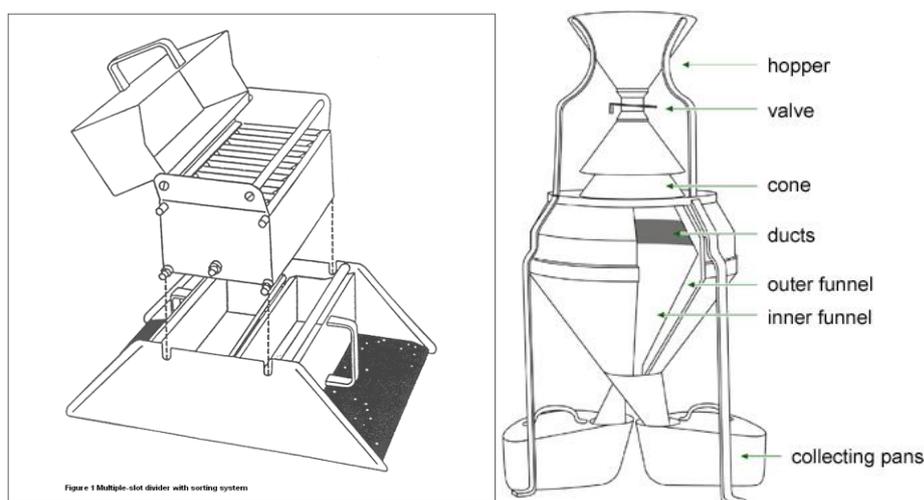


## METHOD 1.0

Effective 1<sup>st</sup> January 2018

### PREPARATION OF THE SAMPLE FOR ANALYSIS

1. Test and reserve samples must be prepared in such a way that the amounts weighed out, as provided for in the methods of analysis, are homogeneous and representative of the contractual samples as received.
2. All the necessary operations must be performed in such a way as to avoid, as far as possible, any change in, or contamination of, the sample. Grinding, mixing and sieving should be carried out as quickly as possible with minimal exposure of the sample to the air and light. Over-grinding should be avoided. Mills and grinders likely to heat the sample appreciably should not be used. Nevertheless, where some loss or gain of moisture is unavoidable, allowance should be made for such changes. Manual grinding is recommended for feeding stuffs, which are particularly sensitive to heat. Care should also be taken to ensure that the apparatus itself is not a source of contamination by trace elements.
3. If the contractual sample as received consists of more than one unopened package or container, then, by means of a dividing apparatus, a portion must be obtained from each of them to prepare a reserve sample (see point 7.). The remainder to be used for the test sample shall be thoroughly mixed together.
4. **Apparatus**
  - 4.1 *Mechanical mill*, easy to clean and capable of grinding grains and feeding stuffs without generation of excessive heat and without causing appreciable change in moisture and volatile matter, until the sample passes completely through a sieve of appropriate aperture size.
  - 4.2 All splitting and dividing of sample material shall be done using a well cleaned dividing or quartering apparatus until a test portion of suitable size is obtained.



5. If the test sample is appreciably moist or if for any reason the preparation cannot be carried out without significant changes in the moisture content of the test sample, determine the moisture content before and after preparation using method 2.1.

**6. Procedure**

It is emphasised that care must be taken to ensure that apparatus is not a source of contamination.

**6.1 Grinding**

**6.1.1 General**

**6.1.1.1** Grinding of some samples may lead to a loss or gain of moisture and volatile matter and, if appropriate, allowance for this should be made.

**6.1.1.2** Grinding should be as rapid as possible and exposure to the atmosphere should be minimised. If necessary, first break or crush the pieces to a suitable size for grinding.

**6.1.1.3** Use the first twentieth of the test sample to complete the cleaning of the mechanical mill and to establish the fineness of grinding and then reject it. It is essential that the sample be thoroughly mixed before each stage of the procedure.

**6.1.2 Fine Samples**

**6.1.2.1** If the test sample passes the 1.00mm sieve completely, mix it thoroughly. Divide the mixture successively using the dividing or quartering apparatus until a test portion of suitable size is obtained.

**6.1.3 Coarse Samples**

**6.1.3.1** If the test sample does not pass the 1.00mm sieve completely, but passes the 2.80mm sieve completely, mix the total amount thoroughly and prepare a sub-sample of suitable size by successive divisions (min. 500 gr for proximate analysis and min. 1500 gr for contaminant and/or undesirable substances analysis).

