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Standard for Fertilizers
Part 10 – Methods of Sampling of Fertilizers

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SON

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Foreword

This National Industrial Standard was first published in 1979. Nigerian Industrial Standards are required to be periodically reviewed so as to reflect changes in technology. This second edition was prepared by Standards Organisation of Nigeria (SON) Technical Committee on Agro-Chemicals. The purpose is to acquaint manufacturers, purchasers and users of fertilizers with the methods of sampling of the product for the purpose of analysis and quality control.

It is important that a representative sample be taken from the lot since some fertilizers are mixtures of unlike materials with a marked tendency towards segregation. Sampling problems are intensified when fertilizers are in bags or piles. This standard specifies methods of taking a representative sample from the lot.

While the solid reduction process is expatiated in this edition, the standard apparatus for the reduction of size of composite samples called the “Gated Riffle” is introduced.

In the preparation of this standard, reference was made to international and other national standards. Cognizance was also given to prevalent practices in Nigeria.

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1. Scope

This standard prescribes methods of drawing representative samples from bagged, bulk and liquid fertilizers.

2. General Requirements of Sampling

In drawing samples, the following precautions and directions shall be observed**.**

(i) The sampling instruments shall be clean and dry.
(ii) The material being sampled, sampling instrument and containers for samples shall be protected from adventitious contamination.
(iii) To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible.
(iv) The sample shall be placed in a clean, dry and air tight glass or other suitable containers.
(v) The sample container shall be of such a size that is almost completely filled by the sample.

(vi) Each sample container shall be sealed air-tight after filling and labeled with full details of the sample, the date of sampling and other particulars of the consignment.
(vii) Samples shall be stored in a cool dry place.

3. Sampling of Bagged Solid Fertilizer

3.1 Scope

Method for obtaining a representative sample from a lot of fertilizer packaged in containers or bags containing not less than 5 kg is described.

3.2 Scale of Sampling

1. **Lot**

In this procedure, a lot is defined as the amount contained in a single consignment or delivered under a single invoice, of materials of the same grade and type, drawn from a single batch of manufacture. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and each batch shall constitute a lot. If this results in a lot being greater than 100 tons, more than one sample must be taken. Each sample so taken will represent 100 tons or less of product. In the case of a consignment drawn from a continuous process, 100 tons of the material shall constitute a lot.

2. Sample Size

The number of containers to be selected from a lot shall depend on the size of the lot and shall be as specified in Table 1.

Table 1 - Number of Containers or Packages to be selected from a Lot

Lot Size, (N)	No. of containers to be selected, (n)

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<28	2
28 – 64	3
65 – 100	4
101 – 300	6
301 – 500	7
501 – 800	8
801 – 1300	9
1300 and above	10

3. **Mode of Selection:** These containers shall be selected at random from the lot.

3.2.4 Randomness of Selection: In order to ensure randomness of selection, a random number table shall be used. In case such a table is not available, the following procedure shall be adopted.

Arrange all containers in a systematic manner, and starting from any container, count them as 1, 2, 3…….r, ('r' being the integral part of N/n). Every r^{th} container counted shall be withdrawn and all such containers shall constitute the sample.

3.3 Preparation of Composite Sample

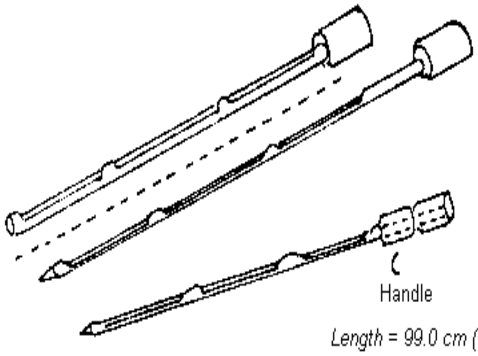
3.3.1 General: A composite sample that is a representative of the lot shall be obtained from the selected containers with the aid of sampling probe. If the container does not permit the use of the instrument, then the quartering method can be applied.

