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STUDIES IN THE HISTORY AND DEVELOPMENT OF THERMOGRAVIMETRY
I. Early development

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The internationally accepted definition of thermogravimetry is 'a technique whereby the weight of a substance, in an environment heated or cooled at a controlled rate, is recorded as a function of time or temperature ' [1]. Thus, it follows that the basic requirements of the technique are a source of heat, the measurement of temperature and a means of weighing.

A source of heat

The method of constructing and operating furnaces on a laboratory scale has been practised since the studies of alchemy [2]. In the context of thermogravimetry, however, the great need was for the development of a method of heating furnaces which could not only be accurately controlled to specific temperatures but could also provide a temperature-time programme to within guaranteed requirements. This was made possible, in the main, by the introduction, early in the 20th century, of electrically operated furnaces. The manner in which they became universally accepted is probably typified by reference to the experiences of the National Physical Laboratory (NPL), Teddington, Middlesex, UK. Thus, in the 1902 Annual Report of the NPL, mention is made of several electric furnaces in the Physics Division capable of operating up to 1200°. It is also reported that Sir Andrew Noble donated £1.000 for the installation of electric furnaces (110V DC) in the Physics Division. One of the few references to temperature control is contained in

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Akadémiai Kiadó, Budapest

the NPL Annual Report of 1904. This was a mercury expansion regulator and relay which maintained the temperature of a large annealing oven at 100°. Mention is also made, in the same Report, of the construction of tubular electric furnaces capable of continuous operation at temperatures in excess of 2000°. By 1909, the NPL had decided to replace all gas fired furnaces, with the exception of those used in the foundry, by electric furnaces on the grounds of cleanliness, economy and accuracy of control and in the Annual Report for the same year (1909) reference is made to numerous enquiries concerning the construction of furnaces for a variety of purposes. Thus, by the early 1900's electric furnaces were rapidly becoming established as the most convenient controlled heat source in laboratories [3].

The measurement of temperature

The fact that until the 18th century there was little advance in sophisticated sources of heat was largely due to the difficulty in obtaining accurate pyrometric measurements at high temperatures. Although the first air thermometer was constructed by Santorio Santorre (1561–1636) in Italy in the early 17th century [4] the development of most methods of temperature measurement currently in use had to await the 19th century. The first device to achieve a high degree of accuracy was the air thermometer [5] which was intensively investigated by Regnault [6] with particular reference to the boiling point of sulphur. Numerous investigations were carried to higher temperatures: for example in 1892 Holborn and Wien [7] extended the air thermometer scale to 1450° and in 1907 comparisons were made to 1680° [8].

Contemporaneously, the thermocouple was being developed. In 1821, the thermoelectric effect was first observed by Seebeck [9] and was applied to the measurement of high temperatures by Becquerel [10] using platinum and palladium and by Pouillet [11] who employed iron and platinum. The literature on thermocouples during the latter half of the 19th century is extensive and often confusing and it is clear that there were great difficulties in successfully applying the thermoelectric effect to temperature measurement, not the least of these being, particularly in the early years, the lack of mirror galvanometers for measuring emf.

Regnault, who was one of the outstanding workers in the field in the mid-19th century, believed that the thermocouple could not be made into an accurate temperature measuring device. Nevertheless, Becquerel revived the technique [12] and Avenarius [13] and Tait [14] were successful in relating the thermoelectric properties of various metals. Le Chatelier finally developed the thermocouple into an accurate temperature-measuring device in 1886 [15]. In spite of this, there were still accounts appearing in the literature of difficulties. For example, even as late as 1898, Stansfiled found that a batch of rhodium contained 10% iridium [16]. It is surprising to find mention of such impurities, at so late a date, particularly the gross contamination of rhodium, one of the easier of the platinum metals to purify.

Of the other temperature-measuring systems developed during the 19th century only the resistance thermometer [17] and optical pyrometer [18] have found any applications in thermogravimetry.

