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Investigation of the Basic Performance of Analytical Balances

Proposal of a microbalance performance evaluation
method that includes the installation environment

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Proposal of a microbalance performance evaluation method that includes the installation environment

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Abstract

Operators of analytical balances sometimes question the accuracy of weighing data depending on the ambient conditions of the location where the balance is installed. In recent years, it has become necessary to provide evidence of weighing performance due to regulations such as fine particle standards (PM2.5) and those of the USP defining minimum sample weights. We propose a new method to evaluate weighing performance in the various ambient conditions where the microbalances are used and also report on the results of this method.

Keyword

Micro-gram, weighing environment, minimum weighing

1. Introduction

Electromagnetic equilibrium digital balances were introduced to the market over 40 years ago. Analytical balances with typical specifications (capacity: 200 g, minimum display: 0.1 mg) are used in many weighing applications. Semi-microbalances (capacity: several hundred g, minimum display: 0.01 mg), microbalances (minimum display: 0.001 mg (1 μg)), and ultra-microbalances (minimum display: 0.0001 mg) are now also available. The resolution (capacity divided by minimum display) of analytical balances continues to improve and now exceeds 10 million to 1. One μg is a minute mass (1 millionth of a one yen coin), and it is clear that the weighing precision is affected by environmental disturbances. This includes not only temperature and humidity changes but also gravitational acceleration, atmospheric pressure, and vibration. However, even when the causes of disturbances to the weighing environment were clear, determining the actual effects of these disturbances on weighing performance was difficult and considered virtually impossible in industry because the many disturbance parameters change over time, making them difficult to quantify.[1]

Due to these issues, it was impossible to determine whether unstable weighing values are due to a problem with the analytical balance itself or environmental disturbances. Obviously, if the cause cannot be determined, it is impossible to come up with a plan to deal with the problem. This leaves the user of the balance feeling unsure about weighing and its results, and problem remains unresolved. With this situation in mind, we proposed a measurement environment evaluation tool that can help solve the abovementioned problems in the locations where analytical balances are used. Using this tool, we successfully revealed true weighing performance in the actual environments where balances are used. We also made it possible to determine weighing performance from a series of evaluation procedures and to propose improvements for the weighing environment with the ultimate goal of improving weighing performance.

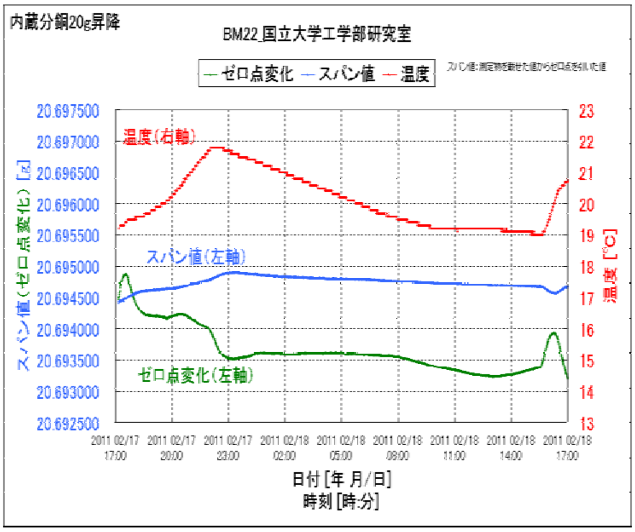
2. The new Measurement Environment Evaluation Tool (AND-MEET)

We propose AND-MEET[2] as a procedure to evaluate the setup environment of weighing instruments such as digital balances. In AND-MEET about 3,000 weighing data points are recorded as an internal weight in the weighing instrument is loaded and unloaded repeatedly over a long period of time. The zero point of when the internal weight was unloaded is subtracted from the acquired internal weight value to calculate the span value over a day. The standard deviation of 10 adjacent span values acquired chronologically is calculated as the repeatability. When the data is graphed with the repeatability value on the vertical axis and the time on the horizontal axis, the weighing performance (repeatability) over 24 hours can be confirmed, for example. In addition to weighing data, temperature, humidity, and atmospheric pressure data is recorded by embedded sensors. When a graph is created with time as the horizontal axis, interaction between the abovementioned repeatability and environment changes can be evaluated.

