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Note

A SIMPLE BUT EFFECTIVE METHOD OF REDUCING NOISE IN MICROTHERMOBALANCES IN FLOWING GASES UP TO 1700 K

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The unacceptable levels of noise and shift in zero point are two important limitations in adopting commercial microbalances to high temperature thermogravimetric investigations in flowing gaseous atmospheres at or near atmosphere pressure. In a previous publication [1], it was shown that the zero point shift could be reduced to an acceptable value if a symmetric, twin hangdown tube arrangement was adopted. As the high level of noise is due to thermal convection in the hot zone arising from temperature gradients near the sample, it was thought that any means of effective reduction of temperature gradients in the vicinity of the sample should reduce the noise. Cahn and Peterson [2] achieved this for air by reducing the diameter of the hangdown tube, while Cox et al. [3] have obtained low noise by incorporating special types of baffles. But their design of baffles is not practical for higher temperature ($T > 900$ K) applications. A combination of simple baffles and appropriate size for the hangdown tubes was found to reduce the noise of our microthermobalance in flowing gases up to 1700 K. The results are presented here.

RESULTS

Details of the microthermobalance are similar to those described in an earlier publication [1], except for the hangdown tube assembly and the twin furnace. As in the previous case, two identical hangdown assemblies including a twin furnace (Pt—20 Rh wire wound) were built around the Sartorius microbalance (model 4102). A schematic diagram of a hangdown assembly is shown in Fig. 1. Two recrystallized alumina tubes of suitable size, closed at one end, were positioned as shown in Fig. 1c, d such that an effective tube diameter of about 19 mm inner diameter around the sample crucible was provided. A set of three 1 mm thick, 16 mm diameter Pt—20 Rh discs were used as the baffle system (Fig. 1g). The flow of gases (viz., He, N_2 and CO_2) through the balance system was regulated with the help of a capillary gas flow meter arrangement [1] (0.5—10 $dm^3\ h^{-1}$). For a given geometry of the hangdown tube assembly, the temperature distribution in the sample region will be greatly influenced by the thermal diffusivity of the gas. If the noise is

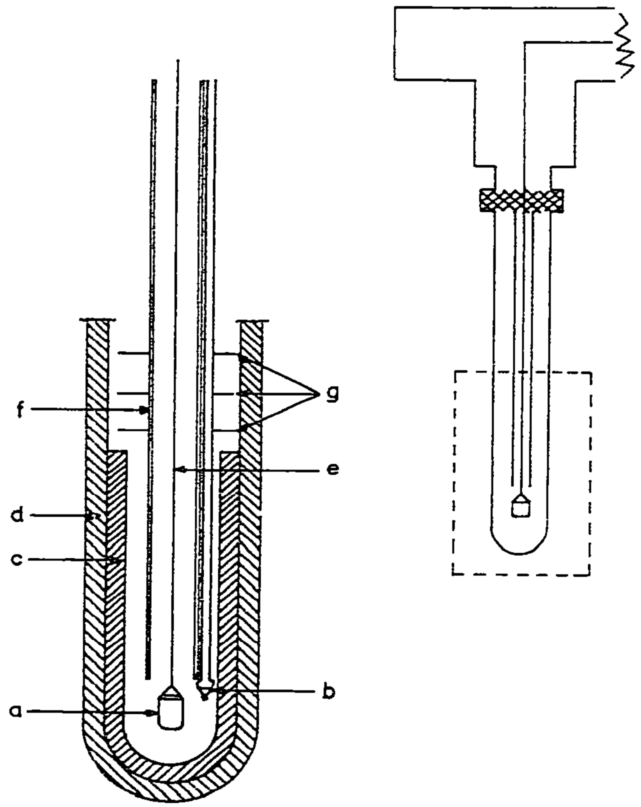

Fig. 1. A hangdown tube assembly. (a) Platinum sample container, (b) thermocouple, (c) inner alumina tube, 19 mm inner diameter; (d) outer alumina tube, 25 mm inner diameter, (e) Pt—Rh alloy suspension wire, 0.1 mm diameter; (f) central alumina tube, 6 mm inner diameter; (g) Pt—Rh alloy discs baffle-system.

the result of thermal convective movement of the gas in this region, then the noise for a given geometry should increase with the decrease in the thermal diffusivity of the gas. As shown in Table 1, carbon dioxide has the least thermal diffusivity among the three gases used in the present experiments

TABLE 1

The effect of type of gas and the configuration of the hangdown assembly on peak-to-peak noise

Gas	Thermal diffusivity (10^{-4} m^2 s^{-1}) (298 K)	Peak-to-peak noise (μg) *			
		I	II	III	IV
CO_2	7 7	150	60	30	4
N_2	19.7	60	25	~1	~1
He	158.0	~1	~1	~1	~1

* I, Without baffles and inner tube.
II, Without baffles, with inner tube.
III, With baffles, without inner tube.
IV, With baffles and inner tube.

