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Special Review

STUDIES IN THE HISTORY AND DEVELOPMENT OF THERMOGRAVIMETRY
III. The influence of Kotaro Honda and the Japanese school

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The emergence of chemistry in Japan is briefly discussed, followed by a detailed description of the Honda thermobalance and the results obtained. The paper concludes with a short biography of Honda, together with a collection of photographs depicting Honda's private and professional life.

Keywords: thermogravimetry, Japanese school, life of Honda

Introduction

The Japanese school can rightly be credited with producing the first apparatus that constantly recorded mass changes which occurred whilst samples were subjected to a programmed temperature rise. Details were reported in 1915 [1] by Kotaro Honda (see Appendix), working at Tohoku Imperial University.

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Before examining this classic paper in detail, it is worthwhile considering briefly the emergence of chemistry in Japan, for it was not until as late as 1837 that chemistry enjoyed recognition as a science independent of medicine. In that year Yoan Udagawa (1798–1846), physician, botanist, chemist and Official Scholar of the Tokugawa Government, published the first Japanese chemistry book, based on Lavoisier's chemistry and entitled 'Semi Kaiso' (Foundations of Chemistry). This book was an adaptation of William Henry's 'An Epitome of Chemistry', the first edition of which was published, in London, in March 1801 [2]. Based on a lecture course given by Henry, this was a slim volume in which students were not only introduced to the works of Lavoisier, Fourcroy, Nicholson, Pearson and others, but were also given a number of simple practical experiments to try for themselves.

At the time of the publication of 'Semi Kaiso' Dutch was the only foreign language permitted by the Tokugawa Government, but the second edition of 'Epitome' (June, 1801) was translated into German by Johann Bartholomäus Trommsdorf (pub. Chemische Probirkabinet, Erfurt, 1801) which, in turn, was translated and enlarged by Adolphus Ypey into a Dutch Version entitled 'Chemie voor Beginnende Liefhebbers' (pub. Amsterdam, 1803). It was the latter translation which formed the basis of 'Semi Kaiso' [3], together with Udagawa's own experimental evidence and consultation of 24 other books, including a Dutch translation (1797) of Lavoisier's 'Traité élémentaire de Chimie' [4].

Towards the end of the 19th century there was a deliberate government policy of sending scientists to Europe to broaden their knowledge and gain experience. Partly as a result of this, in 1868, immediately after the Meiji Restoration, the government founded the Imperial Institute of Chemistry and subsequently nine Imperial Universities, commencing with Tokyo in 1877. The first chair of analytical chemistry was established in 1918 at the Imperial University of Tohoku, the first professor being Matsusuke Kobayashi.

Thus a span of 78 years covers the period from the birth of chemistry in Japan to the beginnings of the development of thermogravimetry.

The Honda thermobalance

To revert to Honda's paper. In the opening remarks Honda comments on the difficulties associated with the 'ordinary method' of following a chemical change taking place in a compound at high temperatures, i.e. heating the compound to various temperatures, cooling to room temperature and weighing. He continues...'it is highly desirable to measure, if possible, the change of weight at high temperatures without cooling to the room temperature. For this purpose, the author has constructed a thermobalance, which admits us to follow continuously the change in its weight at gradually varying temperatures'. [1, p.97]. There are three significant points in these opening remarks.

(1) Honda appears to be the first worker to commit to print the admission that it is difficult to follow reactions at high temperatures by the technique of heating to various temperatures, cooling and weighing. This difficulty was almost certainly appreciated by earlier workers but it was probably not admitted because they had no solution to the problem.

(2) The manner in which Honda uses the term 'thermobalance' ('the author has constructed a thermobalance' and the title of the paper, 'On a Thermobalance') implies that the word had been in existence for some time, yet this is the first occasion that such an apparatus is described and also the first occasion that the term appears in print. It has yet to be superseded and is now the internationally accepted term [5].

The reason why Honda coined the term 'thermobalance' has, so far, remained unanswered by Western thermal analysts, probably because it was felt that to attempt an explanation required a knowledge of the Japanese language, which they did not possess. In addition, neither has an explanation been offered by Japanese workers.

In an attempt to resolve this problem the matter was discussed (in 1976) by one of the present authors (CJK) with the following group of Japanese thermal analysts who also possessed additional qualifications:

– Prof. Dr. Hiroshi Watanabe, who knew Prof. Honda personally.

– Prof. Heikichi Saito, an intimate friend and scientific colleague of Honda.

– Prof. Yasutoshi Saito, who speaks good English and is the son of Heikichi Saito.

– Prof. Ryohei Otsuka, a close friend of Yasutoshi Saito and an ex-student of Heikichi Saito. He speaks good English and has a good understanding of the language.

At the start of the discussion it was mentioned that Honda invented many items of scientific apparatus and always took particular pleasure in inventing terms for them. His knowledge of English was also quite extensive. At this juncture, it may be interesting to speculate whether Honda discussed the matter or, indeed, gained any inspiration from Prof. Gustav Tammann at Göttingen University, under whom he studied during the period 1907–11, and who had recently coined the term 'Thermal Analysis' [6].