A means of weighing

The balance is so old that peoples of ancient civilisations attributed its origin to the Gods. The ancient Egyptians had mastered the art of weighing as early as circa 2800 BC [19] but for a very long time the balance was used merely for weighing substances such as in the making up of recipes. This period ended around the middle of the 17th century when chemists began to use the balance to provide quantitative results. For example, Lemery [20] established that 12 1/2 oz of mercury was obtained from 1 lb of cinnabar and Robert Boyle demonstrated (erroneously) that if tin is burnt in a closed vessel, then the weight of the system increases [21]. The use of the balance in a quantitative fashion gradually progressed until, by the end of the 18th century, publications by workers such as Black [22] and Lavoisier [23] confirmed the quantitative applications of the balance.

This era also saw the true beginnings of thermogravimetry since it was becoming apparent that weight changes which occurred on heating materials to specific temperatures could yield much useful information. Such was the state of progress of equipment during this period that experiments of this nature could only be carried out in a rather crude fashion. Nevertheless, they are of sufficient fundamental importance to merit a brief description of two examples: First, in 1780, Bryan Higgins, (*Appendix 1a*)* in his notable work 'Experiments and observations...on calcareous cements' [24] studied the effect of heating chalk and limestone at different tamperatures using

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* In order to avoid disrupting the narrative, biographical notes of various workers who made key contributions to the development of thermogravimetry are given in Appendices at the end of this article.

the melting points of various materials to indicate the temperatures achieved. Thus, he describes heating chalk 'to a temperature barely sufficient to melt copper' and further, defines white heat as 'the temperature at which cast iron melts completely'. It is also interesting to note that Higgins heated his samples in static and flowing air and commented on the effect this had on the nature of the end-product.

The second example is given by Josiah Wedgwood (*Appendix 1b*) who, in his work which led ultimately to the production of the Wedgwood Pyrometer, needed to know the effect of heat on various clay samples. He obtained the percentage weight losses on heating a day sample, in a progressive fashion, to the 'heat of boiling water', 'that of melted lead'. 'low red heat', and 'a strong one, such as copper melts'. It was Wedgwood's appreciation of the severe limitations of these descriptive temperatures ('red, bright red and white heat are indeterminate expressions') that inspired him to manufacture his pyrometers [25]. He also used the technique of evolved gas analysis, since he noticed that on heating clay samples to specific temperatures a certain quantity of gas was evolved. This he collected in 'bladders tied to the mouths of the vessels in which the pieces were heated' and from the few experiments he carried out on these evolved gases found them to consist of 'common air mixed with fixt air. They all precipitated lime-water and ... none of them were inflammable' [25].

In spite of the success of the Wedgwood Pyrometer, the technique of relating the physical property of a material to the boiling point or melting point of an element or compound was still in use a little over a century later. It was at this time (the late 19th century) that clay mineral chemistry became of some significance. Problems of clay mineral identification by means of microscopy were made additionally difficult due to their exceedingly small particle size but the importance of weight changes occurring at specific temperatures was beginning to be appreciated. Thus, in 1886, Ellis Lovejoy [26] published the loss in weight of clay minerals with reference to the melting point of different materials and found, for example, that kaolin decomposed near the melting point of antimony (634°).

Apparently, Lovejoy knew nothing of the work of Le Chatelier who (as mentioned above) also in 1886, described the successful application of the thermoelectric effect to the development of the thermocouple [15]. In fact, one of his first uses of the thermocouple was to study clay minerals, and in 1887 he describes heating kaolin and pyrophyllite in a furnace which took a mere 10 minutes to reach 1000°. Temperatures were measured by means of a platinum/10% rhodium-platinum thermocouple, which was standardised against the boiling or melting points of various materials viz water, sulphur, selenium and gold [27].

The year 1886 has considerable significance in the development of thermogravimetry since, in addition to the successful development of the thermocouple, it was also the year generally attributed to the introduction of the microbalance. Engaged on investigating the weight and origins of water films forming on glass and other materials, Warburg (*Appendix 1c*) and Ihmori, (*Appendix 1d*) working at Freiburg University, realised the need for weighing small accounts of materials to a high degree of accuracy. Accordingly, they constructed a microbalance, shown in Fig. 1 [28].

