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BIOGRAPHICAL MEMOIR

OF

WILLIAM FRANCIS HILLEBRAND

1853-1925

BY

FRANK WIGGLESWORTH CLARKE

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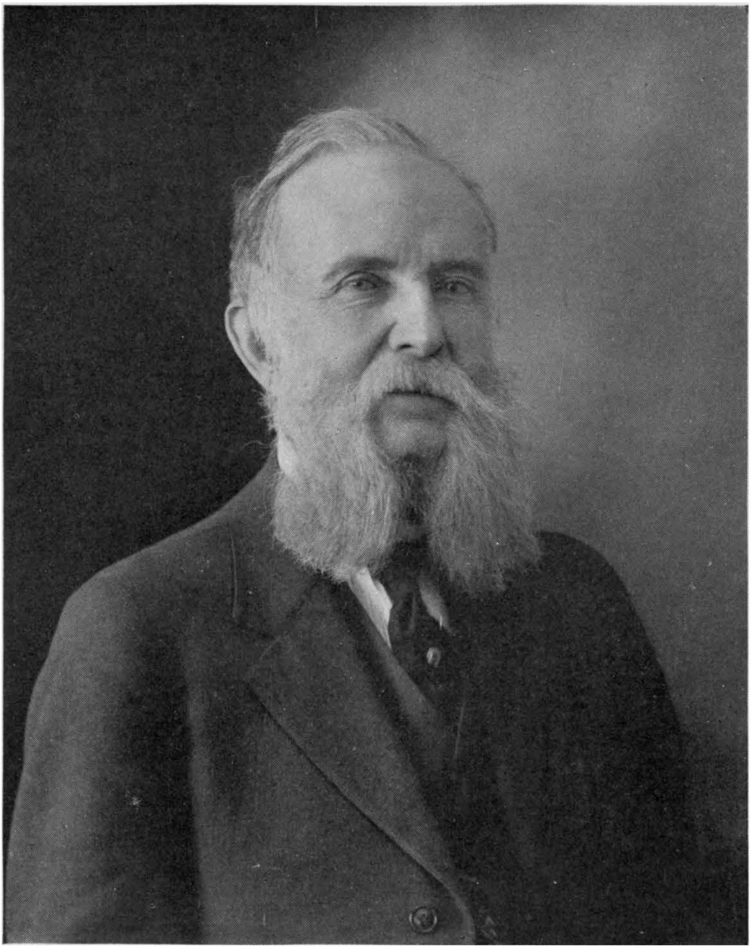

W. F. Hillebrand

BIOGRAPHICAL MEMOIR OF WILLIAM FRANCIS HILLEBRAND

BY FRANK WIGGLESWORTH CLARKE

The following memoir falls naturally into three parts. First, an outline of his life, prepared by Hillebrand for the use of the National Academy of Sciences. Second, a discussion of Hillebrand's career as a chemist, by the present writer. This includes a few lines of preface, covering some omissions in the autobiography. Third, a bibliography prepared by Hillebrand himself.

AUTOBIOGRAPHICAL NOTES

BY W. F. HILLEBRAND

I was born at Honolulu, December 12, 1853. My father, William Hillebrand, was born at Nieheim, near Paderborn, Westphalia, Germany. His father held a judicial position. Educated for the medical profession at Göttingen, Heidelberg, and Berlin, my father, one of four brothers, and the oldest, I think, on account of his health, took a sea voyage to Australia late in the 40's, and thence, at a date unknown to me, went to Manila, where he practiced his profession for a time, but becoming very ill, had to seek another climate. He embarked for San Francisco on a brig with little expectation of living to reach his destination. He did, however, and was advised to seek the Sandwich Islands, where he must have arrived about 1851. By the exercise of great care he recovered his health completely. In Honolulu he met and married Anna Post, stepdaughter of Wesley Newcomb, a well-known conchologist. From this union sprang two sons, myself and Henry Thomas, the latter over 14 years younger than myself.

In the practice of his profession my father became perhaps the most prominent physician in the Islands, and filled several positions of importance, besides being one of the Privy Council of King Kamehameha the Fifth. An ardent botanist and horticulturist, he explored the Islands thoroughly and introduced from various parts of the world many plants and trees new to the kingdom. His garden became the show place in Honolulu from the horticulturist's point of view. In 1865 he was sent, my mother and I accompanying him, to investigate labor conditions in China, Java, and India, with a view to securing a supply of efficient labor for the sugar plantations of the Islands. While on this trip he dispatched two shiploads of Chinese coolies from Hongkong, which was the beginning, on a considerable scale, of what became a strong influx. While on this trip he made large collections of living plants and birds, most of which reached their destination in fair shape and not a few became thoroughly at home in their new habitat. I have very vivid recollections of helping to feed and care for the birds and deer on the seven weeks' trip from Hongkong to San Francisco by sailing vessel, a somewhat rough and gloomy voyage.

In the late 70's, while living in Madeira, he inaugurated the emigration of Portuguese laborers with their families from that island to Honolulu—a movement which, rendered difficult at the start by the opposition of the Portuguese Government, took on considerable proportions when the first emigrants sent back favorable reports.