3.3.2 Sampling Instrument

(i) A slotted single-tube trier (figure 1) or double-tube trier (figure 2) with solid core tip constructed of stainless steel or brass. Stainless steel is required for samples on which micro-nutrients are to be determined. The tubes shall be loosely fitted into each other through out their entire length so that it is possible to rotate one tube within the other. A suitable handle shall be provided for this purpose. In one position, the two openings coincide and admit the fertilizer into the hollow inner tube. A locking arrangement shall be provided to hold the tube together in any desired position.

Length, exclusive of handle = 92.0 cm (minimum)
Slot length = 80.0 cm (minimum)
Slot width = 1.9 cm (minimum)
Inner diameter = 1.6 cm (minimum)

Figure 1: Single-Tube trier



Length = 99.0 cm (minimum)
Slot lengths = 32.0 cm each (minimum)
Slot width = 1.9 cm (minimum)
Outside diameter = 2.2 cm (minimum)

Figure 2: Double-Tube trier

(ii) A corrosion resistant U-shaped trough, slightly longer than the trier (figure 3). This trough may be used to transfer trier sample cores to an intermediate container avoiding spillage or loss.

(iii) Pressure-sensitive patches for sealing sampling holes in bags.

(iv) Intermediate container for holding the cores until all cores have been taken. A large bucket or empty bag may serve this purpose.

(v) Sample container of 1 litre capacity or as required and constructed of corrosion-resistant material with a moisture proof barrier, or fabricated from material that will not permit moisture to enter or leave the sample.

(vi) Pressure-sensitive tape to be applied as a final seal to the sample container. Removal of the seal is forbidden prior to the receipt of the sample in the laboratory.

3.3.3 Sampling Procedure

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(i) Roll or flip over the bag to be sampled one or more times to ensure that the product is free flowing. Then lay bag flat in a horizontal position.

(ii) With the single tube trier, Insert horizontally so that the trier extends diagonally from corner to corner with the slot down (Figure 4a). Rotate one-half turn to bring the slot into the up-position, shake bag slightly to fill the trier, and remove carefully so as not to lose any of the core samples: or

With the double tube trier, insert horizontally as before but with the trier closed and the slot up. Open the trier and shake the bag slightly to fill (Figure 4b). Close the trier and remove it from the bag.

(iii) The core hole should be covered immediately with a pressure-sensitive patch to prevent any spillage of product. The lot sampled should be identified on the patch with the inspectors sample number. This same sample number should appear on the inspection report.

(iv) Transfer each trier sample core to the U-shaped trough (Figure 5), and then to the intermediate container.

(v) The sample submitted to the laboratory must weigh between 500 and 1000 grammes. If the net sample weight is more than 1000 grammes, reduce by riffling method (3.4.3) or quartering method (3.5.2) to an amount between 500 and 1000 grammes. If it weighs less than 500 grammes, take two or more cores from each bag to develop the composite sample.

(vi) Transfer the final sample to the sample container and apply all pertinent information to the label of the sample container.

(vii) Seal the container with the pressure-sensitive tape.

(viii) Forward the sample and report to the laboratory for preparation and analysis.