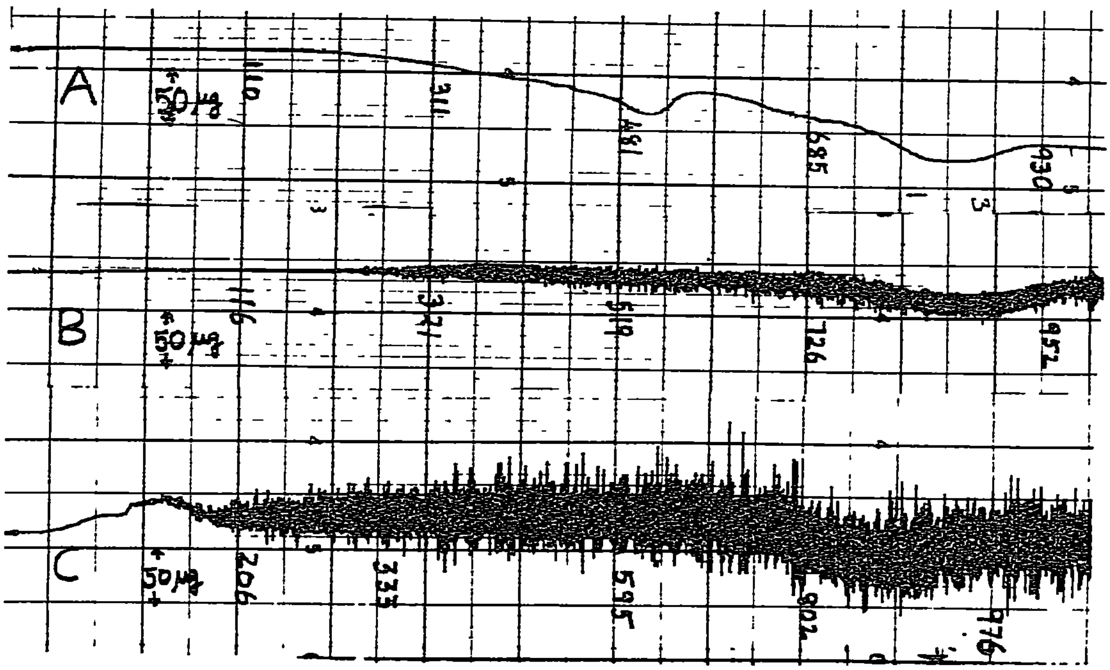

Fig. 2. Peak-to-peak noise (in μg) vs. temperature. (A) Helium; (B) nitrogen; (C) carbon dioxide. Without baffle system but with inner alumina tube in position. Gas flow rate, 5 dm^3 h^{-1}; heating rate, 10 K min^{-1}.

and the noise observed in carbon dioxide was also the highest (Fig. 2C).

A number of experiments were then carried out in flowing carbon dioxide to determine the relative effectiveness of the reduction of the diameter of the hangdown tube and/or the baffles. The results at a heating rate of 10 K min^{-1} and a gas flow rate of 5 dm^3 h^{-1} are presented in Fig. 3. It can be seen that the reduction of the hangdown tube diameter from 25 mm to 19 mm by introducing an inner tube (Fig. 3C), as well as the placement of the baffle assembly (Fig. 3B), reduces the noise significantly. In the present arrangement, baffles appear to reduce the noise relatively more than the reduction in the tube diameter alone. When both were incorporated (Fig. 3A) the peak-to-peak noise was reduced from about 200 μg to less than 5 μg (Fig. 3D). Thus with the hangdown tube arrangement shown in Fig. 1 we could operate the microthermobalance in flowing air, nitrogen, carbon dioxide or helium up to 1700 K, at a heating rate of 10 K min^{-1}, with a maximum noise less than 5 μg (usually about $\pm$1 μg). In agreement with Cahn and Peterson [2], the gas flow rates (0.5—10 dm^3 h^{-1}) had insignificant effect on the noise.