In considering the possibilities for a new term, it was agreed that Honda would realise the necessity of incorporating the word 'balance' and that, in addition, the other essential feature of the apparatus, namely a furnace i.e. heat, must also be included. Honda would then have been confronted with the problem of combining these two factors into a single term. 'Heat Balance', he probably thought, would not convey the correct meaning but, perhaps the alternative 'Thermal Balance' would be more appropriate. It is at this point that the train of thought must be related to the Japanese language. 'Balance', in Japanese is 'tenbin' and 'Thermal' is translated as 'netsuteki' which, when combined, give the term 'netsuteki tenbin'. However, 'netsuteki tenbin' does not roll easily off the Japanese tongue and would be entirely unacceptable. However, if instead of 'Thermal' the prefix 'Thermo-' were used, the translation of 'Thermo-' is 'netsu-' and the term then becomes 'netsutenbin' which is orally acceptable in the Japanese language.

(3) Clearly, in the absence of the originator of the term 'thermobalance' any explanation is purely conjectural, but the Japanese scientists present at this discussion considered the above to be a reasonable explanation.

– It is, at first sight, remarkable that such an apparatus should be devised from no apparent earlier foundation. There are, certainly, no explicit statements regarding development from earlier studies. However, a slight clue emerges when Honda compares the route of the thermal decomposition of chromic anhydride with the results obtained in an earlier paper by measuring the magnetic susceptibility of this compound at different temperatures [7]. Reference to this paper gave no indication as to a possible origin of the thermobalance but did indicate that Honda had previously been considerably involved in research into magnetic susceptibility measurements. Initially, he had constructed an apparatus for these studies incorporating a torsion balance [8] but finding this somewhat unsatisfactory, subsequently constructed a more robust apparatus [9]. This apparatus, which is described in detail, is undoubtedly that on which he based the

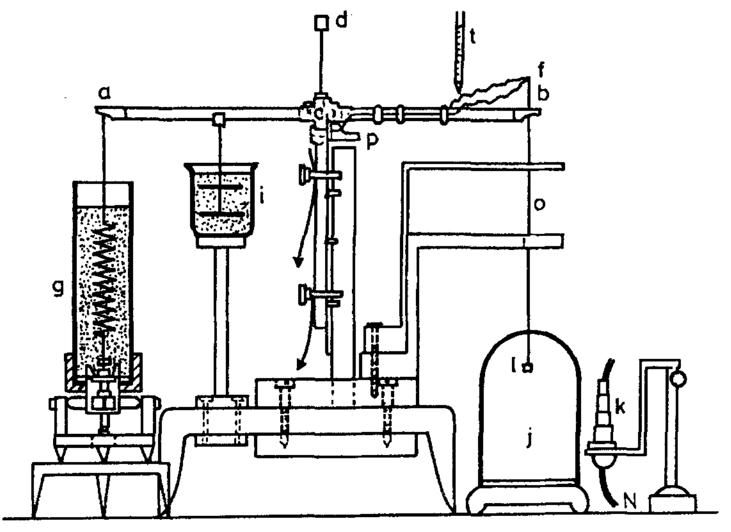

Fig. 1 Magnetic susceptibility apparatus according to Honda and Takagi, 1911 [9]

design of his thermobalance and diagrams, which are reproduced in Figs 1 and 2, show quite clearly the similarities with the thermobalance depicted in Figs 3 and 4.

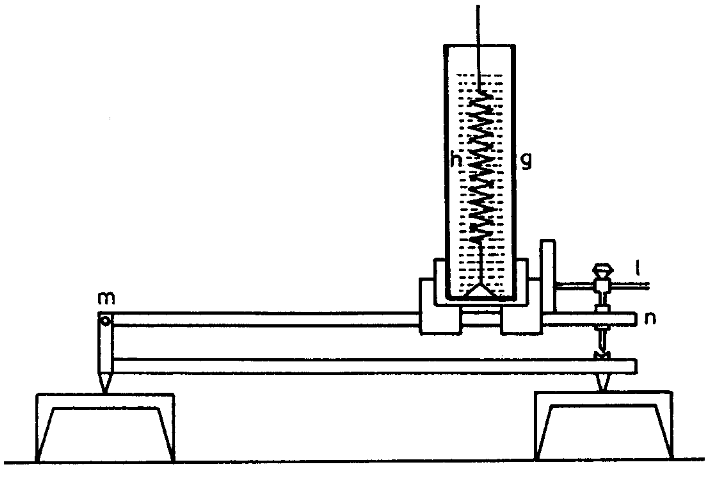

Fig. 2 Magnetic susceptibility apparatus according to Honda and Takagi, 1911 [9]

