. They are forced to learn the technique on the job, and learn fast. I do not know why this situation exists, but it does. It has for long time. Can anyone argue that an analyst is unprepared to analyze materials if he or she lacks sufficient training in the theory of XRF or in specimen preparation?

Many X-ray scientist want to learn more about specimen preparation. Almost two thousand of them have attended a workshop on specimen preparation at the Denver X-ray Conference over the past 20 years. Attendance has consisted of an eclectic mixture of scientists from industry and universities. Their experience ranges from none to more than 20 years. As chair of this workshop for 20 years I noticed some people have attended more than 3 times. This attendance record is proof that both beginners and experienced analysts are looking for this kind of information.

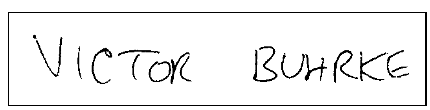
Three books which contain practical information and bibliographies are; “A Practical Guide for the Preparation of Specimens for X-ray Fluorescence and X-ray Diffraction” by Buhrke, Jenkins, and Smith (published by Wiley-VCH), another is “Principles and Practice of X-ray Spectrometric Analysis” by E. P. Bertin (published by Plenum), and lastly an “Introduction to X-ray Powder Diffractometry” by Jenkins and Snyder (published by Wiley lnterscience).

The study of materials is a fast moving science. Do we need more major technical advances in specimen preparation (like those in 1957 from Claisse, and the inventor of the shatter box (hockey puck) back in the late 50s) to keep pace with the advances in materials science? I think we do. What improvements in specimen preparation have occurred since the late 50s? I can not identify any as important as these two.

A sophisticated theoretical treatment of analytical X-ray data requires the specimen meet certain requirements, i.e., homogeneous, infinitely thick, flat, free of contamination, and of the proper size and shape to fit into the specimen holder. Claisse recognized that chemical reactions which occur during fusion convert the phases present into borate, glass-like disks which are homogeneous and are shaped properly to permit easy placement of the specimen in the specimen chamber of the spectrometer. The shape and hardness of the glass like disk (specimen) makes it convenient to handle but the most important result of fusion is the elements are distributed homogeneously. A lack of homogeneity of elemental distribution is a serious problem in XRF analysis and often difficult to eliminate. Fusion helped mitigate this problem... to a certain extent. Fusion is, however, not a cure all. It can cause the vaporization of certain elements. Fusion is also time consuming and expensive when compared to the simple method of pressing a powder into the form of a pellet. Regardless of these shortcomings, the discovery of fusion was a major positive contribution to XRF analysis-. Particle size homogeneity (not elemental distribution homogeneity) is also necessary when analyzing powders such as cements by XRF analysis. A grinder known as a shatter box (hockey puck) developed in the late 1950s enabled the analysis of cements because it not only ground the sample quickly but more importantly it produced specimens with particles of a uniform size. An accurate analysis of cements was not possible until the advent of the shatter box grinder. We are fortunate to have fusion and the shatter box available.

In many cases current specimen preparation techniques are antediluvian when compared to the current state of the software and equipment used in today's X-ray laboratory. The time is right for someone to do an analysis of the various methods in current use in XRD and XRF to tell us whether we are in good shape or if we are heading for trouble. I hope someone will do a literature search on this subject and write a paper on it. Such a paper could serve as the first step in a journey to find better ways to prepare a specimen. How good is the method of preparing powder specimens with trace amounts of material? How have the methods improved over the past 10 years? Are current techniques faster, easier, able to detect smaller amount, less expensive, easier to perform, etc.?

Future advances in XRD and XRF analysis depend upon a willingness of manufacturers, universities, and industry to devote time trying to improve upon existing methods of specimen preparation. These methods would also benefit from a conscientious effort on the part of authors and editors to include this practical information in all publications. Further progress should be expected and demanded by the X-ray analyst. If the literature does not supply practical information about specimen preparation where can one get it? Let the editors of the journals know that you would like to this information included in every paper accepted for publication.



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