3. The actual results of AND-MEET

3-1. An environment with temperature changes

Fig. 1 shows actual results from AND-MEET at a university. This graph shows data for an analytical balance with a minimum display of 1 μg, with the zero point and span value (internal weight value minus the zero point) shown on the left vertical axis. Note that the internal weight of the balance is about 20 g and the span value indicates this value. The horizontal axis is the time axis and represents a 24-hour period from 5 PM on February 17 to 5 PM on February 18 of this year. The temperature change is shown on the right vertical axis and shows that there was a change of about 3 °C between 19 and 23 °C over the day. Furthermore, one scale interval for the zero point and span value (the internal weight value minus the zero point) is 500 μg. The recording of data for the graph started immediately after the digital balance was set up and powered on. The graph shows that the temperature rose 3 °C in 5 hours after the power was switched on. In this period, range of up/down variation of the zero point was about 1.5 mg and the span value changed +0.5 mg. The temperature slowly dropped −3 °C over the next 12 hours in a linear fashion. Accompanying this change, the zero point variation dropped and the span value also slowly dropped −300 μg. It was determined that during the six hour period between 10 AM and 4 PM on February 18 when the temperature variation was the smallest, the zero point dropped by −300 μg but the span value variation was as small as about −100 μg



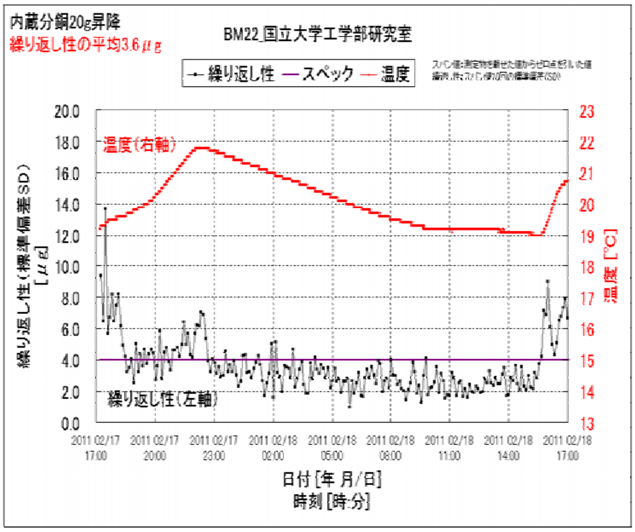


Fig. 1. Changes in temperature, zero point, and span value over 24 hours

Fig. 2. Changes in span value repeatability and temperature

Fig. 2 shows repeatability (reproducibility: σn-1) of 10 adjacent span values calculated from the data of the graph in Fig. 1. The repeatability data for every 10 points is presented as a single point, and a line connects the adjacent repeatability points. Repeatability immediately after setup is poor at about 10 µg/20 g. When the temperature was rising, the value was 4 to 6 µg, which is a relatively poor value. Thereafter, the temperature dropped comparatively slowly and the repeatability was good at 2 to 4 µg. The temperature was most stable from 10 AM to until just after 3 PM on Sunday, February 18, with the repeatability at 2 to 3.5 µg. This proves that the performance achieved sufficiently satisfies the product catalog specifications of σ=4 µg/1g.

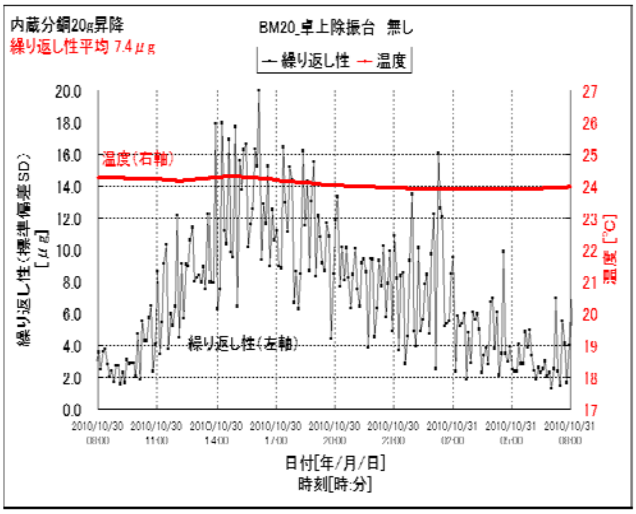
The balance was set up in a room in the engineering department of a national university in Tokyo that conducts material research related to energy. When actually visiting the site, we learned that there is a large high-temperature treating furnace in the research room that operates each day from 5 PM to 10 PM. This was why that room temperature rose every evening.