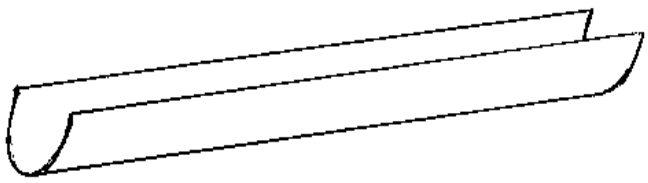

Figure 3: U-Shaped Trough

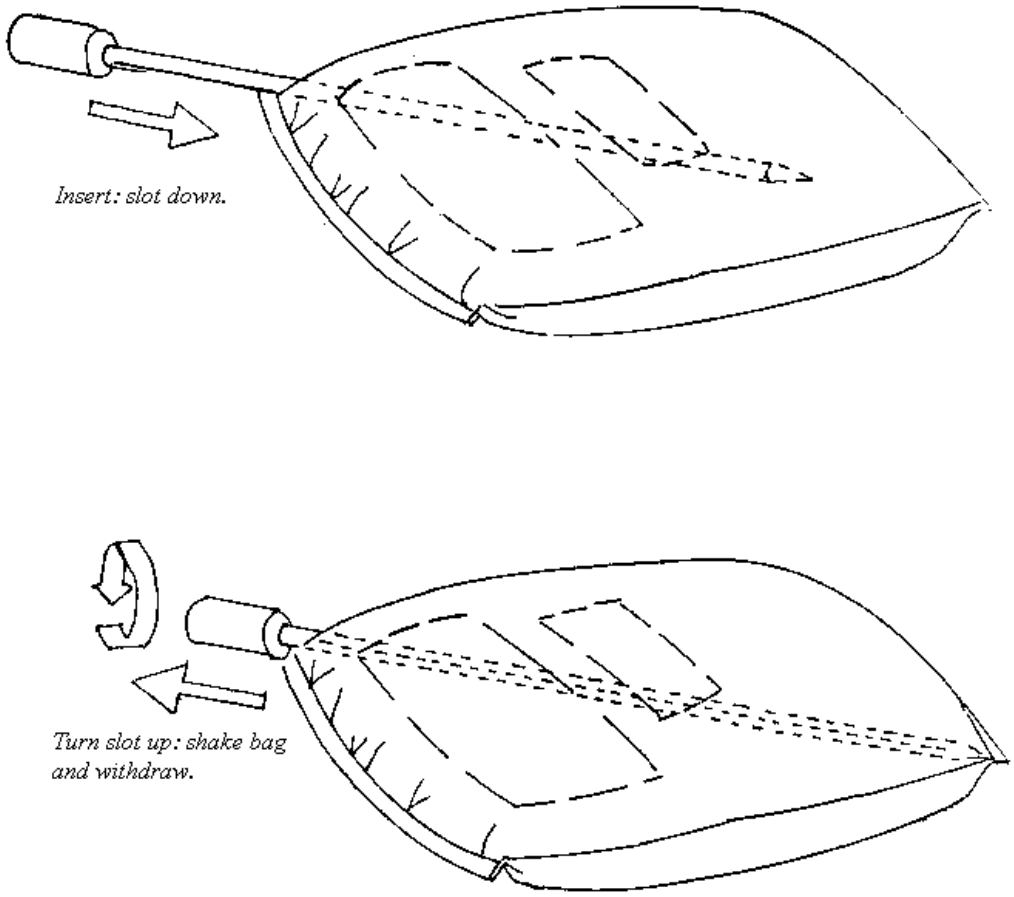

Figure 4: Bag sampling technique

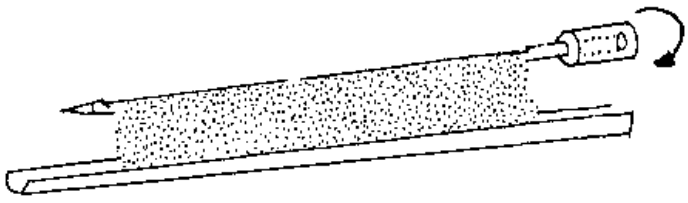

Figure 5: Transfer of core sample from single-tube trier into U-shaped trough.

3.4 Solid Reduction

3.4.1 General

If the composite sample collected is more than 1 kg, it would become necessary to reduce the size by the solid reduction process. For this purpose, a gated riffle is used and certain basic procedures followed to ensure that the reduced portion is still a representative of the original sample.

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Where the apparatus is not available, the quartering method can also be applied.

Note: *This apparatus may be used for taking composite samples where the fertilizer container does not permit the use of sampling probe and for preparation of reduced or test samples.*

3.4.2 Apparatus

(i) Gated Riffle (Figure 6): Riffle should have a minimum of 16 slot openings (8 per side). Slots should be a minimum of 12mm wide. The riffle should include two receiving pans of corrosion-resistant material. Receiving pans must fit riffle from end to end of partitioned section.