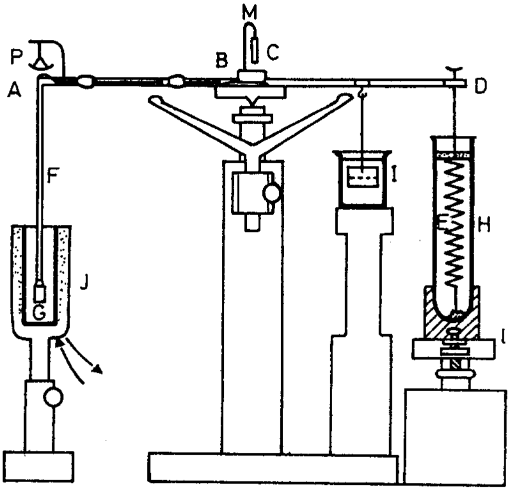

Fig. 3 The Honda Thermobalance

A paraphrase of Honda's description of the thermobalance shown in Fig. 3 and its operation follow (with the present authors' comments in parentheses): AB CD are the two arms of the balance beam, constructed of silica glass because of its low expansion coefficient. These two arms are fixed to a wooden block BC which rests on an agate plane with an agate knife-edge. This block also holds a vertical mirror M to reflect the image of a vertical scale into an observing telescope. A thin porcelain tube F is fixed vertically to the end of the beam at A and a small cylindrical sample container G made of platinum or magnesia hangs from the lower end of F by means of three platinum wires. From point D a very weak spiral spring E is stretched vertically and attached to the bottom of the Dewar flask H which is filled with oil. Honda claimed that this arrangement ensured that the spring remained at a constant temperature and also prevented beam oscillations during heating (whilst the damping effect of this arrangement cannot be denied, it is a little hard to believe that the changes in temperature to which the spring would have been subjected in a laboratory would have made any measurable difference to the mass record of this relatively unsophisticated piece of apparatus).

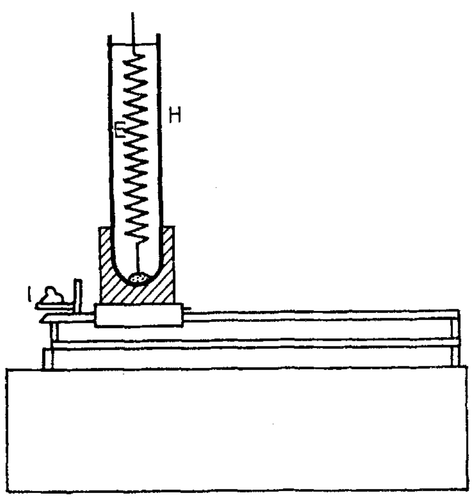

Fig. 4 Detail of the Honda Thermobalance

The Dewar flask can be raised or lowered by means of a lever system (Fig. 4), the degree of movement being read on the calibrated screw head 1. Since the damping effect of the spiral spring alone is not sufficient, another damper I is suspended from the arm CD. To measure the sample temperature, one junction of a platinum/rhodium-platinum thermocouple is suspended into the cylindrical vessel G through the porcelain tube.

The thermocouple wires then extend from G to B there to form the cold junction. Thin copper wires extend from BC to contacts and in the form of very weak spirals. These contacts are then connected to a galvanometer. The temperature of the cold junction is read from a mercury thermometer and the corresponding correction always applied. The thermocouple is calibrated against the melting point of pure metals.

Heating is accomplished by means of a platinum wire furnace J wound non-inductively on a thick porcelain tube (non-inductive furnace windings find favour in present-day equipment since they have a longer life than straight-wound furnaces and obviate any anomalous results when dealing with paramagnetic samples).

To improve the uniformity of temperature in the furnace, a thick nickel tube fits inside the furnace winding. (No mention is made concerning the length of the hot zone but the stand on which the furnace rests (Fig. 3) can be raised or lowered, presumably to allow correct positioning of the sample in the hot zone as well as to charge the crucible with the sample to be investigated.)

The sample, weighing approximately 0.6 g was placed in the crucible, the balance beam was then adjusted to the horizontal position by adjusting the height of the Dewar flask and the zero position of the balance was read by means of the scale and the telescope. The sensitivity of the balance was determined by placing a 10 mg weight in the small pan p at one end of the balance beam and observing the deflection. Observation showed that 1.7 mm deflection corresponded to a weight change of 1 mg.

The furnace temperature was raised by applying a gradually increasing current. (No indication is given in Honda's paper as to how this was achieved, but discussions by one of the present authors (CJK) with Profs Heikichi Saito [10] and Hirotaro Kambe [11] confirmed that this was achieved by means of a manually operated variable transformer.)

Since the velocity of a chemical change is usually very small, the heating must be carried out very slowly and when the change began to take place, the temperature was kept constant until the weight of the sample ceased to alter. This required usually from 1 to 4 hours and the time taken to reach 1000°C was 10–14 hours. (In discussing the manual temperature control, Kambe [11] made the point that workers using the thermobalance had to adjust their liquid intake to enable them to endure an uninterrupted 10–14 hour stint at the variable transformer.)

In spite of the damping system described earlier, slight beam oscillations were observed at temperatures in excess of 300°C. However, these did not cause serious errors in reading the weight scale, since the heating rate was slow.

Honda points out that if a greater weighing accuracy is required, the null-point method is used. This is carried out by raising or lowering the Dewar flask to maintain the balance at the null-point, the degree of movement of the Dewar flask, i.e. the mass change, being determined by means of the calibrated screw mechanism employed in varying the height of the Dewar flask.

The results obtained by Honda on several compounds are detailed in his paper, although there is no indication whether he used the deflection or null-point system of weighing. The results are of sufficient historical importance to be reproduced and commented upon here.

Manganous sulphate $MnSO_4{\cdot}4H_2O$

Figure 5 shows the thermogravimetric (TG) curve obtained by Honda. This indicates that the water of crystallization is given off in two distinct steps; between 70° and 110°C three molecules of water are evolved whilst the remaining molecule is released between 230° and 260°C. At 820°C the anhydrous manganous sulphate decomposes resulting in the formation of Mn_3O_4 at about 850°C. This agrees well with the results obtained by Dollimore [12].

