INCREASE OF CONSTANT WEIGHT RATES OF WEIGHING BOTTLES AND EVAPORATING DISHES IN A PHARMACOLOGICAL EXPERIMENT

by

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Improving the constant weight rates of weighing bottles and evaporation dishes is beneficial to drug quality control. When different quantities of weighing bottles were dried under the same conditions, the greater the quantity is, the lower the constant weight rate will be. When quantities are controlled to be the same, weighing bottles that have been cooled for 50 minutes have higher constant weight rates than those cooled for half an hour. Without being affected by other factors, weighing bottles' constant weight rates are higher when the bottles are dried at the temperature of 180 °C than 105 °C. The rate increases when the bottled are dried for 8 hours instead of 5 hours. After soaking for 48 hours in the solution of 2% HNO_3 + 6% HCl, the weighing bottles and evaporating dishes gain higher constant weight rates. Being more efficient, faster, economical and practical, this experiment has improved the method to increase constant weight rates of weighing bottles and evaporating dishes, without the need of auxiliary equipment, and thus increased the accuracy of drug weighing results.

Key words*: drug quality, weighing bottles and evaporating dishes, drying time, drying temperature, constant weight rate*

Introduction

Drug quality control is done through meticulous inspection of drug quality during the process. It should be strictly implemented in accordance with the standard operation provisions. The experimental equipment shall also meet the criteria of related provisions [1-3]. Weighing bottles and evaporating dishes are commonly used equipment in lab. They are used to measure water content and weight loss after drying. They are also used to hold extract and

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non-volatile matters, especially when the small and high-precision data are required in the test. Thus, the weight changes of weighing bottles and evaporating dishes before and after heating are very important [4-6]. The control of water content in a drug will affect both of its quality and stability. According to related regulations, weighing bottles and evaporating dishes shall be dried until they reach constant weights during operation. However, such method will is not only time-consuming but may also affect the experimental results. Therefore, new alternatives need to be found to improve the constant weight rates of weighing bottles and evaporating dishes [7-9]. During drug production, water content is an important factor in drug quality test. The level of water content will directly affect a drug's quality and expiration date. Therefore, strict control and water content test must be done during the production and quality control of drugs [10]. The conventional method to detect water content sets the reference temperature at 105 °C ±3 °C. A sample test needs to take 2- 3 hours, or even 3-4 hours to finish. During the process, every half an hour, the sample will be moved from the oven to the precision scale [11]. This operation will have to repeat until the last two results of the sample's weights no longer change. The constant result means that the sample is completely dried. By reading results from precision scales and calculating artificially, we can conclude the solid content of the sample. Though oven method can accurately determine the solid content of a sample, it is not efficient [12, 13] enough to adapt to the fast-paced commercial production. By investigating the influencing factors, this experiment aims to improve the method for better constant weighting rate of the weighing bottles and evaporating dishes [14, 15].

Materials and methods

Equipment for measuring the constant weight of the weighing bottle

Low-form glass weighing bottle (40 mm × 25 mm), desiccator, silica gel, blast drying oven, electronic analytical balance (1/10000), multi-channel temperature tester.

Equipment for measuring the constant weight of evaporating dish

Ceramic evaporating dish, desiccator, silica gel, blast drying oven, electronic analytical balance (1/10000), multi-channel temperature tester.

Verification of the blast drying oven

Control accuracy of constant temperature

The constant temperature is set at 105 °C for control accuracy (or at other temperatures ranging from 50 °C to 250 °C). Thermometers that are within 1° of accuracy (calibrated) or other instruments (calibrated) are put into the oven, placed on the partition board vertically below the thermoscope. When the temperature of the drying oven rises to 105 °C, we should wait for another 20 minutes, before measuring the temperature of corresponding positions on each partition. The temperature of each partition should be within 105 °C ±2°C.

Temperature uniformity test between each layer of partition

As with the constant temperature control accuracy test, the difference between the maximum and minimum temperatures measured by the baffle plate should be within ±2 °C.