(ii) Sample Container: As in 3.3.2 (v)

(iii) Pressure-Sensitive Tape: As in 3.3.2 (vi)

3.4.3 Riffling Method

(i) Ensure that all equipment is clean.
(ii) Set riffle in a level position, not tilted in any direction.
(iii) Place the two receiving pans in position beneath the riffle.
(iv) Transfer the composite sample to the hopper of the riffle.
(v) Open the gate fully and allow the entire sample to flow into the pans beneath the riffle, forming two equal portions.
(vi) If required, steps (iv) and (v) above may be repeated by selecting alternating equal portions (pans) until the content of the collection pan is between 500 and 1000 grammes.
(vii) Transfer the final sample to the sample container and specify all information on the label of the sample container.
(viii) Seal the container with pressure-sensitive tape.
(ix) Forward the sample and report to the laboratory for preparation and analysis.
(x) Clean all equipment before storing for re-use.

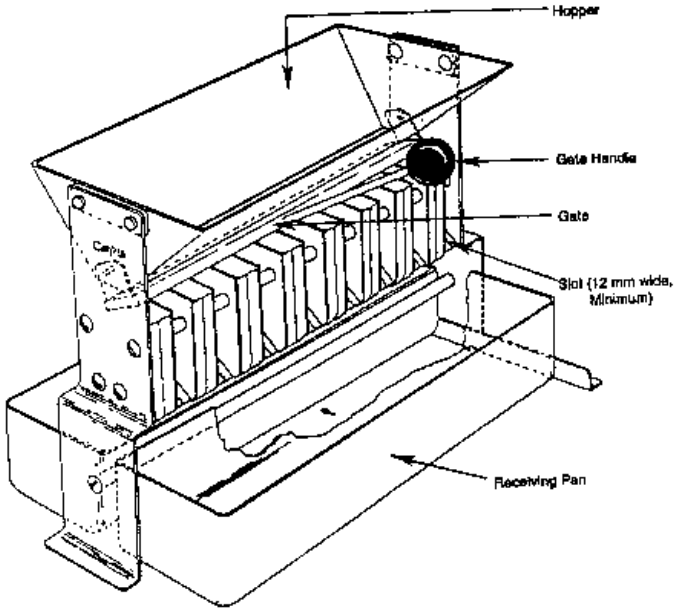

Figure 6: Riffle with cutoff gate and two pans.

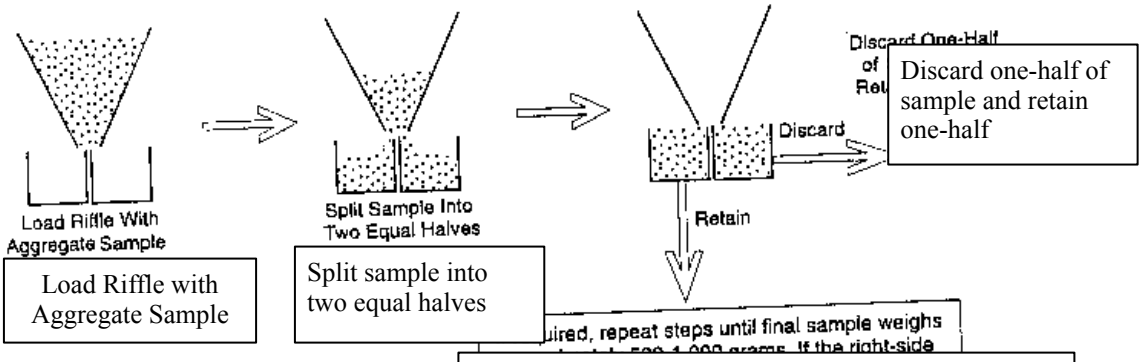

Figure 7: Sample riffling steps to redu
to approximately 500 - 1000

3.5 Quartering Method

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3.5.1 Scope

This method shall be used in drawing representative composite sample if the fertilizer container does not permit the use of sampling probe and for reduction of composite sample if the solid reduction apparatus is not available.

3.5.2 Procedure

Spread the material to be sampled or reduced on a level, clean, dry, and hard surface. Flatten it out and divide into four equal parts. Remove any diagonally opposite parts. Mix the two remaining parts to form a cone. Flatten out the cone and repeat the operation of quartering until the required sample size is obtained.

