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The impact of feedback on the determination of masses and forces in controlled atmospheres

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Abstract

Examples of various developments are shown which have led to the present balances used for application in controlled atmospheres. The development started from the beam balance and the spring balance. The geometrical configuration, selection of materials and choice of indication resulted in a diversity of balances, adapted to physicochemical experiments in vacuo or in gases and vapours under predetermined temperatures and pressures. The introduction of feedback control, applicable to all basic modifications of mechanical balances, resulted in improved performance with reduced demands on mechanical design. It has facilitated the simultaneous observation of different variables, e.g. mass and torque or force components. The elements of automatic weight compensation in a closed loop are discussed.

Feedback control represents the basis of free magnetic suspension which enables the weighing of an object in a closed container with an external balance and the measuring of forces on a body in controlled motion. It also enables signals for sample temperature and gas pressure to be transmitted through the wall of the reaction chamber. New developments in this special field are briefly discussed.

INTRODUCTION

If we exclude mass determination by vibration [1], three basic methods are in common use for weighing and force measurement: comparison of weight, i.e. the beam balance; the use of elastic forces, i.e. the spring balance and torsion balance; and the use of electromagnetic or electrodynamic forces, i.e. the electrical balance and suspension balance.

Instruments according to the first and second principle can work as simple autonomous systems or in the compensation mode, which corresponds to a closed loop with the operator as controller. With the beam balance, the operator adds or removes weights, shifts a sliding weight or varies the torque of a torsion wire. In order to weigh in controlled atmospheres, the motion of a torsion head can be easily affected through a rotatable seal, or a set of weights may be operated by electromagnets. But compensation by electromagnetic or electrostatic forces is by far more elegant and easily lends itself to automation with the aid of feedback control.

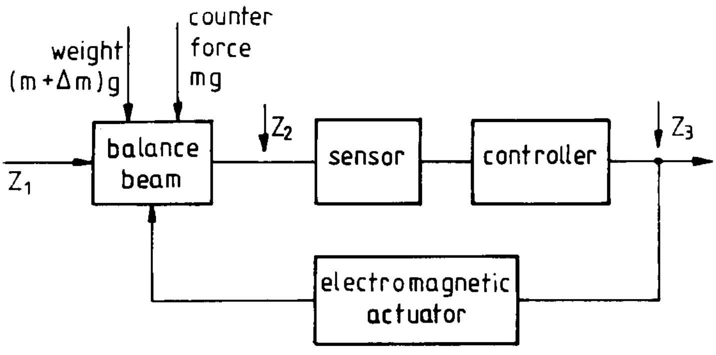

Fig. 1. Closed loop of an electromagnetic balance.

Self-compensating balances with photoelectric position sensors

According to Fig. 1, the closed loop of an electromagnetic balance contains a comparator (e.g. a balance beam), a deflection sensor, a controller and an electromagnetic actuator. The input is a weight or a force; the output may be a current. Disturbances Z_1 and Z_2 correspond to an additional force such as a Knudsen force or the effect of a difference in arm length, the zero shift or the sensor and/or controller, and a bias in the indication. Disturbance Z_3 originates in drift of the conversion factor of the actuator. The sensor can be based on diverse physical effects, such as electrostatic or electromagnetic induction, and various optical methods, including interferometry and polarization. With the aid of proportional, derivative and integral control, a very high open-loop gain is attained, resulting in the suppression of certain systemic errors, very good dynamic properties and last, but not least, infinitesimal deflection of the balance pan.

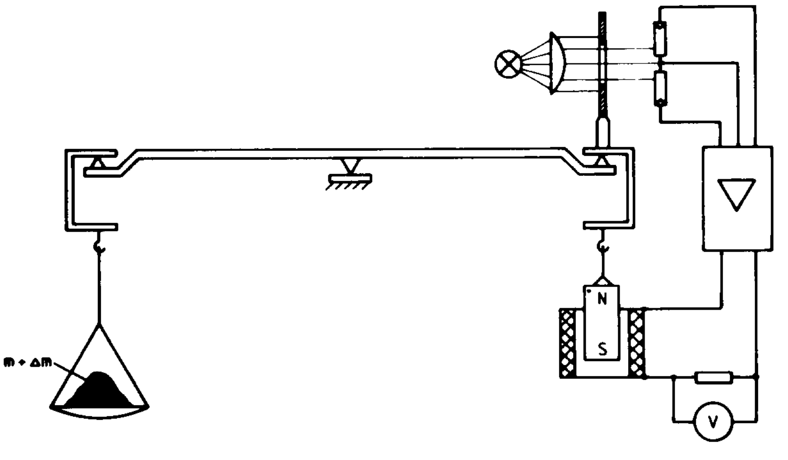

Fig. 2. Scheme of an electromagnetic beam balance.