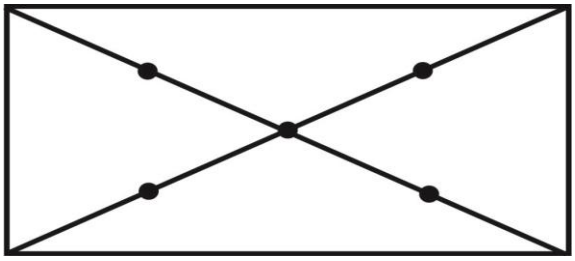

Figure 1. The four points for temperature measurement on each partition to test temperature accuracy

Test the temperature uniformity between different positions of single-layer separator

As with the constant temperature control accuracy test, measurements were made at the four positions as shown in fig. 1, for each layer of baffle, and the difference between the maximum and minimum measured temperature of each layer of baffle should be within ±1 °C.

Temperature stability test

A multi-channel thermometer (calibrated) or other thermometers (calibrated) was used in the test. The instrument's division value was less than 1 °C. It could be put anywhere inside the oven with the measuring temperature set (50-250 °C). When the temperature in the oven reached the measuring temperature, we waited for another 2 hours, before reading the results for every 4 minutes. The results were read for 6 times in total within 20 minutes. The average value was taken as the reference temperature.

The temperature was then measured every two hours for five times. The difference between the temperature and the reference temperature must meet the following criteria: If the set temperature is less than 200 °C, the difference should be no greater than 2 °C. If the set temperature exceeds 200 °C, the difference should be no more than 1% of the set temperature.

According to tab. 1, four experimental indicators were used to test the temperatures, and they are: the temperature accuracy, temperature uniformity among partitions, temperature uniformity at different points of one partition, and temperature stability. The standard reference temperature was set at 105 °C. The results of temperature accuracy show that: the temperature difference of Partition 1 is 0.3 °C, that of Partition 2 is 0 °C, which are in line with the ±2 °C difference of the criteria. As for the temperature uniformity among partitions, five tests were made to measure the temperature on the partition. The maximum temperature difference is 0.6 °C, and the minimum temperature difference is –0.7 °C. The differences between the maximum and minimum temperatures are 1.3 °C. The results also conform the experimental criteria of ±2 °C. In terms of temperature uniformity at different points of one partition, five tests were conducted on the Partitions 1 and 2, respectively. The maximum temperature difference is 0.4 °C, and the minimum temperature difference is –0.4 °C. The differences between the maximum and minimum temperatures are 0.5 °C and 0.6 °C, respectively. The results meet the experimental criteria of ±1 °C. With regard to the temperature stability, Partitions 1 and 2 were tested five times for each. The maximum temperature difference was 0.3 °C and the minimum temperature difference was –0.1 °C, which were in line with the 1% criteria difference between the actual and the set values.

Verification of electronic analytical balance

Five different positions, fig. 2, were selected on the electronic analytical balance (Shanghai Hengping FA1104 Electronic Analytical Balance). A 100 g standard weight was weighed twice on each position of the balance. The results were shown in tab. 2.

According to the results, the maximum error is 0.0001, so the eccentric loading error of the balance is within the error range (±0.0005 g).

Table 1. Temperature difference under different indicators

Indicators	Set temperature [℃]			Actual temperature [℃]	Variation [℃]
Temperature accuracy	105		Partition 1	104.7	–0.3
			Partition 2	105.0	0
Temperature uniformity	Between each partition	105	Partition 1	104.7	–0.3
				105.1	0.1
				105.6 (max.)	0.6
				105.4	0.4
				104.3 (min.)	–0.7
	Single layer partition between different positions	105	Partition 1	104.8	–0.2
				104.6 (min.)	–0.4
				105.1 (max.)	0.1
				105.0	0.0
				104.7	–0.3
			Partition 2	105.1	0.1
				105.0	0.0
				105.4 (max)	0.4
				104.8 (min)	–0.2
				105.2	0.2
Temperature stability	105			104.7	Average (105)
				104.9	
				105.0	
				105.1	
				105.0	
				105.1	
				105.2	0.2
				105.0	0.0
				105.3	0.3
				105.1	0.1
				104.9	–0.1

Weighing bottle

A weighing bottle is a cylindrical glass bottle with a grinding stopper. It is often used in the technique of weighing by difference, which involves repetitive weighing of a weighing bottle containing a quantity of solid reagent on an analytical balance. Weighing bottles are also used in cooking out samples (drying method to determine water content).

