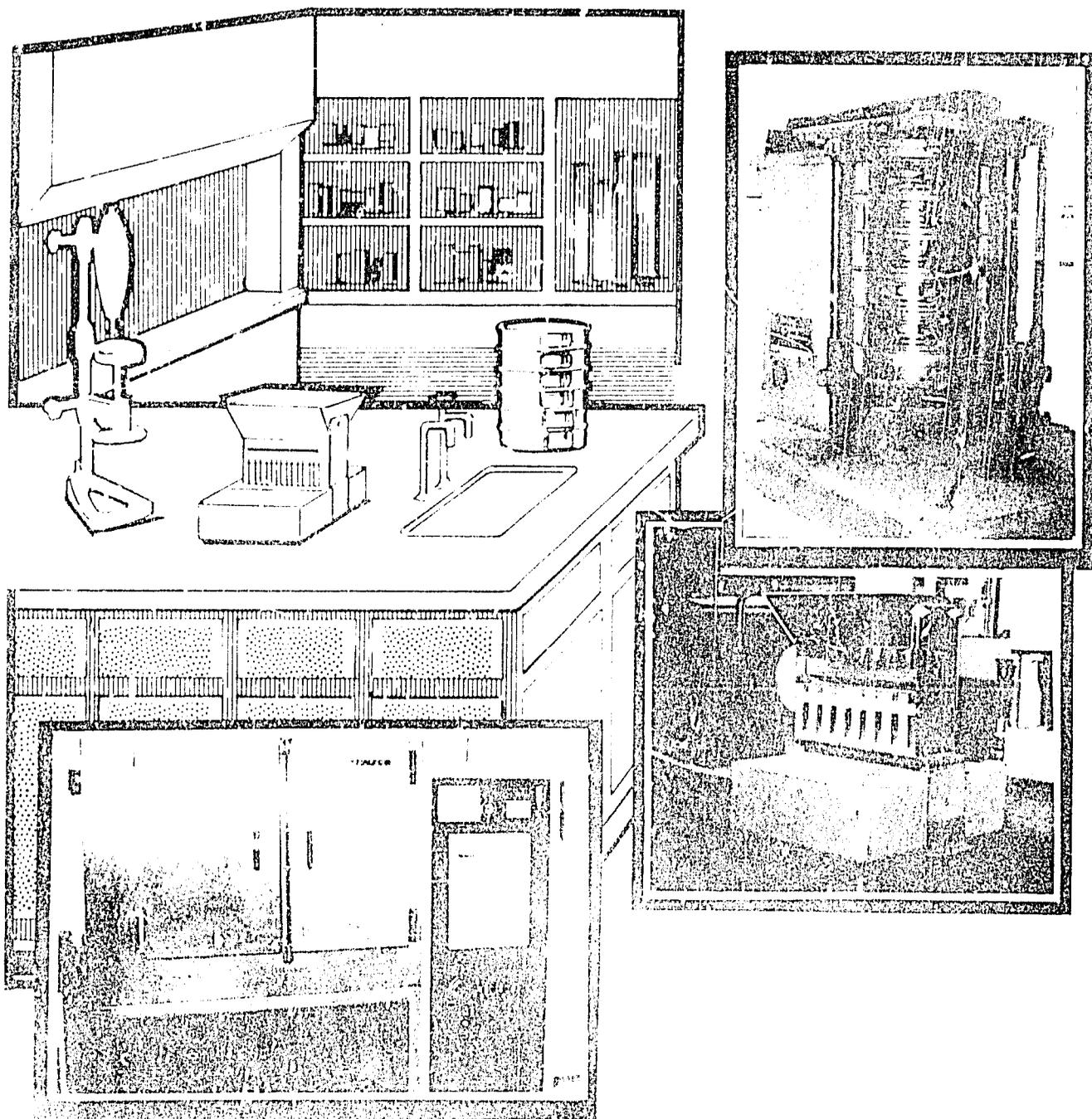
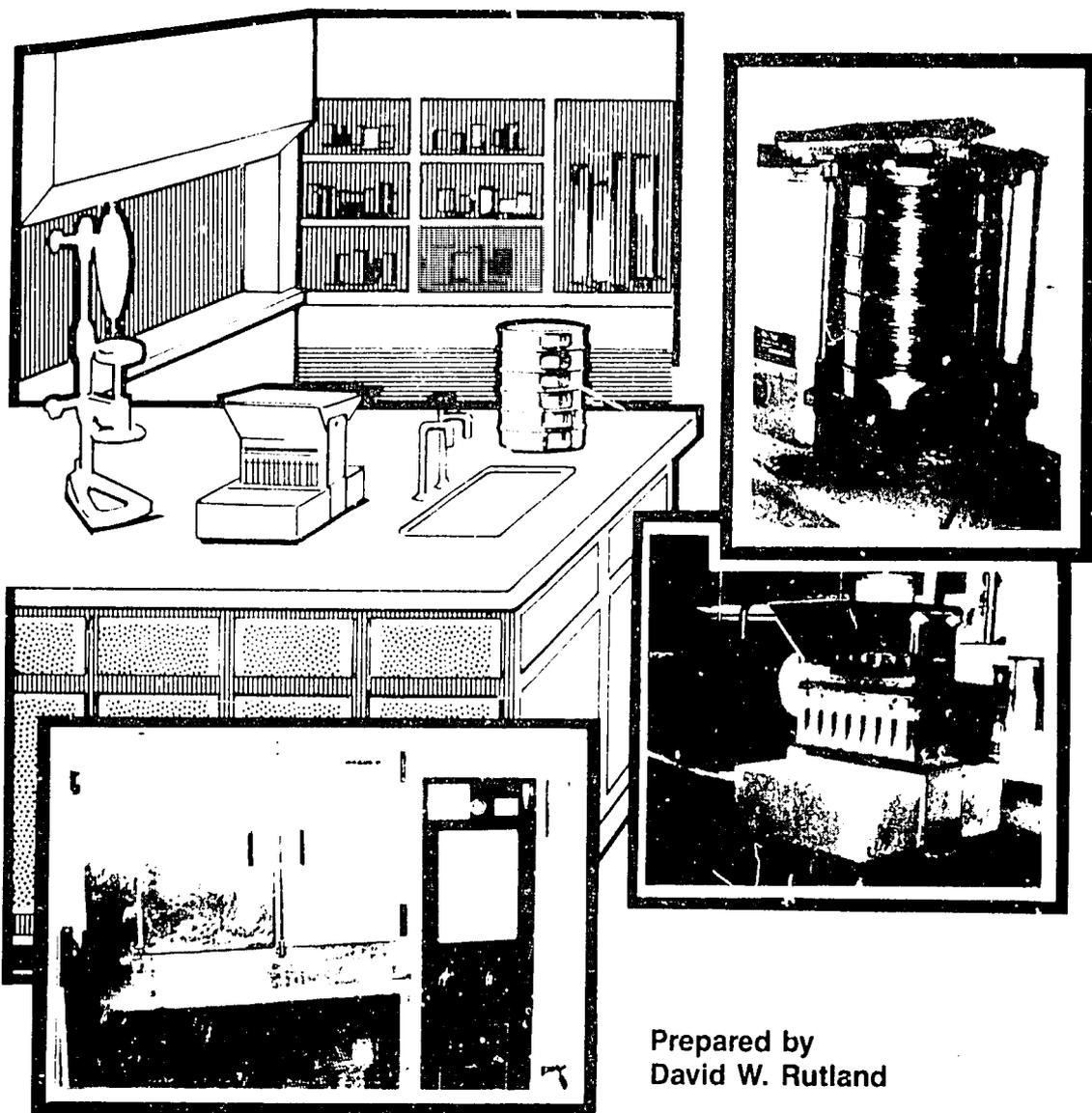


# Manual for Determining Physical Properties of Fertilizer



International Fertilizer Development Center

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# Abstract

The International Fertilizer Development Center (IFDC) has prepared a manual outlining methods for the determination of physical properties of fertilizer, which includes procedures used by IFDC to evaluate the physical quality of commercial and experimental fertilizer products and raw materials. Many of the procedures included in the manual were adopted from the Tennessee Valley Authority (TVA)/National Fertilizer Development Center (NFDC) and other organizations involved in the measurement of physical properties. IFDC does not endorse these methods as being standards.

The physical properties of fertilizer that are included in the manual are critical relative humidity, moisture absorption-penetration characteristics, flowability, chemical compatibility in blends, caking tendency, size analysis, physical compatibility in blends, angle of repose, bulk density, apparent density, true density of solids and liquids, viscosity, granule crushing strength, abrasion resistance, impact resistance, handling strength, sphericity, conditioner adherence, dustiness, surface area, disintegration rate, and porosity.

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# Foreword

Physical properties of fertilizers are important in processing, handling, transportation, bagging, storage, and application. Recognizing this importance, IFDC maintains and operates a well-equipped fertilizer physical properties laboratory. This manual is a compendium of the procedures used in this laboratory to perform physical quality evaluations of commercial and experimental fertilizer products and raw materials. IFDC has decided to publish this manual in view of the fact that to its knowledge there is no other such manual presently in print.

IFDC wishes to thank the Tennessee Valley Authority (TVA)/National Fertilizer Development Center (NFDC) for valuable assistance in establishing a fertilizer physical properties laboratory. Special thanks goes to Mr. George Hoffmeister, formerly with TVA, and Mr. Frank Johnson. Many of the procedures included in the present manual were adopted from NFDC and originally included in *TVA Procedures for Determining Physical Properties of Fertilizers* (Special Report No. S-444). Over the past several years, IFDC has adopted procedures from other organizations and also devised several of its own procedures. The TVA Special Report No. S-444 is out of print and has been replaced by NFDC's *Physical Properties of Fertilizers and Methods for Measuring Them* (TVA Bulletin Y-147). While this is a very valuable bulletin, it is not a procedural manual.

Although the procedures included in this manual have proved useful in carrying out IFDC's research and development work, they are not "official standards," and IFDC does not endorse any of them as such. None of the procedures have any guarantees and therefore should not be used legally as a claim for physical deficiencies or in advertisement for claims of superior quality.

IFDC shall not be responsible for any incidental or consequential damages resulting from the use of the procedures in this manual whatsoever.

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## List of Abbreviations and Symbols

Weight		Linear Measurement		Volume		Time	
kg	kilogram	m	meter	L	liter	h	hour
g	gram	cm	centimeter	mL	milliliter	min	minute
mg	milligram	mm	millimeter	m <sup>3</sup>	cubic meter	s	second
lb	pound	μm	micron	cm <sup>3</sup>	cubic centimeter		
wt	weight	ft	foot, feet	ft <sup>3</sup>	cubic feet		
		in	inch				
		dia	diameter				
				<b>Area Measure</b>			<b>Other</b>
				cm <sup>2</sup>	square centimeter	cP	centipoise
				r	radius squared	rpm	revolutions per minute

*Handwritten mark*

## Laboratory Absorption-Penetration Test

Hygroscopicity is the degree to which a material will absorb moisture from the atmosphere. Hygroscopicity of fertilizers is important when considering (1) conditions under which a bulk pile can be stored and (2) material flowability during handling and field application.