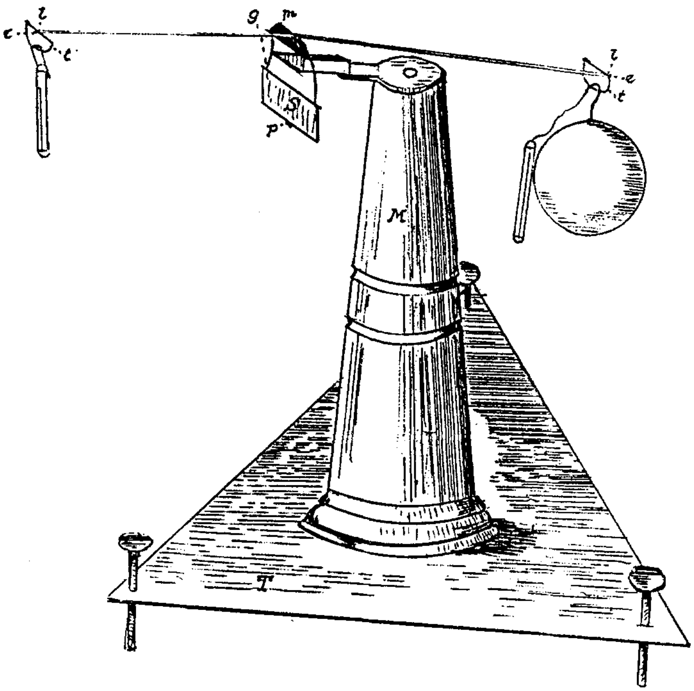

Fig. 1 The Warburg & Ihmori Microbalance

The balance beam is a glass tube, 8 cm long and a little over 1 mm thick and sealed at both ends. The knife edges are small pieces of a hollow ground razor blade cemented onto the beam with sealing wax. The ends of the centre knife edge (*m*) rest on two points on a brass plate which is attached to a brass pillar (*M*). The pillar is fixed to a base plate (*T*) supplied with three levelling screws. Onto both ends of the centre knife edge the curved glass tube (*g*) is attached, which carries the reading mirror (*S*) (a selected and silvered microscope cover glass). The weight of the beam and attachments is 0.21 g and the beam is so arranged that the knife edges at the centre and both ends lie as near level as possible. Onto the end knife edges are attached the 'roof-shaped' brass plates (*l*) which are provided with loops (*t*) made of thin platinum wire and onto which loads are hung. The weight of the plates plus the platinum loops is 0.024 g. The mirror scale is read by means of a telescope with a vertical scale, the distance between the mirror and the scale being 272 cm. The position of the centre of gravity of the balance beam was usually regulated by cementing small pieces of platinum at (*p*) so that the sensitivity of 30 scale divisions was equivalent to a load of 0.6 g on each side. A significant increase in sensitivity could not be achieved without a loss in reliability. The largest deflection observed experimentally did not exceed 50 scale divisions and at this limit, the deflection in scale divisions was proportional to the excess weight added.

The successful introduction of the microbalance served as a stimulus for following the weight changes which occur on heating materials. Notable amongst those who pursued this line of work was Walther Nernst (*Appendix 1e*) who, in 1903, described the construction of a microbalance, based on the torsion principle, for work on the determination of molecular weights at very high temperatures [29]. This paper gives only the briefest details concerning the balance, but in a further publication, with Riesenfeld, (*Appendix 1f*) in the same year [30], the equipment is fully described and illustrated (Fig. 2).