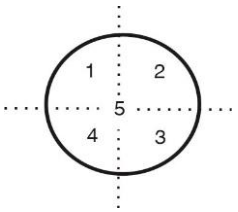

Figure 2. Schematic diagram of different positions on the Electronic Analytical Balance

Evaporating dish

Evaporating dish is a vessel used for evaporating concentrated solutions. It can be used for the evaporation, concentration and crystallization of liquid substances; (extract, non-volatile, total solid).

Constant weight

Unless otherwise specified, constant weight refers to the weight of the test article when it has gone through two consecutive drying or burning, with less than 0.3 mg of weight difference. The test article shall not be weighed for the second and subsequent times after being dried to the constant weight without additional drying for 1 hour under the specified

conditions. The test article shall not be weighed for the second time after being burned to a constant weight until it is burned for another 30 minutes.

Table 2. The weight values measured by the electronic analytical balance

Positions	1	2	3	4	5
Values [g]	100.0000	100.0000	100.0001	100.0000	100.0000
	100.0001	100.0000	100.0000	100.0000	100.0000

Results and discussion

The constant weight of the weighing bottles

The weighing bottles were cleaned and numbered, before being put into the blast drying oven (DHG-9070A, Shandong Olaibo Instrument Co., LTD.) for drying (set temperature: 105 °C; drying time: 5 hours). Then, the bottles were moved out of the oven to put the lids on. Next, they were put into a desiccator (desiccants were effective and the lid of the desiccator well-sealed with Vaseline) for cooling down to the room temperature (cooling time was changed based on the number of weighing bottles). The bottles were accurately weighed, until difference between the two results were narrowed to less than 0.0003 g.

Influencing factors of the test

Relationship between the number of weighing bottles and the cooling time.

Different numbers of weighing bottles in the desiccator require different cooling times to reach the room temperature. More weighing bottles will release more heat, thus requiring longer time to cool. Based on this reality, the bottles will not be weighted until they reach the room temperature. Following the aforementioned procedure in section *The constant weight of the weighing bottles*, we operated with 8, 12, and 22 weighing bottles, respectively, and waited for 30 minutes for them to cool down before measuring their weights. The results are shownin tabs. 3-5.

Table 3. Weight and difference of eight weighing bottles before and after drying at 105 °C for 5 hours

Serial number	First weight [g]	Second weight [g]	Variation [g]
1-1	17.3511	17.3509	0.0002
1-2	18.6431	18.6430	0.0001
1-3	18.3607	18.3606	0.0001
1-4	18.6031	18.6030	0.0001
1-5	17.8535	17.8533	0.0002
1-6	19.0940	19.0938	0.0002
1-7	17.2452	17.2452	0.0000
1-8	17.8170	17.8169	0.0001

According to tab. 3, after drying for 5 hours at 105 °C and cooling for 30 minutes, three weighing bottles out of the eight bottles had weight difference of 0.0002 g, the other five bottles had the difference of 0.0001 g. All variations were within the normal range (<0.0003 g).

After drying at 105 °C for five hours and cooling for 30 minutes, two out of the twelve weighing bottles had the weight difference of 0.0001 g and the ten bottles had the difference of 0.0000, which were less than that of the first group (eight weighing bottles), and both met the requirements (<0.0003 g).

Table 4. Weight and difference of twelve weighing bottles before and after drying at 105 °C for five hours

Serial number	First weight [g]	Second weight [g]	Variation [g]
2-1	17.7504	17.7504	0.0000
2-2	19.1976	19.1976	0.0000
2-3	18.0731	18.0730	0.0001
2-4	18.2472	18.2472	0.0000
2-5	17.6939	17.6939	0.0000
2-6	18.2584	18.2584	0.0000
2-7	17.7468	17.7468	0.0000
2-8	18.7515	18.7514	0.0001
2-9	18.3173	18.3173	0.0000
2-10	19.7500	19.7500	0.0000
2-11	18.4830	18.4830	0.0000
2-12	18.9283	18.9283	0.0000

Table 5. Weight and difference of 22 weighing bottles after drying at 105 °C for 5 hours and cooling for 30, 40, and 50 minutes, respectively

