Applied Inorganic Analysis

The 1929 first edition of *Applied Inorganic Analysis* [1] was a classic of its time, commonly referred to as the "analyst's bible." Co-authored by two Chief Chemists of the National Bureau of Standards, Gustav Ernst Fredrik Lundell and William Francis Hillebrand, the book represented the authors' comprehensive knowledge and broad practical experience. The 1953 second edition [2] was a thorough revision by Harry Aaron Bright and James Irvin Hoffman, who were Chief of the Analytical Chemistry Section and Assistant Chief of the Chemistry Division, respectively. Both editions of *Applied Inorganic Analysis* presented critical expositions of the chemical methods of analysis as applied to a broad range of complex matrices including metals, alloys, rocks, minerals, ores, ore concentrates, and glass. Each of the two editions stood alone as the authoritative single-volume reference for inorganic analysis for many years, and even today remains a valuable resource for pre-instrumental, classical methods of chemical analysis.

The story of the development of this remarkable book began in 1878 after the young American chemist William Francis Hillebrand completed a course in metallurgy and assaying at the mining academy in Freiberg, Germany. Fully intent on returning to America and impressed with the opportunities for young chemists as assayers, he took this course in order to supplement the training in mineral analysis that he had received from Bunsen, under whom he had earned his Ph.D., magna cum laude, at the University of Heidelberg, in 1875. Upon returning, he failed to find suitable work in the East, so he made his way in 1879 to Leadville, Colorado, where he became the third partner in a small assaying firm. Samuel F. Emmons, an occasional customer who was in charge of the Rocky Mountain Division of the newly formed United States Geological Survey (USGS), offered Hillebrand a job as a chemist. Considering this offer to be the opportunity of a lifetime, he quickly accepted, remaining in the Denver laboratory of USGS until 1885, when he was transferred to the Washington USGS laboratory. During his 28 years at USGS, Hillebrand made more than 400 complete (for that day) analyses of various rock samples [3].

In 1897 Hillebrand wrote a 50-page introduction to USGS Bulletin No. 148 [4] on the methods of analysis of silicate rocks, and this was translated into German and published in Germany in 1899. In 1900 this material was rewritten and enlarged to 114 pages and issued as an independent document, USGS Bulletin No. 176 [5]. This brought a flock of commendatory letters, all confirming what L. V. Pirsson said: "We must now look upon you as the final authority in silicate analysis." Another edition, which included the analysis of carbonate rocks, appeared in 1907 as Bulletin No. 305 [6]. This also was translated into German. An expanded edition was published in 1910 as Bulletin No. 422 [7], and the series culminated in 1919 as Bulletin No. 700 [8], a book of 285 pages. Even in the late nineteenth century and early twentieth century, the problem was not so much the lack of a suitable method for the final determination of an element as it was the lack of preceding separation methods that made an accurate determination possible, an occasional occurrence today. The significance of the analytical methods in this series of bulletins lay in Hillebrand's success in developing a workable separation scheme for materials as complex as silicate and carbonate rocks.

This book is expected to remain the key reference work for highly accurate classical analytical methods, and although these methods are less commonly practiced today, analytical chemists will continue to appreciate the theory, chemical knowledge, and artistry inherent to their application.

In 1908, Hillebrand became the second Chief Chemist of the NBS. In 1917 he succeeded in bringing G. E. F. Lundell from Cornell, where Lundell was an assistant professor of chemistry. In 1923 Hillebrand and Lundell together began writing *Applied Inorganic Analysis,* a project that Hillebrand had been advised to do 10 years earlier. A 900-page book was planned in five parts: I. General Considerations; II. The Determination of the Elements; III. Silicate Rock Analysis; IV. Carbonate Rock Analysis; and V. Miscellaneous Methods of Analysis. Parts III and IV were essentially a reprint of USGS Bulletin 700. The aim of the book

throughout was to present analytical methods that would work in the real world of complex matrices. In the introduction to the first edition, Lundell stated, "A fair criticism of much of the work that has been published on methods for the determination of the elements is that a great deal is claimed on the basis of experiments that have been carried out in pure solutions, and very little, if anything, is said as to how the methods are to be applied or what results can be expected in analyses of the more or less complex materials in which the elements are found. . . . The aim throughout the book therefore has been to stress the preparation of the solution for the determination that is to be made, rather than to describe processes that can be used with certainty only in the specific applications for which they were devised." [2] Herein lies the true greatness of this book. In addition to the broad range of matrices covered, the separation methods are discussed and developed in such a way that the knowledgeable analyst can adapt them to other complex matrices. Unfortunately, Hillebrand died in 1925 at a time when only about one-third of the book had been completed. Lundell went on to complete the book in 1929 using Hillebrand's detailed notes and his own already considerable expertise