4. Sampling of Bulk Fertilizer in Heaps or Wagons and from Conveyor Belts

4.1 Scale of Sampling

Samples shall be taken from each heap, wagon or conveyor belt as in 4.2.3 (i), 4.2.3 (ii) or 4.2.3 (iii) as the case may be.

4.2 Preparation of Composite Sample

4.2.1 Sampling Equipment

(i) **Trier -** Any of the triers specified in Table 2 may be used. Also see Figure 8a.

(ii) **Sampling Cup:** This is for sampling fertilizers from conveyor belts or troughs. The sampling cup is illustrated in Figure 8b with slot dimensions of 2 x 20 cm. It can be fabricated from a non corrosive material using the dimensions in Figure 8b.

4.2.2 Sample Size

(i) **From Storage Piles** – Ten vertical cores shall be taken from sampling points as shown in Figure 9, with any of the triers specified in 4.2.1(i).

(ii) **From Truck or Wagon Load -** Ten vertical cores shall be taken from sampling points as shown in Figure 10, with any of the triers specified in 4.2.1(i).

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(iii) **From Conveyor Belt** – Estimate the time lapse for the entire transfer operation and calculate the approximate time interval needed for a ten equal-time-spaced passes. Using the sampling cup specified in 4.2.1(ii) with the slot perpendicular to the falling stream, make ten passes through stream at a uniform rate and at the ten time interval calculated.

Table 2 – Trier Specifications

Trier	Length (cm)	O.D (mm)	I. D. (mm)	Compartments	
				Number	Size (cm)
Missouri	150	29	22	8	7.6
552 Grain	160	35	29	11	8.9
Missouri "D" (see Fig. 8a)	132	32	25	1	113

4.2.3 Sampling Procedure

(i) **From Storage Piles** - Using any of the triers specified in Table 2, take one from each sampling point as designated for coned or rigid piles. If the pile is one sided as shown in Figure 9b, take one core from areas 1 and 6, and 2 cores each from areas 2, 3, 4 and 5. Mix thoroughly to obtain a composite sample.

(ii) **From Truck or Wagonload:** Using any of the triers specified in Table 2, take one core from each sampling area as designated in Figure 10, and mix thoroughly to obtain a composite sample.

(iii) **From Conveyor Belt:** Estimate the time lapse for the entire transfer operation and calculate the approximate time interval needed for a ten equal-time-spaced passes. Using the sampling cup, (Figure 8), with the slot perpendicular to the falling stream, make 10 passes through the stream at a uniform rate and at the 10 time intervals calculated. Mix thoroughly to obtain a composite sample.

5. Test and Reference Samples

5.1 Preparation of Test and Reference Samples

(i) Each of the composite samples obtained in 3.3.3 and 4.2.3 shall be reduced or divided into three equal portions, not less than 500g each,

by the solid reduction process or quartering method described in 3.4 and 3.5 respectively. Each of these portions shall constitute the test sample.

(ii) Each test sample shall be transferred immediately to a suitable container (as described in 3.3.2(v)) provided with a tight-fitting stopper or lid so that the original composition of the fertilizer remains unchanged. Each container shall be properly labeled, giving all the particulars of the consignment.

