

## Note

# Some comments on the history of vacuum microbalance techniques

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## Abstract

Based on the reviews of Behrndt, Iwata, Duval, Jenemann and Gast, we present a chronology of vacuum microbalance techniques in table form. The earliest discoveries of weights and balances are dated to about 3000 B.C. In the 19th century, balances were operated in vacuum for meteorological purposes. Thermogravimetry began with Talabot who, in 1833 in Lyon, equipped a laboratory with thermobalances for quality control of Chinese silk. At the beginning of the 20th century, microbalances were placed in high-vacuum apparatus.

## HISTORY

Although microweighting in vacuo and at variable temperatures are recent techniques, their origins go back to the time when the kilogram was standardized [1] and possibly to the beginning of vacuum techniques. We can recommend the following reviews on the development of chemical balances [2], microbalances [3–7], and thermogravimetry [8–11]. A survey of current commercially available instruments is included in the book written by Mikhail and Robens [12]. Concerning vacuum microbalance techniques, the volumes by Czanderna and Wolsky [13] and by Lu and Czanderna [14] should be consulted. For details of the proceedings of the VMT Conferences, the reader should see refs. 15–18. Monographs on thermogravimetry are mostly presented in the context of thermal analysis. A variety of conferences deal with this subject, for example, those organized by the ICTA (now ICTAC) [19] or by national thermoanalytical societies. Specialized journals are *Thermochimica Acta*, the *Journal of Thermal Analysis* and *Netsu Sokutei*.

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TABLE I  
A historical record of vacuum balances and thermobalances

Year	Invention/investigation	Name	Ref.
<3000 B.C.	Discovery of weights	(E. Mediterranean) (Egypt, Negade period)	30
≤3000 B.C.	Discovery of a balance beam	Hesi-re	31, 32
≈2650 B.C.	Drawing of a balance		22
27 B.C.	Mass loss of limestone	Vitruvius	33
1450	Gravimetric hygrometer with wool	Nicolaus Cusanus	34
≈1470	Gravimetric hygrometer with a sponge	Alberti	35
≈1490	Gravimetric hygrometer with cotton, buoyancy and air density measurements	Leonardo da Vinci	36
1685	Gravimetric hygrometer with vitriolic acid	Gould	37, 38
1833	Thermogravimetry of silk	Talabot	39, 40
1854	Susceptibility measuring method	Plücker	41
1861	Meteorological vacuum balance	Regnault	42, 43
1875	Bunge vacuum balance	Bunge	44
1886	Microbalance, glass beam	Warburg, Ihmori	45
1889	Susceptibility measuring method	Gouy	46
1895	Electromechanical balance	Ångström	47
1901	Glass fibre balance	Salvioli	48
1903	Inclination torsion microbalance	Nernst	49–51
1903	Adsorption measurement	Giesen	52
1909	Triangular quartz-rod buoyancy balance	Steele, Grant	53, 54
1909	Electrostatic suspension of particles, $10^{-15}$ g	Konstantinowsky	55
1910	Susceptibility measurement	Pascal, Cheneveau	56, 57
1919	Photographic recording balance	Bunge, Kuhlmann	2
1912	Electromagnetic compensating vacuum balance	Urbain	58
1912	Triangular quartz-rod beam-zero balance	Gray, Ramsay	59
1912	Electromagnetic compensating vacuum balance	Emich	60

1914	Quartz fibre suspension sorption measurement, 10 <sup>-10</sup> g	Petterson	61, 62
1914	Quartz rod beam	Aston	63
1915	Thermobalance	Honda	18, 64
1920	Coefficient of expansion	Honda	65
1923	Hydrostatically compensated thermobalance	Guichard	66, 67
1926	Quartz spring vacuum balance	McBain, Bakr	68
1927	Etching	Inamura	69
1929	Quartz fiber suspension of the beam	Strömberg	70
1929	Susceptibility measurement in vacuum	Aharoni, Simon	71
1930	Quartz spring vacuum balance with electromagnetic force compensation	Oshima, Fukuda	72
1931	Quartz balance with pointer	Donau	73
1933	Photographic recording thermobalance	Skramovsky	
1936	Vapour pressure, Knudsen method	Mochida, Shibata, Niwa	74, 75
1937	Magnetic suspension	Holmes	76
1940	Quartz balance with rider	Barret et al.	77
1943	Automatically compensating electrodynamic vacuum microbalance, Helmholz coils, permeation measurements	Vieweg, Cast	78
1944	Automatic quartz-rod torsion balance	Gulbransen	79
1945	Adamel photographic-recorded thermobalance	Chevenard et al.	80
1946	Electromagnetic compensating glass-beam balance	Gregg, Wintle	81
1947	Electromagnetic suspension	Clark	88
1948	Automatically compensating electromagnetic microbalance, measurement of dielectric properties	Gast, Alpers	82
1949	Particle size distribution	Hara	84
1949	Vacuum torsion balance	Rhodin	85, 86
1950	Thermocouple in the sample	Kinjo, Iwata	87, 88
1950	Gravimetric surface area and pore size determination	Glasson	89
1950	Electromagnetic suspension balance	Beams	90, 91
1953	Electromagnetic registration	Kinjo, Iwata	92
1953	A.R.A.M./D.A.M./Setaram vacuum thermobalance	Eyraud	93, 94
1953	Sartorius differential thermogr. analysis	de Kaysen	95