Of course, we could never recommend setting up a micro-balance in a room with an operating heat treating furnace, but we explained the AND-MEET results and convinced the professors that the micro-balance performed up to standards during weighing in the afternoon when the furnace was off.

3-2. When the building vibrates (with or without an anti-vibration table)

We will now describe the effects of a typhoon on repeatability. Fig. 3 and 4 are graphs showing temperature changes and span value repeatability when a typhoon occurred. This data was acquired on October 30, 2010. The rough weather that accompanied Typhoon 14 (975 hPa) continued until dawn the next day. While the temperature change was slight (0.5 °C or less), Fig. 3 shows that repeatability, which started at 3 µg, gradually worsened and reached an average of 14 µg. After this, it gradually improved. Data from another microbalance recording at the same time nearby shows that repeatability was stable at about 3 µg excluding a few time periods over several hours.

One balance was on an anti-vibration table and the other was not. Fig. 4 shows the data from the balance on an anti-vibration table.



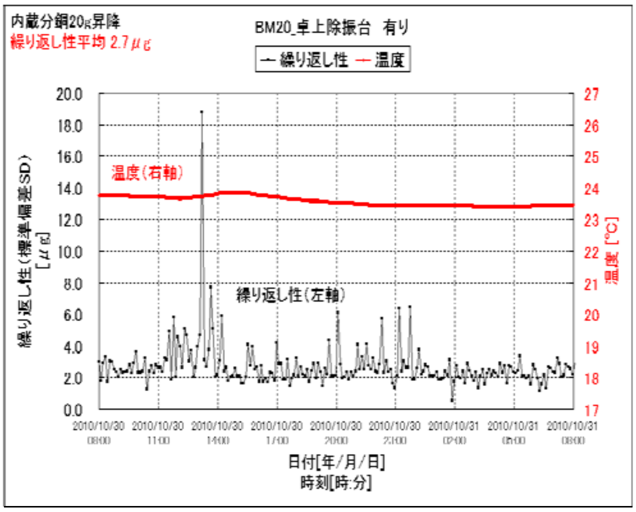


Fig. 3. Passing typhoon, no anti-vibration table

Fig. 4. Passing typhoon, anti-vibration table used

While the following is speculation, we believe that the typhoon caused a gradual variation in atmospheric pressure so the repeatability of the span value was not affected, even though zero drift was. However, the pressure changes from the typhoon were accompanied by winds that shook the building. The people in the room did not feel the shaking of the building but the shaking affected the highly sensitive microbalance greatly. The results of Fig. 3 and 4 show that the anti-vibration table protected the analytical balance from the minute vibrations of the building, which resulted in more stable weighing values.

3-3. The effect of air blown from an air conditioner

Most research rooms where analytical balances are set up use air conditioning to control the room temperature. While this controls the temperature, this does not always produce positive results for balances. If you use air conditioning to proactively stabilize the temperature and look at the results carefully, you can see that in order to maintain a constant temperature the air conditioner constantly causes minute changes in temperature. The analytical balance is sensitive enough to track these changes and this produces small fluctuations in the weighing values.

Fig. 5 and 6 are graphs of data from microbalances set up in a room with an air conditioner. The output from the temperature sensor inside the balance showed rippling of about 0. 1 °C and the zero point changed along with this. The results here show that while the temperature change for the day was small at 0.5 °C, the repeatability was about 5 µg and varied greatly. Conversely, when a balance was covered with a table-top breeze break and the open space of the room was narrowed to prevent blown air from the air conditioner from directly hitting the balance (Fig 7 and 8), even though the temperature change was large at 2 °C over a day, there were no ripples in the temperature and the accompanying zero point changes, with the repeatability falling to an average of 4 µg or less. In addition, it was determined that the poor repeatability at 6 AM on July 13 seen in Fig.6 was due to an earthquake (off the shore of Fukushima at 5:47 PM, Magnitude 5.3) over 200 km from where the weighing was performed in Saitama Prefecture.