In 1870 my mother, brother, and I left the Islands, I to enter Cornell University preparatory to completing my education in Germany. A year later my father followed us and spent a year in studying botanical collections, chiefly with Prof. Asa Gray at Harvard, as a preliminary to entering upon the preparation of a Flora of the Hawaiian Islands. In the summer of 1872 we all took ship for Europe by way of Scotland; thence to Hamburg, Bremen, Braunschweig, Cologne, and Bonn, and eventually Heidelberg, where I entered the University in November, 1872. The family remained in Heidelberg for a few years, but my mother's health became bad and all but myself sought the milder climate of Madeira, and later Teneriffe, whence, as my mother's health became better, they returned to Europe and lived in Switzerland, and then in Heidelberg for a time. During most of this time my father was engaged in writing the Flora. It was at Heidelberg that he died, on July 13, 1886, just after he had placed the text of the work in the hands of the printer. This work it was my pleasure and duty to edit and see through the press, with the assistance of my father's friend, Prof. E. Askenasy, of Heidelberg. Some of the foregoing information and some additional data are to be found in the editor's preface to the Flora.

My early education was acquired at Oahu (Punahou) College, near Honolulu, under the chief guidance of Prof. W. D. Alexander, with exception of the two years, 1862 and 1863, at the College School in Oakland, California, at the head of Webster Street on 12th. My grandmother and step-grandfather Newcomb were then living in Oakland, and I had already visited them in 1861 without my parents. It may here be remarked that my grandmother was descended from the Wells family, of Colchester, Conn. My maternal grandfather, William Stoughtenburgh Post, whom I never saw, was a descendant of a Quaker family of Long Island by way of Hyde Park, Dutchess County, New York.

Latin, Greek, French, elementary mathematics, composition, and spelling comprised essentially my school curriculum, which studies were continued at Cornell, with German added. My father was a fine musician as well as botanist, but I did not inherit has tastes, although I loved to accompany him on his excursions through the dense forests and up the lofty mountains of our island home, and was zealous in aiding his search for new and rare plants and flowers. Not seldom did I make such botanical all-day excursions by myself or with a boy companion, sometimes at considerable risk. Botany as a subject of study interested me not at all, and for music I had but an indifferent ear and taste, though fond of it if not ultra classical and to me devoid of melody.

Up to the time of reaching Bonn, in the summer of 1872, I had shown no bent or aptitude, unless a liking for geography, travel, and history can be so called, for any line of study that pointed toward a choice of a profession or career. Mathematics I abhorred, and English composition was something that there seemed little hope of my ever becoming master of.

My inability to get a good working understanding of mathematics has been a serious handicap throughout my professional career. My actual study of English was limited to the merest elements, my father laying much greater stress upon the classics. While the study of the latter has been of much assistance and of no little cultural benefit, I can not too much deplore the scant consideration that was given to English. My father, though himself a master of English, I think was inclined to believe that my later home would be in Germany, notwithstanding that all my youthful associations were with Americans and my predilections for America. This bias arose from the great preponderance of Americans over Europeans in Honolulu, from the fact that all my early teachers were Americans, that the language of the family was English, and that I had frequent association and correspondence with my mother's family in California and none with my father's, except two of his brothers—one in California, the other in Honolulu, who married American wives. Out of all this grew a strong intention to be and remain an American myself.

On arriving in Germany in the summer of 1872 it became necessary to choose a career. Having seen much of a physician's life through my father and step-grandfather Newcomb, medicine offered no charms. Law was still less to my liking, for the reason that I lacked utterly the mental qualifications of a successful lawyer. The engineering professions were excluded from consideration by reason of my weakness in mathematics. The outlook was unpromising. One day my father suggested chemistry—a possibility that had never occurred to me. In Honolulu I had learned some of the simplest elements of the science and had enjoyed the study. My father's suggestion appealed to me and I decided then and there to become a chemist, or at least to make a try at becoming one. Fortunately for me, in those days a considerable knowledge of mathematics was not considered essential for a chemist. I matriculated at Heidelberg in November, 1872, and studied under Bunsen, Kirchhoff, Kopp, Blum, Leonhard the younger, and Karl Klein, and listened to an occasional course under Treitschke and one or two others. After five semesters I took my degree (Ph. D.) *summa cum laude,* March 2, 1875, and with the three first named as my examiners continued on what we here would call postgraduate work under Bunsen.

I must not fail here to pay tribute to my friend and close associate at the time, Thomas H. Norton. He it was who prompted me to prepare with him for examination in advance of the time contemplated; and for three months before examination we spent all our spare time together, quizzing each other in German every night until midnight, then for recreation indulging perhaps in a game of chess, at which I was the unfailing loser. Norton was more mature than I and of superior mental power, and it was to his unfailing help that my success at that time was largely due. With him during the next semester I did my first research work, and had justice been done our joint paper "On Metallic Cerium, Lanthanum, and Didymium" would show him as the chief author. My second research, on the specific heats of the metals named, was an outgrowth of the first and was done alone. Both papers were published in Poggendorff's Annalen and the second was copied in translation by the Philosophical Magazine.

After two semesters of this research work I studied for three semesters at Strassburg, chiefly organic chemistry under Fittig and microscopical petrography under Rosenbusch. The organic branch of chemistry did not appeal to me and I made less use of my opportunities than would otherwise have been the case. Nevertheless, with Fittig's help, I determined the constitution of quinic acid and published also a short paper on the crystallography of one of its derivatives, assisted herein by Prof. Paul Groth, who kindly placed the facilities of his laboratory at my service.

Fully intent on returning soon to America and impressed with the opportunities for young chemists as assayers in the western mining regions, I decided to finish my studies by a course in metallurgy and assaying at the mining academy of Freiberg in Saxony. This, as it turned out, was a most wise move, for supplementing the experience in mineral analysis gained with Bunsen it opened the way to an employment which fitted me in no small degree for my earlier work subsequently with the U. S. Geological Survey.