**6.1.3.2** Carefully grind this sub-sample in the well cleaned mill until it passes through the 1.00mm sieve completely. Mix thoroughly. Divide the mixture successively using the dividing or quartering apparatus until a test portion of suitable size is obtained.

**6.1.4 Very Coarse Samples**

**6.1.4.1** If the test samples do not pass completely through the 2.80mm sieve, then, in case of proximate analysis carefully grind a sub-sample of circa 1 kg in the well cleaned mechanical mill until it passes the 2.80mm sieve completely. In case of analysis for contaminants and/or undesirable substances, the complete contractual sample (e.g. 10 kg of maize for mycotoxin analysis) shall be ground in the well cleaned mechanical mill until it passes the 2.80mm sieve completely. Mix thoroughly.

**6.1.4.2** Divide the ground test sample, or sub-sample as the case might be, successively by means of the dividing apparatus until a test sample of suitable size for all the determinations required, is obtained (min. 500 gr for proximate analysis and min. 1500 gr for contaminant and/or undesirable substances analysis). Grind this sample material in the well-cleaned mechanical mill until it passes the 1.00mm sieve completely. Mix it thoroughly. Divide the mixture successively using the dividing or quartering apparatus until a test portion of suitable size is obtained.

**6.1.4.3** Laboratories equipped with high-throughput grinders can do the grinding of the contractual sample in one run if the ground material passes the 1.00 mm sieve completely. In such case mix the ground material thoroughly. Divide the mixture successively using the dividing or quartering apparatus until a test portion of suitable size is obtained.

## **6.2** *Samples difficult to grind*

**6.2.1** If the condition (such as hardness, oiliness, etc.) of a test sample not passing through the 1.00 mm sieve makes grinding difficult, take a test portion immediately after the preliminary mixing described in 6.1.3.1, or after the preliminary grinding procedure described in 6.1.3.2.

**6.2.2** Determine the moisture and volatile content.  
Dry the sample until, after crushing with the pestle and mortar, or by other means, it passes the 1.00mm sieve completely. Again determine the moisture and volatile matter content of the prepared test portion so that the results of analyses may be corrected to relate to the sample in its original condition as regards moisture and volatile matter content.

**6.3** When a microscopic examination for the presence of undesirable substances is required, it is recommended that the test portion is crushed and ground only to such an extent that facilitates the examination. Grinding to pass 1.00mm may lead to difficulties in identifying the undesirable substances.

## **6.4** *Samples for which there are special requirements*

**6.4.1** For determinations requiring special degrees of fineness, further grinding may be necessary. In such cases, prepare another test sample as described in 6.1, 6.2 or 6.3, but having the required degree of fineness.

**6.4.2** In some cases it may be necessary to avoid breaking or damaging the test sample, for example, for the determination of pellet hardness.

**6.4.3** If the test sample is fatty, the test sample may be prepared by warming and mixing. In some cases preliminary extraction of fat may be necessary.

**6.4.4** If microbiological examination is required, the sample shall be handled in sterile conditions and in such a way that the microbial condition does to change.

## **7. Retention and Storage of Samples**

**7.1** A suitable sized portion of the contractual sample, as received, must be retained, for a period as set out in Gafta 124 Sampling Rules, Section 1 Sub-Section 9, Retention of Samples. The retained sample must be stored at such a temperature that will minimise compositional changes.

**7.2** A portion of the processed samples must also be retained for at least one month, and these must be stored at such a temperature that will minimise compositional changes. Samples intended for the analysis of vitamins or substances, which are particularly sensitive to light, should be placed in brown glass containers.

## **8. Correction Factor**

### **8.1 General**

If there is likely to be a loss or gain of moisture and volatile matter during the grinding or mixing operations, it is necessary to use a correction factor to relate the results of analyses to the sample in its original condition as regards moisture and volatile matter content. A similar concept applies if a preliminary extraction of fat is carried out.

### **8.2 Calculation**

The correction factor C is given by the equation:

$$C = \frac{100 - U_0}{100 - U_1}$$

Where

$U_0$  is the moisture content, expressed as a percentage of mass, of the laboratory sample as it was, before grinding or after a non-moisture loss grinding.

$U_1$  is the moisture content, expressed as a percentage of mass, of the laboratory sample after grinding in the case of a moisture loss grinding.

### **8.3 Correction of Results**

Multiply the results of analyses by the correction factor C.