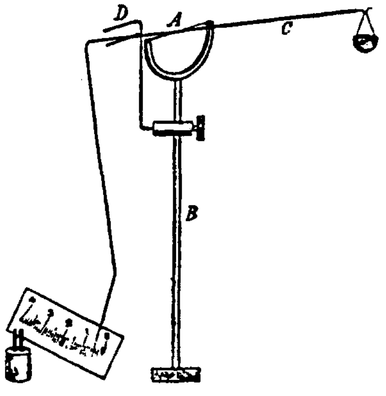

Fig. 2 The Nernst Microbalance

To determine the extent to which the beam deflection of the balance is proportional to load, Nernst and Riesenfeld also describe the technique of adding small pieces of platinum wire of known weight to the balance pan and observing the scale deflections. They found that variations in scale deflections between each piece of platinum wire to be no greater than 0.2% and considered this acceptable for their use. They also comment on the extreme care needed when loading the balance to prevent damage to the quartz fibre.

As an example an the suitability of accuracy of their balance for microanalysis, Nernst and Riesenfeld give details of determining the carbon dioxide content of very pure Iceland spar. This experiment has been quoted by Duval [31] and others as the first thermogravimetric determination. Careful translation of the experimental details shows this not to be true since Nernst and Riesenfeld write as follows: ... 'a crystal, dried in a desiccator, was weighed into the weighing capsule which was then heated by means of a strong Bunsen flame to bright white heat. The capsule was at first covered with a platinum lid to prevent loss by decrepitation. Later, the lid was removed in order to drive off the remaining carbon dioxide'. Their next sentence shows quite definitely that weighing was discontinuous since they continue: –'The capsule was then, again, brought to the balance (Sodann wurde das Schälchen wiederum auf die Waage gebracht)' [32]. Thus, it is clear that Nernst and Riesenfeld did not carry out thermogravimetric experiments, as accepted by present day definition [1].

These workers expressed no views on the technique of weighing the sample continuously whilst it was being heated and it is difficult to say whether they even appreciated the advantages and significance of this technique. Nevertheless, they approached this concept later in the same paper when using the microbalance for determining the atomic weights of the rare earths. They commence by converting the rare earth oxide to the sulphate by heating with sulphuric acid and then 'the sulphate thus obtained was put onto the capsule which was dried at low red heat for about 10 minutes in an air bath. The air bath consisted of a brass cylinder of 3 cm diameter and height. In this cylinder were two holes, one for a thermometer and into the second the capsule was inserted ... The capsule can be lowered into the hole with the aid of a platinum wire ...'(No mention is made of the significance of the thermometer or of any temperature readings taken with it). 'Since the RE sulphates are strongly hygroscopic, the capsule was removed from the cylinder, at accurately measured half-minute intervals, onto the balance and the weight determined' [33].

It is significant that no attempt was made to weigh the sample whilst it was being heated in the air bath, even though they describe lowering the capsule into the air bath with the aid of a length of platinum wire and the