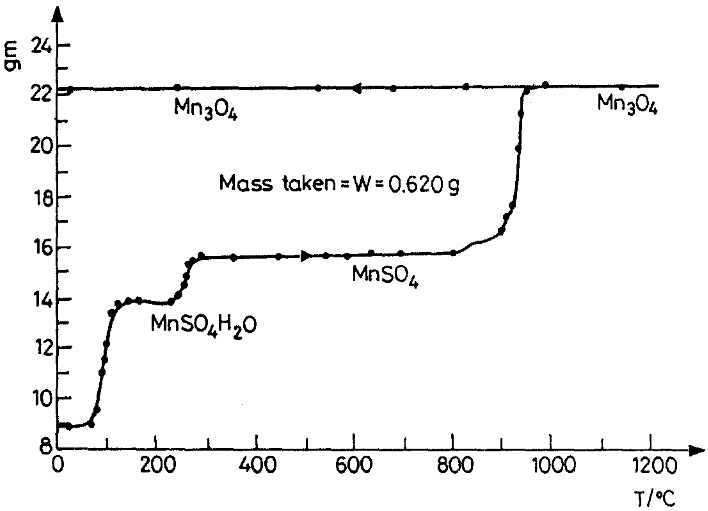

Fig. 5 Thermal decomposition of $MnSO_4{\cdot}4H_2O$ [1]

Calcium sulphate $CaSO_4{\cdot}2H_2O$

The TG curve for $CaSO_4{\cdot}2H_2O$ is reproduced in Fig. 6 from which it can be seen that dehydration commences at about 130°C and is complete by about 260°C. The initial sample mass used by Honda was 0.535 g and the total mass loss was 0.116 g (Theoretical 0.112 g). In addition Honda quotes the 'mean velocity of separation', i.e. the rate of mass loss, dm/dt as 0.0992 g per hour.

It is clear that, in this paper, Honda's intention was to describe his thermobalance in detail and to demonstrate its usefulness by several examples without attempting any detailed interpretation of the results. However, if such an interpretation is attempted, some interesting information can be obtained. Consider, for example, the results obtained on $CaSO_4{\cdot}2H_2O$: firstly, in describing the operation of his thermobalance, Honda does not indicate whether he obtained a linear heating rate. This can be deduced as follows: The rate of mass loss is given as 0.0992 g per hour and the total loss is 0.116g. Thus the total mass loss is achieved in 70 minutes.

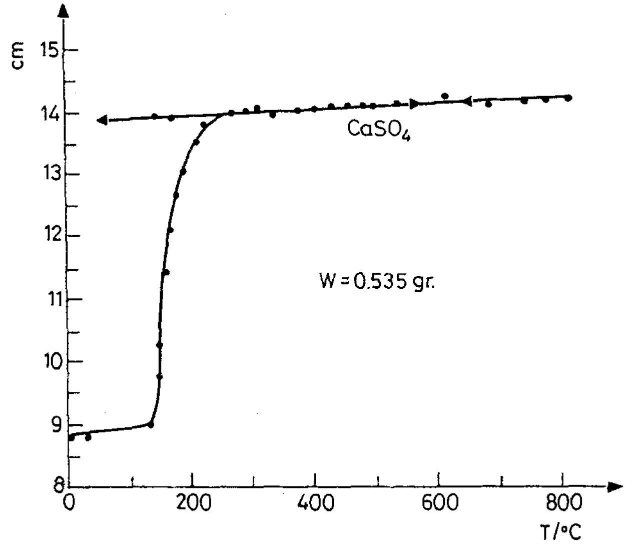

Fig. 6 Thermal decomposition of $CaSO_4 \cdot 2H_2O$ [1]

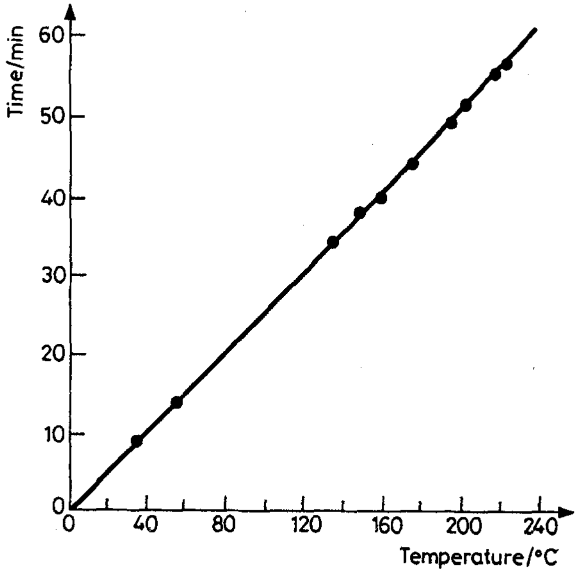

Fig. 7 Relationship between time and temperature in the thermal decomposition of $CaSO_4 \cdot 2H_2O$ (see Fig. 6)

Reference to Fig. 6 shows that the total mass loss occurred at 300°, i.e. 300°C was reached in 70 minutes. If, we assume, ambient temperature to be 20° C then a temperature rise of 280°C was achieved in 70 minutes i.e. Honda used a heating rate of 4 deg·min^{-1}, the temperatures relating to the points on Honda's TG curve can be estimated and correlated with time, with the following results:

Point number on Graph	T / °C	Estimated time / min
2	36	9
3	56	14
4	136	34
5	150	38
6	150	38
7	150	38
8	160	40
9	176	44
10	196	49
11	204	51
12	220	55
13	224	56
14	248	62
15	272	68

A plot of time *vs*. temperature is given in Fig. 7 from which it can readily be seen that Honda obtained a remarkably linear heating rate, at least up to 300°C, notwithstanding the fact that the temperature gradient was obtained by a manual increase of current to the furnace. Unfortunately, Honda does not give a 'mean velocity of separation' for the TG curves of materials decomposing at higher temperatures, i.e. calcium carbonate and chromium trioxide. Thus it is not possible to determine whether the linear heating rate was maintained at temperatures above 300°C.