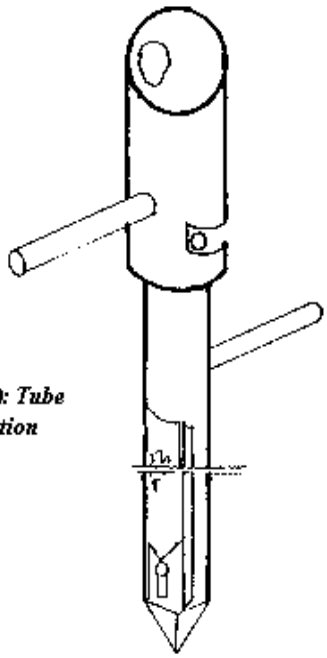

Figure 8a(i): Tube before insertion

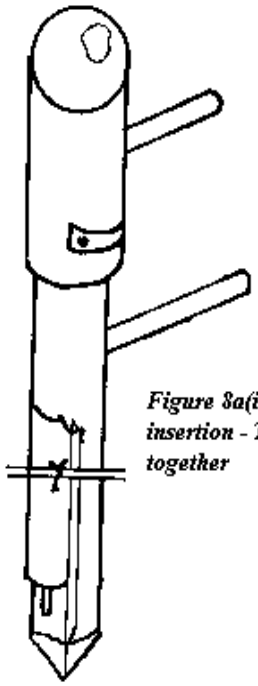

Figure 8a(ii): Tube after insertion - T handle together

FIGURE 8a: MISSOURI "D" TUBE

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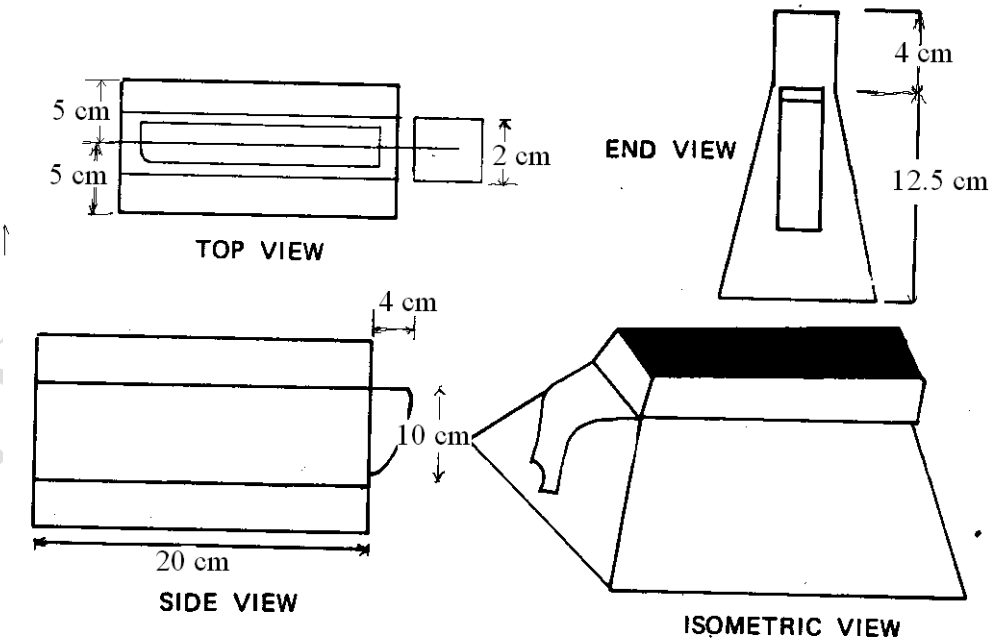

Figure 8b: Sampling Cup

(iii) Each test sample container shall then be sealed with the seals of both the manufacturer or dealer and the purchaser or the inspecting officer, as the case may be.

(iv) One sample so sealed shall be marked for the purchaser or the inspecting officer, as the case may be, for analysis: and another marked for the manufacturer or the dealer as the case may be. The third sample shall constitute the reference sample to be used in case of dispute. It shall be kept at a place agreed to between the purchaser or the inspecting officer and the manufacturer or the dealer, as the case may be.

6. Sampling of Liquid Fertilizer

6.1 Sampling in the Absence of Free Ammonia

6.1.1 Sampling from Storage Tank and Delivery/Recirculation Line

6.1.1.1 Sampling Equipment

(i) Weighted sampling bottle and steel jacket as illustrated in Figure 11.
(ii) Glass or polyethylene sample receiver.

6.1.1.2 Sample Size

A minimum of 250mL shall be taken.

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6.1.1.3 Sampling Procedure

(i) **From Top Opening of Storage Tank –** Lower weighted sampling bottle into well while the bottle fills. Transfer sample to the sample receiver, seal tightly and forward to the laboratory for analysis.

(ii) **From Delivery or Recirculation Line** – Flush delivery line with the material at least twice the volume of the delivery line: or re-circulate material for at least 15 minutes through the recirculation line before taking sample. Fill the sample receiver from the delivery or recirculation line with about 250mL of liquid sample, cap tightly and forward to the laboratory for analysis.