In the fall of 1878 I returned to the United States and spent the following nine months in Ithaca, N. Y., where my grandparents were living, and in New York City, looking about for a situation. On a visit to Philadelphia I first met Professor Genth and his assistant, Edgar F. Smith. Professor Genth's fame as a mineral analyst was well known to me, and on that account I felt specially drawn to him, our tastes being so similar in respect to mineral analysis. I should perhaps say here that at Heidelberg I had met and seen much of Edward S. Dana. He and I in 1873 took a foot trip through the Eifel and to the Kaiserstuhl, in the Rhine Valley, and thus my attention became first drawn to mineralogy, in which Dana had already had a good deal of experience.

In the spring of 1879 the press had a good deal to say about the new organization of the Geological Survey under Mr. Clarence King. This seemed to be a better opening than a university position or one in the industries, which at that time employed few chemists; so I wrote to Mr. King, transmitting recommendations from Bunsen and President White, of Cornell University. No answer came until months later, when at Leadville Mr. King wrote from Salt Lake City that it had not been determined until then whether or not there would be an opening for a mineral chemist. The decision was now in the negative. An interesting incident of the spring was a trip to Trenton, N. J., at the invitation of Mr. Henry Villard, the widely known railroad reorganizer, whom I had come to know in Heidelberg, where his family sojourned for some time. The trip was made in a special car, with a few other invited guests, for the purpose of witnessing what was perhaps the first test of firing a boiler by a jet of "atomized" oil.

In Lansingburgh, where I spent some time with friends, I met a Mr. Ballou from Leadville, Colo., where he and a brother operated an ore-sampling plant. His advice helped me to reach a decision that I had been tending toward; so late in June 1879, I went to Denver, where I sought advice from Senator Hill, to whom I had an introduction. He himself had nothing to offer, and I soon went to Central City and Georgetown. At Central I met an old Freiberger, brother of a student I had known in Heidelberg, who was assayer at an outlying mine, and one day accompanied him to the mine and helped him with his day's work, and so learned how different the conditions of assaying were from them as I had learned the art in Freiberg. My friend and everyone talked of little else but Leadville, where a number of Freibergers were then employed in mines and smelters, among them one or two whom I had known at Freiberg. It seemed the place to go to, so I soon found myself there, and within a few days formed a third member in the firm of William S. Ward and Robert E. Booraem, Ward wishing to retire and remain for a time as a silent partner. Both he and Booraem retired in the course of the year and I continued the business alone till July, 1880.

During the year I had met Mr. S. F. Emmons, in charge of the Rocky Mountain Division of the U. S. Geological Survey, who came occasionally to my office for an assay. About the time of the great miners' strike, when business became bad, Mr. Emmons asked me if I would like to take a position as chemist in his division of the Survey. Thus was offered me from a clear sky the very position I had sought when applying to Mr. King. Naturally I accepted the offer and for a month lived with Mr. Emmons and his two geologist assistants, Cross and Jacobs, in a comfortable cottage which had been rented while they were on field service in the Leadville district, the main office being in Denver. During this time I collected samples of ores at the smelters for later examination in the laboratory, which was to be created in Denver. For some time after reaching Denver I was occupied in planning and then supervising for Mr. Richard Pearce the building of a one-story brick structure on the bank of Cherry Creek where Lawrence Street crosses it. The site was on the southeast corner of the block on which the building stood. This laboratory Mr. Pearce rented to the Survey and in it I worked first with Antony Guyard and later with L. G. Eakins. From July 3, 1880, till November, 1885, I remained in Denver; then was transferred to the Washington laboratory, which had been established in 1883, under F. W.

Clarke, Chief Chemist. On July 1, 1909, I entered on duty at the Bureau of Standards in Washington, as Chief Chemist, succeeding after an interval my predecessor, William A. Noyes.

From 1892 to 1910 I filled the chair of General Chemistry and Physics in the National College of Pharmacy, which, while I was still with it, became affiliated with the George Washington University. From it I received the honorary degree of Doctor of Pharmacy.

BIOGRAPHICAL SKETCH

The foregoing autobiography is obviously incomplete in several respects. It ends with his transfer to the Bureau of Standards, a deficiency which is amended by an excellent bibliography, prepared by Hillebrand himself, which gives a list of his publications down to the end of 1923. Three later titles have been added by the present writer. Another lack is characteristic of Hillebrand's modesty—for nothing is said of the honors he received in recognition of his scientific achievements. Of this, more later. September 6, 1881, Hillebrand married Martha May Westcott, of Perrysburg, Ohio. He died on February 7, 1925, of heart failure, following an operation which, however, was not the cause of his death. His widow and two sons, William Arthur and Harold Newcomb, are still living. William is an electrical engineer at Palo Alto, California; Harold is professor of English in the University of Illinois. The two sons are worthy of their father.

So far we have a clear outline of Hillebrand's ancestry, his education, and his training as a chemist—a training which he received under masters of the first rank, whose teaching was an inspiration. In this he was most fortunate. His career as a chemist may now be considered in four sections, namely, his work in Germany, in Colorado, in the Geological Survey at Washington, and last of all in the Bureau of Standards.

The original work done by Hillebrand in Germany is represented by four published papers. Two of these, in the field of organic chemistry, serve to illustrate the breadth of his training; the other two, the joint work of Hillebrand and Norton, laid the foundation of his reputation. Metallic cerium, lanthanum, and "didymium" were for the first time isolated in reguline form and their specific heats were determined. This research fixed the valency of the metals and established their position in the classification of the elements. That the "didymium" of 1876 has since been proved to be a mixture of several closely related metals does not detract from the fundamental significance of the investigation.