The second additional piece of information that can be obtained from a close study of the TG curve of $CaSO_4 \cdot 2H_2O$ concerns the detailed mode of decomposition of this material. A cursory examination of Honda's curve, would suggest that with a sample mass of 0.535 g and a heating rate of 4 deg·min^{-1}, the two molecules of water are lost in one step, which would agree with more recent work where a single-stage dehydration was obtained with samples of 0.300 g and heating rates of between 0.5 and 6 deg·min^{-1} [13]. It is highly probable that Honda knew of the existence of $CaSO_4 \cdot 1/2H_2O$ but he makes no attempt to interpret his curve as a two-stage dehydration process, merely commenting that 'the water of crystallization begins to escape at about 130°C and the whole is given off at about

260°C [1, p. 101]. However, if from Honda's TG curve (Fig. 6), the first derivative (d*m*/d*t*) is plotted, the result obtained is shown in Fig. 8. This shows quite clearly the two peaks, which would correspond to the loss of 1 1/2 molecules and 1/2 molecule of water respectively.

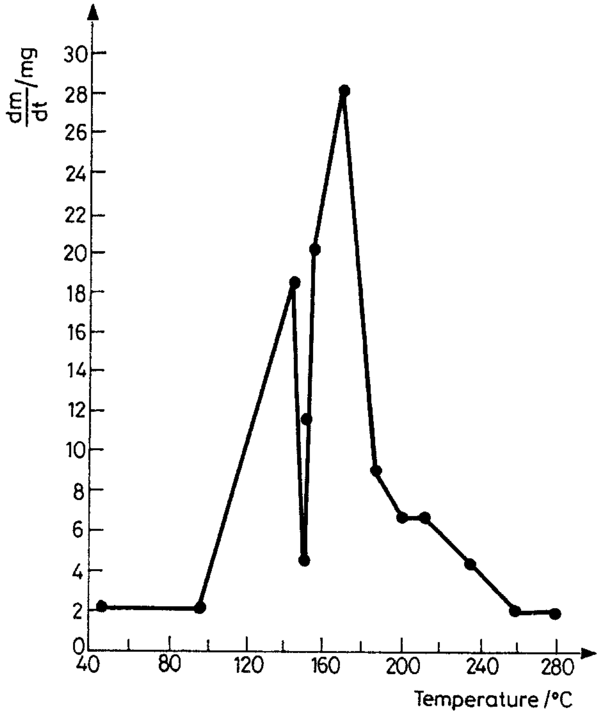

Fig. 8 Rate of mass loss (d*m*/d*t*) *vs.* temperature of $CaSO_4 \cdot 2H_2O$ (see Fig. 6)

Calcium carbonate $CaCO_3$

The thermal decomposition of calcium carbonate, as obtained by Honda, is reproduced in Fig. 9 and shows a single stage process in which decomposition commences at approximately 500°C and terminates at about 880°C. This result agrees fairly well with the later work of Newkirk and Simons who, using a sample mass of 0.344 g, a heating rate of 5 deg·min^{-1}, and a dynamic nitrogen atmosphere, found that the decomposition starts and finishes at 574° and 840°C respectively.

Chromic anhydride CrO_3

The rather complicated decomposition pattern of CrO_3 is shown in Fig. 10. Not surprisingly, the mass changes associated with the decompositions CrO_3 – Cr_6O_{15} and Cr_5O_9 – Cr_2O_3 do not agree particularly well with the theoretical.

Nevertheless, Honda claims that the results obtained confirm his earlier experiments on the same system using high temperature magnetic susceptibility [7].

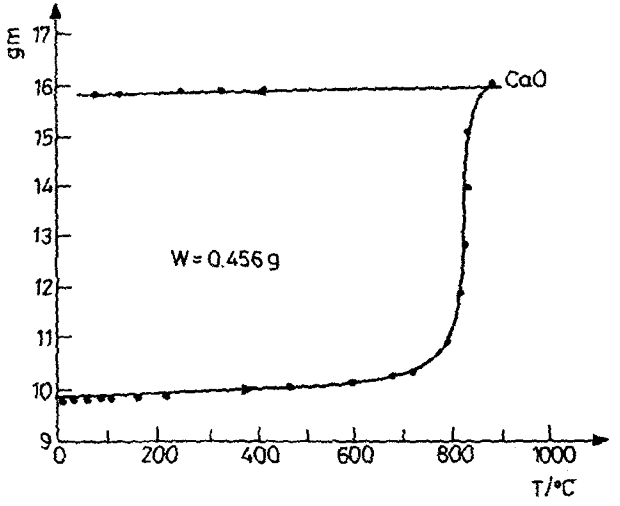

Fig. 9 Thermal decomposition of $CaCO_3$ [1]