6.1.2 Sampling Liquid Fertilizer in Containers

6.1.2.1 Sample Size

The number of containers to be selected from a lot shall depend on the size of the lot and shall be as specified in Table 3. The mode and randomness of selection shall be as specified in 3.2.3 and 3.2.4.

Table 3 - Sample Size for Liquid Fertilizer in Containers

Lot Size (N)	**Number of Containers to be selected (n)**
1 - 3	All containers
4 - 20	Not more than 4
21 - 60	Not more than 6
61 - 100	Not more than 8
101 - 400	Not more than 10
More than 400	Not less than 20

6.2 Sampling of Ammoniacal Solution

6.2.1 Apparatus

(i) **Container**: Polyethylene reagent bottle of about 1 litre capacity with buttress-type cap.

(ii) **Sample Flow Control Apparatus:** Constructed from the following fittings: 3.81 x 0.63 cm reducing bushing, 0.63 cm tee, 0.63 cm nipple nose needle valves with hose connections. All fittings except valves can be either aluminum or stainless steel (Figure 12).

6.2.2 Sampling procedure

(i) Attach the valve directly to the tee junction which is then attached to the reducing bushing through the nipple. To both valves attach 0.63cm internal diameter Tygon tubing of 30cm length to sample valve and sufficient length to vent valve to reach disposal area of container. To the free end of sample tubing, attach 7.5cm length of 0.63cm glass or stainless steel tubing inserted through No. 4 rubber stopper. To the exit of the metal tube, attach an additional 15cm length of Tygon tubing. Make certain all connections are tight. This apparatus can be attached directly to the tankers, but requires additional coupling, which varies with the installation, to attach to storage tanks. 3.7cm “quick coupler” suffices in most cases.

(ii) Prepare the sample bottle in the laboratory by adding approximately 500mL of water, replacing the cap and weighing accurately to ± 0.1 g. Attach the sampling apparatus to the tank and with the sample valve closed, flush the line through the vent valve. Partially collapsing the sample bottle, insert the sample tube with stopper and fit tightly. With the sample tube dipping below the surface of the water in the bottle, throttle the vent valve to maintain a small flow of the solution and partially open the sample valve, collecting approximately 100mLof the sample. (Bottle should not expand to full size during this process). Close the sample valve, remove the sample tube, partially collapse the bottle, and cap tightly. Reweigh to ± 0.1 g accuracy and calculate the weight of sample. Cool to 20^{o}C. Transfer to 1 or 2 litre flask. Dilute to volume with water, mix thoroughly and take aliquots for analysis.

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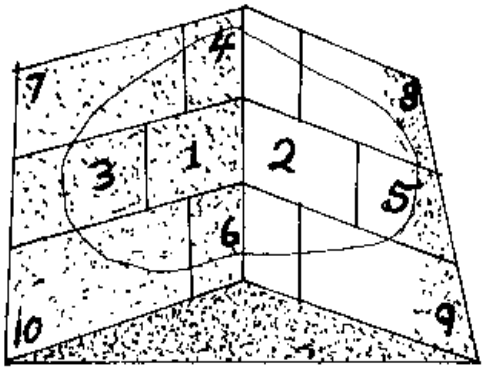

Figure 9a: Sampling points for coned or ridged pile.

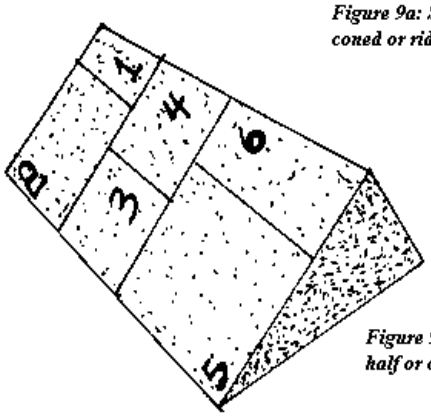

Figure 9b: Sampling points for half or one sided pile.

FIGURE 9: SAMPLING POINTS IN STORAGE PILE.