Hillebrand's career in Colorado began as an assayer in Leadville. We may assume that his work there was done with his usual accuracy, but it added nothing to his permanent reputation. When, however, the United States Geological Survey established a laboratory in Denver, Hillebrand was called to it as a chemist, and his serious work in geochemistry began. To him the appointment was the opportunity of his lifetime, and he surely made the most of it. There were minerals to be analyzed and described, such as the zeolites of Table Mountain and the Cryolite of Pikes Peak, and in this work he had the valuable cooperation of Whitman Cross, the distinguished petrographer. There was also one new mineral to his credit, the ferric tellurite, emmonsite. His analyses of the rocks of Leucite Hills were of more outstanding importance and mark the beginning of an epoch in the history of chemical petrology. The older analyses of igneous rocks had been, as a rule, exceedingly crude; only their more important constituents were determined, while even such common elements as titanium, phosphorus, and manganese were either overlooked or ignored. Hillebrand's analyses were of much higher order, as regards both accuracy and completeness. A new standard was set from which Hillebrand never departed.

In 1885 the Denver laboratory was discontinued and the chemical work of the Geological Survey was concentrated in Washington. Thereafter, for twenty-four years, Hillebrand was my intimate colleague. As Chief Chemist, I was able to relieve him of much purely routine work, to throw opportunity for research in his way, and to encourage him in all his investigations. The work that he published was, however, all his own, and he alone is entitled to credit for it. It naturally followed the same lines as were followed in Denver, but with greater variety and a much larger field to cover. Rocks and minerals were analyzed and methods of analyses were tested and improved. Incidentally a number of new minerals were recognized and described, and their titles can be found in the bibliography which concludes this memoir. One of them, however, deserves special notice here, as showing Hillebrand's generosity in dealing with colaborers. He was engaged upon a study of the alunite-jarosite group when he received from Prof. S. L. Penfield a sample of supposed jarosite from a new locality. It resembled ordinary jarosite in all external particulars, but proved on analysis to be entirely new. Instead of the potassium of normal jarosite it contained lead, and so was given the name plumbojarosite, and in publishing his description of it he made Penfield, who had had no suspicion of its true character, joint author of his paper. Few men would have shown so much liberality. The men who worked with Hillebrand always received full recognition for their services. Some mines in Utah have in recent years yielded plumbojarosite in quantities sufficient to make it a profitable ore of lead. If it had not been for Hillebrand's discovery of the species, it might have been discarded by the miners as worthless. That actually happened in one instance, until a member of the Geological Survey pointed out the real value of the ore and overcame the skepticism of the too practical miners. We have here a good example of the economic significance of a purely scientific research.

It would be easy to give the mere details of Hillebrand's analytical work, but that would furnish no real evidence of his influence upon other analysts. While in the Washington laboratory of the Geological Survey he made more than 400 *complete* analyses of silicate rocks, on the standard of completeness which he established in Denver. Many more analyses of similar character were made by his associates in the laboratory, following his example and often aided by his guidance. It is not necessary here to discuss technicalities, but the broad significance of this work may be shown statistically as follows:

In his great compilation and classification of analyses of igneous rocks, Washington[1] has tabulated 8,602 analyses pub-

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[1] H. S. Washington: U. S. Geological Survey Prof. Paper No. 99, 1917.

lished or made between the years 1884 and 1913, both inclusive. These analyses are divided into two classes, as "superior" and "inferior," a distinction based upon a rigorous system of weighting. Six thousand four hundred and fifty-five analyses are rated as superior, and of these 1088, or one-sixth of the whole number, were made in the laboratories of the Geological Survey. This alone is enough to show Hillebrand's influence upon the work of his immediate associates. The "inferior" analyses of Washington's classification are characterized by incompleteness, bad summation, or by obvious errors in the determination of essential constituents in a rock; and very few such analyses are charged against the chemists of the Survey. Even these analyses are often justifiable. An analysis should be an answer to a definite problem; and if that answer is given by a partial analysis, nothing more is demanded by the petrographer for whom it was made. He states his problem and the answer is given. A complete analysis may be desirable, but it is not necessary.

In 1894 Hillebrand published a paper entitled "A plea for greater completeness in chemical rock analysis."[2] Thus, by precept as well as by example, his influence upon other analysts was widely extended. The analyses of igneous rocks made since Hillebrand began his work have been much more thorough. This improvement is shown in the work of other American chemists, and the admirable analyses made by British and Australian analysts are fairly comparable with his. Petrographers who were formerly satisfied with incomplete analyses now demand something better; and what were thought to be insignificant traces of the minor constituents of a rock are now seen to be useful. The study of igneous rocks is much more exact than when Hillebrand began his labors, and the evidence of the thin section is reinforced by the chemical analyses.

In 1897 Hillebrand wrote a fifty-page introduction to Bulletin No. 148 of the U. S. Geological Survey on the methods of analysis of silicate rocks. The rest of the bulletin was a compilation of the analyses so far made in the Survey. This introduction was soon translated into German and published

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[2] Journ. Amer. Chem. Soc., vol. 16, pp. 90-93.

in Germany. In 1900 it was rewritten and enlarged to 114 pages and issued as an independent document, Bulletin No. 176. Another edition, which included the analysis of carbonate rocks, appeared in 1907 as Bulletin No. 305. This also was translated into German. It was republished in 1910 as Bulletin 422, and the series culminated in 1919 as Bulletin No. 700, a book of 285 pages. With each edition Hillebrand's reputation as an analyst was increased and his influence upon both chemists and petrographers was greatly broadened. Other papers on the determination of various elements were published at intervals between the appearance of his bulletins. During the last year of his life Hillebrand was engaged upon the preparation of a more general treatise upon inorganic analysis. This task, unfortunately, he did not live to finish. He left, however, notes sufficient to enable his collaborator, Mr. G. E. F. Lundell, to undertake its completion.

