

SOME COMMENTS ON THE HISTORY OF THERMOGRAVIMETRY

C. EYRAUD¹, E. ROBENS², P. ROCHAS³

¹ Laboratoire Lyonnais d'Automatique et de Génie des Procédés,
Université Claude Bernard, 43, boulevard du 11 Novembre 1918,
F-69622 Villeurbanne Cédex (France)

² Institut für Anorganische und Analytische Chemie der Johannes-
Gutenberg-Universität, Postfach 3980, D-6500 Mainz (F.R.G.)

³ Institut Textile de France, F-69 Lyon (France)

SUMMARY

In a review of the authors about the history of vacuum microbalance techniques (including a chronology in table form), also published in *Thermochimica Acta*, (ref. 1) it was pointed out, that also some corrections are to be made on the history of thermogravimetry. The first findings of balances are dated to about 3000 B.C. A gravimetric hygrometer is mentioned by Nicolaus Cusanus in 1450. In the 19th century chemical reactions at variable temperatures were investigated using balances. The very beginning of the thermogravimetry is antedated to the work of Talabot who in 1833 at Lyon equipped a laboratory with thermobalances for quality control of Chinese silk.

HISTORY

The history of chemical balances has been written in the comprehensive work of Jenemann (ref. 2). Concerning microbalances we rely on the detailed review of Behrndt (ref. 3), Iwata (ref. 4) and recently of Gast (ref. 5). Earlier reviews on microbalances were made by Emich (ref. 6), Gorbach (ref. 7) and Cunningham (ref. 8). The history of thermogravimetry and thermobalances we find in Duval's book (ref. 9). A survey on commercially available instruments is included in the book of Mikhail/Robens (ref. 10). Concerning vacuum microbalance techniques the books of Czanderna/Walsky (ref. 11) and Lu/Czanderna (ref. 12) should be consulted. With respect to the origin of methods and instruments we need, however, some revisions and supplements.

A very basic revision should be made on the invention of the balance itself. The oldest balance beam made of limestone, about 85 mm long, was bought (and not found) by Sir Flinders Petrie in Upper Egypt (ref. 13). Regarding the material and its elaboration he is likely right in assigning this object to the predynastic Amratic period (Negade). His absolute dating of the Egyptian



Figs. 1,2: Talabot's thermobalances at Lyon for quality control of Chinese silk

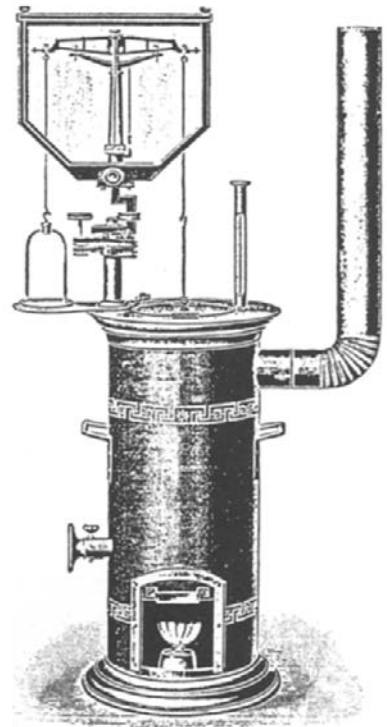


Fig. 3: Sauter conditioning apparatus (ref. 14)

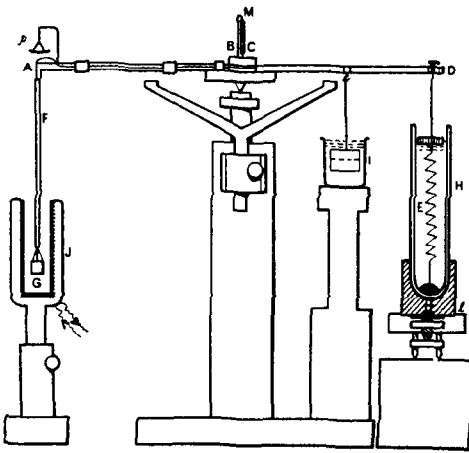


Fig. 4: Honda thermobalance

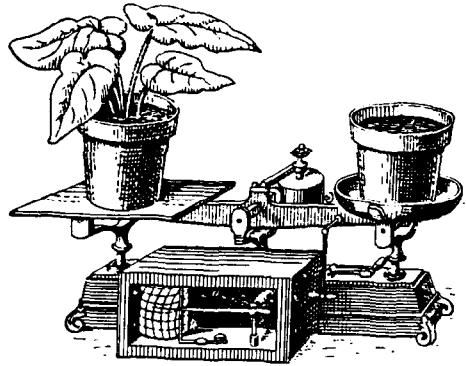


Fig. 5: Plant-physiological recording Roberval balance

history, however is incorrect, as proved e.g. using the radiocarbon method (ref. 15). The beginning of the Egyptian dynasties is about 3000 B.C. and not 5000 B.C., as he assumed. Thus, the balance beam can be dated somewhat older than 3000 B.C. (ref. 16). Regarding an early drawing of a balance beam of the same type in the tomb of Hesi-re (3rd dynasty), we know that such balances were used at about 2650 B.C. (ref. 17). Of course, the invention might have been much earlier, but we have no properly dated findings.