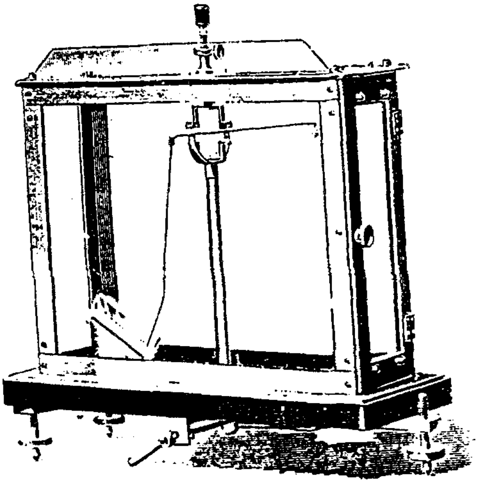

Fig. 3 The Nernst microbalance ex Spindler & Hoyer catalogue List 36

fact that it would have been a simple matter to attach the opposite end of this wire to the balance. Perhaps the idea never occurred to them or perhaps they dismissed any such attempt as futile, bearing in mind the high sensitivity of the balance, the care needed when loading the balance to prevent damage to the quartz fibre (a fact mentioned earlier in this account) and the disturbing influence that factors such as convection would have had on such an experiment. That the Nernst microbalance was a success is evidenced by the fact that the Germany instrument manufacturer Spindler and Hoyer, of Göttingen produced a commercial version which, in 1905, sold for 80 Marks. A later version is shown in Fig. 3 (taken from Spindler and Hoyer, List 36. Probable date of publication 1912, price not given*). The apparatus was also featured at the World Exhibition in St. Louis, 1904. In 'German International Exposition, St. Louis, 1904, Official Catalogue, Exhibition of German Empire', (page 366), the following entry appears: 'Prof. Walter Nernst, Göttingen, Burgerstr. 50. Furnace with Iridium bulb for determining the vapour density of metals, sensitive balance of quartz filaments for the same purpose'. This appeared under the heading 'Apparatuses for determining

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* Catalogue kindly made available by Dr F Greenaway, Science Museum, London

molecular weights'. No further information was given nor was there a picture. The exhibit was in the Palace of Industry in the Chemical Exhibition.*

A student of Nernst at Göttingen, Otto Brill (*Appendix 1g*) used the same technique, i.e. heating samples in a furnace for short periods and then removing them for weighing, to investigate the dissociation of magnesium carbonate and the alkali earth carbonates [34]. Probably due to Nernst's influence, Brill also failed to carry out a true thermogravimetric experiment. His technique was to hang a small platinum crucible from a length of platinum wire, the crucible was then raised and lowered into a wire-wound platinum furnace by attaching the wire to a suitable stand. However, unlike Nernst and Riesenfeld, Brill measured the temperature by means of a thermocouple positioned near to the crucible and the samples were heated in a dynamic carbon dioxide atmosphere. From the results obtained for the decomposition of calcium carbonate he also found that strontium and barium carbonates decomposed in a similar fashion to calcium carbonate forming the corresponding oxides via a single stage decomposition process. On the other hand, magnesium carbonate formed a series of stable intermediate basic carbonates before finally decomposing to form magnesium oxide. In addition to determining dissociation temperatures, Brill applied his technique to the analytical determination of the alkali earth carbonates in admixture. A 2–3 mg sample was heated in the furnace in a carbon dioxide atmosphere for 10 minutes at 920°. The crucible and contents were removed from the furnace, cooled and weighed, reheated in the furnace for 10 minutes at 1170° and again removed from the furnace, cooled and weighed. The calcium, strontium and barium contents of the mixture were calculated by assigning the weight loss at 920° to the evolution of carbon dioxide from calcium carbonate and the weight loss at 1170° to the evolution of carbon dioxide from strontium carbonate. Barium carbonate is not decomposed under these conditions, but by igniting the residue with a weighed amount of borax the resultant weight loss is due to the carbon dioxide evolved from the barium carbonate. This procedure adopted by Brill for analysing mixtures was the forerunner of the technique of Automatic Thermogravimetry proposed by Duval in 1947 [35] and later applied to mixtures of alkali earth carbonates by Erdey and co-workers [36]. After consid-

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* The authors are indebted to Mrs Marie H. Roberts, Chief Librarian, Reference Department, St Louis Public Library, 1301 Olive Street, St Louis, Missouri 63103, for the information concerning the German International Exposition, St Louis, 1904.

erable experimental work, Brill felt sufficiently confident of his results to notify F. A. Kahlbaum that their product labelled 'Purest Calcium Carbonate, Kahlbaum' in fact contained 0.6% $MgCO_3$.

In some further work on the atomic weight of thorium [37], Brill, again, used a technique close to thermogravimetry since he comments that ashing over a Bunsen flame causes errors which can be easily overcome if a small electric furnace is used and the platinum crucible is suspended in the furnace from a suitable support. Had that he chosen to suspend the crucible from the balance into the furnace rather than from a support!