In addition to his analyses of igneous rocks, Hillebrand made many analyses of minerals, and especially of those which contained the rarer elements, such as vanadium, uranium, columbium, tantalum, and the metals of the "rare earths." These anaylses are of the most difficult kind and were made with Hillebrand's characteristic thoroughness and accuracy. He also made important analyses of mine waters and of mineral springs. The spring known as Ojo Caliente, near Taos, N. M., is noteworthy on account of the unusual content of fluorine. A spring water rich in zinc from the zinc region around Joplin, Mo., is also remarkable. Both of these waters were analyzed by Hillebrand.

Among Hillebrand's analyses of minerals, there is one group which has proved to be of extraordinary although unsuspected importance. They led to the discovery of terrestrial helium—an element which was previously known only by its lines in the spectrum of the solar corona. The history of that discovery is briefly as follows:

In the summer of 1887 the present writer obtained from the owner of a feldspar quarry in Connecticut a remarkable crystal of uraninite. It was unusually large, with brilliant luster and apparently of a high degree of purity. It was turned over to

Hillebrand for investigation and a portion of the crystal was broken off for analysis. The first step in the process of analysis was the solution of the powdered material in sulphuric acid, and during solution a slow evolution of gas was noticed. An ordinary routine analyst would have called the gas carbon dioxide and thought no more about it; but this was not Hillebrand's procedure. Carbon dioxide would have been given off rapidly when solution began, but the gas from uraninite was emitted continuously until solution was complete. What was it? In order to answer this question Hillebrand, with the cooperation of Dr. William Hallock, collected some of the gas in a Geissler tube and found it to give a brilliant nitrogen spectrum. Some of it, sparked with hydrogen, formed ammonia, and with oxygen it gave nitric acid. Nitrogen alone of all known gasses fulfilled these conditions, and so it was recorded as nitrogen. This result was so surprising that the investigation was carried further, and samples of uraninite and its varieties were obtained from Norway, Texas, Colorado, and other localities, and *complete* analyses, 18 in all, were made of them. In most of them, but not in all, the evolution of gas was observed; but apart from that fact the series of analyses was by far the most thorough investigation of the composition of uraninite that had ever been made, and it has not since been equaled.[3]

In 1895 Raleigh and Ramsay discovered argon, an essential constituent of the atmosphere, and Ramsay at once began a search for other sources of it. His attention was called to Hillebrand's paper, and from a sample of cleveite (a variety of uraninite from Norway) he isolated the gas and determined its chief properties. A sample of it was sent to Sir William Crookes, who examined it spectroscopically and found and measured the same lines that were given by the helium of the sun. Terrestrial helium was discovered, and since then many other sources of it have been found. At first the discovery was one of purely scientific interest, but helium now is obtained from the natural gas of Kansas and adjacent States in suf-

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[3] For Hillebrand's complete paper, see Am. Jour. Sci., ser. 3, vol. 40, 1890, p. 384; also, in U. S. Geol. Survey Bull. No. 78, 1890.

ficient quantities to be used, instead of the inflammable hydrogen, for inflating balloons.

Very soon after the public announcement of Ramsay's discovery I received the following letter from him:

University College, London,
Gower St., W. C., *March* 25, 1895.

My Dear Professor Clarke:

I come to you with a request which I hope you will grant. It is to procure for me a considerable quantity of the uraninite from which Hillebrand obtained his nitrogen. I don't know what to ask for, for I don't know how plentiful the uraninite is. As much as you can conveniently let me have. Of course I will pay expenses. I have obtained from cleveite a mixture of argon and helium! There is hardly any nitrogen, and I am afraid that Hillebrand must have deceived himself. The nitrogen I find amounts to 0.06 a per cent or so of the gas, and may easily have been derived from the water. The gas shows almost all the argon lines—not quite all, suggesting that air argon contains some unknown constituent—and in addition a fair number of lines, some brilliant, some less so, but one of astonishing brilliancy, of wave length 587-49, exactly the wave length of D_3 of the solar corona, which has been ascribed to helium.

A note has been read today at the French Academy, and I have sent in an account to the R. Society. I am off to Paris on Thursday, so I am very hurried. My time is occupied in preparing a lecture for the French Chemical Society. Of course I was led to investigate this gas, so as to obtain more clues to argon compounds, and my next work with argon will be to try the action of uranium oxides on it. But helium is terribly tempting, so I am afraid argon will come second best off for the present.

Kind regards.

Ever sincerely, W. Ramsay.

This letter was referred to Hillebrand, who at once wrote to Ramsay the following letter, of which the original draft is now in my possession:

U. S. Geological Survey,
Washington, D. C., *Apr.* 4, '95.

Prof. Wm. Ramsay.

