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SUB-SAMPLING BY ROTARY SPLITTING

Requirements for ensuring representative sub-samples

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Application Note: 38

Background

The purpose of rotary splitting a sample is to make sub-samples of the bulk sample into smaller increments that are representative of the bulk sample. A rotary sample splitter is the preferred equipment for sample division as it is capable (with correct use and procedures) to obtain representative sub-samples from a dry free flowing solid material.

Rotary splitting is based on the two guiding principles of sampling; take *many increments* and do so whilst the *material is in motion*. Splitting needs to be conducted in such a manner that adherence to these requirements can be demonstrated. Work by Allen & Khan, (1970) illustrates that a minimum of 30 revolutions at a carousel peripheral velocity of <0.6 meters/second (Gy, 1979) are required to provide adequate precision in rotary splitting of a sample (Figure 1). The main parts, including the feeder control and feeder switch of a rotary splitter are shown in Figure 2.

In a rotary splitter, particles will move radially along the inclined sides of the divider until the distance between the divider sides is sufficient for them to fall into the cup. The discharge from the vibratory feeder chute should be just above the position of the collection cup diameter.

The aperture size in rotary splitting is defined as the inside diameter of the collection vessel (cups) for cylindrical collection cups. The inside cup diameter must be three times the nominal top size of the material being split (Figure 5). For example, if the diameter of a cup in a 10-way rotary splitter is 100 mm, the maximum nominal top size that should be split is: $100/3 = 33.3\ mm$.

Some rotary splitters can be purchased with or field retrofitted with a revolution counter. Figure 4 shows a rotary splitter fitted with a counter to track the number of revolutions made in splitting a sample. In addition, the carousel and vibratory feeder speed must remain constant during the splitting event. Never change the speed of either once splitting has commenced as this will create a bias in the sampling which will most likely show up as excessive split error. Each revolution equates to one increment received in all the sample receiving containers. Therefore if 55 revolutions are required to completely split the sample, 55 increments were taken.

The advantage of a spinning riffler is that it is designed to produce multiple splits of a sample, either as 8, 10 or 12 sub-samples and that these sub-samples are proportional to the original sample mass, *i.e.*, 1000 g sample split 10-ways should give 100 g masses per cup in a 10-way split. It is quite easy, therefore, to establish Quality Control (QC) tests to confirm that the weight of each sub-sample is within acceptable limits of the expected weight. Here a split error is calculated for each cup and the mass loss from the splitting event is also determined.

If split error for at least one of the cups is >2 %, adjust the speed of the carousel and or the feeder, and try again (slower speeds improve performance). By trial and error, a set of ideal speeds can be determined for many various types of material split, and if records are likewise maintained, they may be set each time the splitter is used to split similar materials.

By running the table at higher speeds, the sample tends to bounce off the rotating cup containers, and lands outside of the sample collection containers. Optimal rotation will need to be determined based on the criteria of < 2 % split error and < 5 % mass loss.

Selecting the correct splitter for the sample types to be sub-sampled depends on the volume of sample that needs to be split as well as the nominal top size (NTS) of the sample. Splitters vary in size and the hopper feeder can also only handle certain particle sizes. To ensure that splitting is accurate, the following should be adhered to in a rotary splitting event:

- The larger the NTS (see Appendix for calculation of NTS), the larger the initial mass needs to be otherwise the split error increases.
- The splitter size used is dependent on the size of the initial mass as the hopper needs to be big enough to hold the sample and if the mass is too small for the splitter the split error will increase.
- The hopper feeder size needs to be taken into consideration because if the nominal top size of the material is too large it will cause the feeder to choke.
- The rate of vibration of the feeder.
- The rate of rotation of the turn table:
  - The minimum number of turns required per split is 35 revolutions (Allen, 2003).
  - The peripheral velocity of the turntable must be <0.6 meters/second.

Splitting is accurate and repeatable provided the limits of the splitter are adhered to and the factors above are always considered. The factors that affect the split accuracy is the mass of the sample, sample top size, intensity of vibrations and speed of splitter cups. Therefore, the higher the nominal top size the larger the sample mass required to obtain an accurate split.

Figure 6 shows as rotary splitter in operation. It is important that the rotary splitter is level with the horizontal (a spirit level can be used to confirm this), the support onto which the rotary splitter is placed is strong and stable enough to hold the splitter without any tilting. Figure 7 to Figure 9 shows various unwanted practices that will create a bias in rotary splitter sub-sampling. Also relevant is that during splitting the sample material must not be forced to move quicker through the splitter – the material should always be allowed to flow freely.