This data is a good example by which we were able to understand the effects of blown air from an air conditioner on weighing values. Furthermore, it was determined that to further improve the repeatability value of 4 μg acquired for Fig. 8, it is necessary to strengthen measures to block breezes.

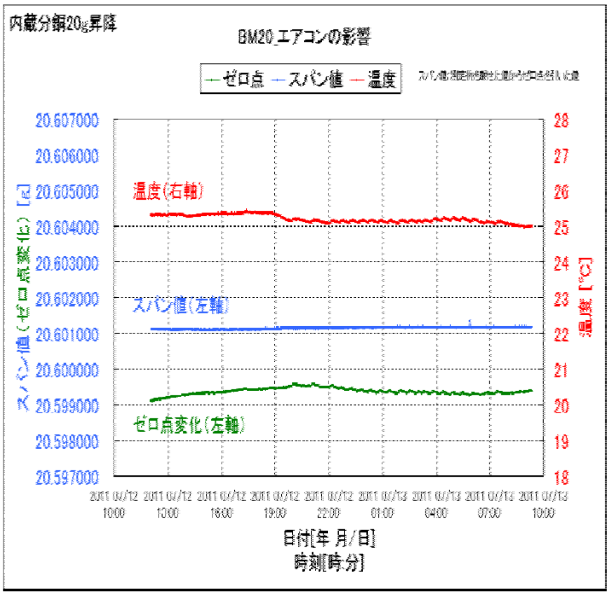


Fig. 5 Before countermeasures for air conditioner

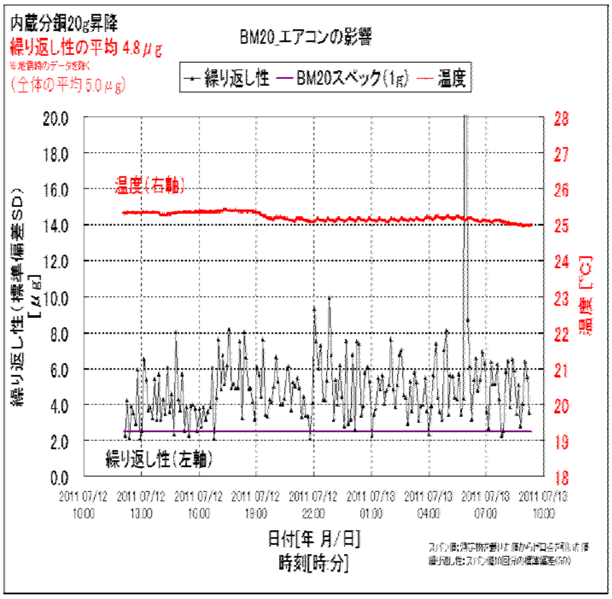


Fig. 6 Before countermeasures for air conditioner

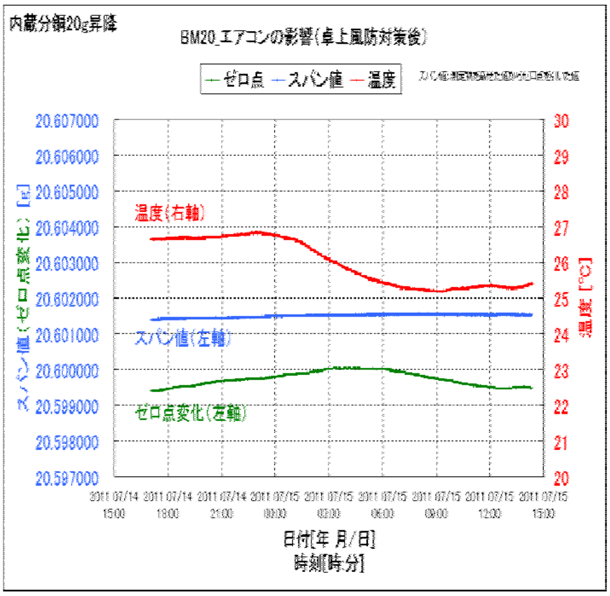


Fig. 7 After countermeasures for air conditioner

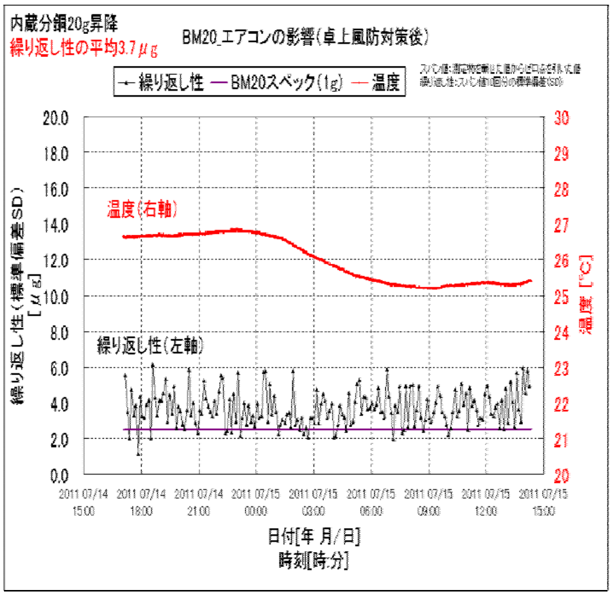


Fig. 8 After countermeasures for air conditioner

3-4. The effects of humidity changes

Fig. 9 and 10 show the data when investigating the introduction of a micro-balance as a mass comparator at a location where the business of calibrating weights is planned.

The balance was set up in a room covered in thick concrete. After the balance was set up, AND-MEET was started along with the air conditioner. The temperature change was a mere 1 °C, and the data had a very good span value repeatability of 1.8 µg. However, the zero point variation was high at +5 mg/day, which caused the user some uneasiness. This zero point drift was due to a sudden humidity change caused by switching on the air conditioner, which can be seen in Fig. 10. The humidity changes occurred because it was thought that it would be better to switch on the air conditioner when AND-MEET started. As the air conditioner continued to run, the zero point drift abated and ideal repeatability data was also obtained.