Dear Sir: A week ago Dr. Hallock, who was so good as to aid me some years since in a certain portion of my examination of the supposed nitrogen from uraninite, wrote me a few lines suggesting the identity of that gas with argon and calling my attention to peculiarities noticed by us at that time in its spectrum. Two days later a New York paper published a telegraphic dispatch announcing your identification of the gas from cleveite as argon with admixed helium, and today I have been permitted to read your letter on the same subject to Prof. F. W. Clarke. Dr. Hallock's words made comparatively little impression upon me, but since the announcement in the *New York Journal* I have gone over in my mind the whole subject, and even meditated preparing a fresh lot of the gas from some of the Connecticut material, with the intention of having it examined by an expert. In the meantime I waited with interest for further details of your discovery in the hope that the newspaper might not have been altogether correct in the implied statement that the gas contained *no* nitrogen, for while I had about come to the conclusion, for reasons given below, that my gas might well have contained argon, I was very unwilling, and I still am so, to believe that the brilliant spectrum of nitrogen observed by me could have been due to atmospheric nitrogen introduced at some stage of the manipulations. It was this brilliant nitrogen spectrum, coupled with the formation of ammonia and nitric acid, which has led me to believe firmly, notwithstanding certain other facts, that the gas could at most contain no more than small amounts of other constituents than nitrogen. At the same time I have always considered that the gas was well worthy of further examination, and I have often regretted not having at that time turned over some of it to an expert in spectroscopic work.

The circumstances and conditions under which my work in this line was done were unfavorable; the chemical investigations had consumed a vast amount of time and I felt strong scruples about taking more from my regular routine work. I was a novice at spectroscopic work of this kind and was thereby led to attach too little importance to certain observations which in the light of your discoveries deserved the utmost consideration. Among other things, it puzzled me that the formation of ammonia should have proceeded so very slowly, and that only comparatively small amounts of the gas should have been converted at the expiration of even several days' passage of the current. But, having had no previous experience, I was unable to judge whether this was especially abnormal or not; it might have been due to insufficient current or to some other cause. A similar observation applies to the contraction on sparking with oxygen, which was only carried far enough to prove a contraction and to obtain the tests for nitric acid.

It doubtless has appeared incomprehensible to you, in view of the bright argon and other lines noticed by you in the gas from cleveite, that they should have escaped my observation. *They did not.* Both Dr. Hallock and I observed numerous bright lines on one or two occasions, some of which apparently could be accounted for by known elements, as mercury or sulphur from sulphuric acid, but there were others which I could not identify with any mapped lines. The well-known variability in the spectra of some substances under varying conditions of current, degree of evacuation of the tube, led me to ascribe similar causes for these anomalous appearances and to reject the suggestion, made by one of us in a doubtfully serious spirit, that a new element might be in question. The various degrees of doubt with which the announcement of the discovery of nitrogen in uraninite were at first received were, moreover, little calculated to inspire one with a desire to announce a new element on the basis of such imperfect spectroscopic observations as had been made, and I finally came to the conclusion that the bright lines—since to the best of my recollection they were not constant or always the same in the two or three samples of gas examined—were probably not due to any original constituent of it. For this reason I most unfortunately made no reference in my published paper to an unusual appearance of the spectrum, which I so much the more regret because I have thereby laid myself open to criticism on the score of careless observation.

Do not mistake my purpose in entering thus at length upon explanations. I have not the slightest thought of claiming or hinting at a prior discovery. I merely wish to absolve myself in your mind, in part at least, from the charge of gross carelessness. While the observation was not lacking, unfortunately for me the same can not be said of the interpretation of those observations.

I take pleasure in sending you, under separate cover, by registered mail, the final remnant of that portion of my uraninite from Glastonbury, Connecticut, 3½ grams in weight, capable of affording about 70 c. c. of gas, of which the analysis is given under V in Table I and on which the special experiments detailed on the last few pages of my paper were made. I also inclose three crystals and fragments of crystals from the same locality aggregating considerably more in weight. Their composition is unquestionably the same as that of the 3½ gram lot. I will write at once to a gentleman in Connecticut who was in possession of more of the same material and endeavor to obtain some for you, but am not at all sure of success.

The material in the small tube contains about 2.4 per cent of the gas, but I must warn you that it is a matter of many days to effect anywhere near complete decomposition of this variety by hot dilute sulphuric acid. Fusion with an alkaline carbonate will effect complete liberation in a short time. It is possible that the gas from this variety may really contain nitrogen in some quantity—at least I may be excused for hoping so. Possibly also the gas from bröggerite, which I examined spectroscopically, may be richer in nitrogen than that from cleveite which you examined. True cleveite, as shown by Lindström's original analysis and my No. XVII, differs in composition from bröggerite in some important respects, and is much more readily soluble in acids than bröggerite, just as the latter is vastly more soluble than the Connecticut varieties of uraninite. Hence I can not refrain from suggesting that it were well to examine as many of the varieties as possible in order to ascertain whether or not the gas differs in composition. If the easily soluble forms have been derived by oxidation from original forms like those of Connecticut, richer in uranium dioxide and gas, as well as much less soluble, it may very well be that the composition of the gas varies quantitatively if not qualitatively. That it is a mixture in one case you seem to have shown.

Above all, it seems to me that a density determination of the gas is needed. The high summations of some of my complete analyses as well as those in which the gas was not estimated (see especially Tables I and II), referred to by me on page 75 of my paper, are *certainly* not to be accounted for by ordinary errors of weighing or by impurities of reagents. I make this assertion with the utmost confidence. They *would* be explained if the gas had a density about half that of nitrogen. While this would appear to be impossible if argon is the chief constituent of the gas, may it not be that argon is really subordinate? Your observation, that the gas from cleveite does not exhibit certain lines which atmospheric argon shows, might be regarded then as confirmatory of this view instead of indicating, as you wrote to Professor Clarke, that atmospheric argon contains something else than argon; for with dilution it is to be expected that certain argon lines would disappear. May not helium, or helium and other elements with densities less than nitrogen and constituting an infra-lithium group, make up the bulk of the gas? It seems not at all an unreasonable supposition that we may have to deal here not with a single element, but with two or more related members of a family or group. If such a specifically lighter gas or gases are present, the percentages given by me must all be reduced to correspond, whereas if argon is the chief constituent they must be largely increased and the summations thus made to depart still more from 100.