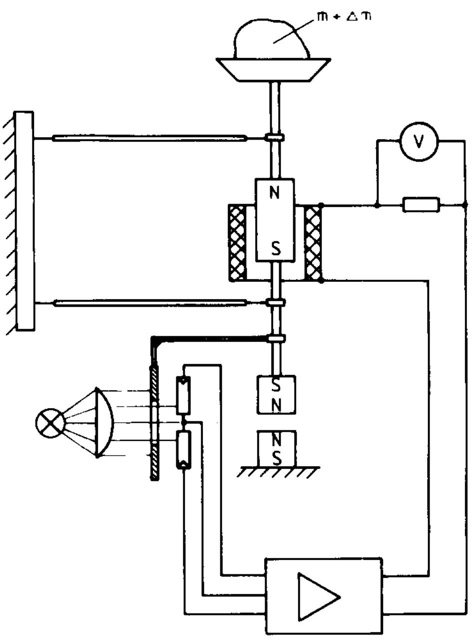

Fig. 3. Beamless self-compensating balance.

Automatically compensating balances can be realized according to Fig. 2. The balance pan is suspended from the left end of the balance beam, while the right end supports a bar magnet and a slit diaphragm which controls the ratio of the luminous fluxes from the light source to each of a pair of diodes. A signal arises which is proportional to the deflection of the beam and forms the input of a controller, whose output feeds a coil around the magnet. Thus, the balance is kept in equilibrium and the current which flows through the coil is proportional to the change in mass Δm. Similar arrangements have been used in the balance of Eyraud [2], an early vacuum balance made in France, and in related microbalances produced by Setaram and others. The actuator can be exchanged for an electrodynamic device, e.g. a moving coil system. The Cahn balance is an example of this modification [3].

Automatic compensation balances can be built without a balance beam, as shown in Fig. 3. The pan is guided on a vertical path by two levers with elastic joints. A force coil and a bar magnet generate the compensating force and the position is again detected by a photoelectric sensor. The system needs an upward directed force to compensate the weight of the balance pan and its support. This auxiliary force can result from magnetic repulsion. If we replace the electromagnetic actuator of this balance by an electrodynamic one, with a moving coil in the air gap of a magnet, the well-known electrodynamic balance results, which is built by many corporations for utilization in laboratories and in production.

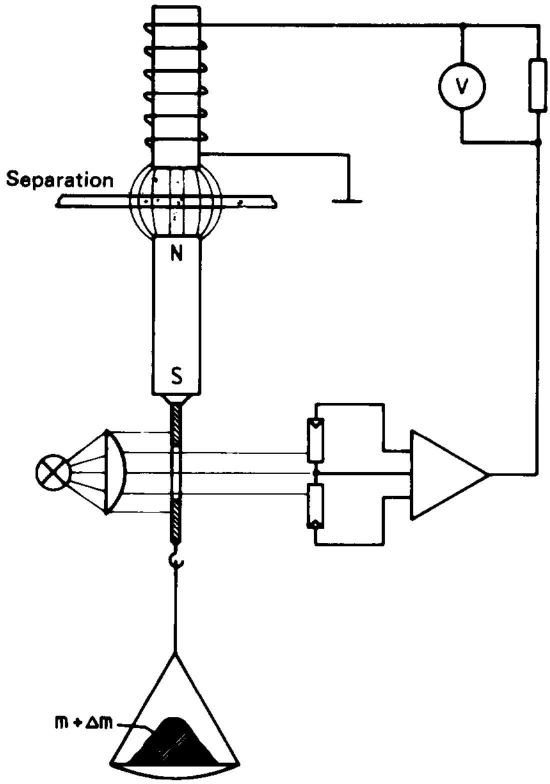

Fig. 4. Scheme of a magnetic suspension balance.