TABLE 1 (continued)

Year	Invention/investigation	Name	Ref.
1954	Magnetic susceptibility	Hirone et al.	96
1955	Low/high-temperature vacuum balance	Gregg	97
1955	Suspension in alternating electrical field	Straubel	98
1956	MOM derivatograph	Paulik et al.	99
1956	Aminco thermo-gravimetric high-pressure/vacuum with spring balance	Campbell et al., Muller	100, 101
1957	Quartz crystal balance	Sauerbrey	102
1958	Combination of DTA and TGA	Splitek	103
1958	Automatic vacuum thermobalance	Tomonari et al.	104
1959	High-pressure thermobalance	Rabatin, Card	105
1959	Sartorius vacuum microbalance	Gast	106–108
1959	Electromagnetic suspension	Gast	109
1959	NETZSCH thermogravimetric apparatus with pan on top		121
1960	Liquid density measurement	Schöneck, Wanninger	110
1960	Cahn vacuum microbalance	Cahn, Schultz	111, 112
1962	Sartorius Gravimat	Sandstede, Robens	113
1970	Vibration vacuum balance	Gast	114
1970	Compensation by light pressure	Zinnow, Dybwad	115
1971	Autom. quartz-fiber ultramicrobalance	Czanderna, Wolsky, Rodder	13, 116
1971	Simultaneous measurement of weight and torque by hybrid compensation	Gast	117
1972	Weighing in alternating electric field, $10^{-13}$ g	Straubel, Böhme, Robens	118
1975	Wireless transmission of values from sample	Gast	119
1978	Glass ceramics beam	Loimar, Ulrich	1
1982	Simulation of high heating rates	Dunn et al.	120

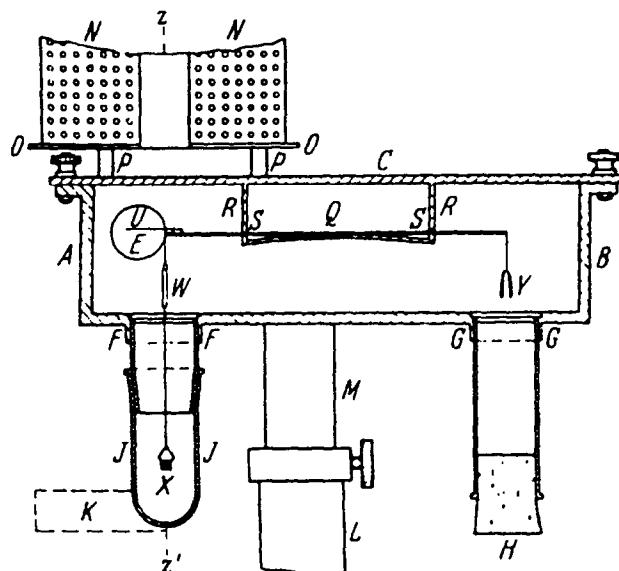


Fig. 1. Emich electromagnetic compensating vacuum microbalance: Z, solenoid with coil N; W, permanent magnet; X, sample pan, Y, counter-weight; Q, quartz tube beam; diameter, 0.7 mm; length, 82 mm; U, quartz fibre pointer; E, ocular micrometer.

With respect to the origin of methods and instruments, some revisions and supplementary reviews are required. Because of incorrect absolute dating of Egyptian history, drawings and discoveries of weighing equipment were thought to be older than they really are [20]. The oldest balance beam, a small instrument made of limestone originates from Upper Egypt [21] and is assigned to the pre-dynastic Negade period (3700–3200 B.C.).

From an early drawing of a balance beam of the same type in the tomb of Hesi-re (3rd dynasty), we have proof of the use of balances at about 2650 B.C. [22]. Thus, we may date the balance beam to somewhat earlier than 3000 B.C. From that time, there are also discoveries of weights in Egypt, in other parts of the eastern Mediterranean, and in India. Of course, its invention may have been much earlier, but there are no properly dated findings.

The Egyptians used the balance in metal manufacturing [23]. An early thermogravimetric investigation is mentioned in Vitruvius' report on lime burning [24]. Gravimetric hygrometers loaded with wool, cotton or a sponge were first described in the 15th century by Nicolaus Cusanus, Leo Battista Alberti and Leonardo da Vinci [25]. Leonardo claimed to have observed big deflections of a balance loaded with weights of equal mass but different volume during a thunderstorm, as a consequence of density changes in the air. He recommended this buoyancy balance 'as a means to see, when the weather will break' [26, 27].