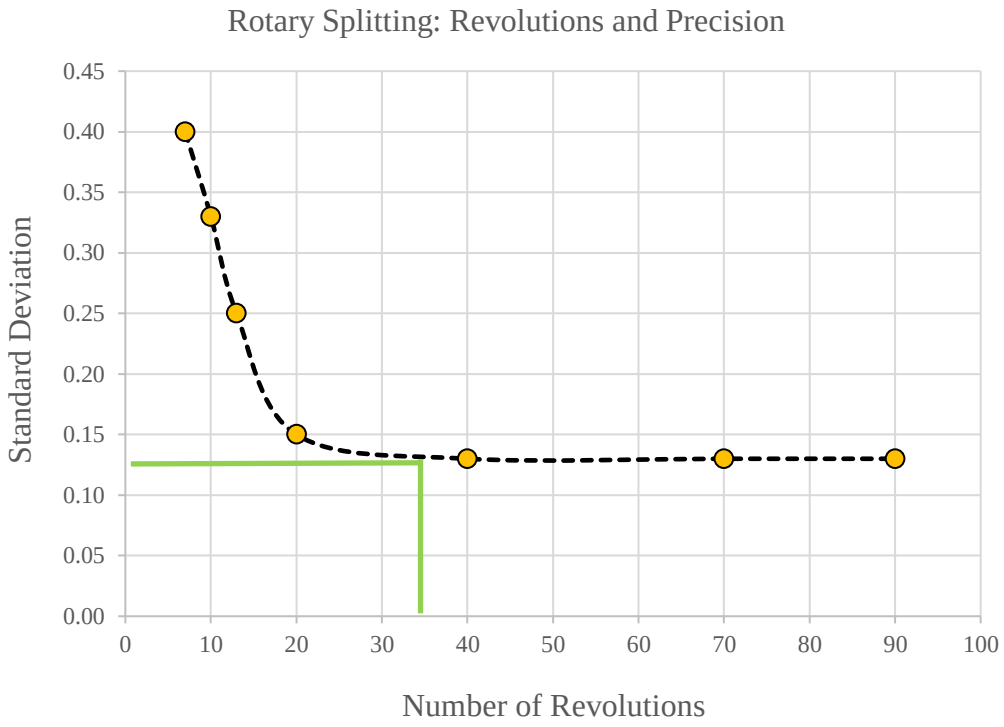

Figure 1. Work by Allen & Khan, (1970) illustrates that a minimum of 35 revolutions is required to provide adequate precision in rotary splitting of a sample.

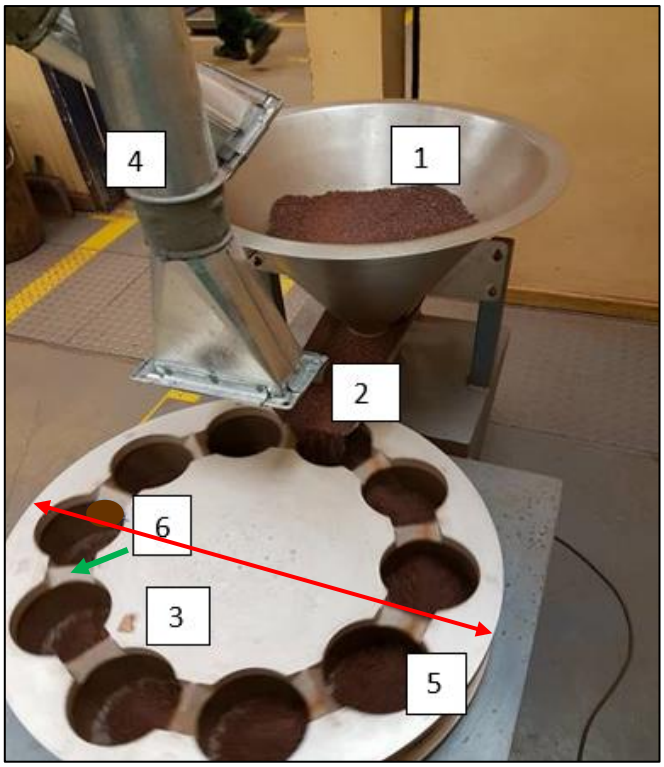

Figure 2. The main parts of a rotary splitter: 1. Cone shaped hopper, 2. Vibratory feeder, 3. Carousel, 4. Dust extraction unit, collection cup, 6. Inclined divider (green arrow). Red line shows the diameter of the carousel.