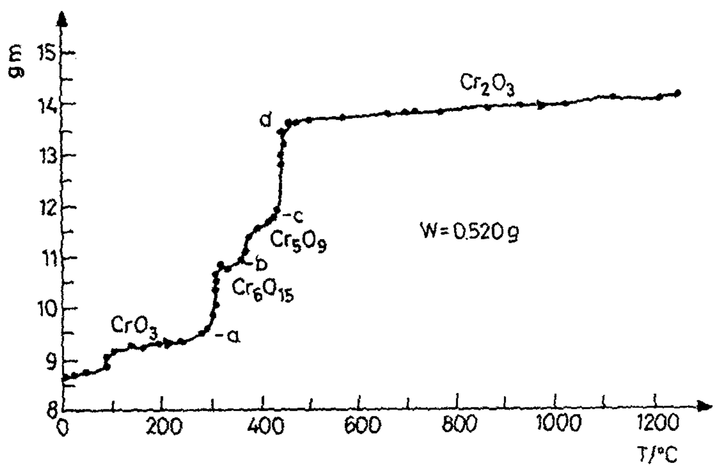

Fig. 10 Thermal decomposition of CrO_3 [1]

Photographs of a commercial version of Honda's thermobalance are shown in Figs 11 and 12 (photographs supplied by Prof. R. Otsuka and reproduced by kind permission of Prof. K. Funaki, Tokyo Institute of Technology). Reference to Fig. 3, a line drawing taken from Honda's original paper [1] shows two damping devices on the right-hand side of the balance beam, whereas only one damping device is apparent in Fig. 11. According to Saito [10] it was decided to remove

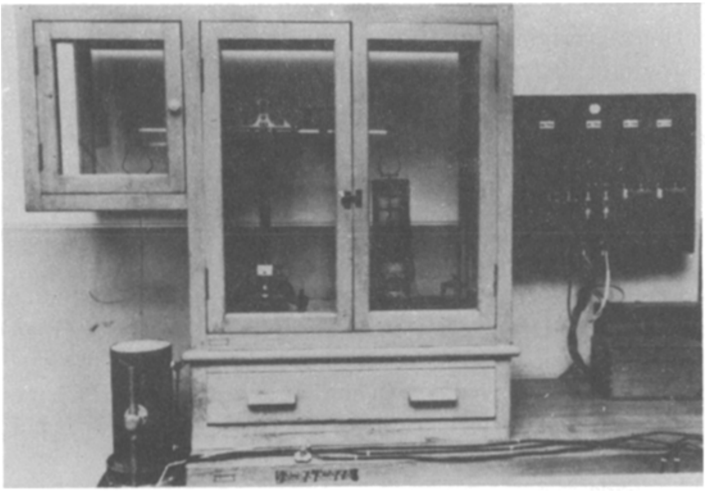

Fig. 11 A commercial version of the Honda Thermobalance (ex Prof. Funaki)

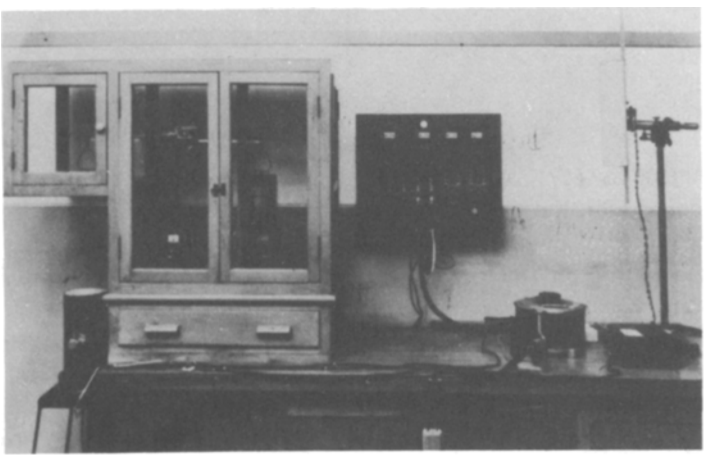

Fig. 12 A commercial version of the Honda Thermobalance showing the variable transformer

the second damper, in order to improve sensitivity. In Fig. 11, on the extreme right of the balance case, the method of null point weighing, achieved by varying the height of the Dewar flask with the aid of a screw mechanism, is clearly seen, whilst in Fig. 12 the variable transformer is seen on the laboratory bench, the lack of any mechanical contrivance confirming comments earlier in this paper [10, 11] that the furnace temperature was manually controlled. According to Saito [10] no photograph of Honda's prototype thermobalance exists, neither was it preserved for posterity. It was, however, the prototype that was used to obtain the results reported in Honda's original publication [1]. (Recent evidence has confirmed that the Honda Thermobalance depicted in Fig. 11 is the only extant example in the world. Since the recent death of Prof. Funaki, this unique apparatus is housed in the museum of the Tokyo Institute of Technology.)

The Honda thermobalance depicted in Figs 11 and 12, was made commercially by Narusekagaku, Sendai. Production commenced in 1920, Saito taking delivery of the first apparatus. Several hundred were made until production ceased in about 1954 [10].

Honda was a worker of sufficient experience to appreciate the considerable impact this paper would have. Nevertheless, natural modesty permitted him merely to conclude his paper with the words 'The investigation also shows the great convenience of using such a balance in similar investigations in chemistry'.