Fertilizer materials vary in their ability to withstand physical deterioration (wetting and softening) when exposed to humid atmosphere. Even materials with about the same critical relative humidity (CRH) often behave differently as a result of differences in "moisture-holding capacity"; thus, determination of CRH is not sufficient in itself to indicate the hygroscopicity of a fertilizer.

In this procedure, the hygroscopicities of fertilizers or blends are compared by imposing various periods of humid exposure on samples contained in completely filled, open-top glass cups. Determinations are made of (1) rate of moisture absorption per unit of exposed surface, (2) depth of moisture penetration (visible wetting), (3) moisture-holding capacity, and (4) integrity of the wetted granules.

### Apparatus

1. **Laboratory Temperature-Humidity Chamber**—With horizontal airflow; approximate internal dimensions of 94 cm wide, 51 cm deep, and 66 cm high. Temperature range from  $-18^{\circ}\text{C}$  to  $+93^{\circ}\text{C}$ . Relative humidity range of 20%-96%.
2. **Sample Cups (Figure 1)**—Cylindrical glass, 6.8 cm in diameter by 20 cm deep.
3. **Balance**—Top-loading, with a minimum 2-kg capacity and a resolution of 0.1 g.

### Sample Preparation

No special preparation is required other than obtaining a representative sample.

### Procedure

A preweighed sample cup is filled level full with the fertilizer to be tested. The filled sample cup is accurately weighed on the balance. The weighed sample is placed in the laboratory temperature-humidity chamber. Standard conditions are 80% relative humidity (RH) and  $30^{\circ}\text{C}$ . However, lower and higher relative humidities and temperatures can be used.

The sample cups are removed from the chamber for weighings and examinations after periods of 3, 7, 24, 48, and 72 h. Determinations are made as follows:

**Moisture Absorption**—The samples are weighed, and the weight gain is calculated and expressed as milligrams/square centimeter of exposed surface.

$$\text{MA, mg/cm}^2 = \frac{\text{FW}-\text{OW}}{\text{ES}} \times 1,000$$

where,

MA = moisture absorption,  $\text{mg/cm}^2$

FW = final weight of sample, g

OW = original weight of sample, g

ES = exposed surface,  $\text{cm}^2 = \pi r^2 = 36.3 \text{ cm}^2$

**Moisture Penetration**—The depth of moisture penetration is measured in centimeters by observation through the glass walls of the cup. The moist portion of the fertilizer usually is readily distinguishable by its difference in color. Check analyses of various strata in the cups have shown that ordinarily there is essentially no moisture penetration below the visibly affected layer; therefore, the visual observations can be taken as a reliable measure of depth of moisture penetration.

**Moisture Holding Capacity**—The moisture-holding capacity is calculated from the previously accumulated absorption and penetration data as follows:

$$\text{MHC, mg/cm}^3 = \frac{\text{MA}}{\text{MP}}$$

where,

MHC, mg/cm<sup>3</sup> = moisture-holding capacity, mg/cm<sup>3</sup>.

MA = moisture absorption, mg/cm<sup>2</sup>.

MP = moisture penetration, cm.

then,

$$\text{MHC, \%} = \frac{\text{MHC, mg/cm}^3}{(\text{OW/V}) \times 1,000} \times 100$$

where,

MHC, % = moisture-holding capacity, %.

MHC, mg/cm<sup>3</sup> = moisture-holding capacity, mg/cm<sup>3</sup>.

OW = original weight of sample, g.

V = sample cup volume, cm<sup>3</sup>.

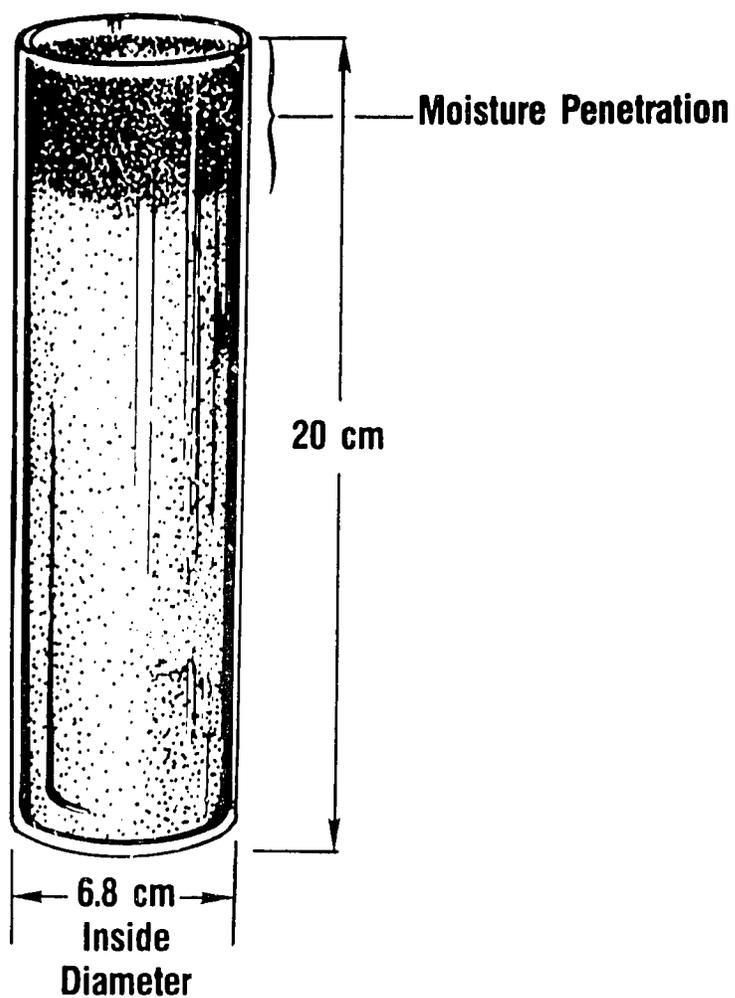
**Integrity of the Wetted Granules**—The physical condition of the wetted portion of the sample is evaluated by pressing granules between the fingers. The granule integrity is rated as excellent, good, fair, or poor. This test is carried out on a second, duplicate cup of fertilizer to avoid interference with weight determinations.

## Typical Values

Fertilizer		72 Hours Exposure at 30°C, 80% RH					Granule Integrity of Wetted Area
		Moisture Absorption	Moisture Penetration	Moisture-Holding Capacity			
Type	Grade	(mg/cm <sup>2</sup> )	(cm)	mg/cm <sup>3</sup>	%		
Prilled urea	46-0-0	350	15.0	23	3.0	Fair	
Granular urea	46-0-0	350	15.0	23	3.0	Good	
Granular ammonium sulfate	21-0-0	94	0	-	-	Excellent	
Prilled ammonium nitrate	34-0-0	1,000 <sup>a</sup>	50 <sup>a</sup>	20	2.2	Poor	
Granular diammonium phosphate	18-46-0	175	1.5	117	11.7	Excellent	
Granular mono-ammonium phosphate	11-55-0	90	1.0	90	9.0	Excellent	
Granular triple superphosphate	0-46-0	235	1.8	131	11.7	Excellent	
Granular potassium chloride	0-0-60	135	2.4	56	5.4	Good	

a. Values obtained by extrapolation.

(TVA Method, Modified)



**Figure 1. Glass Cylinder Used in Measuring Moisture Absorption and Penetration in a Fertilizer.**

## Critical Relative Humidity

Critical relative humidity (CRH) is that humidity of the atmosphere above which a material will absorb a significant amount of moisture and below which it will not.

For each fertilizer compound or mixture, there is a maximum relative humidity to which the fertilizer can be exposed without absorbing moisture from the air. Determination of this value is necessary when controlled-humidity storage areas are being designed for a material. The value is of interest also as an indication of the degree of protection that is likely to be required during handling. In the case of mixtures, determination of CRH is one check on compatibility; some mixtures have an intolerably low CRH, much lower than the CRH of either component alone.

The procedure for determination of CRH involves exposure of small fertilizer samples to progressively higher relative humidities in a variable-humidity chamber. The lowest humidity that initiates significant absorption of moisture is determined by frequent weighings of the sample; this is taken to be the CRH.

### Apparatus

1. **Laboratory Temperature-Humidity Chamber**—With horizontal airflow; approximate internal dimensions of 94 cm wide, 51 cm deep, and 66 cm high. Temperature range from -18°C to +93°C. Relative humidity range of 20%-96%.
2. **Aluminum Cups**—Approximately 5 cm in diameter by 0.6 cm deep with a tight-sealing lid.
3. **Balance**—Analytical with a minimum 100-g capacity and a resolution of 0.0001 g.

### Sample Preparation

Samples usually are exposed "as received" without any special treatment. Parallel tests are made with crushed granules (minus 850  $\mu$ m) if it appears that granule surfaces may be "case hardened" or of nonhomogeneous composition.

### Procedure

A 2- to 4-g sample is placed in a preweighed aluminum cup. The cup with contents is accurately weighed on the analytical balance. The weighed sample, in a tightly covered sample cup, is preheated to the dry-bulb temperature of 30°C.

This preheating precludes condensation of moisture on the sample, which might occur if a cold sample were exposed to a high humidity. After heating, the cover is removed from the sample cup and exposure is begun. The humidity for this first exposure usually is chosen to be about 10 percentage points below the expected critical humidity of the sample. Exposure is continued for 3 h, with weighings of the sample on an analytical balance at the end of each hour. The remainder of the procedure, outlined below, depends on whether or not the sample absorbed moisture progressively during this first exposure.

1. **If Sample Did Not Exhibit Progressive Absorption of Moisture**—The humidity in the cabinet was below the critical humidity of the material. Therefore, humidity in the cabinet is raised by five percentage points for the next exposure period and the sample is reexposed an additional 3 h as described above.
2. **If the Sample Exhibited Progressive Absorption of Moisture**—The humidity in the cabinet was above the critical humidity of the material. Therefore, humidity in the cabinet is lowered by five percentage points for the next exposure period and a fresh sample is exposed.

By repetition of the above general procedure, the critical humidity of pure materials usually can be established within about  $\pm 5\%$  relative humidity. Exposure times less than 3 h can be used, but the degree of confidence is reduced somewhat.