Apparently the next step in development, but in reality more than 40 years old, is the free magnetic suspension balance, published by Beams [4] and also by Clark [5] and taken up recently by Luce and Gast [6]. As illustrated in Fig. 4, an electromagnet attracts a ferromagnetic body, e.g. a permanent magnet carrying a slot diaphragm and the balance pan. The diaphragm is part of an optical position sensor which controls the current through the winding of the electromagnet. This current is proportional to the deviation of the total weight from an initial value, which depends on the vertical position of the sensor. The lower magnet with diaphragm and pan can be included in a vessel, whose wall has to be non-magnetic, between the poles of the two magnets. Thus the sample is weighed inside the container which can be thermostated and evacuated or filled with gases or vapours.

Balances with inductive sensors and amplitude modulation of the carrier frequency

The next examples of balances, operating in a closed loop, utilize inductive position detectors. In 1943, the author was commissioned by his

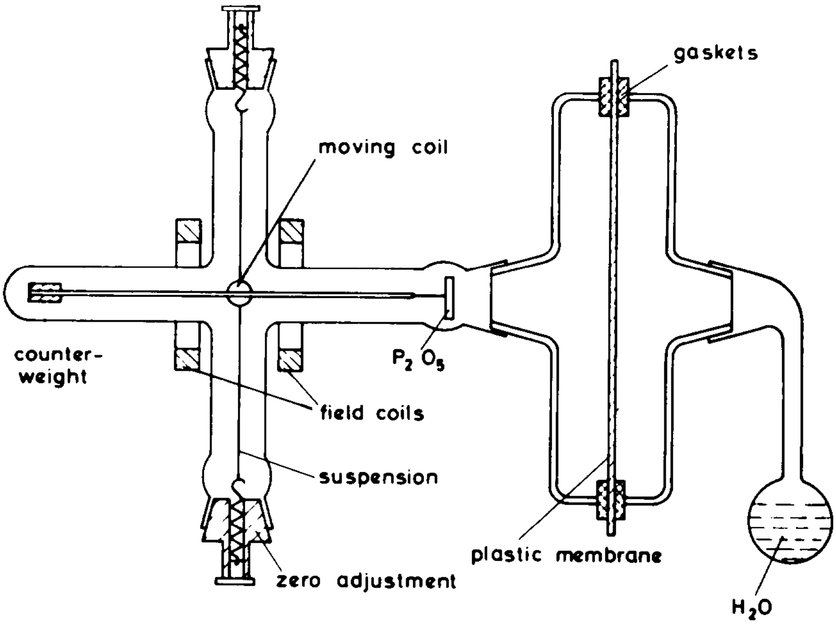

Fig. 5. Permeation measurement with the electrodynamic vacuum microbalance.

chief, Professor Vieweg, to improve the existing measuring methods for the permeation of water vapour through plastic foils. The following solution was chosen. As can be seen in Fig. 5, saturated water vapour diffuses through the examined membrane into the casing of the balance. The interior of the casing is kept at a negligible partial pressure of H_2O by a pill of phosphorous pentoxide, which is attached to the beam of the vacuum microbalance. Such a balance was not available at that time and it had to be developed and built. Being rather unprejudiced, the author designed the balance primarily for the task in question. It had to be easy to handle, reliable over periods of weeks, automatically recording and, as far as possible, insensitive to adsorption. The principle of automatic compensation was applied. An electrodynamic torque motor was used, consisting of a pair of Helmholtz field coils rigidly mounted outside the vacuum chamber, and a moving coil attached to the balance beam inside the chamber. The moving coil was suspended in taut bands of platinum–nickel alloy. They served as electrical connections and could be twisted from the outside by ground-in metallic stoppers in order to tare the balance [7].

With a constant current I_1 exciting the field coils and an arbitrarily variable and measurable current I_2 flowing through the moving coil, this system represents a dynamometric electrobalance, if the direction of the balance beam is optically detected and kept horizontal by adjusting the current I_2. The mass increment Δm follows the equation

$$\Delta m = k_1 I_1 I_2$$

where k_1 is an instrument parameter. But the system was first rendered self-compensating in the following way. The combination of field coils and moving coils represents a variable transformer. If the field coils are excited by an alternating current I_3, a signal u is induced in the moving coil

$$u = k_2 I_3 \alpha$$

where k_2 is an instrument parameter and α the deflection of the balance beam. In order to close the automatic compensation control loop, the signal u must be converted into the direct current I_2 which flows through the moving coil as mentioned above, while the field coils are continuously excited with the direct current I_1. This means that I_1 and I_3 flow simultaneously through the Helmholtz coils, which is feasible by means of a choke and a capacitor, forming a separating filter, as can be seen in Fig. 6. Now, it is necessary to derive the current I_2 from the signal u which is proportional to amplitude and sensitive to phase. This could be achieved by phase-sensitive rectification and subsequent amplification. Choosing a high frequency of I_3 and utilizing resonance, a very high gain of the system was obtained.