Cahn and Peterson [2] have concluded that the noise could be reduced in flowing air if the inner diameter of the hangdown tube is made 16 mm or less. Cox et al. [3] have recently shown that the noise could be reduced by suitable baffle design, even in carbon dioxide atmosphere. Both studies were limited in temperature range ($T <$ 1200 K). In the present study it is shown that a combination of hangdown tube of optimum diameter and a simple baffle system reduces the noise to 1 μg level even at 1700 K. Although the

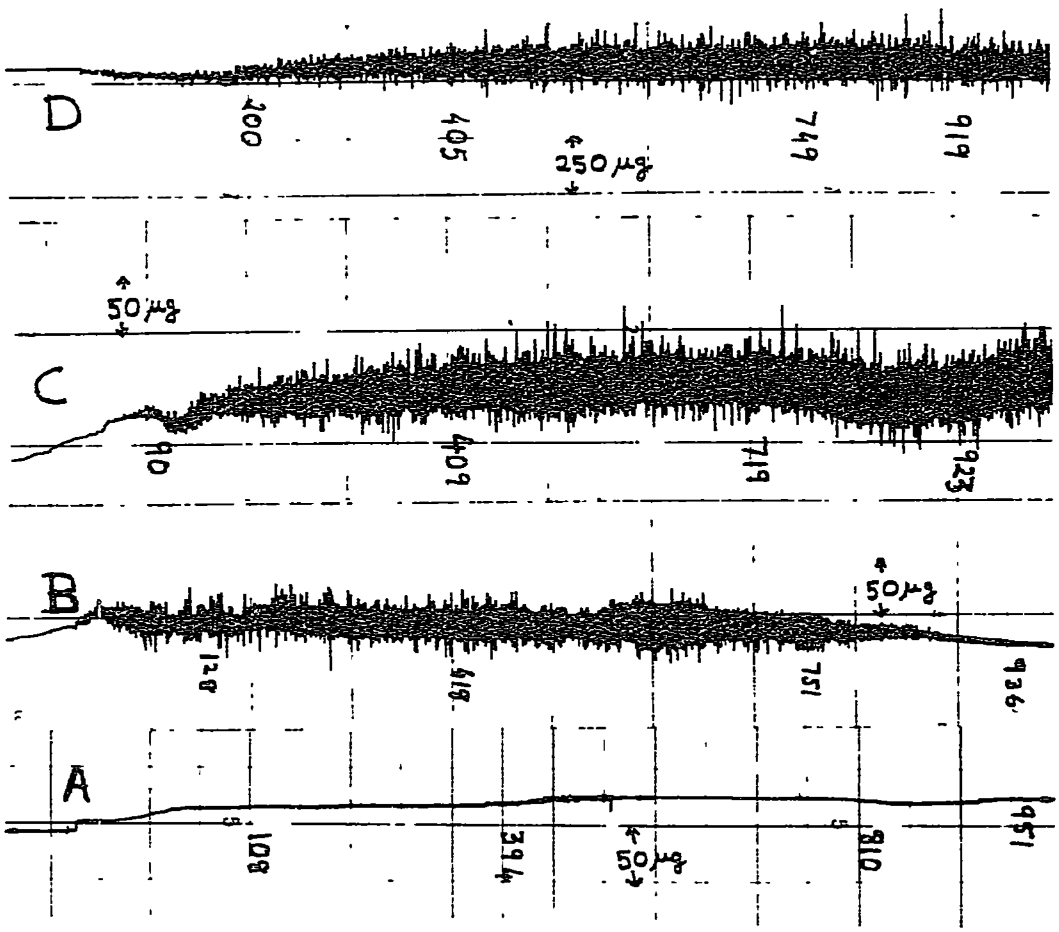

Fig. 3. Peak-to-peak noise (in μg) vs. temperatures in flowing carbon dioxide. (A) With the baffle system and the inner alumina tube in position; (B) without the inner alumina tube, only baffles, (C) only inner tube in position; (D) without the baffles as well as the inner tube in place. Gas flow rate, 5 $dm^3\ h^{-1}$; heating rate, 10 K min^{-1}.

noise level followed the thermal diffusivity of the flowing gas (Table 1), no quantitative relationship could be derived.

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