I send with this, under separate cover, a copy of my paper to which I have frequently referred, for your greater convenience in case you should have had access to it only in the *Chemical News,* where it appeared in disconnected form, running through several numbers.

My naturally great interest in this matter must be my excuse for writing at such length. I shall await the results of your further researches with the greatest impatience. Should you deem it desirable to make public any part of this letter in connection with your own work, you are fully at liberty to do so. I remain,

Very cordially yours,

W. F. HILLEBRAND.

This letter of Hillebrand's was followed by further correspondence, and the two following letters by Ramsay explain themselves:

12 ARUNDEL GARDENS, W., *April* 21, 1895.

MY DEAR SIR:

I have to thank you most cordially for your very kind and courteous letter of the 4" April. I never like to have to impeach the work of another; and I am sorry that our results do not agree. But agree they do not. The gas evolved from the sample of cleveite of Norwegian origin which I obtained from a London mineralogist consists of a mixture of hydrogen, argon, and helium; there is no nitrogen. This cleveite is, however, very easily decomposed by weak acid; in half an hour the whole mineral is disintegrated and mostly in solution. I find your sample (for which my warm thanks) much more difficult to decompose. In fact, a quantitative estimation is still in progress. I can tell you nothing as yet about *its* gas.

The density of the original gas from my sample is 1.55 (H-1); and the percentage of hydrogen is

$$\frac{3.7 \times 100}{4.7}$$

roughly. Tomorrow I shall carry out an accurate determination of the amount of H_2. This gives the density of the mixture of He and A as 3.6. I can't guess at the amount of A present, but its lines are distinct enough, in spite of what Cleve says in the Comptes rendus. I am inclined to suspect that the substance evolved is a hydroxide of helium, and possibly a hydroxide of argon; for after metallic palladium has done its best in removing hydrogen, the gas still shows a curious spectrum in which He is not conspicuous, and it is only after explosion and sparking with O that the He and A lines become very marked. There is another odd thing. The gas gives a band spectrum when the tube is filled at a fair pressure (I should judge 10 mm.), and the He and A lines come out strong only when the pressure is considerably reduced.

I hope tomorrow to remove He from a fair quantity of the gas (about 2½ liters) and then to weigh the residue directly instead of calculating its density indirectly. I shall also find the ratio of its sp. hts. tomorrow. I am writing all this in a provisional way; please don't take all that I say as absolutely established—confirmatory experiments must still be made. But, as a first approximation to truth, possibly to be modified later, it may stand as I have written it. It may interest you to know that English pitchblende, of which a good deal is now found in Cornwall, gives very little gas. I haven't examined it yet.

I am anxious to acknowledge your letter and kind present, otherwise I should have delayed a day or two and been able to tell you more about it. I had to give two lectures in Paris at the end of March, immediately after finding He, and my wife wanted me in Scotland for 10 days; so I have just returned to London. This accounts for the apparent small progress made. I have to give a paper on Thursday next, and will quote from your letter, as you kindly allow me to do. This is the first chance of English publication, for all our societies have been having Easter holidays.

Believe me, yours very truly,

W. RAMSAY.

UNIVERSITY COLLEGE, LONDON,
GOWER ST., W. C., *May* 30, 1895.

DEAR DR. HILLEBRAND:

I have much pleasure in sending you a tube of helium from your own uraninite, and am also glad to tell you that your gas contains a considerable proportion of nitrogen—at least 10 p. c. None of this is visible in the tube I send you, however, for it has all been removed by sparking with oxygen in presence of caustic soda. There was no leakage, so I am very sure that you were right in your original statement. Not all minerals give off nitrogen; so you were exceptionally unfortunate in having lighted on one which does.

Considerable progress has been made with the whole investigation since I wrote you last. The density of the gas from my sample of cleveite was 3.9; but I am beginning to doubt whether it was entirely free from nitrogen, and I am repeating a determination, starting from a fresh sample. My doubts are increased by the different results I get with the gas from bröggerite, of which Professor Brögger of Christiania was kind enough to send me a large stock (120 grams). Its density is 2.2, and I am not yet quite certain whether it is pure as it can be made. However, this last density has been checked several times, and though I shall try again, after a long sparking—the coil is rattling beside me while I write—I have no expectation to change the result. Two at least of the easily visible helium lines in the red are identical with good argon lines, and one in the orange. And this leads to the possibility that what we have called argon may contain one of the constituents of helium. I am at work on this point.

We have now got helium from a great many minerals; almost all of those, but not all, contain uranium. But none of these forms a *source*, except your specimen, the original Norwegian cleveite which I bought here and bröggerite. The quantities are insignificant. I may mention thorite, orangite, samarskite, monazite, as among the richest; but the yield is ridiculously small in all cases.

So far as we have gone, we have been able to make no spectroscopic distinction between helium from all these sources. All lines shine out in all of them and with about equal brilliancy.