In the evacuated casing, with frictionless suspension and high resistance in the circuit of the moving coil, there is little physical damping. In order to obtain adequate dynamic behaviour, a direct current amplifier with proportional and derivative action was included in the signal path. Figure 7 shows the amount of water permeating through a disc of polystyrene in the steady state of diffusion, measured with the first prototype of these balances.

In September 1944, the three existing instruments were destroyed, but a new one was built by Erika Alpers in 1946 for ponderometric measurement of permitivity and loss factor [8], and another, whose photograph is shown

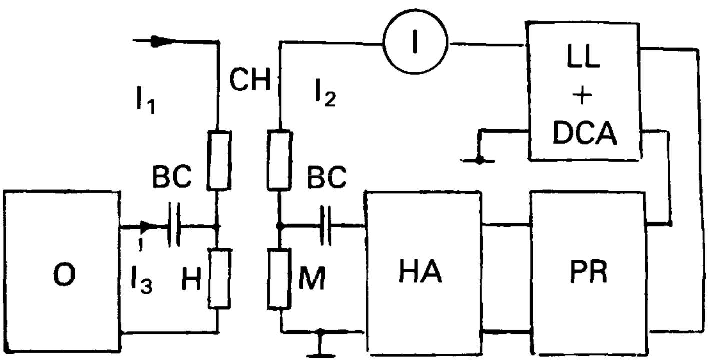

Fig. 6. Block diagram of the electrodynamic vacuum microbalance: I_1, exciting direct current; O oscillator; H, Helmholtz coils; M, moving coil; CH, chokes; BC, blocking capacitors; HA, high frequency amplifier; PR, phase-sensitive rectifier; LL, low pass and lead network; DCA, direct current amplifier; I, indicating instrument.

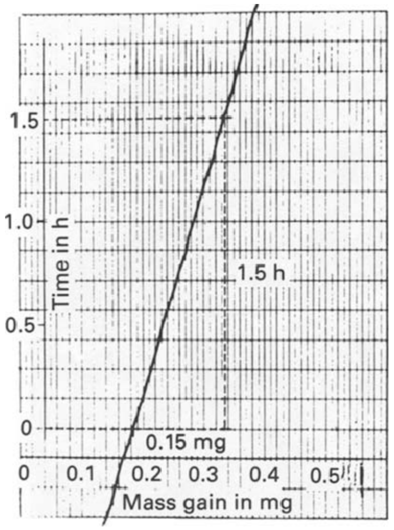

Fig. 7. Permeation of water vapour through polystyrene at 25°C.

in Fig. 8, was built in 1949 for a laboratory in Australia. A direct line of development leads from these prototypes to the microbalances produced by Sartorius for use in air and in controlled environments. Figure 9 shows the system of an ultramicrobalance which is now equipped with a permanent magnet but still utilizes the transformatoric sensor with the moving coil as secondary winding, simultaneously used as a torque generator.

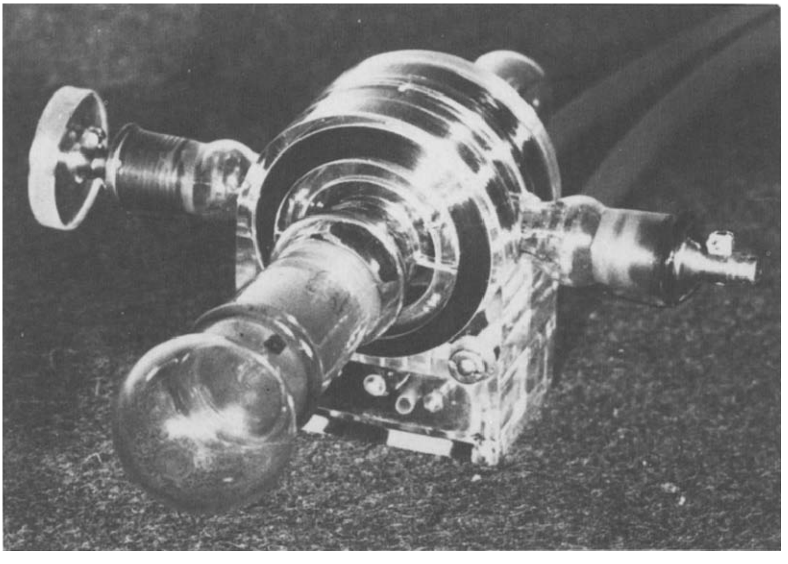

Fig. 8. Early electrodynamic vacuum microbalance.