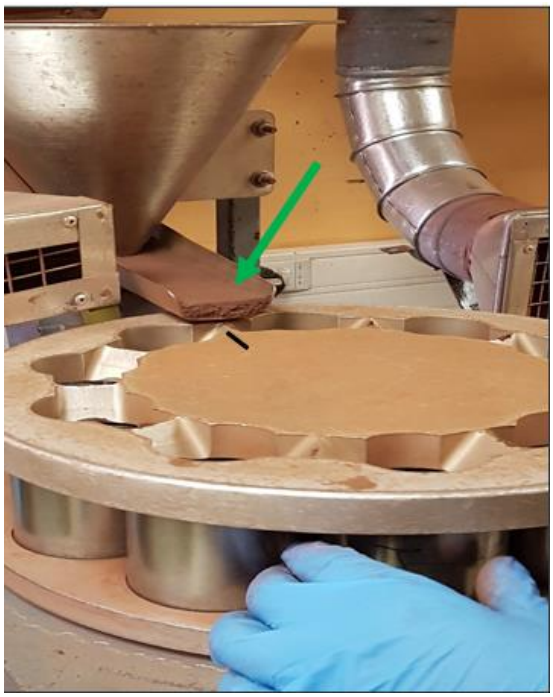

Figure 3. Before commencing the rotary split, stop the vibratory feeder when the material on the feeder is evenly spread (mono layer) on the vibrating feeder (green arrow). This allows an even start and split of the material on commencing the splitting event. The black line is to indicate the starting position of the carousel.

Figure 4. Turntable (carousel) feeder control and feeder switch on a rotary splitter. Some rotary splitters can be field retrofitted with a revolution counter. Here 87 revolutions were recorded to split a sample.

Figure 5. The diameter of the collection cups must not be less than three times the nominal top size of the sample to be split.

Figure 6. A rotary splitter in operation.

Figure 7. A 12-way rotary splitter. The splitter rests on a durable and level frame.

Figure 8. Left: Operator forcing the sample material onto the vibratory feeder. This is not allowed as the sample always needs to be under free flow. Right: The base that the rotary splitter rests on is not level (red arrow), meaning that the splitter itself is not level - this may cause a bias in the splitting of a sample.

Figure 9. Rotary splitter place onto a wooden pallet.
A more stable base is required. Note that the hopper is skew.

Procedure for use of Rotary Splitter

Initial Set up

1. The rotary splitter must be placed onto a solid and level base, i.e., either a concrete floor or steel stand. Under no circumstances must it be placed onto a wooden or plastic pallet.
2. Place a spirit level onto the centre of the carousel to ensure that the rotary splitter is level with the horizontal.
3. Make a visible permanent mark on the carousel (as shown by a drawn black line in Figure 3, pg., 5) to indicate the starting and ending position for measurement of the time for one revolution.
4. Position the carousel so that the black line is aligned with the vibratory feeder.
5. Switch the **carousel** on.
6. Switch on the **vibratory feeder**.
7. Record the time (T_R) in seconds for one revolution of the turntable using the black line as a guide.
8. Calculate the **peripheral velocity** v and record the result on a worksheet:

$$v = \frac{2\pi r}{T_R} \qquad [1]$$

Where r, is the radius of the carousel in meters, and T_R is the time in seconds taken for one revolution, and π=3.14. For example, a carousel of diameter 1.07 meters (the radius is half the diameter, r = 1.07 x 0.5) takes 5 seconds for one revolution. The peripheral velocity is:

$$v = \frac{2(3.14)(1.07\ meters\ x\ 0.5)}{5\ seconds}$$

$$v = 0.671\ meters/second$$

Since 0.671 m/s is greater than the velocity of 0.6 m/s, the carousel is turning too fast. Reduce the carousel speed to a lower setting, measuring again the time for one revolution and then recalculate the peripheral velocity. Accept the carousel speed setting once the velocity is <0.6 m/s This setting can be used in all subsequent sample splits. Alternatively, using equation [1], the velocity can be *predicted* by replacing the time by a larger value, e.g., if the time is doubled to 10 seconds, the velocity is halved to 0.336 m/s., which is below 0.6 m/s and therefore acceptable.

$$v = \frac{2(3.14)(1.07\ meters\ x\ 0.5)}{10\ seconds}$$

$$v = 0.336\ meters/second$$

9. Allow the carousel to rotate again and record the time for one revolution. Note the value obtained and record result on the worksheet.