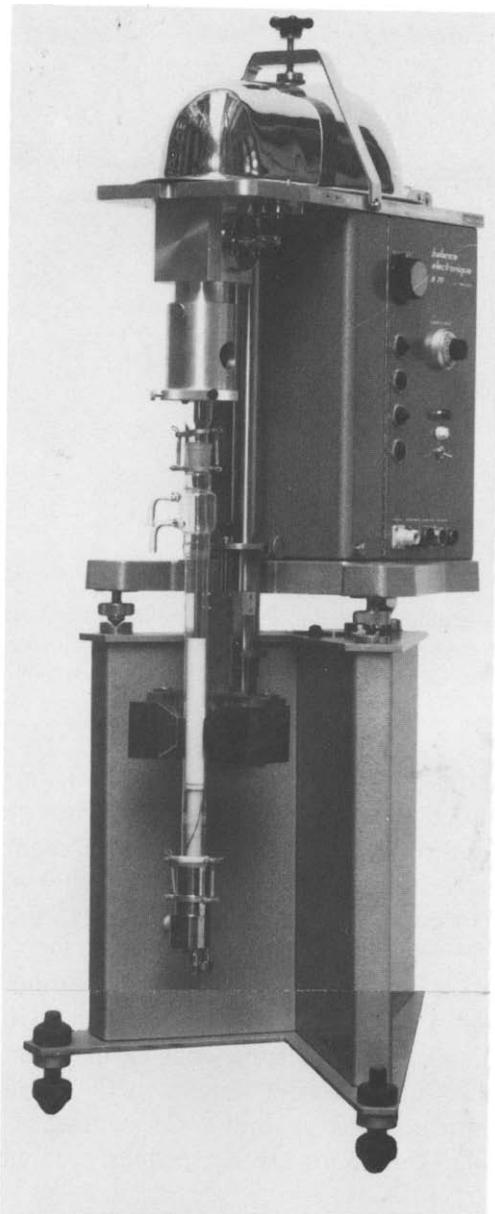


Fig. 2. Eyraud recording vacuum thermobalance (Setaram).

Thermogravimetry began in 1833 when Talabot equipped a laboratory in Lyon (France) with thermobalances for quality determination of Chinese silk [28]. Independently, in 1915, Honda seems to be the very first to use the expression ‘thermobalance’ for his instrument [29]. The first microbalance was built in 1886 by Warburg and Ihmori.

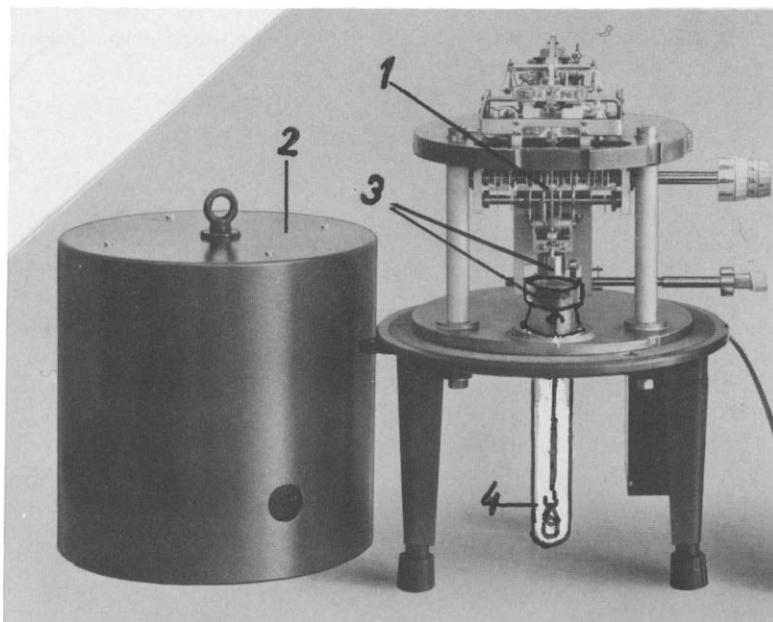


Fig. 3. Gast electromagnetic suspension balance (Sartorius): 1, electrodynamic compensating balance with mechanical weight load; 2, balance thermostat; 3, magnetic suspension system; 4, sample vessel.

In the 19th century, balances were operated in vacuum for meteorological purposes. The first vacuum microbalance, already with electromagnetic compensation, was made in 1912 by Emich (Fig. 1). In 1953, the company A.R.A.M. manufactured Eyraud's electrodynamic recording balance (Fig. 2). The most favorable technique for mass determination in *vacuo* is realized using electromagnetic suspension of the sample, the balance being outside the sample vessel (Fig. 3).

Table 1 lists important developmental steps in vacuum balances and thermobalances, and gives their first application to various types of investigation.

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