In Figure 1 moisture absorption data obtained in tests of two products are plotted versus time of exposure. The data for the oil-prilled urea illustrate behavior typical of relatively pure fertilizer salts. There was a definite absence of moisture absorption at 70% relative humidity and a definitely sustained absorption at 75% relative humidity; thus, the CRH of this product lies between 70% and 75%. Data for an 18-46-0 grade diammonium phosphate are plotted also in the figure; these are typical of data obtained with fertilizers that contain minor amounts of soluble impurities in addition to the major soluble phases. The CRH of such a material is less definitely defined than that of a pure salt. From the figure it is evident that 65% relative humidity was below the CRH of this material, since only an insignificant amount of moisture was absorbed in 3 h. Relative humidity of 75%, on the other hand, was definitely above the critical since moisture absorption was large and continuous throughout the 3-h period. Relative humidity of 70%, however, was intermediate in its effect on the sample, since it caused absorption of about 2% moisture during the first 2 h but no further absorption in the third hour. The "practical value" for the CRH of such a product must be decided on the basis of the use to which the value is to be put. For example, in a storage area from which material is to be bagged, it would be desirable to keep the relative humidity at 65% or lower, since absorption of even 2% moisture would result in caking in bags. On the other hand, exposure to 70% relative humidity and consequent absorption of 2% moisture might be tolerable in a bulk-handling operation where the material is to be used immediately. Exposure to relative humidity of 75% or above would cause rapid moisture absorption and wetting in almost any storage or handling operation.

### Typical Values

Fertilizer		Critical Relative Humidity Range at 30°C, %
Type	Grade	
Prilled urea	46-0-0	70-75
Granular urea	46-0-0	70-75
Granular ammonium sulfate	21-0-0	75-85
Prilled ammonium nitrate	34-0-0	60-65
Granular diammonium phosphate	18-46-0	70-75
Granular monoammonium phosphate	11-55-0	70-80
Granular triple superphosphate	0-46-0	75-85
Granular potassium chloride	0-0-60	70-80
Granular potassium sulfate	0-0-50	80-85

(TVA Method)

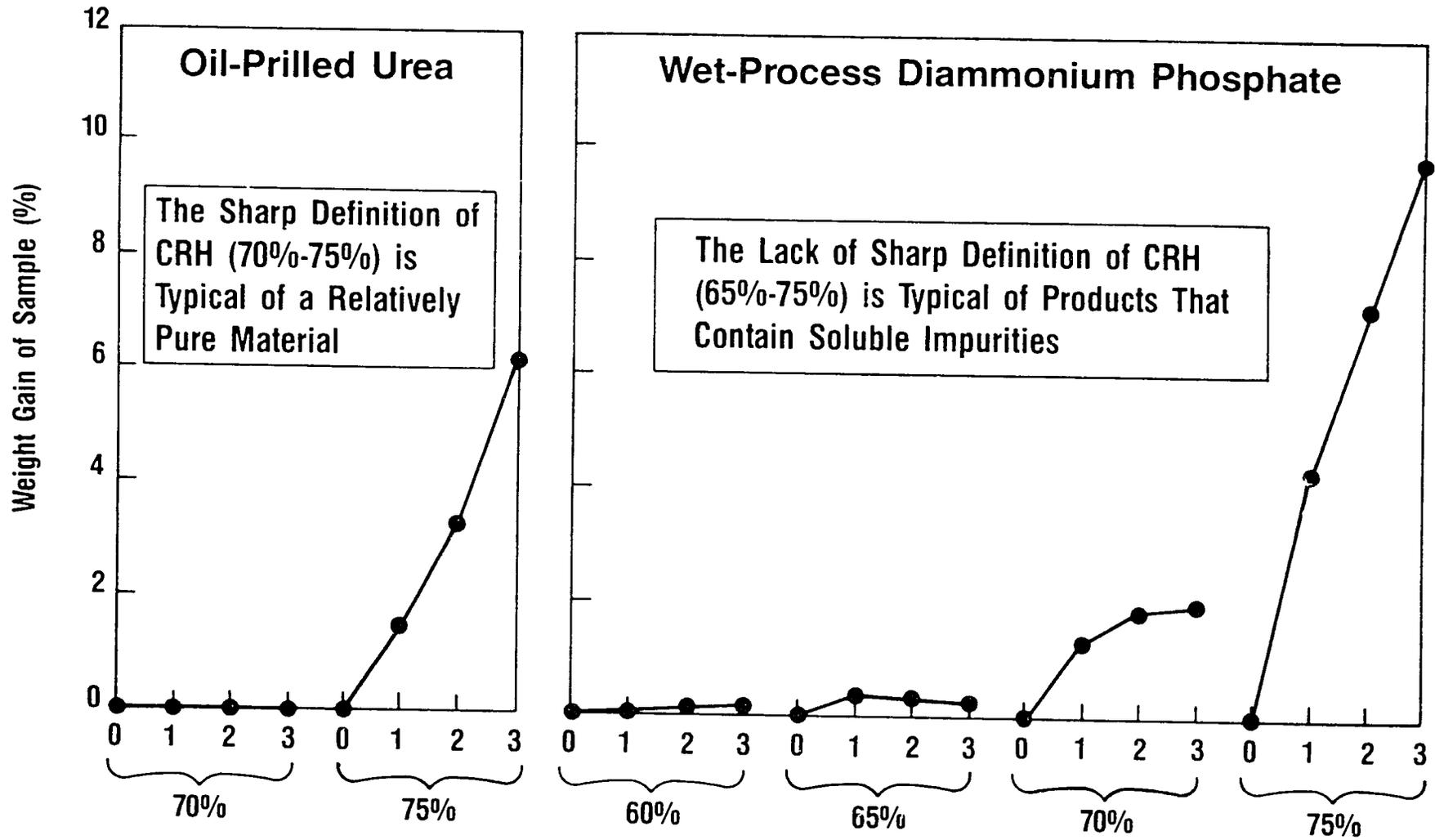


Figure 1. Hours of Exposure at Indicated Relative Humidity (30°C).

## Flowability

Flowability is the ability of a material to remain flowable under humid conditions. Flowability is important when considering the movement of material in conveyor systems and fertilizer applicators.

### Apparatus

1. **Rotary Drum (Figure 1)**—25 cm in diameter by 15 cm deep, inclined (20°); equipped with shallow (1-cm) removable lifting flights. On the back wall of the 20°-inclined drum, four concentric circles are etched. The first should be etched at the level that a 500-cm<sup>3</sup> sample will assume. The other three circles should be etched at the level that 375-, 250-, and 125-cm<sup>3</sup> samples will assume.
2. **Motor Drive Assembly**—With variable-speed controller.
3. **Laboratory Temperature-Humidity Chamber**—With horizontal airflow; approximate internal dimensions of 94 cm wide, 51 cm deep, and 66 cm high. Temperature range from -18°C to +93°C. Relative humidity (RH) range of 20%-96%.

### Sample Preparation

No special preparation is required other than obtaining a representative sample.

### Procedure

A 500-cm<sup>3</sup> sample of the test fertilizer is first placed in the inclined (20°) rotary drum. The rotary drum (with motor drive assembly) is placed in the laboratory temperature-humidity chamber and positioned such that the horizontal airflow is directed into the open face of the drum at an angle of 45°. The variable-speed controller is kept outside the chamber and connected to the motor drive assembly by means of a 110-volt line run through a port on the side of the chamber.

The laboratory temperature-humidity chamber has been preset to 90% relative humidity at 30°C. The drum is then rotated at 12 rpm, and measurement is made of the time during which the material remains free flowing.

As the fertilizer becomes moist, it will adhere to the sides of the drum. Free-flow times are measured as the times required for the material to become 25%, 50%, and 75% nonflowable. This determination is made by noting the elapsed times as the 375-cm<sup>3</sup> (25% nonflowable), 250-cm<sup>3</sup> (50% nonflowable), and 125-cm<sup>3</sup> (75% nonflowable) marks become visible above the sample line.

### Typical Values

Fertilizer		Flowability (Minutes at 30°C and 90% RH)		
Type	Grade	25% Nonflowable	50% Nonflowable	75% Nonflowable
Prilled urea	46-0-0	5	10	15
Granular urea	46-0-0	5	11	17
Granular ammonium sulfate	21-0-0	175	220	280
Prilled ammonium nitrate	34-0-0	7	12	16
Granular diammonium phosphate	18-46-0	70	130	180
Granular monoammonium phosphate	11-55-0	80	165	190
Granular triple superphosphate	0-46-0	180	240	300
Granular urea-based NPK	19-19-19	10	20	30
Granular ammonium sulfate-based NPK	8-24-8	90	130	155
Granular ammonium nitrate-based NPK-MgO	12-6-22-3	37	43	48
Granular urea-based NPK-MgO	12-6-22-3	31	38	40

(TVA Method, Modified)

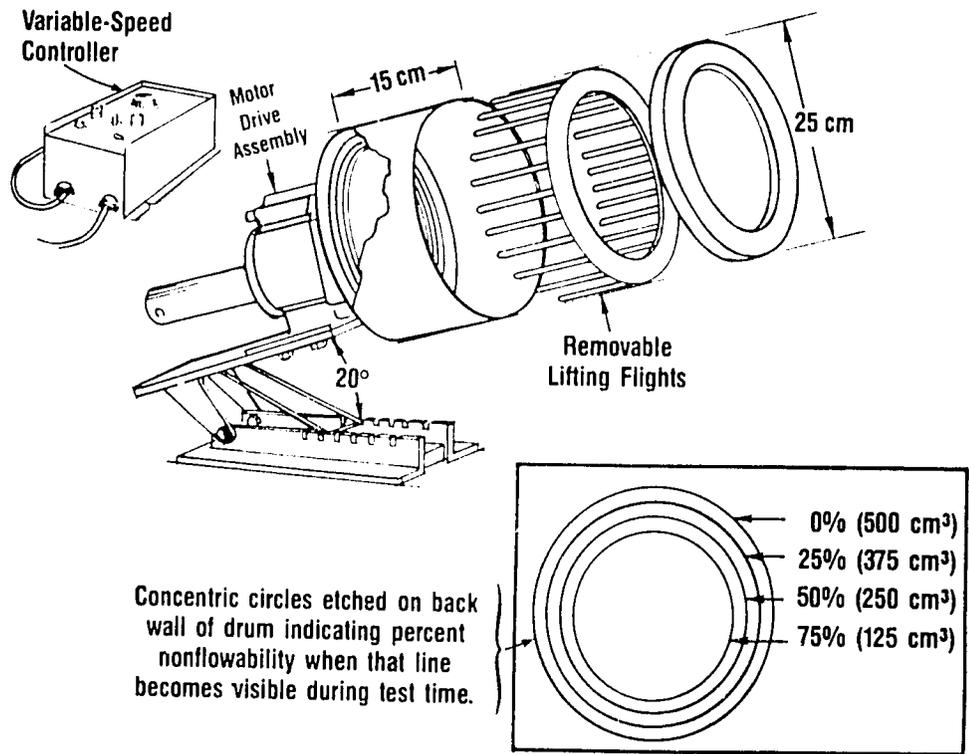


Figure 1. Rotary Drum for Measuring Flowability of Fertilizer When Exposed to Humid Conditions.