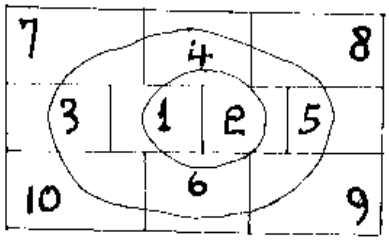

FIGURE 10: SAMPLING POINTS IN TRUCK OR CAR.

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6.3 Sampling of Anhydrous Ammonia

6.3.1 General

This method of obtaining samples of anhydrous ammonia is based on the assumption that the material contains only a small amount of impurity, primarily water. It is recommended that duplicate samples be taken from each tank or vessel sampled.

CAUTION: *Use extreme care in handling anhydrous ammonia. Suitable gas masks and rubber gloves are required*.

2. Apparatus

(i) Sample tube of thermal shock resistant glass calibrated to contain 100mL and graduated in 1mL in 0.05mL divisions, 2 - 4mL in 0.1mL division, 4-10mL in 0.5mL division, and 10-100mL in 1.0mL divisions.

(ii) Rubber stopper with tight fitting piece of glass tube 0.60cm internal diameter and 5.8cm long, bent at its exit from the outer end of the stopper to let gases escape.

(iii) Sample carrier, constructed of plywood or aluminum.

(iv) Protective equipment: rubber or other non-porous gloves, goggles or approved gas mask.

6.3.3 Sampling Procedure

(i) Place two dry and clean sampling tubes in the sample carrier and connect the sampling line connection assembly to the unloading valve of the tank or vessel.

(ii) Open the valve slowly and flush the sampling line by venting 3-4 litres of ammonia. Close the sample line valve, remove the vented stoppers from the tube and fill the tube to the 100mL mark in such a manner that condensing moisture will not enter the sample tube. Also note the vessel, container or line pressure of the material sampled.

(iii) Insert the vented stopper in the sampling tube and repeat the above steps to fill the second tube.

(iv) Tag the collected samples for identification and submit to the laboratory for analysis.

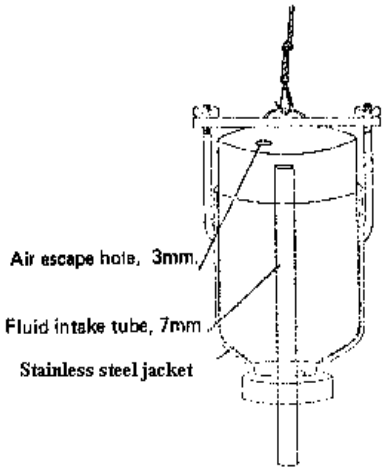

Figure 11: WEIGHTED SAMPLING BOTTLE

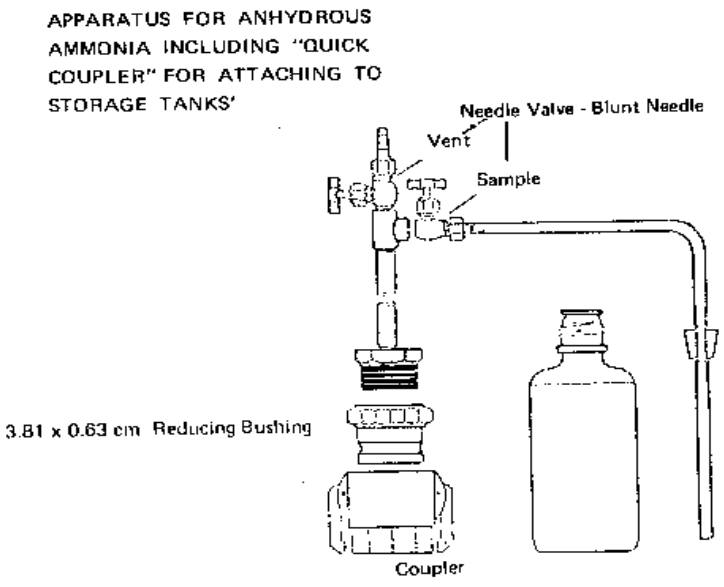

FIGURE 12: SAMPLING