Sample Splitting

1. The sample must be completely dry before splitting.
2. Before splitting a sample, ensure that all cups, vibratory feeder, hopper bin and carousel are meticulously clean and have no remnants of the previous sample.
3. Place the cups on the carousel after weighing each cup. Note that the cups are not equal in weight. The cup mass (M_1 ….M_{10}) should be written onto each cup using a marking pen so that they do not have to be weighed each time when subsequent samples are to be split. Record the cup masses onto a worksheet.
4. The mass of the cups should be verified monthly and records kept of the weights.
5. Weigh the total sample to be split and record the mass as the initial mass.
6. Carefully add the sample into the hopper bin, ensuring no spillage of sample occurs in this step.
7. Ensure that the entire sample is transferred into the hopper.
8. Switch the **carousel** on.
9. Switch on the **vibratory feeder** to allow sample material to move onto the vibratory feeder and to cover its total area with sample material. This allows an even distribution of the sample onto the vibrating feeder so that a monolayer of sample is formed (Figure 3).
10. It is important to note that once the carousel rotation is turned on, it may be necessary to adjust the rotation speed by sight, to where one thinks particles will not bounce around when hitting the cup edge and or the inclined divider.
11. Stop the **vibratory feeder** when the material on the feeder is evenly spread on the vibrating feeder as shown by the red arrow as seen in Figure 3.
12. Re-transfer all the contents that may have fallen into the cups back into the hopper bin.
13. Now start the carousel and then the vibrating feeder.
14. Allow the sample to split to completion.
15. Switch off the vibratory feeder.
16. Switch off the turntable.
17. Measure the mass of each cup containing the split sample increment.

18. Calculate the mass loss:

$$\%\ Mass\ Loss = 100\ x\ \frac{Initial\ Mass\ -Final\ Mass}{Initial\ Mass}$$

The *Final Mass* is the sum of the mass of sample less the mass of the cups (Table 1). Mass loss must be <5 %. If the mass loss >5 %, it is possible that sample was lost in transfer to the hopper bin and or are bouncing off the edge of the cups or inclined feeder between the cups.

19. Calculate the mean sample mass using equation [2], followed by the split error per cup from equation [3]:

$$Mean\ Sample\ Mass = \frac{Sum\ of\ all\ individual\ sample\ masses}{Number\ of\ cups\ (n)} \qquad [2]$$

$$Split\ Error\ per\ cup = \frac{|Sample\ Mass\ in\ cup\ 1 - Mean\ Sample\ Mass\ |}{Mean\ Sample\ Mass} \qquad [3]$$

Where, n is the number of cups in the rotary splitter. The | | bars mean that the absolute value of the difference must be used, i.e., if a negative sign/value is obtained, ignore the sign.

> *[A 5% split error is acceptable for sizes >6 mm and <2% for those < 6 mm. If only one of the splits is >2 %, the whole sample is recombined and split again, until the split error per cup is <2%. However, these criteria need to be established by experiment for the sample types received for splitting].*

An example of mass, split error and mass loss that is required for quality control of rotary splitting is shown in Table 1.

Table 1. A set of results for a 10-way rotary splitter showing split error per cup and mass loss for the spitting event. Here split errors are >2%, therefore a resplit is required to achieve <2%.

		Initial Mass (g)=	*300.40*		
Cup No.	**Sample & Cup Mass (g)**	**Cup Mass (g)**	**Sample Mass (g)**	**% Split Error**	
1	286.2	255.100	31.10	3.60	
2	272.3	241.000	31.30	4.26	
3	289.2	259.600	29.60	1.40	
4	286.3	257.200	29.10	3.06	
5	282.1	251.500	30.60	1.93	
6	288.0	256.800	31.20	3.93	
7	275.5	244.900	30.60	1.93	
8	281.6	252.800	28.80	4.06	
9	290.2	261.700	28.50	5.06	
10	284.5	255.100	29.40	2.07	
			Total Mass	300.20	Outcome
			% Mass Loss	0.067	True
			Max. Split % Error	5.06	False

20. Once the entire sample has been split and the split error per cup is < 2% and mass loss < 5 %, if two increments are required for assay, take two opposite cups and transfer into suitable labelled sample bags.
21. The remainder of the material can be combined for later assay should this be required.

References

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