## Moisture Penetration Test

This test is devised to determine whether (1) moisture continues to penetrate a material for an extended period of time under cycling relative humidity conditions or (2) a crust forms on the material that effectively stops moisture penetration. Moisture penetration into fertilizers is important when considering (1) conditions at which a bulk pile can be stored and (2) material flowability during handling and field application.

- Apparatus**
1. **Plastic Cylinder (Figure 1)**—Clear, approximately 20 cm inside diameter by 150 cm high.
  2. **Controlled Temperature-Humidity Chamber**—With horizontal airflow, internal dimensions of approximately 4.0 m wide, 5.5 m deep, and 4.0 m high. Temperature range from 5°C to 40°C. Relative humidity (RH) range of 45%-98%.

**Sample Preparation** No special preparation is required other than obtaining a representative sample.

**Procedure** The plastic cylinder is filled with the sample, leveled with a straight edge, and tapped several times on the side. The void created by tapping is then filled with the sample and leveled with a straight edge. This is repeated until a void is no longer created by tapping. The approximate sample quantity required is 50 kg.

The sample is then subjected to a constant temperature (30°C) but cycling relative humidities (12 h at 68% followed by 12 h at 93%) for a period of 60 days.

The depth of moisture penetration is measured in centimeters by observation through the clear wall of the cylinder. The results are also graphed to illustrate moisture penetration versus time (days). Additionally, granule integrity is determined qualitatively by handling the surface portion of the sample after exposure for 60 days and rating the granule strength as excellent, good, fair, or poor.

### Typical Values

Fertilizer		60 Days Exposure at 30°C and 12 h Cycles of 68% and 93% RH	
Type	Grade	Moisture Penetration	Granule Integrity of Wetted Area
		(cm)	
NP granulated mixture	28-28-0	12	Poor
NP bulk blend	28-28-0	43	Good
NPK granulated mixture	17-17-17	26	Poor
NPK bulk blend	17-17-17	45	Good

(IFDC Method)

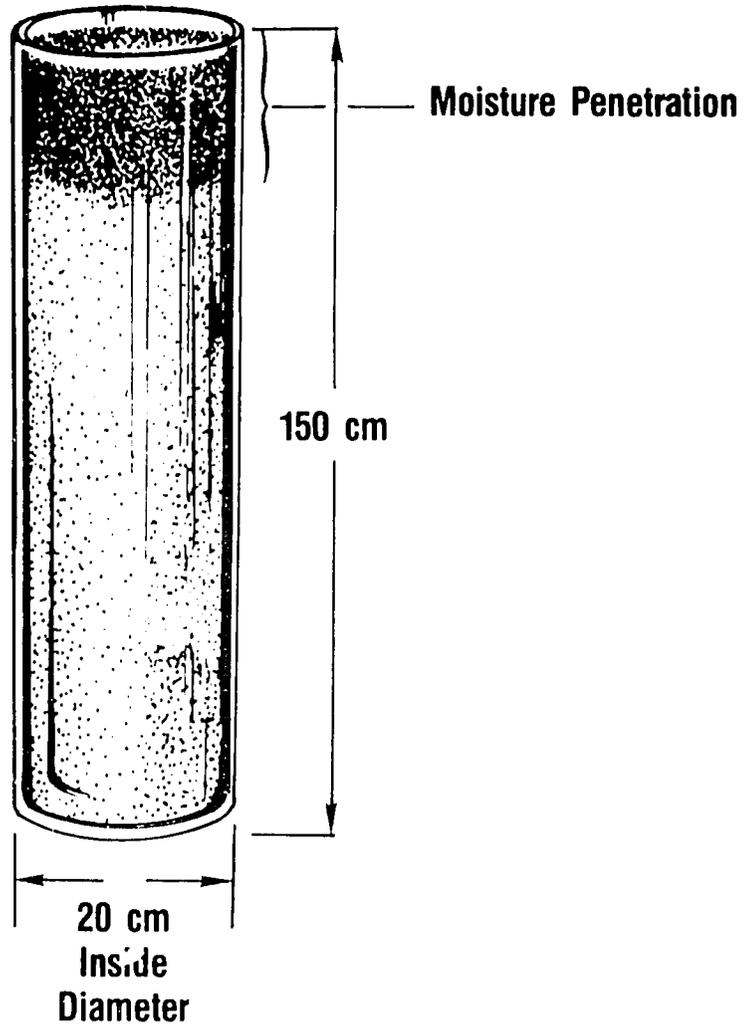


Figure 1. Plastic Cylinder Used in Measuring Moisture Penetration in a Fertilizer.

## Chemical Compatibility in Blends

Chemical compatibility in blends is the ability of two or more materials to remain dry and free flowing when blended together. Incompatibility is evidenced by wetting, caking, gas evolution, and/or particle disintegration. Compatibility of materials is important in any bulk-blending or NPK granulation system.

### Apparatus

1. **Glass Bottle**—200-mL capacity.
2. **Oven**—With a minimum temperature range from 25°C to 50°C.

### Sample Preparation

The ingredients that are to constitute the test blend are combined and blended such that the total sample will occupy about two-thirds of the glass bottle.

### Procedure

The freshly made blend sample is tightly capped in the glass bottle and placed in the oven at 30°C for 30 days. The storage temperature and period may be changed as deemed necessary. The blend is inspected daily, and any wetting, caking, disintegration, or gas evolution is noted. General guidelines for rating compatibility are as follows:

Visual Observation	Rating
Dry and free flowing	Compatible
Damp, but still free flowing	Predominantly compatible
Damp and nonflowing	Predominantly incompatible
Wet and nonflowing	Incompatible

### Typical Values

The chemical compatibility of some common fertilizers is shown in Figure 1.

(TVA Method, Modified)



## Caking Tendency (Large-Bag Method)

Caking tendency is the tendency of a fertilizer to agglomerate or lump in either bulk or bag storage and is affected by one or more of the following: (1) moisture content, (2) particle size, (3) particle hardness, (4) presence of conditioners, (5) storage temperature, (6) storage pressure, (7) storage time, (8) curing time, and (9) material composition. Excessive caking can cause problems in handling and field application.

- Apparatus**
1. **Controlled Temperature Chamber**—With horizontal airflow, internal dimensions of approximately 4.0 m wide, 5.5 m deep, and 4.0 m high. Temperature range from 5°C to 40°C.
  2. **Balance**—Platform, with a minimum 100-kg capacity and a resolution of 0.1 kg.
  3. **Bags**—Woven polypropylene, with a 0.1-mm (4-mil) polyethylene liner.
  4. **Sewing Machine**—Commercial hand-held type.
  5. **Sieve**—With 12.5-mm (0.5-in) openings, approximately 30 cm wide by 60 cm long.
  6. **"Dummy" Weights**—Capable of applying a pressure of 0.28 kg/cm<sup>2</sup> to the test samples (pressure approximately equivalent to that at the bottom of a 20-bag stack of 22.7-kg (50-lb) bags of most fertilizers).
  7. **Plywood Board**—With 10- to 15-mm thickness. Large enough to cover the total test surface.

**Sample Preparation** No special preparation is required other than obtaining a representative sample.

**Procedure** A layer of "dummy" bags (dimension of layer large enough to accommodate all the test bags) is placed on the floor of a room where the temperature is maintained at 30°C.

Since test materials differ in bulk density, the weight of material used in bagging is adjusted to provide filling of the bags to their design volume.

The bagged test samples are stacked on top of the layer of "dummy" bags. The bag dimensions are measured for each test sample. A 10- to 15-mm thick plywood board is placed on top of the test samples (dimensions large enough to cover all test bags). The total test surface area is determined as the sum of the contact areas of the bags with the plywood board.

A deadweight pressure of 0.28 kg/cm<sup>2</sup> is applied by placing "dummy" weights on top of the plywood. The amount of "dummy" weight is determined by multiplying the surface area of the test layer by 0.28 kg/cm<sup>2</sup>. This pressure is equivalent to that placed on the bottom bag of a 20-bag stack of 22.7-kg bags.

The standard procedure considers 1-, 3-, and 6-month storage periods. Other storage periods may be used. Test samples are inspected as follows:

1. "Dummy" weights are removed, and test bags are inspected for bag set without moving them from their original position. The inspector feels the bags and records bag set as none, light, medium, or hard.
2. The test bags then are dropped once (on thin side) from a 1-m height, and the material is screened very gently to determine the percentage of plus 12.5-mm lumps.
3. Hardness of lumps is rated by crushing in the hand. Ratings of light, medium, or hard are given. Light lumps are those that can be broken easily with the hand; medium lumps are those that can be broken when pressed against a hard

surface with the hand; and hard lumps are those that cannot be broken without using mechanical means (crusher, grinder, hammer, mallet, etc.).

### **Evaluation**

It is not possible to give a universal criterion as to what degree of caking is tolerable because requirements differ widely in different areas and for different uses. Where machine application is employed, complete freedom from lumps is highly desirable; however, a small percentage (up to perhaps 15%) of only light lumps may be acceptable. For hand application, where labor is available and each bag can receive individual treatment, a higher percentage of lumps and the presence of lumps of medium hardness may be tolerated.

(TVA Method, Modified)

## Caking Tendency (Small-Bag Method)

Caking tendency is the tendency of a fertilizer to agglomerate or lump in either bulk or bag storage and is affected by one or more of the following: (1) moisture content, (2) particle size, (3) particle hardness, (4) conditioners, (5) storage temperature, (6) storage pressure, (7) storage time, (8) curing time, and (9) material composition. Excessive caking can cause problems in handling and field application.

### Apparatus

1. **Controlled Temperature Chamber**—With horizontal airflow, internal dimensions of approximately 4.0 m wide, 5.5 m deep, and 4.0 m high. Temperature range from 5°C to 40°C.
2. **Graduated Cylinder**—With 2,000-mL capacity.
3. **Bags**—A 0.05-mm (2-mil) polyethylene inner liner, two 1-ply center paper bags, and a 0.1-mm (4-mil) polypropylene outer bag. Filled dimensions are approximately 9 cm thick by 18 cm long by 12 cm wide.
4. **Sieve**—With 12.5-mm (0.5-in) openings, approximately 30 cm wide by 60 cm long.
5. **Storage Rack (Figure 1)**—With slots tall enough to hold 10-12 test bags and a 60-kg "dummy" weight. The width and depth of the slots should be just large enough to hold bags.
6. **Hoist**—Portable or overhead, capable of lifting "dummy" weights for insertion and removal of test bags.
7. **"Dummy" Weights**—Capable of applying a pressure of 0.28 kg/cm<sup>2</sup> to the test samples (pressure approximately equivalent to that at the bottom of a 20-bag stack of 22.7-kg (50-lb) bags of most fertilizers). With the bag dimensions specified, a "dummy" weight of 60 kg is required.
8. **Plywood Boards**—Thickness between 10 and 15 mm, 20 cm long by 15 cm wide.