In the 19th century balances were used to investigate chemical reactions at different and variable temperatures. The very beginning of thermogravimetry, however, should be dated to 1833. In this year Talabot equipped a laboratory at Lyon with thermobalances for quality control of Chinese silk (ref. 18, Figs. 1,2). Independently in 1915 Honda seems to be the very first to use the expression "thermobalance" for his instrument (Fig. 4, ref. 19). After a short time, commercialized instruments were offered from several companies (Fig. 3).

If we generalize thermogravimetry as a generic term for continuous weighing also at constant temperature, we should consequently include gravimetric sorption measurements and gravimetric hygrometry. An example of physiological investigations of water turnover is shown in Fig. 5. The beginnings of hygrometry are found in the 15th century, when Nicolaus Cusanus, Leo Battista Alberti and Leonardo da Vinci proposed or even realized hygrometers using a balance loaded with wool, cotton or a sponge

(ref. 20). Leonardo claimed to have observed big deflections of a balance loaded with weights of equal mass but different volume during a thunder-storm. He assigned the deflection to density changes of the air, and he recommended this buoyancy balance "as a means to see, when the weather will break" (refs.21).

REFERENCES

- 1 E. Robens, C. Eyraud, P. Rochas: Some comments on the history of vacuum microbalance techniques. To be published in: S.A.A. Jayaweera (ed.): *Thermochimica Acta* (1990).
- 2 For reference see especially p. 775 of H.R. Jenemann: *Die Entwicklung der mechanischen Präzisionswaage*. In: M. Kochsiek (ed.): *Handbuch des Wägens*, 2. ed. Vieweg, Braunschweig 1985, p. 745-779.
- 3 K.H. Behrndt: *Die Mikrowaagen in ihrer Entwicklung seit 1886*. *Z. angew. Phys.* 8 (1956) 9, 453-472.
- 4 S. Iwata: Über die Entwicklung der Thermowaage, besonders in Japan. Vortrag am Chemischen Inst., Universität Bonn, 6.6.1961.
- 5 Th. Gast in: S.A.A. Jayaweera (ed.): *Thermochimica Acta* (1990).
- 6 F. Emich: Einrichtung und Gebrauch der zu chemischen Zwecken verwendbaren Mikrowaagen. In: E. Abderhalden (Hrsg.): *Handbuch der biochemischen Arbeitsmethoden*, Bd. 9, Berlin 1919, 55-147.
- 7 G. Gorbach, *Mikrochemie*, N.F. 14 (1936) 254.
- 8 B.B. Cunningham, *Nucleonics* 5 (1949) 62.
- 9 C. Duval: *Inorganic Thermogravimetric Analysis*. 2. Ed., Elsevier Amsterdam 1963, pp. 3-24.
- 10 R. Sh. Mikhail, E. Robens: *Microstructure and Thermal Analysis of Solid Surfaces*. Wiley, Chichester 1983.
- 11 A.W. Czanderna, S.P. Wolsky: *Microweighing in Vacuum and Controlled Environments*. Elsevier, Amsterdam 1980.
- 12 C. Lu, A.W. Czanderna: *Applications of Piezoelectric Quartz Crystal Microbalances*. Elsevier, Amsterdam 1984.
- 13 R. Hall, Petrie Collection, London, personal communication.
- 14 Sauter catalog, Ebingen 1928, pp. 124 ff.
- 15 F.A. Hassan, *J. Near Eastern Studies* 39 (1980) 203-207.
- 16 H.R. Jenemann: 10000 Jahre Waage?. In print.
- 17 F.G. Skinner: *Weights and Measures, their ancient origins and their development in Great Britain up to 1855*. Science Museum, London 1967, p. 7.
- 18 C. Eyraud, P. Rochas: *La thermogravimétrie à la condition des soies de Lyon, une aventure méconnue*. Paper presented at the "International Microbalance Techniques Conference", 20. April 1985, Dijon. *Thermochimica Acta* (1986).
- 19 K. Honda, *Science Reports of the Tôhoku Imperial University, Sendai Serie I*, 4 (1915) 97-105, 2610.
- 20 E. Robens, C.H. Massen, J.J. Hardon: *Studies on historical gravimetric hygrometers*. To be published in: S.A.A. Jayaweera (ed.): *Thermochimica Acta* (1990).
- 21 E. Gerland, F. Traumüller: *Geschichte der physikalischen Experimentierkunst*. Leipzig 1899, Reprint: Olms, Hildesheim 1965, pp. 107-108.