Serial number	Cooling for 30 minutes			Cooling for 40 minutes			Cooling for 50 minutes		
	First weight [g]	Second weight [g]	Weight variation [g]	First weight [g]	Second weight [g]	Weight variation [g]	First weight [g]	Second weight [g]	Weight variation [g]
3-1	18.5223	18.522	0.0003	18.5225	18.5224	0.0001	18.5224	18.5223	0.0001
3-2	17.7251	17.7246	0.0005	17.7255	17.7252	0.0003	17.7252	17.7251	0.0001
3-3	18.4021	18.4018	0.0003	18.7080	18.7077	0.0003	18.4022	18.4021	0.0001
3-4	18.4390	18.4386	0.0004	18.9519	18.9517	0.0002	18.4391	18.4388	0.0003
3-5	17.3256	17.3251	0.0005	18.4770	18.4768	0.0002	17.3257	17.3254	0.0003
3-6	19.4577	19.4574	0.0003	18.5232	18.5231	0.0001	19.4580	19.4577	0.0003
3-7	20.7077	20.7076	0.0001	18.0363	18.036	0.0003	20.7079	20.7076	0.0003
3-8	18.9517	18.9513	0.0004	17.6323	17.6319	0.0004	18.9518	18.9515	0.0003
3-9	18.4766	18.4764	0.0002	18.6545	18.6544	0.0001	18.4768	18.4765	0.0003
3-10	18.5228	18.5226	0.0002	17.6138	17.6135	0.0003	18.5230	18.5229	0.0001
3-11	18.0361	18.0357	0.0004	17.7440	17.7438	0.0002	18.0360	18.0359	0.0001
3-12	17.6322	17.6319	0.0003	18.9316	18.9314	0.0002	17.6321	17.632	0.0001
3-13	18.2320	18.2319	0.0001	17.4454	17.4451	0.0003	18.2320	18.2317	0.0003
3-14	18.6542	18.6541	0.0001	18.5106	18.5103	0.0003	18.6543	18.654	0.0003
3-15	17.6135	17.6133	0.0002	18.9836	18.9836	0.0000	17.6135	17.6132	0.0003
3-16	18.6720	18.6717	0.0003	18.2804	18.28	0.0004	18.6722	18.6721	0.0001
3-17	17.7437	17.7433	0.0004	18.4025	18.4022	0.0003	17.7435	17.7432	0.0003
3-18	18.9312	18.9309	0.0003	18.4393	18.4391	0.0002	17.4450	17.4448	0.0002
3-19	17.4450	17.4447	0.0003	18.3258	18.3257	0.0001	18.5106	18.5104	0.0002
3-20	18.5103	18.5099	0.0004	18.4581	18.458	0.0001	18.9831	18.9831	0.0000
3-21	18.9833	18.9829	0.0004	18.7080	18.7077	0.0003	18.2801	18.28	0.0001
3-22	18.2802	18.2799	0.0003	20.7078	20.7077	0.0001	17.9314	17.9314	0.0000

The results showed that after cooling for 30 minutes, the weight difference between the first and the second time was greater than 0.0003 g in eight cases, equal to 0.0003 g in eight cases, and less than 0.0003 g in six cases. After cooling for 40 minutes, the weight difference between the first time and the second time was greater than 0.0003 g in two cases, equal to 0.0003g in eight cases, and less than 0.0003 g in twelve cases. After cooling for 50 minutes, the weight difference between the first and the second time was equal to 0.0003 g in ten cases, and less than 0.0003 g in twelve cases.

The results showed that the experimental error was the smallest when 22 weighing bottles were dried at 105 ℃ for 5 hours and cooled down for 50 minutes.

Constant weight of evaporating dishes

Evaporating dishes were cleaned before being put in the blast drying oven for drying. After drying the dishes were taken out of the oven and put into a desiccator (desiccants were effective and the lid of the desiccator well-sealed with Vaseline) for cooling down to the room temperature. Cooling time was changed based on the number of weighing bottles. The bottles were accurately weighed, until difference between the two results was narrowed to less than 0.0003 g.

Influencing factors of the test

Drying temperature

According to the previous method, five evaporating dishes were weighed at 105 ° C and 180 ° C, respectively. The drying time was five hours and the cooling time was 30 minutes, tab. 6.