An interesting fact is that gas from meteoric iron, after removal by hydrogen and hydrocarbons, consists mainly of argon and the least trace of helium. The argon spectrum is good, but the helium a mere shadow, but an undoubted one. Let me caution you to be economical with the vacuum tube. The helium spectrum fades on running the tube for long. I suppose the platinum sparked from the electrodes carries it on to the glass. If the tube is run for 5 or 6 hours continuously you will see no more helium. So in exhibiting it to your friends, which you will doubtless do, don't give them more than a minute apiece, or make them learn to look through a spectroscope at something else first, before you turn on the helium. Keep the current as weak as you can to get good definition, and it will last all the longer.

Please give my kind regards to Professor Clarke and assure him that it is only want of time which has delayed my answering his very kind letter. I am in the midst of a commotion and can hardly snatch a moment to write letters.

Yours very truly,

W. Ramsay.

Hillebrand's accuracy in his identification of nitrogen in the gas from uraninite is thus fully recognized. Hillebrand furnished the clue which led to Ramsay's great discovery. If argon had been known when Hillebrand made his analysis, he might have carried his investigation further and found helium himself. But—

> "Of all sad words of tongue or pen,
> The saddest are these—it might have been."

On July 1, 1909, Hillebrand entered upon his duties as Chief Chemist of the Bureau of Standards. His work in this new position was largely administrative, although he still found some time for original research, in which there was a distinct overlapping from his work in the Survey. He was much interested in the study of minerals containing vanadium, as may be seen from a glance at his bibliography. In 1900 he published a paper upon carnotite and its associated minerals, and in 1913 he described other vanadates from Peru, Colorado, and Utah. The last paper that came from his pen related to the same group of minerals and was published in 1924, not long before his death.

We now come to a line of work which began in the Geological Survey and was afterwards splendidly developed in the Bureau of Standards. Early in 1892 Hillebrand received from the Colorado Scientific Society five samples of an ore containing zinc, which had been a source of trouble to the analysts and assayers of Colorado. They worked by various short-cut commercial methods for the determination of zinc, and their results, as obtained by different methods, did not agree. The zinc in the samples sent to Hillebrand was determined by Mr. L. G. Eakins, under Hillebrand's direction, and by the use of the slower and more accurate methods employed in the Survey. In this way it was found which of the commercial methods was the most trustworthy, and something was learned as to their sources of error—a great help to the metallurgists of Colorado. In 1905, under the auspices of a special committee of the American Chemical Society, of which Hillebrand was chairman, the subject of the earlier investigation was taken up on a much larger scale. A uniform sample of a zinc ore was prepared and analyzed. A large number of analysts repeated the work, using different and specified methods, and the most accurate procedure was definitely determined.

In 1906 another step was taken in the standardization of analyses. A large sample was prepared of an argillaceous limestone used in the manufacture of Portland cement. This was analyzed by Hillebrand in the Geological Survey, and by Dr. C. E. Waters in the U. S. Bureau of Standards. These concordant analyses were repeated by many other analysts, but with widely variant results; these results were critically compared, their sources of error pointed out, and the cement industry was given greater chemical exactness. Still later three samples of iron were taken, analyzed at the Bureau of Standards, and then distributed to iron masters for comparison with the analyses made by their chemists.

Under Hillebrand's administration as Chief Chemist of the Bureau of Standards, the standardization of analyses was carried much further; and now the Bureau distributes annually about 5000 samples, representing 70 different substances. Of Hillebrand's personal share in this phase of the Bureau's activities his colleague, Dr. Waters, speaks as follows:[4]

> "He took the greatest interest in the standard samples and he did everything in his power to increase their number and usefulness. He

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[4] See Waters' admirable notice of Hillebrand in *Science* for March 6, 1925.

always jealously guarded the integrity of these samples and would not countenance including among their number any materials the composition of which was not known with an accuracy great enough to satisfy him."

Of Hillebrand as Chief Chemist of the Bureau I can do no better than to quote Dr. Waters, who says:

"He was a kindly chief, ready to discuss the problems and worries of his subordinates, not given to making the facile excuse that he was too busy to talk. He gave to every man his due credit; he sought and obtained promotions in rank and salary for those who, in his opinion, were deserving. Always modest about his own attainments, he gave his associates full credit for whatever the chemistry division of the Bureau accomplished. He took his administrative duties seriously and suffered undue worry lest he should be found wanting. A man in his position must many times make decisions relating to the use of Government funds and be tempted to divert them from their specifically authorized use to some other he may think more worthy. Dr. Hillebrand 'leaned backward' in his uprightness and would not countenance any violation of the letter or the spirit of the law. His honesty in these matters was but a further expression of the integrity of purpose that was the great guiding principle of his life, the principle that made him perform a routine analysis with the greatest care, that made him give to any task the best that was in him."

In 1906 Hillebrand was President of the American Chemical Society. It was a difficult year in the history of the Society, for there were serious dissensions with which the President had to deal. Some of the industrial chemists felt that their interests were slighted; that the Journal of the Society published too little that concerned them, and there was some fear that they might break away and form an independent organization. This danger was averted by the leaders of the Society by establishing a second journal, the *Journal of Industrial and Engineering Chemistry*. A forward step was taken with Hillebrand as the leader.

In addition to the American Chemical Society, Hillebrand was a member of the National Academy of Sciences, the American Philosophical Society, the American Society for Testing Materials, the Washington Academy of Sciences, the Geological Society of Washington, and the Göttingen Gesellschaft, and fellow of the American Association for the Advancement of Science. In 1916 he was awarded the Chandler Medal by

Columbia University. Of this Dr. Waters says: "His address on that occasion, 'Our analytical chemistry and its future,' is well worth reading by any chemist who may be disposed to regard analytical work as uninteresting and not worthy of a man's best effort." That is true of every honestly conducted scientific research, no matter how humble it may be.

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