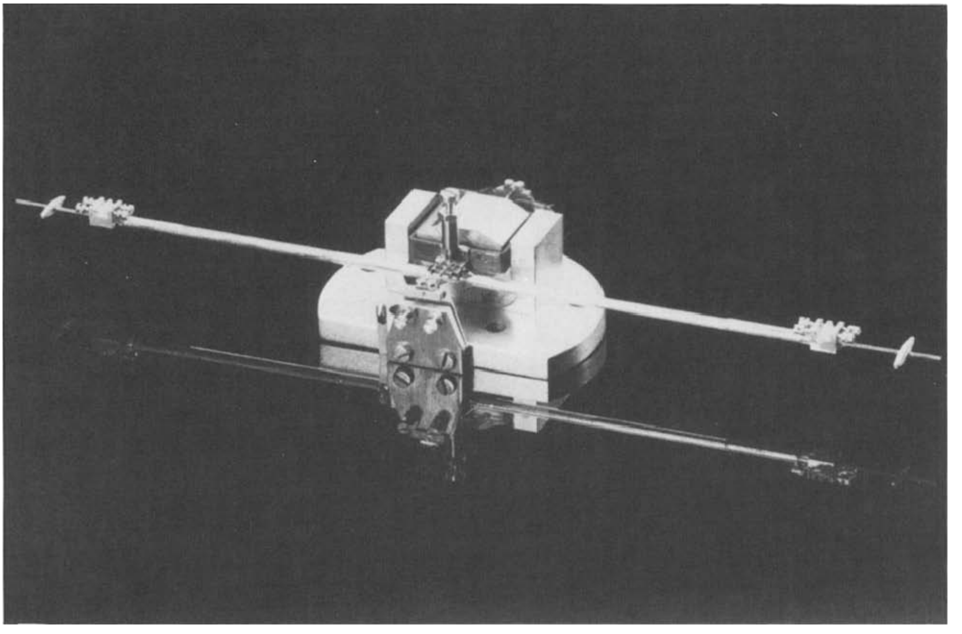

Fig. 9. System of an ultra microbalance.

Simultaneous measurement of weight and torque

A rather difficult task was presented in the measurement of molecular weights according to Knudsen. Two vapour jets from a cylindric casing which contained the sample, generated a torque which had to be recorded simultaneously with the loss in weight. From the measured values the velocity of the jets can be calculated. From this magnitude and the temperature of the sample, the molecular weight is determined. The requirements were: drift in mass determination less than 1 μg over some hours; load capacity 15 g; and useful resolution in torque measurement, 10^{-5} dyne cm = 10^{-12} N m. A taut-band-suspended balance beam was used, shown in Fig. 10, with the winding printed on a silica plate fused to the girders at the rear end, and a crosshead for two suspension bands brazed to the front end. The suspension bands carried a lower crosshead, to which a vertical torsion band was attached. This band led down to a shaft which supported the sample by means of a gimbal joint and a sturdy wire. The shaft was equipped with a metal vane, playing between two pairs of electrodes. The mass rate was measured by automatic electrodynamic compensation, with the printed winding being exposed to the magnetostatic field of a pair of horseshoe magnets and to the superimposed high-frequency field of two coils mounted in the lumen of the magnets.

Simultaneously, the torque could be measured by automatic electrostatic compensation with the aid of the vane in the combined static and high-fre-