Perhaps surprisingly, the paper discussed above is Honda's sole publication in the field of thermogravimetry. This fact has been criticised by subsequent workers, who made major contributions to thermogravimetry in later years [15, 16], but it deserves to be emphasised that Honda was, first and foremost, a metallurgist who constructed his thermobalance merely to assist in one small part of his research interests i.e. the study of chemical changes at high temperature.

Appendix

Kotaro Honda [17] and Fig. 13 was born on 23 February, 1870 in Yahagimachi, Aichi, the third son of a wealthy farmer, Heizaburo Honda, who spent a considerable sum of money on irrigation work in Yahagimachi.

After graduating, in 1897, from the Department of Physics, Tohoku Imperial University, he became a lecturer in that department in the same year. Between 1907 and 1911 Honda visited Europe, spending the majority of his time in Germany, where he studied at Göttingen University under Prof. G. Tammann. (He subsequently received an honorary doctorate's degree from Göttingen in 1934.) He stayed in Paris for a short time during 1910 and in 1911 paid a brief visit to England. Whilst in England he went to Cambridge University where Prof. Sir James A. Ewing invited him to accept a visiting professorship in metallurgy, but the Japanese government refused to allow him to accept Ewing's invitation. Consequently, after buying some souvenirs in London and indulging some sightseeing, which included a visit to Westminster Abbey, he returned to Japan, where he became professor of physics at Tohoku Imperial University in 1911. His studies on iron led to his winning, in July, 1916 The Imperial Academy Award and in April of the same year, largely due to Honda's efforts, a research institute was established at the University. He was its first director and it initially bore the title of The Second Division of the Temporary Institute for Physical and Chemical Re-

Fig. 13 Kotaro Honda

search. Originally, investigations were carried out in the fields of the physical metallurgy of steels and the magnetic properties of metals, (KS-steel and KS-magnetic steel were two products of this work) and it rapidly became appreciated that the work of the Institute was of national importance. Accordingly, the Institute was established on a permanent basis in 1919, its terms of reference being the study of iron and steel. It was also re-named The Institute for Iron and Steel, with Honda remaining as Director of Research, and was the first of many research institutes in Japan to have a formal affiliation with a university. A photograph of the Institute building at this time is shown in Fig. 14. Under Honda's direction, the reputation of the Institute continued to increase and in 1922, the field of research was extended to include light metals, alloys and various metallic compounds. This expansion was accompanied by a further change of title to The Research Institute for Iron, Steel and Other Metals, Tohoku University and at this stage Honda reliquished the post of Director to become the Institute's first President. After the Second World War a new building was constructed (Fig. 15) and in honour of his achievements a statue of Honda was erected outside the main entrance, (one of us, CJK, is indebted to Prof. Dr. Hiroshi Watanabe past Director of the Institute, for providing photographs for Figs 14 and 15).

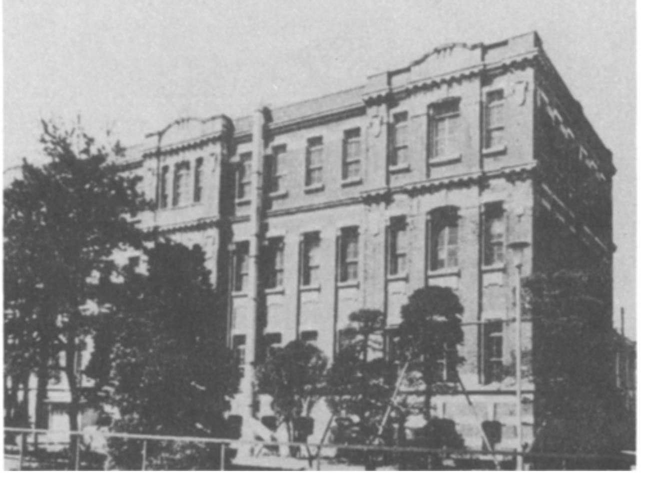

Fig. 14 The Institute for Iron and Steel, Tohoku University, circa 1920

Fig. 15 The Research Institute for Iron, Steel and other Metals, Tohoku University, circa 1950

A 'Memorial Room' is retained at the Institute, containing many of Honda's personal effects. Whilst associated with the Institute, Honda was provided with 'grace and favour accommodation' at 1-6-11 Tsuchidoi, Sendai.

In September, 1975, the Japan Institute of Metals founded a Metals Museum at Aoba Aramaki, Sendai, which houses several items of interest relating to Honda. One is a series of press cuttings reporting on his death (in 1954) and the photographs accompanying these reports leave little doubt as to his standing in science. For example, the size of the funeral cortège bears a similarity to that which, in Britain, would normally be associated with Royalty. A further item of interest is a photograph of Honda's birthplace. Since, to the best of the authors' knowledge this has never appeared in a western publication, it is reproduced in Fig. 16 (one of use, CJK, is deeply indebted to Dr. Yunoshin Imai, Emeritus Prof. Tohoku University and Director of the Metals Museum, Sendai for kindly providing a photograph of the press cutting showing Honda's birthplace).