The second edition of *Applied Inorganic Analysis* was begun by Lundell in the late 1930s, at which time he was the accepted dean of the world's inorganic analytical chemists. However, he had not completed the book at the time of his death in 1950. The job of finishing the second edition was handed over to Harry Aaron Bright (then Chief of the Analytical Chemistry Section), and James Irvin Hoffman (then Assistant Chief of the Chemistry Division). It is indeed fortunate that the task fell to these two very capable men. Hoffman came to NBS in 1919 and distinguished himself with atomic weight determinations and the development of several significant chemical processes that were essential to the American effort in World War II. In 1938, he co-authored with Lundell *Outlines of Methods of Chemical Analysis* [9], which was intended as a companion volume to Applied Inorganic Analysis, and this book became a classic in its own right. Harry Aaron Bright joined the staff at NBS in 1913 as a laboratory assistant and worked closely with Lundell until Lundell retired in 1948. After Lundell's death, Bright became the recognized authority for the chemical analysis of metals and alloys in the U.S. Together, Bright and Hoffman made a thorough revision of the first edition of *Applied Inorganic Analysis,* adding significant new material, much of which had been developed at NBS by themselves together with R. K. Bell, R. Gilchrist, J. L. Hague, H. B. Knowles, B. F. Scribner, J. K. Taylor, and E. Wichers. There was scarcely a page of the old edition that was not improved either by the omission of out-of-date material, by the inclusion of new references and footnotes, or by extensive additions to the text itself. For example, in the intervening years between the editions, R. Gilchrist had developed a completely new separation scheme for all the precious metals in the absence of gold, and this replaced the separation method of E. Wichers which had appeared in the first edition. In order to prevent the book from increasing to an unwieldy size, Part V of the first edition on the analysis of glass, bauxite, and other refractories was unfortunately omitted.

That the first edition had become a classic was made plain in the book reviews of the second edition. In his book review which appeared in the August 1953 issue of *Analytical Chemistry,* M. Gilbert Burford commented, "A new edition of this classic work should be welcomed by all chemists, and it is evident that the book will continue to be invaluable." He went on to say, "The authors' hope that 'here and there something helpful in solving a pressing problem may have been presented' should prove to be one of the classic understatements of the year." [10]. In his book review in the February 1954 issue of *The Analyst,* L. S. Theobald wrote: "In the reviewer's opinion, the work is the best of its kind. It is one to be used both in the library and at the bench, and it is indispensable to every chemist engaged in 'Applied Inorganic Analysis'." [11].

The book contains a wealth of durable information that continues to be relevant today. The basic material in Parts I and II on sampling and sample preparation has fundamental importance. Sections 4 and 5 of Part I contain common and special methods of separation, employing both inorganic and organic reagents, which will remain useful in the preparation of samples for instrumental analysis. The extended discussions in Part II for each of the elements no doubt will prove to be the most enduring material in this book. For each element (or small group of elements), there is a detailed discussion of occurrence, importance, general considerations of the chemical behavior that are important in its analysis, dissolution procedures for mineral forms of the element, methods of separation, and methods of determination.

This book is expected to remain the key reference work for highly accurate classical analytical methods, and although these methods are less commonly practiced today, analytical chemists will continue to appreciate the theory, chemical knowledge, and artistry inherent to their application. *Applied Inorganic Analysis* will continue to be a clear window on the past, providing

detailed discussions and procedures for those needing to perform classical analytical determinations. Those fortunate enough to own a copy have a masterpiece and a treasure.

Prepared by Charles M. Beck II.

Bibliography

[1] W. F. Hillebrand and G. E. F. Lundell, *Applied Inorganic Analysis,* John Wiley & Sons, New York (1929).

[2] W. F. Hillebrand, G. E. F. Lundell, H. A. Bright, and J. I. Hoffman, *Applied Inorganic Analysis, Second Edition,* John Wiley & Sons, New York (1953).

[3] W. F. Hillebrand, A Plea for Greater Completeness in Chemical Rock Analysis, *J. Am. Chem. Soc.* **16**, 90-93 (1894).

[4] W. F. Hillebrand, Some Principles and Methods of Analysis Applied to Silicate Rocks, in *Analyses of Rocks with a Chapter on Analytical Methods, Laboratory of the United States Geological Survey, 1880-1896,* Frank Wigglesworth Clarke and W. F. Hillebrand, *U.S. Geological Survey Bulletin,* No. 148 (1897) pp. 15-64.

[5] W. F. Hillebrand, *Some Principles and Methods of Rock Analysis, U. S. Geological Survey Bulletin,* No. 176 (1900).

[6] W. F. Hillebrand, *The Analysis of Silicate and Carbonate Rocks, U. S. Geological Survey Bulletin,* No. 305 (1907).

[7] W. F. Hillebrand, *The Analysis of Silicate and Carbonate Rocks: A Revision of Bulletin 305, U.S. Geological Survey Bulletin 422,* (1910); slightly revised when reprinted in 1916.

[8] W. F. Hillebrand, *The Analysis of Silicate and Carbonate Rocks: A Revised and Enlarged Edition of Bulletin 422, U.S. Geological Survey Bulletin 700* (1919).

[9] G. E. F. Lundell and James Irvin Hoffman, *Outlines of Methods of Chemical Analysis,* John Wiley & Sons, New York (1938).

[10] M. Gilbert Burford, review of *Applied Inorganic Analysis, Second Edition,* by W. F. Hillebrand, G. E. F. Lundell, H. A. Bright, J. I. Hoffman, *Anal. Chem.* **25**, 1279-1280 (1953).

[11] L. S. Theobald, review of *Applied Inorganic Analysis, Second Edition,* by W. F. Hillebrand, G. E. F. Lundell, H. A. Bright, J. I. Hoffman, *Analyst (Cambridge, UK)* **79**, 113-115 (1954).