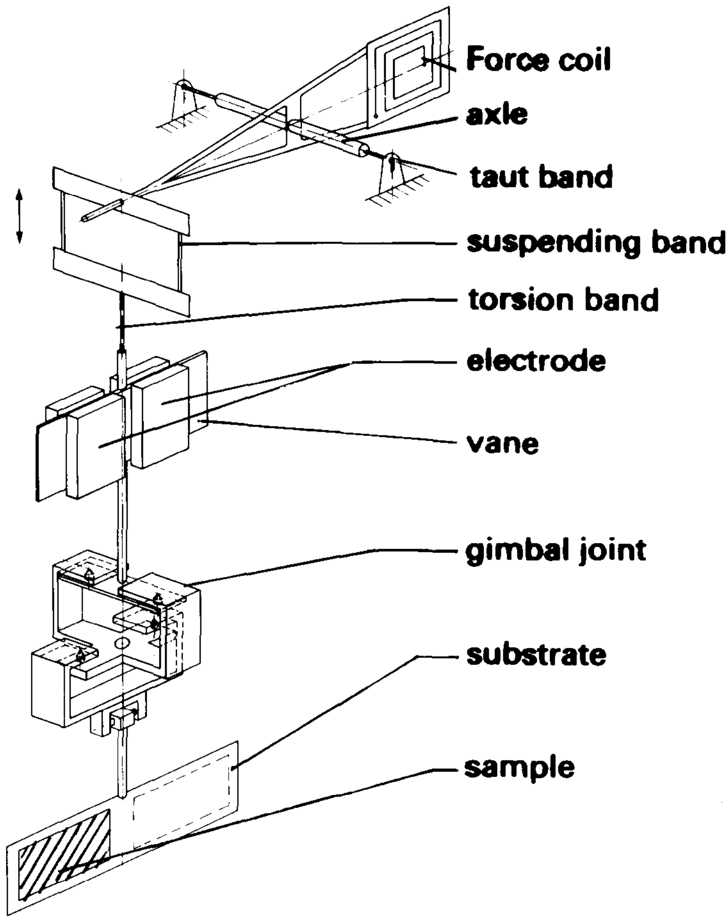

Fig. 10. Combined weight and torque measurement.

quency field in the gaps of each pair of electrodes [9]. The instrument was thoroughly tested and fulfilled its specifications.

Balances with position sensors utilizing frequency modulation

While the balances described above utilized sensors with carrier frequency and amplitude modulation, we shall now consider systems with frequency modulation.

In 1957, the author built a model of the suspension balance whose attracting magnets were discs of hard ferrite. The downward flux of the upper magnet could be controlled by an electromagnet placed on top of it, as shown in Fig. 11; the lower one carried the balance pan and was overlaid with an aluminium foil. This foil influences the inductivity of a flat sensor coil which is cemented to the lower side of the upper magnet and included in the grid resonant circuit of a Huth–Kühn oscillator. The anode current

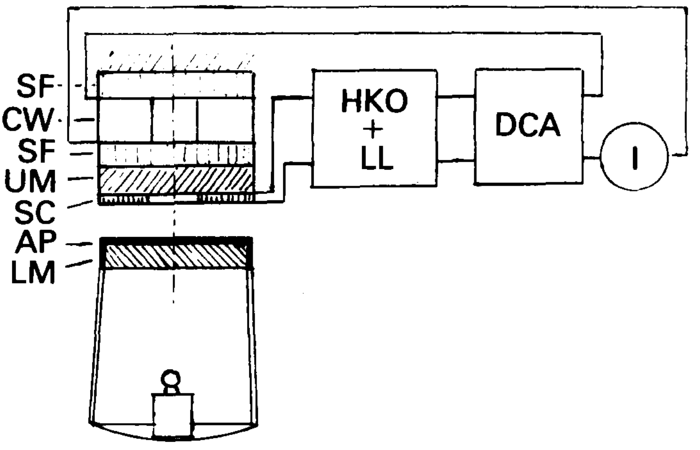

Fig. 11. Suspension balance with ferrite magnets and eddy current sensor: SF, soft ferrite; CW, control winding; UM, upper magnet; SC, sensor coil; AP, aluminium plate; LM, lower magnet; HKO, Huth–Kühn oscillator; LL, low pass filter; DCA, DC-amplifier; I, indicator.

of the oscillator depends on the distance between the sensor coil and the aluminium foil. Therefore an error signal for the distance control including derivative action can be extracted by a load resistance. This system served satisfactorily over three decades for classroom demonstrations. The idea to