### Sample Preparation

No special preparation is required other than obtaining a representative sample.

### Procedure

The bags are charged with 1,800 cm<sup>3</sup> of the test sample. The inner liner and paper bags are folded and securely taped. The outer polypropylene bag is then sealed.

The filled test bags are placed in the storage racks inside the controlled temperature chamber at 30°C. A 10- to 15-mm thick plywood board is placed on top of the test bags. A deadweight pressure of 0.28 kg/cm<sup>2</sup> is applied by placing a "dummy" weight on top of the plywood board.

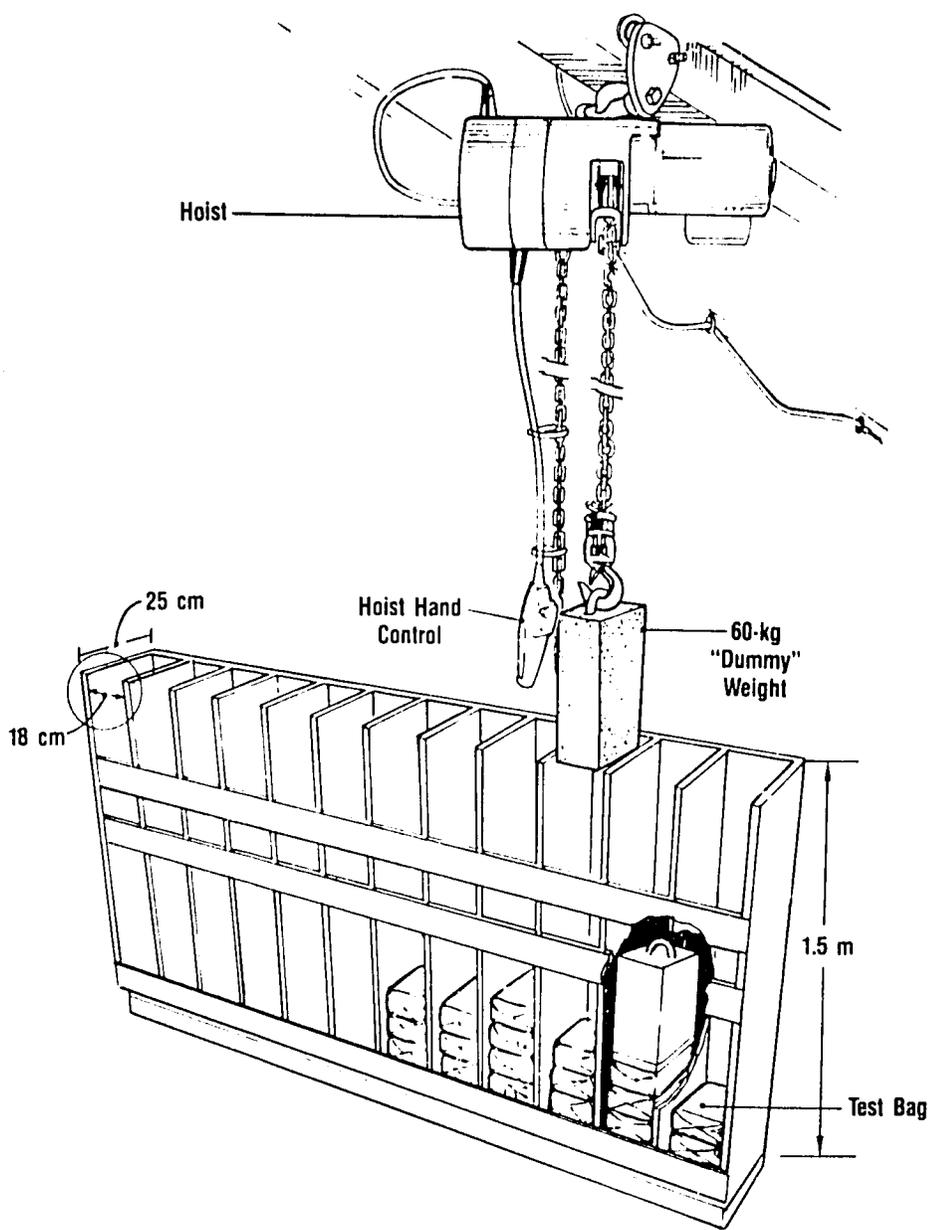
The standard procedure considers 1-, 3-, and 6-month storage periods. Other storage periods may be used. The test samples are inspected as follows:

1. "Dummy" weights are removed, and test bags are inspected for bag set without moving them from their original position. The inspector feels the bags and records bag set as none, light, medium, or hard.
2. The test bags then are dropped twice (once on each thin side) from a 1-m height, and the material is screened very gently to determine the percentage of plus 12.5-mm lumps.
3. Hardness of lumps is rated by crushing in the hand. Ratings of light, medium, or hard are given. Light lumps are those that can be broken easily with the hand; medium lumps are those that can be broken when pressed against a hard surface with the hand; and hard lumps are those that cannot be broken without using mechanical means (crusher, grinder, hammer, mallet, etc.).

**Evaluation**

It is not possible to give a universal criterion as to what degree of caking is tolerable since requirements differ widely in different areas and for different uses. Where machine application is employed, complete freedom from lumps is highly desirable, but a small percentage (up to perhaps 15%) of only light lumps may be acceptable. For hand application, where labor is available and each bag can receive individual treatment, a higher percentage of lumps and the presence of lumps of medium hardness may be considered tolerable.

(TVA Method, Modified)



**Figure 1. Storage Rack to Measure Caking Tendency of Fertilizer Using Small-Bag Method.**

## Size Analysis (Sieve Method)

Particle size of fertilizer products and/or fertilizer raw materials is defined as the particle diameter ranges of the test material. Particle size affects agronomic response; granulation techniques; and storage, handling, and blending properties. These procedures can be used to determine size distributions of materials ranging in size from 25.0 mm to 38  $\mu\text{m}$ . Only water-insoluble materials may be tested in Procedure 2.

### Apparatus

1. **Test Sieves**—20 cm in diameter and 5 cm high with sieve covers and sieve pans. Openings for test sieves range from 25.0 mm to 38  $\mu\text{m}$  (Table 1).
2. **Sieve Shakers**—Dry apparatus (Tyler Model B Ro-Tap<sup>®</sup> or equivalent) for Procedure 1 and wet apparatus (Combs Gyrotory Type HL sifting machine or equivalent) for Procedure 2.
3. **Balance**—Top loading with a minimum 5.0-kg capacity and a resolution of 0.1 g.
4. **Weighing Pan**—Suitable for weighing samples.
5. **Brush**—Either soft, fine wire, or stiff bristle.
6. **Oven**—Capable of attaining a temperature of 105°C (for Procedure 2).

### Sample Preparation

No special preparation is required other than obtaining a representative sample.

### Sample Quantity Requirements

The sample quantity used is dependent upon the type of material, the particle sizes, and the particle-size distribution. The general rule in determining the size of a sample is that it be limited in weight so that no sieve in the series used in the analysis be overloaded. Overloading of the sieves results in blinding of the screen meshes. Generally, the larger the mean particle size the larger the sample required for accuracy and, conversely, the finer the mean particle size the smaller the sample required. Table 2 gives a good guide for selecting the appropriate sample quantity.

### Sieve Selection and Arrangement

Choose a series of sieves of such sizes that the desired data may be obtained (Table 1). The selection is normally made so that, if possible, no one sieve retains more than 50% of the entire sample, no more than 10% of the sample remains on the top sieve, and no more than 10% of the sample passes the bottom sieve.

Nest the selected sieves in order of decreasing aperture size with the largest aperture sieve on the top.

### Procedure 1 (Dry Sieving: Material Predominantly > 150 $\mu\text{m}$ )

Place sieve pan on the bottom of the nest. Riffle, weigh, and transfer a sample onto the top sieve and place a sieve cover on top of the nest. Place the nest of sieves in the dry-sieve shaker, adjust height of nest holder for number of screens used, secure, and shake for 5 min. After completion of shaking period, transfer material from each sieve to weighing pan with brush and weigh to  $\pm 0.1$  g. Record weight from each sieve. Calculate results for each sieve as follows:

$$\% \text{ on sieve} = \frac{\text{Weight on sieve, g}}{\text{Weight of sample, g}}$$

**Procedure 2 (Wet Sieving: Material Predominantly < 150 μm)**

Riffle, weigh, and transfer a sample onto the top sieve, and place a fitted sieve cover on top of the nest. Place the nest of sieves in the wet-sieve shaker, adjust height of nest holder for number of screens used, secure, attach water hose to fitted sieve cover, start water (approximately 0.5 L/min), and shake for 20 min. After completion of the shaking period, dry material for 1 h at 105°C. Transfer material from each sieve to weighing pan with brush and weigh to ±0.1 g. Record weight from each sieve. The material quantity that passes through the bottom sieve with the wash water is considered to be the difference between the original sample weight and the total weight on the individual sieves. Calculate results for each sieve as follows:

$$\% \text{ on sieve} = \frac{\text{Weight on sieve, g}}{\text{Weight of sample, g}}$$

**Typical Values**

Type	Grade	Cumulative % Retained on Indicated Sieve Openings, mm					
		4.75	3.35	2.36	1.70	1.18	0.85
Prilled urea	46-0-0	0	1	4	48	92	100
Granular urea	46-0-0	0	2	65	90	98	100
Prilled ammonium nitrate	34-0-0	0	0	1	74	98	100
Granular diammonium phosphate	18-46-0	0	0	22	79	96	99
Granular monoammonium phosphate	11-55-0	0	1	30	81	97	99
Granular triple superphosphate	0-46-0	0	0	22	79	96	99
"Coarse" potassium chloride	0-0-60	0	0	16	45	80	95
"Granular" potassium chloride	00-60	0	5	38	75	93	99

(IFDC Method)

**Table 1. Fourth Root of Two Series of Sieve Openings<sup>a</sup>**

<b>Size</b>	
25.0	mm
22.4	mm
19.0	mm
16.0	mm
13.2	mm
12.5	mm
11.2	mm
9.5	mm
8.0	mm
6.7	mm
6.3	mm
5.6	mm
4.75	mm
4.00	mm
3.35	mm
2.80	mm
2.36	mm
2.00	mm
1.70	mm
1.40	mm
1.18	mm
1.00	mm
850	μm
710	μm
600	μm
500	μm
425	μm
355	μm
300	μm
250	μm
212	μm
180	μm
150	μm
125	μm
106	μm
90	μm
75	μm
63	μm
53	μm
45	μm
38	μm

a. Taken from Annual Book of American Society for Testing and Materials (ASTM), Section 14, General Methods and Instrumentation, *Standard Specification for Wire-Cloth Sieves for Testing Purposes*, Designation E11-81.