Appendices

Appendix 1a

Bryan Higgins was born 1736-7 of an Irish family. He presented a thesis for the MD degree at the University of Leyden in 1756, wrote several books and patented four inventions. A close friend of Josiah Wegdwood, he obtained from him various items of earthenware apparatus for use in his laboratory. He arranged several courses of chemistry lectures and demonstrations which proved quite popular. His business ventures proved very profitable; in 1793 he was involved with Mr. J. J. Schweppe and his daughter Collette in the manufacture of artificial mineral water and a few years later visited Jamaica to improve the manufacture of sugar and the process of rum making. Perhaps his greatest claim to scientific fame was in accusing (unsuccessfully) Priestly of plagiarism, claiming that the latter's outstanding work on gases owed much to his (Higgins') lectures and demonstrations on the subject. He died in Staffordshire, U.K. in 1820.

Appendix 1b

Josiah Wedgwood, the youngest of 13 children, was born at Burslem, Staffordshire, U.K., on 12 July, 1730. His father died when he was nine and he therefore left school and went to work in the pottery of his eldest brother, Thomas. At the age of 11 he contracted small-pox which left him weak in health. When he was 15 years old he began a five-year apprenticeship to Thomas but ill-health forced him to depart the thrower's bench. He subsequently gained a broader experience of the potter's art and loved experimenting. This experimentation was not appreciated by Thomas, who refused to take him into partnership. He thus worked for a variety of potters and became a Master Potter in 1759. With little capital he started his own business in Burslem. He improved both the works and the working conditions and prospered rapidly. In 1762 he was appointed Queen's Potter. He moved his works to Ertruria (Staffordshire) in 1773 building houses there for his workmen. He was elected a Fellow of the Royal Society on 16 January, 1783.

He married his third cousin on 25 January 1764 and they had seven children. In his latter years he suffered persistent ill-health and finally died, a wealthy man, on 3 January, 1795 in Ertruria.

Appendix 1c

Emil Gabriel Warburg was born on 9 March, 1864 at Altona, Nr Hamburg, Germany. Initially educated at Heidelberg University, he was sufficiently inspired by Kirchoff's lectures on experimental physics to change, from studying chemistry to physics. He subsequently transferred to Berlin University, gained his doctorate there, in 1867, and also qualified as a lecturer. In 1872 he was invited to the post of professor extraordinary at the newly-founded Kaiser Wilhelm University at Strasbourg. In 1876 he became professor of physics at Freiburg University and in 1845 succeeded August Kundt as professor of experimental physics at Berlin University. In 1905 he succeeded Kohlrausch as President of the Physicalisch–Technische Reichsanstalt, a post he held until his retirement in 1922.

Although he never achieved the brilliant success of his contemporary, Wilhelm Roentgen, it has been estimated that about one-fifth of all the professors of experimental physics in Germany studied under Warburg.

He died on 28 July, 1931 at Gut Grunau, Nr Bayreuth.

Appendix 1d

Teizo Ihmori was born on 24 August, 1851 in the province of Hizen Japan (now Saga). When he was 20 years old he went to Tokyo where he first learned German and then taught it at the Tokyo Foreign Language School. In 1877 he became an assistant lecturer in physics in the Medical Faculty of Tokyo University and during this time translated a Germany text book on physics into Japanese which was used as the standard reference book by his students. In 1881 he became assistant professor in the Medical Faculty and also taught elsewhere at the same time. He travelled to German in 1884 and studied at Freiburg University, under Prof. Warburg, from whence he obtained his doctorate in 1886. During this period, it was normal for Japanese scientists to be given 18 months study leave abroad. However, Ihmori managed to persuade the Ministry of Education to extend his leave and he finally returned to Japan in 1887 and immediately took up the post of teacher and Vice-principal of the Fourth High School. In the following year, together with some friends, he founded a private School of Pharmacy, rising to the post of professor in 1893. At this time he also taught at a girls' High School. By all accounts he was a good teacher and popular with his students. However, early in the 20th century he suffered a cerebral haemorrage, from which he never fully recovered. He died in March, 1916 aged 65.