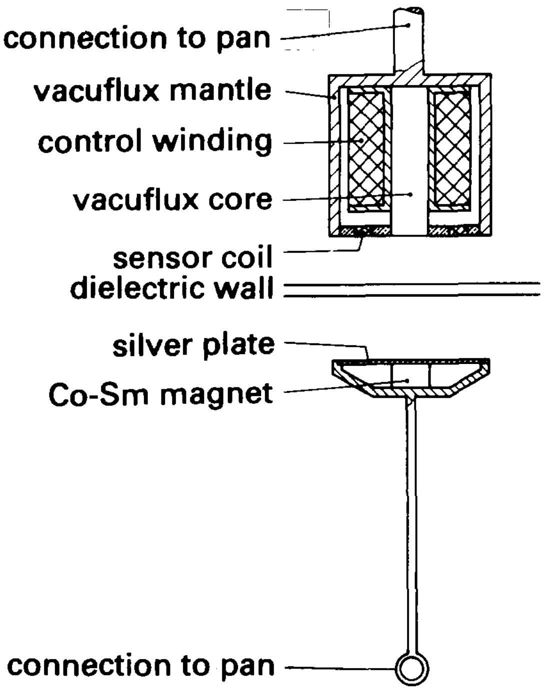

Fig. 12. Magnetic system of the suspension balance.

use it as a coupling between the sample and a balance outside the vessel gave the impulse to develop the "Schwebewaage" [10, 11], manufactured by Sartorius over the years in small series. The coupling of this balance is shown in Fig. 12, in a design which is adapted to modern magnetic materials. A load capacity of 30 g and a useful sensitivity of 1 sc div per 10 μg were obtained. The precision of altitude detection could be improved by a phase-locked loop, linking the control voltage of a VCO to the inductance of the sensor coil.

Superimposed current control in the suspension balance

A very early improvement consisted of superimposed current control, which brings the exciting current of the upper magnet to zero by integral control, with the distance as correcting element. This arrangement not only increases the load capacity, but also reduces the generation of heat in the control winding and establishes a reliable and useful relationship between the weight of the sample and the distance of the magnetic poles. Moreover, it eliminates any reaction forces between control winding and magnets, and thus the winding can be rigidly attached to the supporting structure of the apparatus.

A beamless suspension balance

With a position control loop which includes frequency modulation and a phase-locked loop, the frequency generated by the VCO is reliably linked with high precision to the distance of the magnetic poles. According to the law of attraction between the poles, it also depends on the total weight of the suspended parts. The latter relationship is linear in a rather wide load range, as can be seen in Fig. 13 [12]. The specific resistance of the aluminium, as well as the flux of the lower magnet, depend on temperature. A correction is therefore necessary. If we provide a close thermal coupling between aluminium disc and magnet, the influence of temperature can be eliminated either by shunting the magnet with thermoflux or by digital correction. This suspension balance is simple in design, sturdy and accurate enough for thermoanalytical purposes.

Magnetostatic sensors in the suspension balance

The photoelectric, inductive and capacitive position sensors considered so far imply restrictions with regard to the material and thickness of the walls of the container and, therefore, confine the internal pressure. If reactions under very high pressures are to be investigated, the magnetostatic principle should be taken into account; this has been tested with success by the author with Luce [13] and with Pahlke [14]. According to Fig. 14, two small

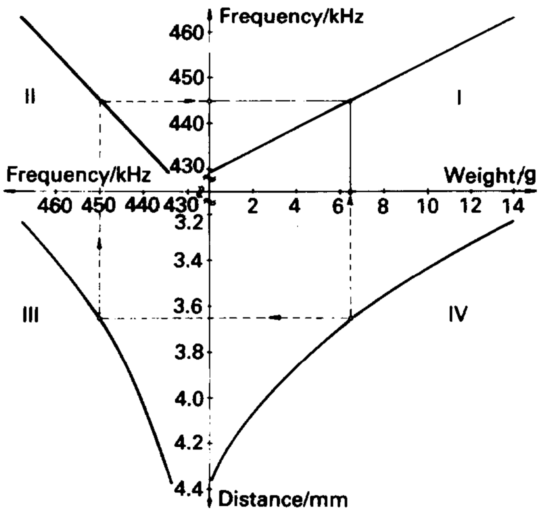

Fig. 13. Relations between weight, distance and frequency in a suspension balance.

bar magnets are attached to the shaft, which connects the suspended bar magnet to the balance pan. They generate a magnetic field of cylindrical symmetry with a horizontal plane of zero radial field strength. This plane travels along the Hall sensors, which are arranged opposite to one another