**Table 2. Quantity of Material Required for Test Sieving<sup>a</sup>**

Screen Opening	Recommended Quantity (g)
25.0 mm	1,600
22.4 mm	1,600
19.0 mm	1,600
16.0 mm	800
13.2 mm	800
12.5 mm	800
11.2 mm	800
9.5 mm	800
8.0 mm	500
6.7 mm	500
6.3 mm	450
5.6 mm	400
4.75 mm	400
4.00 mm	350
3.35 mm	350
2.80 mm	240
2.36 mm	240
2.00 mm	200
1.70 mm	200
1.40 mm	160
1.18 mm	160
1.00 mm	140
850 μm	140
710 μm	120
600 μm	120
500 μm	100
425 μm	100
355 μm	80
300 μm	80
250 μm	70
212 μm	70
180 μm	60
150 μm	60
125 μm	50
106 μm	50
90 μm	40
75 μm	40
63 μm	35
53 μm	35
45 μm	30
38 μm	30

a. The quantity should be that indicated for the sieve corresponding to the dominant size fraction of the sample.

## Size Analysis (Cyclone Method)

Particle size of fertilizer raw materials is defined as the particle diameter ranges of the test material. Particle size of fertilizer raw materials affects granulation techniques, handling properties, and beneficiation efficiency. This procedure is useful in determining size distribution of water-insoluble material in the size range of 10-40  $\mu\text{m}$ .

### Apparatus

1. **Warman Cyclosizer<sup>®</sup>** (Figures 1 and 2)—Consisting of a console cabinet, five cyclones mounted in series, sample container assembly, rotameter, pressure gauge, thermometer, electrical control panel, pump, and water tank (Figures 1 and 2).
2. **Oven**—Capable of attaining a temperature of 105°C.
3. **Balance**—Top loading, with a minimum 150-g capacity and a resolution of 0.1 g.
4. **Beakers**—Two 250-mL capacity, one 3-L capacity, and five 1,000-mL capacity.
5. **Wetting Agent**—Commercial detergent or equivalent.
6. **Test Sieves**—Two, 20 cm in diameter by 5 cm high with openings of 53 and 75  $\mu\text{m}$ , a sieve cover, and sieve pan.
7. **Combs Gyrotory Type HL Sifting Machine**—Or equivalent wet-sieving apparatus.
8. **Air-Comparison Pycnometer**—Beckman Model 930 or equivalent for determination of true density of solids (see IFDC S-114).

### General Description and Method of Operation

The cyclones are mounted with the apex "vertically up" and are arranged in a series such that the vortex outlet of each cyclone is the feed for the next in line. The vortex outlet from the final cyclone discharges to the drain manifold. The apex of each cyclone opens out into a small cylindrical chamber with a discharge valve. This valve is kept closed during the test, and therefore the apex discharge of the cyclone is, in effect, closed. A flow control valve is provided as an integral part of the sample container assembly, and water pumped from the tank through the five cyclones is manually controlled at a specified flowrate as indicated on the rotameter.

To determine the size distribution of a sample, a known weight is slurried with water and transferred to the sample container, which is removed from the holder for this purpose. After the container is loaded with the sample, the sample container valve is closed, effectively sealing the sample within the container, which is then returned to the holder. Water is pumped through the circuit, and the sample is bled into the water stream by slowly opening the sample container valve. This valve is adjusted so that the whole of the sample is released over a period of approximately 5 min, during which time the flowrate is maintained at maximum and there is a preliminary distribution of solids to the cyclones.

The initial distribution of the sample is an approximate size separation, with each cyclone and apex chamber containing an excess of undersize material. Controlled elutriation is then effected by reducing the water flow to a predetermined rate and holding it constant for a specified time while particles smaller than the limiting particle separation size of each cyclone are gradually elutriated to the vortex outlet. Solids smaller than the limiting size of the final cyclone pass out with the waste water.

After the elutriation time has elapsed, the water flow is increased again; as soon as practical thereafter, the solids that have collected in the five cyclones are discharged into separate beakers by opening the cyclone apex valves. The solids are

settled, the water is decanted, and the solids are dried and weighed. The weight passing the final cyclone is determined by difference, and the effective separating sizes of the five cyclones are calculated from the specified limiting sizes and the correction factors for the actual levels of the operating variables.

### Subdivision of the Gross Sample

For a sizing analysis using the Warman cyclosizer, the amount of subdivision required depends on the size distribution of the sample. The weight of the test sample used for the determination should be such that not more than 15 g collects in any one cyclone. To determine an accurate maximum weight for a certain sample, a preliminary sizing may be run. In every case the final sample should be true "split" weight and not a weight adjusted to a set figure by adding or removing small portions.

In the normal course of determining size distributions, one must deal with gross samples in both the wet and dry states. Where samples are received wet, it is strongly advised that they be reduced to the test sample and sized without being dried, with the dry weight of the test sample being calculated by determining the moisture content of a duplicate sample. For the reduction of a gross sample of dry powdered material to a test sample of suitable size, riffle splitting is usually satisfactory.

Techniques for the subdivision of gross samples of slurry are not so well developed as those for dry powders. There are three methods in general use:

1. Wet riffling.
2. Filtration followed by mixing and splitting the filter cake.
3. Withdrawing samples from the slurry while it is being agitated.

All methods, when carefully applied, will usually give satisfactory results.

### Preparation of the Test Sample

The preparation of dry test samples is conveniently handled in a 250-mL beaker. The samples are pulped with 50-150 mL of water to which a little wetting agent has been added. The choice of wetting agent may vary with the type of material, but the usual commercial detergents have been found satisfactory. The pulped sample should be hand-stirred to ensure wetting of the particles.

Wet samples in the form of filter cake, as distinct from slurry, are pulped as described for the dry samples. Slurries need no preparation unless they contain oversize material.

The Warman cyclosizer is designed to operate on material in the subsieve range; all samples should be screened (according to IFDC S-107) on a 75- $\mu\text{m}$  sieve for materials with a true density of 4.0 g/cm<sup>3</sup> or lower and on a 53- $\mu\text{m}$  sieve for materials with a true density higher than 4.0 g/cm<sup>3</sup> (IFDC S-114). After the test sample is pulped, as described above, it is wet screened on the appropriate mesh, and all undersize is washed through into a 3-L beaker (IFDC S-107). The washings are allowed to settle for 1 h, and the sides are occasionally tapped during this time. After settling, the liquor is carefully syphoned or decanted to waste until the volume remaining is approximately 200 mL. (Any solids still in suspension after 1 h will be less than 10  $\mu\text{m}$  and can be safely discarded.) The settled solids can then be transferred to the sample container for the sizing analysis.

### Test Procedure

When the sample has been prepared, select an elutriating flowrate and, by referring to the flowrate correction factor chart (see Figure 6), determine the millimeter reading of the rotameter corresponding to this selected value. Then proceed as follows:

1. With the pump "off," remove the sample container from its holder by turning the container until one of the metal sides is facing you and pull straight upward.

2. Open fully the valve on the sample container and empty out any water. Stand it inverted on the handwheel of the valve.
3. Pour the test sample into the container and wash the remaining solids out of the beaker into the container (Figure 3a).
4. Continue to fill the sample container with clean water until the level is about halfway up the outside taper. Screw up the valve of the sample container until it is closed. At this stage the sample should be sealed within the container and all air eliminated.
5. With the sample container valve closed, return the container to the holder on the Warman cyclosizer by a reversal of Step 1 (Figure 3b).  
Note: It is imperative that you ensure that the sample container is correctly fitted in the holder and that a glass side is facing you before proceeding further.
6. Turn on the water supply to the constant head tank and wait until tank is full (i.e., until the float valve has closed).
7. Ensure that the control valve is closed and switch the pump on at the control panel.
8. Open the control valve slowly and allow the air to be expelled from the pipework.
9. Open the control valve fully (Figure 3c).
10. Starting from No. 1 cyclone, bleed the air from the cyclone by opening the apex valve, allowing the air to be expelled, and then closing the apex valve. Repeat this procedure with cyclones No. 2 through No. 5. The last traces of air are sometimes difficult to remove from the No. 3 cyclone and, in such cases, an alternative procedure can be used. Close both the apex valve and the control valve and allow the residual air to collect in the apex chamber; then open both valves fully and the air will be expelled. Since the vortex outlet of the No. 5 cyclone is open to the atmosphere, it is not possible to remove the central air column; thus, a "flash air column" will always be present.
11. With the control valve fully open, set the timer to 5 min and open the sample container valve slowly (Figure 3d).
12. Manually regulate the container valve, so that by the time the alarm sounds, the sample has been completely discharged into the stream. Avoid sudden surges from the sample container.
13. After the 5 min has elapsed, close the control valve until the flowmeter indicates the required elutriating flow (usually 11-12 L/min). Set the timer to the required elutriating time (usually 60 min).
14. When the alarm indicates that the elutriation time has elapsed, cancel the alarm and turn the control valve to full flow.
15. Starting with No. 5 cyclone, pull the plastic tube from the drain manifold, open the apex valve, and discharge the solids from the apex chamber into a 1,000-mL beaker (Figure 3e).
16. Close No. 5 discharge valve and proceed to No. 4 cyclone and so on in turn to the other cyclones. Note the water temperature.
17. Allow the beakers to stand for at least 20 min and then decant the excess water.
18. For final recovery and weighing, filter the sized fractions on a tared paper and dry them, or simply transfer them to evaporating or petri dishes for drying without filtering.
19. Calculate the percentage passing No. 5 cyclone as the difference between the initial weight (calculated by determining the moisture content of a duplicate sample) and the sum of the weights of the separate fractions.

## Calculations

After the weight percentages retained in the five cyclones have been determined, it is necessary to calculate the effective particle separation sizes under the conditions of the test.

To do this, a correction factor must be determined for each of the four variables and multiplied with the limiting particle separation size for each cyclone.

Thus for each cyclone:

$$d_e = d_i \cdot f_1 \cdot f_2 \cdot f_3 \cdot f_4$$

- where  $d_e$  = effective particle separation size.
- $d_i$  = limiting particle separation size.
- $f_1$  = temperature correction factor for the water temperature of the test (Figure 4).
- $f_2$  = true density correction factor for the particle true density (Figure 5).
- $f_3$  = flowrate correction factor for the actual flowrate used (Figure 6).
- $f_4$  = time correction factor corresponding to the time of elutriation (Figure 7).

In actual calculations, it is generally convenient to determine first an overall correction factor by multiplying the four separate factors together and using this figure to correct the limiting particle separation sizes.