Fig. 16 Honda's Birthplace

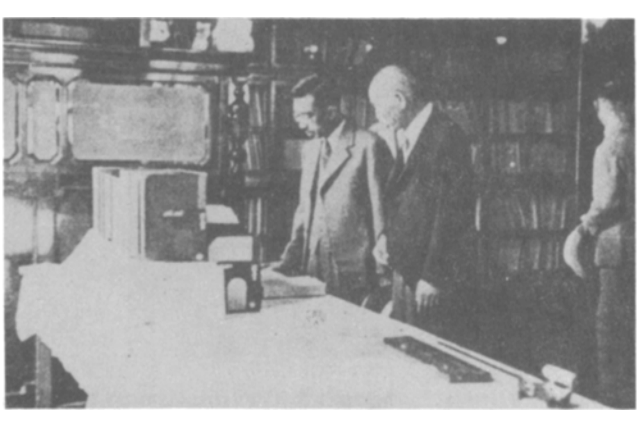

Fig. 17 (L) Honda explaining to Emperor Hirohito the work of the Institute (August 1947)

Fig. 18 (L) Honda in April, 1937. The first recipient of the Cultural Order

Fig. 19 (L) Honda. Photograph taken in Paris, February, 1910

During his professional career Honda received many decorations and awards for his outstanding achievements, the most notable of these being the Cultural Order, in 1937. This Order was conferred personally by Emperor Hirohito and Honda was the first recipient.

Fig. 20 (M) Prof. and Mrs. Honda on their Golden Wedding Anniversary

He married in 1898 and had two daughters. On 22 November, 1952 the Honda Memorial Foundation was established in honour of Honda's achievements in the fields of science and engineering. Unfortunately, he did not live to see the Foundation develop, as he died on 12 February, 1954.

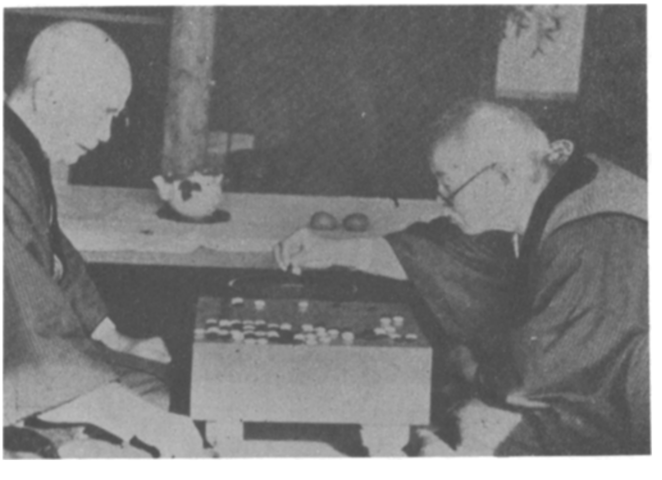

Fig. 21 (M) Honda, in 1950, playing 'Go' with Prof. Tawara, (a well-known physical metallurgist)

Some years ago, one of use, (CJK), visited Japan to carry out work into some aspects of a programme of research into the history and development of thermogravimetry [18] and was presented with two books by Prof. Dr. Hiroshi Watanabe, past President of the Board of Trustees, Honda Memorial Foundation, on behalf of the Foundation. Both books include photographs depicting various aspects of Honda's private and professional life. Since very few, if any, of these have previously appeared in a western publication, it was considered useful and informative to conclude this paper with a selection. The books are entitled 'The

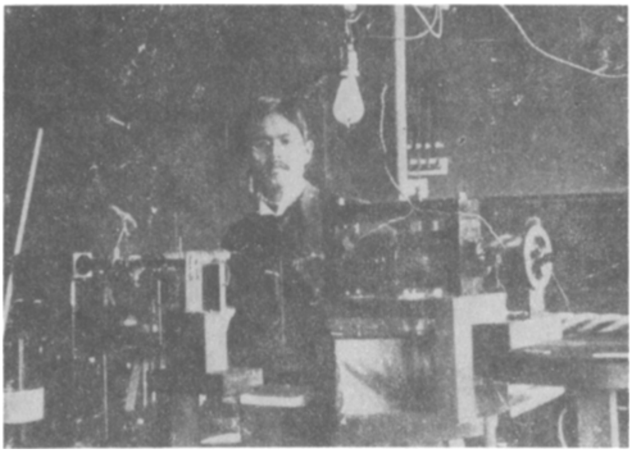

Fig. 22 (M) Honda at Göttingen University in March, 1908

Fig. 23 (M) Honda receiving his honorary doctorate at Göttingen University in 1934

Fig. 24 (M) Welcoming Dr. Einstein (1924) left to right: Prof. Honda, Dr. Einstein, Dr. Aichi, Dr. Kusakabe

Life of Kotaro Honda' Teijiro Ishikawa, 3rd edition, pub. Honda Memorial Foundation, Sendai 1973 and 'A Memorial to Kotaro Honda – Contributions from his Students' pub. Seibundoshinkosha, Tokyo 1955. The suffixes 'L' and 'M' to Figs 17 to 24 indicate their origins to 'Life' and 'Memorial' respectively.

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Zusammenfassung — Es wird kurz die Entwicklungsgeschichte der Chemie in Japan behandelt, gefolgt von einer ausführlichen Beschreibung der Honda Thermowaage und der erhaltenen Ergebnisse. Die Arbeit schließt mit einer kurzen Biographie von Honda, verbunden mit einer Sammlung von Fotographien, welche Honda's privates und wissenschaftliches Leben darstellen.