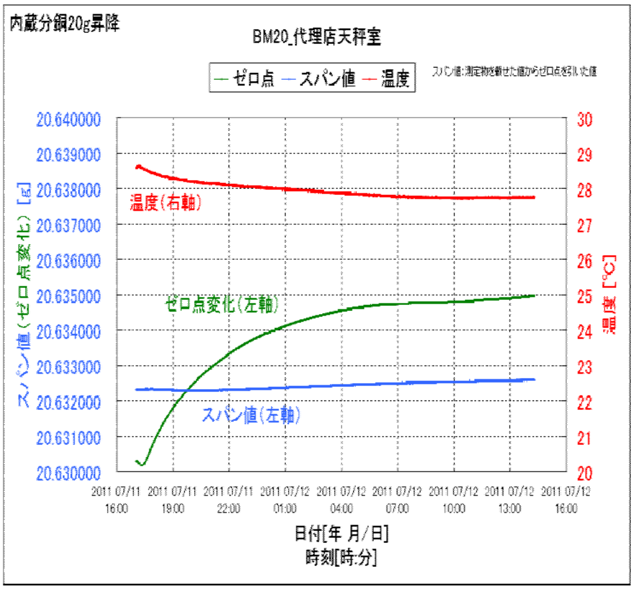


Fig.9 Effect of humidity

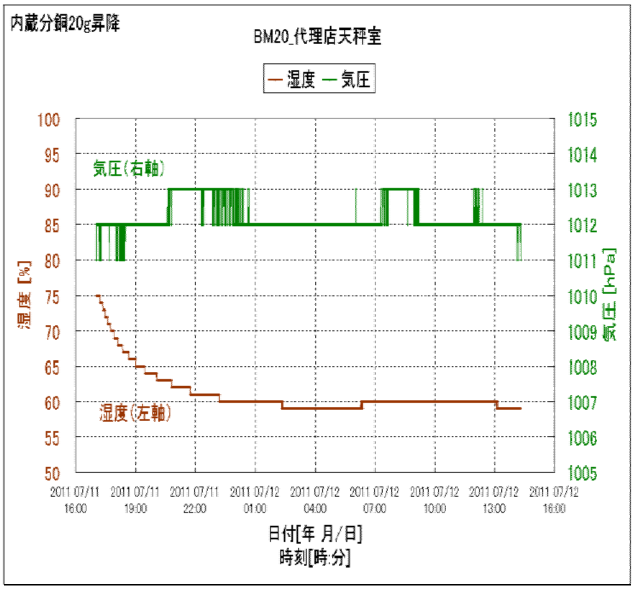


Fig. 10 Effect of humidity

4. Discussion

○ The environmental causes affecting the repeatability of microbalances

The results of AND-MEET clearly indicated the causes of weighing value errors affecting the performance of micro-balances. All the data cannot be presented here due to limitations of space, but concrete examples of the effects of earthquakes, passing low pressure systems, temperature ripples and wind and humidity changes from air conditioner use, the coming and going of people (vibrations, pressure variations), and heat sources near the balance have been presented. Using AND-MEET clearly makes it possible to determine these influences quantitatively and understand them.

○ Determination of the minimum sample weight

Clinical testing and contracted research in the pharmaceutical and food product fields often involve work that requires validation of weighing. In the specimen sampling stage at the start of analytical work, analytical balance repeatability and the minimum sample weight[3], which is calculated from the repeatability, must be determined. To determine the minimum sample weight, it is necessary to confirm the extent to which the balance is influenced by disturbances in the setup environment. AND-MEET is an effective means to verify these influences. Analyzing the AND-MEET results makes it possible to improve the weighing environment and prove the minimum sample weight that can be achieved in that environment.

○ The expanding needs of environmental weighing (PM2.5, etc.)

The problem of lung cancer deaths due to air pollution is increasing. The Manual for Continuous Monitoring of Air Pollution announced by the Ministry of Environment in February of last year describes the measurement of minute particulate matter (PM2.5)[4]. The manual states that a special filter is used to trap particulates, which are then measured at the µg level. The measurement environment must have a temperature of 21.5 ± 1.5 °C and a relative humidity of 35 ± 5%, so a constant-temperature room is required. Such requirements mean that weighing must be done with the micro-balance set up in an environment with a strong air circulation. We have confirmed that AND-MEET is an effective way to evaluate the effects of the blown air. By assessing and evaluating the setup environment of a micro-balance using the AND-MEET results, it has become possible to improve the setup environment and meet the PM2.5 specifications required for the micro-balance.

5. Summary

We believe that a manufacturer that simply makes micro-balances and other special instruments under a certain environment is not fulfilling its market responsibilities. Instruments are strongly influenced by their setup environment and manufacturers of weighing instruments must take proactive measures to deal with this issue. Consequently, A&D offered its measurement environment evaluation tool (AND-MEET) to users, advancing methods to acquire weighing data using automated machines in production lines utilizing balances. By using AND-MEET, users can determine the basic performance of analytical balances in their setup location and infer and investigate the causes for repeatability issues. With AND-MEET, users can also ultimately develop plans to improve weighing environments.

In this way, AND-MEET can be used to assuage the worries of researchers unsure about introducing more sensitive weighing instruments. At the same time, it can be used to investigate the basic performance of weighing instrument products while considering the setup environment.

As with other industrial product markets, developing countries are assuming greater prominence in the weighing market, and the "Made in Japan" brand is reeling from the effects. Nevertheless, Japan still maintains prominence in the markets it has long dominated: "small and light" markets, such as new materials and electronic components, and instrument markets that require greater precision and higher quality. This is evidenced by the wide use of production equipment made in Japan for µg-level weighing on production lines.

Our newly proposed AND-MEET is a fundamental tool to support these still growing industries going forward. We will work to ensure that it is recognized as a way to support locations using analytical balances in various fields ranging from research to production and contributes to further advancements in these fields.

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