(One of us (CJK) is greatly indebted to Dr. Shigeo Iwata for providing these biographical notes. A comprehensive account of the life and work of Teizo Ihmori is given by Iwata in Bull. Soc. Historical Metrol. Japan, 2 (1980) 25.)

Appendix 1e

Walter Nernst was born in Briesen, West Prussia (now Wabrzezno, Poland) on 25 June, 1864. His first ambition was to be a poet and he developed a lifelong infatuation for literature and the theatre, particularly the works of William Shakespeare. Nevertheless, he studied mainly physics and mathematics at several universities before graduating, first in his class, in 1883 from Wurzburg. He gained his doctorate in 1887. A superb craftsman with keenly developed technical skills, he carried out research at several places before settling, for 15 years (1890-1905) at Göttingen. He next succeeded Hans Landoldt at Berlin's Second Chemical Institute, which he soon converted into a physical chemistry institute. In 1922 he succeeded Warburg (*Appendix 1c*) as President of the Physikalisch-Technische Reichsanstalt and from 1924-1933 was Director of the Institute of Experimental Physics at Berlin University. His studies were mainly in the field of thermochemistry, for which he was awarded the Nobel Prize in chemistry in 1920.

In 1904 he invented the Nernst Lamp, which replaced the less efficient carbon-filament by R. E. oxides. He sold the patent right and made a small fortune, although the device was short-lived, due to the introduction of the tungsten filament. He was an enthusiastic carp farmer, arguing on the basis of the second law of thermodynamics that fish were a better investment from an energy standpoint than warm-blooded livestock. He was also a car fanatic, owned one of the first cars in Göttingen at the end of the 19th century, and published several papers on the efficiency of the internal combustion engine. He died of a heart attack in Bad Muskau, Oberlansitz, Germany, on 18 November 1941.

Appendix 1f

Ernst Hermann Riesenfeld was born on 25 October, 1877 at Brieg, Schlesien, Germany. Between 1897 and 1901 he studied at Heidelberg, Breslau and Göttingen and obtained his doctorate from Göttingen in 1901. In 1903 he was appointed assistant in the chemistry laboratories at Freiburg University and between 1913 and 1920 held the posts of Director of the Technological Institute, Freiburg and ordinary professor at Freiburg University. In 1920 he moved to Berlin, where he became professor at the University and subsequently Departmental Director at the Physical Chemistry Institute, a post he held until 1934. He later moved to Stockholm where he remained until his death on 19 May, 1957.

Appendix 1g

Otto Brill was born on 27 September, 1881 in Pardubitz, Böhmen. He studied at the Technische Hochschule, Vienna; Göttingen and Berlin Universities and obtained his doctorate (Dr. Ing) from Vienna in 1904. Early in 1904 Sir William Ramsay had taken delivery of a Nernst microbalance from Spindler and Hoyer, Göttingen. (In a letter to Spindler and Hoyer dated 28 March, 1904, Ramsay acknowledges receipt of the balance and comments that it is working well). Brill used this balance for several studies, including the atomic weight of radium, when he carried out research in Ramsay's laboratory at University College, London. He spent two short periods in this laboratory during the sessions 1906-07 and 1907-08, starting on 23 October, 1906 and finally leaving at the end of June, 1908. Each year he paid a fee of six guineas, plus a registration fee of one guinea but was not working for a London degree. (The authors are indebted to Dr. W. A. Smeaton, lately of University College, London, for information regarding Brill's activities there.)

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Zusammenfassung — Die international anerkannte Definition der Thermogravimetrie lautet: "Ein Verfahren, wobei das Gewicht einer Substanz in einer mit kontrollierter Geschwindigkeit aufgeheizten oder abgekühlten Umgebung als Funktion der Zeit oder der Temperatur aufgezeichnet wird" [1]. Daraus folgt, daß die grundlegenden Voraussetzungen dieses Verfahrens eine Wärmequelle sowie die Messung von Temperatur und eine Möglichkeit zur Gewichtsbestimmung sind.