Table 6. Weight and difference of five evaporating dishes before and after drying at 105 °C and 180°C

Serial number	105 ℃			180 ℃		
	First weight [g]	Second weight [g]	Weight variation [g]	First weight [g]	Second weight [g]	Variation [g]
4-1	61.2553	61.2551	0.0002	61.2552	61.2549	0.0003
4-2	54.0126	54.0123	0.0003	54.0121	54.0119	0.0002
4-3	54.6649	54.6653	–0.0004	54.6650	54.6651	–0.0001
4-4	55.6831	55.6834	–0.0003	55.6831	55.6829	0.0002
4-5	58.4641	58.4636	0.0005	58.4640	58.4636	0.0004

The results showed that after the evaporating dishes were dried for five hours at 105 ℃ and then cooled for 30 minutes, two weight differences between the first and second time were greater than 0.0003 g. After the evaporating dishes were dried were dried for five hours at 180 ℃ and cooled for 30 minutes, one weight difference was greater than 0.0003 g.

Influence of drying time on constant weight

Five evaporating dishes were dried at 105 ° C for 5 and 8 hours, respectively, cooled for 30 minutes and weighed, tab. 7.

Table 7. Weight and difference of five evaporating dishes before and after drying at 105 °C for 5 and 8 hours, respectively

Serial number	5 hours			8 hours		
	First weight [g]	Second weight [g]	Weight variation [g]	First weight [g]	Second weight [g]	Variation [g]
5-1	61.2553	61.2551	0.0002	61.2550	61.2548	0.0002
5-2	54.0126	54.0123	0.0003	54.0126	54.0122	0.0004
5-3	54.6649	54.6653	–0.0004	54.6650	54.6649	0.0001
5-4	55.6831	55.6834	–0.0003	55.6828	55.6828	0.0000
5-5	58.4641	58.4636	0.0005	58.4635	58.4636	–0.0001

The results showed that at 105 °C, after the dishes were dried for five hours, two weight differences were greater than 0.0003 g; after the dishes were dried for eight hours, one weight difference was greater than 0.0003 g.

Before and after treatment with the 2% HNO_3+6% HCl solution

Five evaporating dishes were soaked with a solution of 2% HNO_3 + 6% HCl for 48 hours, then dried at 105 °C for five hours, cooled for 30 minutes, and weighed, tab. 8.

Table 8. Effects of the new method of treating with the 2% HNO_3 + 6% HCl solution

Serial number	Before processing			After processing		
	First weight [g]	Second weight [g]	Variation [g]	First weight [g]	Second weight [g]	Variation [g]
6-1	61.2553	61.2551	0.0002	61.2523	61.2521	0.0002
6-2	54.0126	54.0123	0.0003	54.0118	54.0116	0.0002
6-3	54.6649	54.6653	– 0.0004	54.6650	54.6647	0.0003
6-4	55.6831	55.6834	– 0.0003	55.6820	55.6820	0.0000
6-5	58.4641	58.4636	0.0005	58.4632	58.4631	0.0001

The results showed that two weight differences were greater than 0.0003 g and two were 0.0003 g before the five evaporating dishes were treated with the 2% HNO_3 + 6% HCl solution. After the treatment, there was only one weight difference between the first and second time, which was equal to 0.0003 g.

Conclusions

During the test, the weighing bottles are put into the oven twice following the same sequence and cooled down for same period of time. After the test, we can conclude that, the less number of weighing bottles, the higher the constant weight rate will be, under the condition of clean bottles, effective desiccants, and well-sealed desiccators. For the same number of weighing bottles (12, 22), the longer the cooling time is, the higher the constant weight rate will become.

After the test of evaporating dishes, we can conclude that the evaporating dishes have higher weight differences, especially when it is repeatedly used without special treatment. The reasons are as follows.

- First of all, when the water sample has permanent hardness, it is easy to cause calcium and magnesium ions in the water to form sulfate, chloride and nitrate when drying. The water of crystallization contained in calcium and magnesium sulfate is generally difficult to remove by drying, which leads to higher results.
- Secondly, the chloride and nitrate of calcium and magnesium have strong water absorption, which leads to the changing weighing results and affects the accuracy.
- Although increasing heating temperature or extend the time can enhance the constant weight rate, the problems are still not solved.
- A better alternative is to use the solution of 2% HNO_3 + 6% HCl to soak the dishes for 48 hours before measuring the constant weight. The solution can completely remove the complex substances left in the evaporating dish to ensure its cleanness and constant weight.
- Without the influence of other factors, drying time, and drying temperature also had an impact on the results.

- The constant weight rate can be improved by exerting longer drying time and higher temperature, but this method is less effective than soaking the dishes in the solution of 2% HNO3+6% HCL for 48 hours.

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