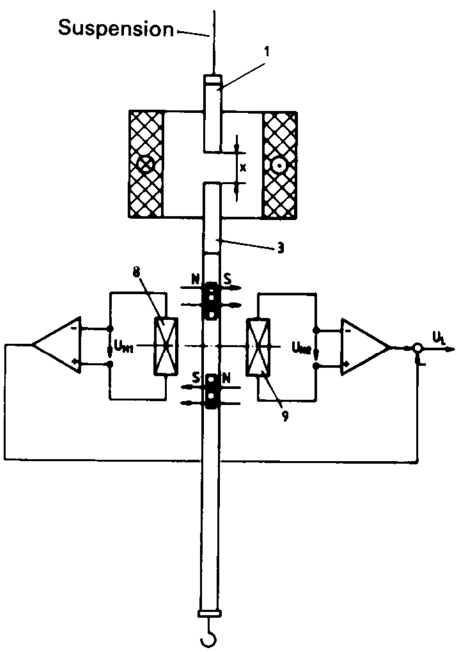

Fig. 14. Magnetic coupling with magnetostatic position sensors: 1, upper magnet; 3, lower magnet; 8, 9, Hall probes.

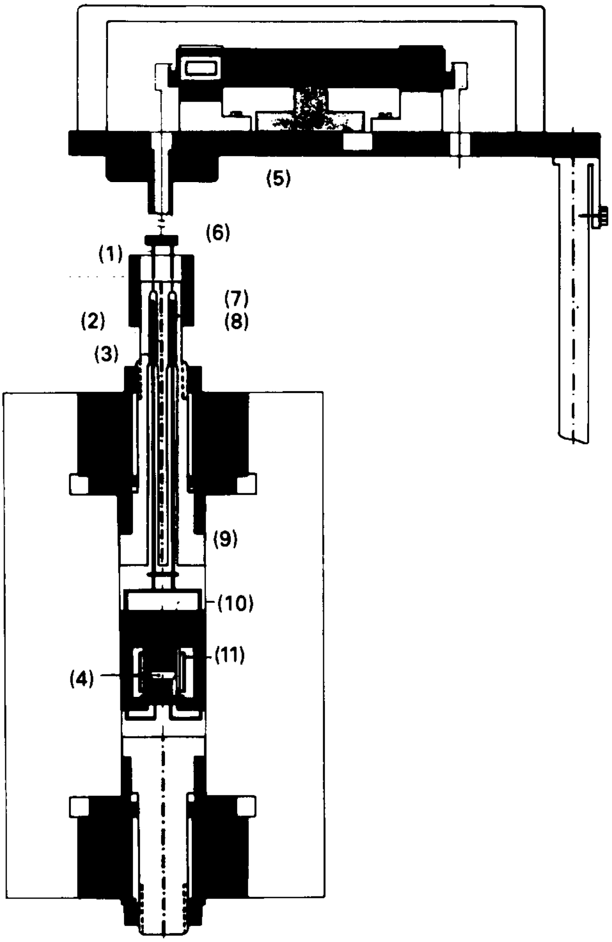

Fig. 15. High pressure suspension balance: 1, upper magnets; 2, non-magnetic shell; 3, floating suspension; 4, sample; 5, microbalance; 6, magnetic coupling; 7, control winding; 8, suspended magnets; 9, autoclave; 10, furnace; 11, reaction chamber.

near the wall of the container. The sum of the Hall voltages is proportional to the deviation of the lower magnet from a chosen position which can be adjusted by shifting the Hall sensors parallel to the axis of the suspended magnet. With the aid of a PD-controller, this magnet is maintained in stable equilibrium. A superimposed control loop keeps the mean value of the control current at zero. The control winding is fixed to the frame of the instrument. Thus far, the suspended magnet can freely rotate around its axis. This degree of freedom has to be suppressed, if a bell-shaped oven is utilized to heat the sample on the balance pan or in a crucible inside an autoclave. In this case, the suspension of the balance pan or crucible must penetrate the walls of the oven through narrow vertical bores which rules out rotations by more than 1 angular degree. Figure 15 shows the answer to this problem. Two pairs of bar magnets are used, which, in addition to the attraction force necessary for the suspension, also produce the restoring torque needed for azimuthal stability. The figure shows the microbalance with an articulated but non-rotable connection to the upper pair of magnets, and to the lower pair of magnets inside the bores of the upper Bridgeman seal. The lower Bridgeman seal permits access to the oven and removal or insertion of the balance pan with the connection to the lower pair of magnets. An accuracy of 1 μg of weight transfer through the described coupling in the pan has been demonstrated. Thus, the importance of feedback for precise weighing under severe environmental conditions has been confirmed.

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