**Example of Calculation Procedure**

Under the "standard" conditions, the cyclones are specified to separate as follows:

- Cyclone No. 1—44  $\mu\text{m}$
- No. 2—33  $\mu\text{m}$
- No. 3—23  $\mu\text{m}$
- No. 4—15  $\mu\text{m}$
- No. 5—11  $\mu\text{m}$

These sizes are the limiting particle separation sizes for a water temperature of 20°C, a particle true density of 2.65 g/cm<sup>3</sup>, a water flowrate of 11.6 L/min, and an infinite elutriation time.

Consider a test sizing of phosphate rock under the following conditions: water temperature, 10°C; particle true density, 3.04 g/cm<sup>3</sup>; flowrate, 11.95 L/min; and elutriation time, 60 min.

Then, from the graphs, the correction factors are as follows:

- Temperature . . . . .  $f_1 = 1.14$
- Particle true density . . . .  $f_2 = 0.90$
- Flowrate. . . . .  $f_3 = 0.986$
- Time. . . . .  $f_4 = 0.985$

The overall correction factor is thus  $1.14 \times 0.90 \times 0.986 \times 0.985 = 0.996$ , and therefore the effective separation sizes are as follows:

- Cyclone No. 1:  $44 \times 0.996 = 43.8$
- 2:  $33 \times 0.996 = 32.9$
- 3:  $23 \times 0.996 = 22.9$
- 4:  $15 \times 0.996 = 14.9$
- 5:  $11 \times 0.996 = 11.0$

For improved accuracy, the calculations may be based on the calibration certificate supplied with the equipment. This procedure is demonstrated in the illustration of the Warman Cyclosizer Result Sheet (Figure 8), on which calculations have been made on the basis of the calibration data shown in the lower left-hand corner of the sheet.

**Auxiliary Method**

1. True Density of Solids—IFDC S-114.
2. Size Analysis (Sieve Method)—IFDC S-107.

(Reprint from Warman Cyclosizer Manual; Modified)

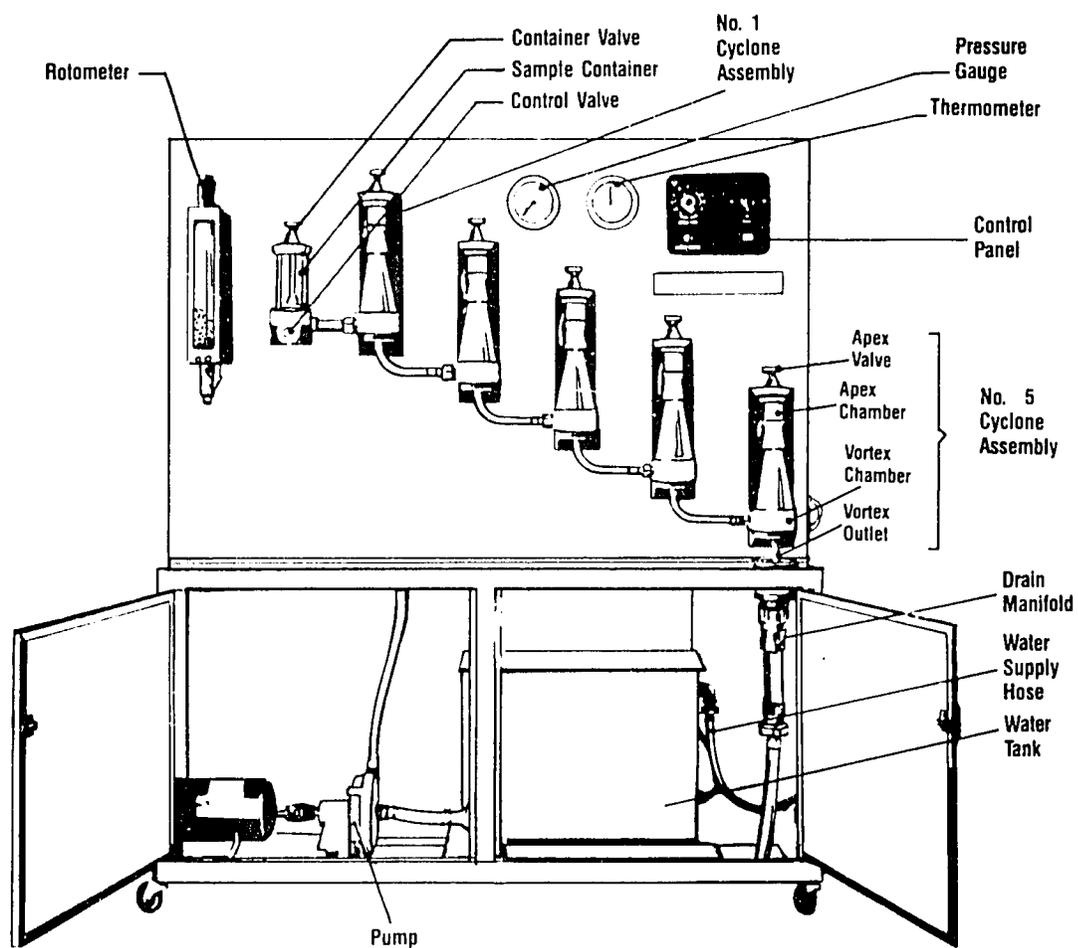


Figure 1. Warman Cyclosizer®.

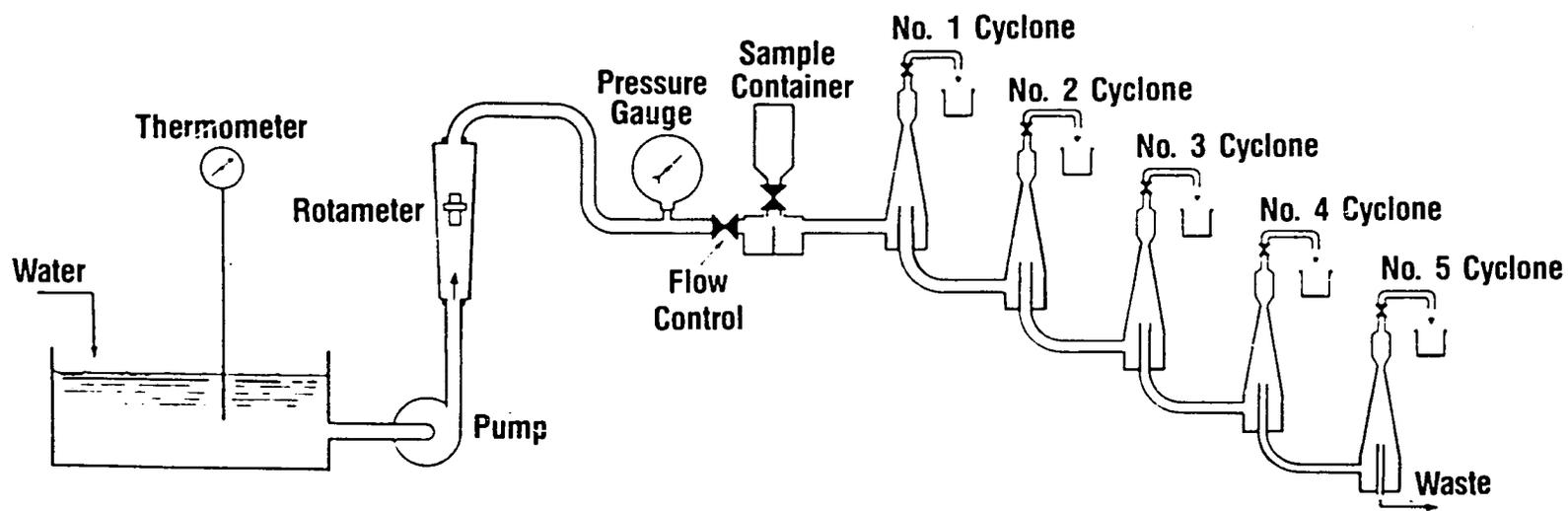


Figure 2. Schematic Diagram of Warman Cyclosizer® .

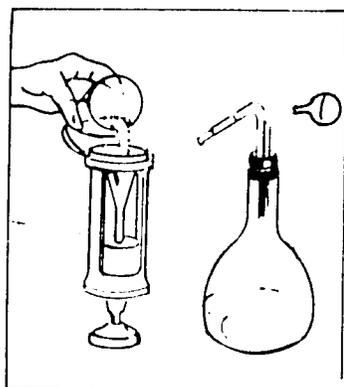


Figure 3a

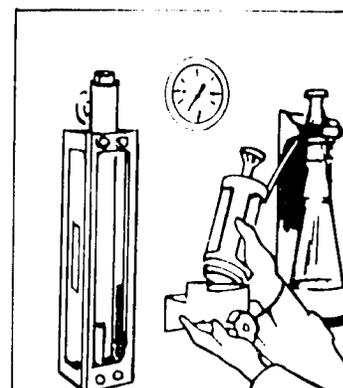


Figure 3b

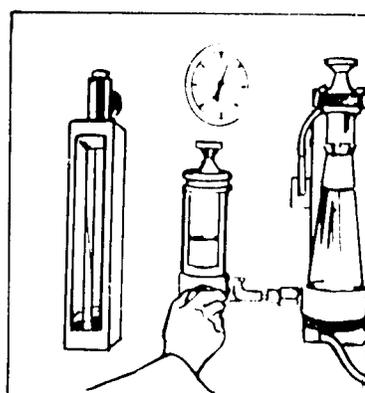


Figure 3c

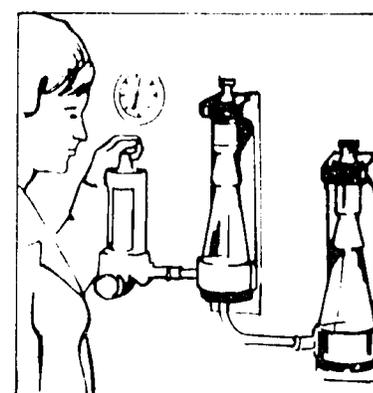


Figure 3d

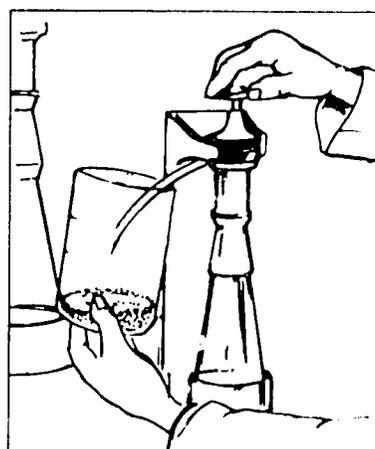


Figure 3e

Figure 3. Step-by-Step Illustration of the Test Procedure.

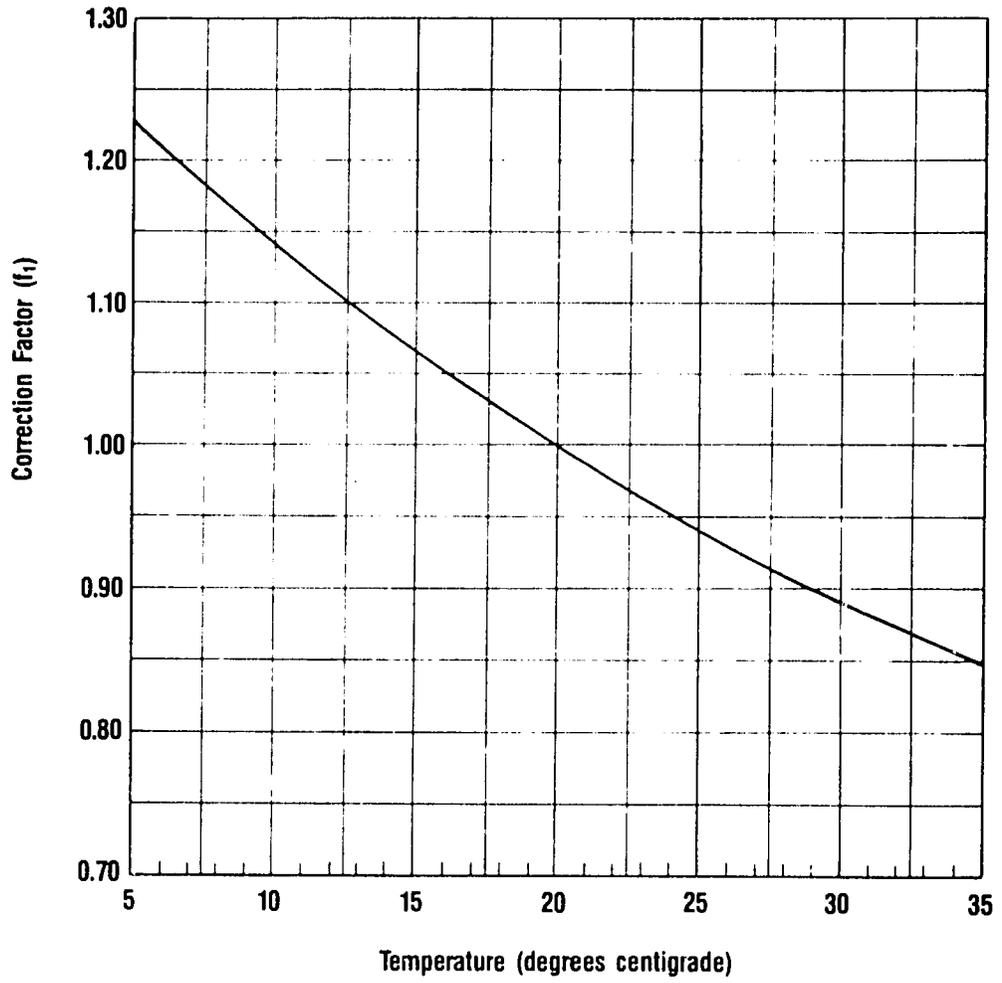


Figure 4. Temperature Correction Factor ( $f_1$ ) Chart.

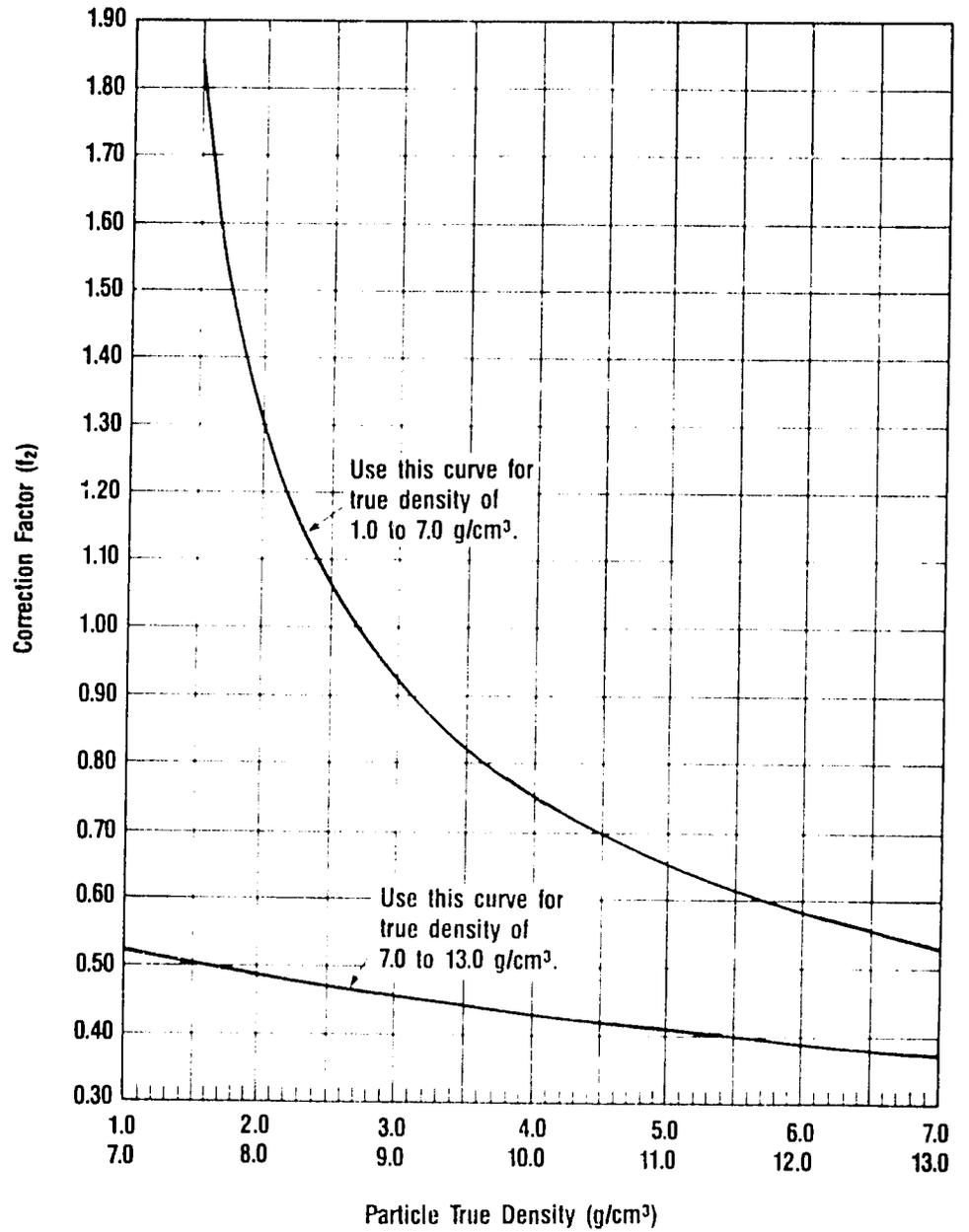


Figure 5. True Density Correction Factor ( $f_2$ ) Chart.

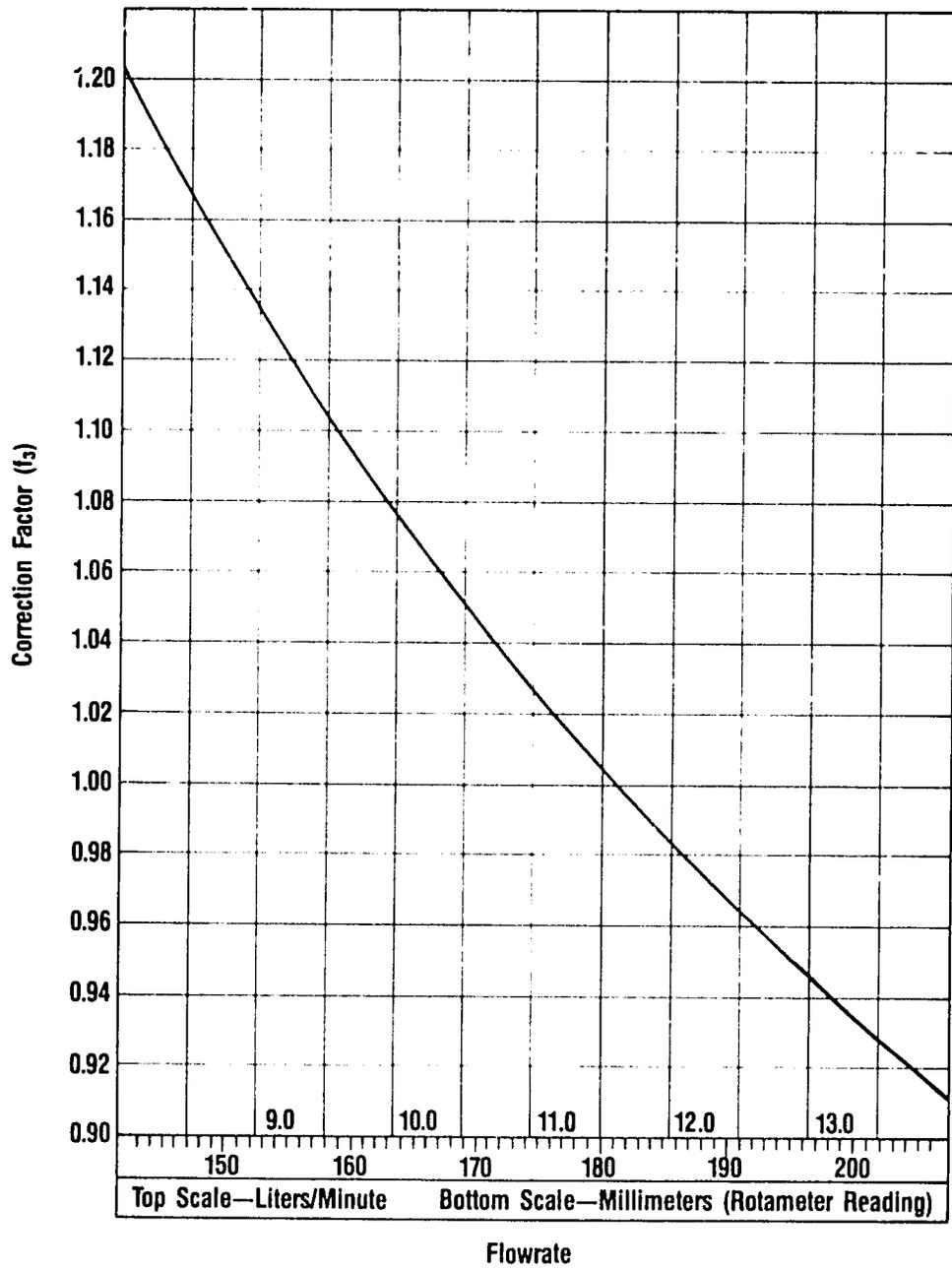


Figure 6. Flowrate Correction Factor (f<sub>3</sub>) Chart.

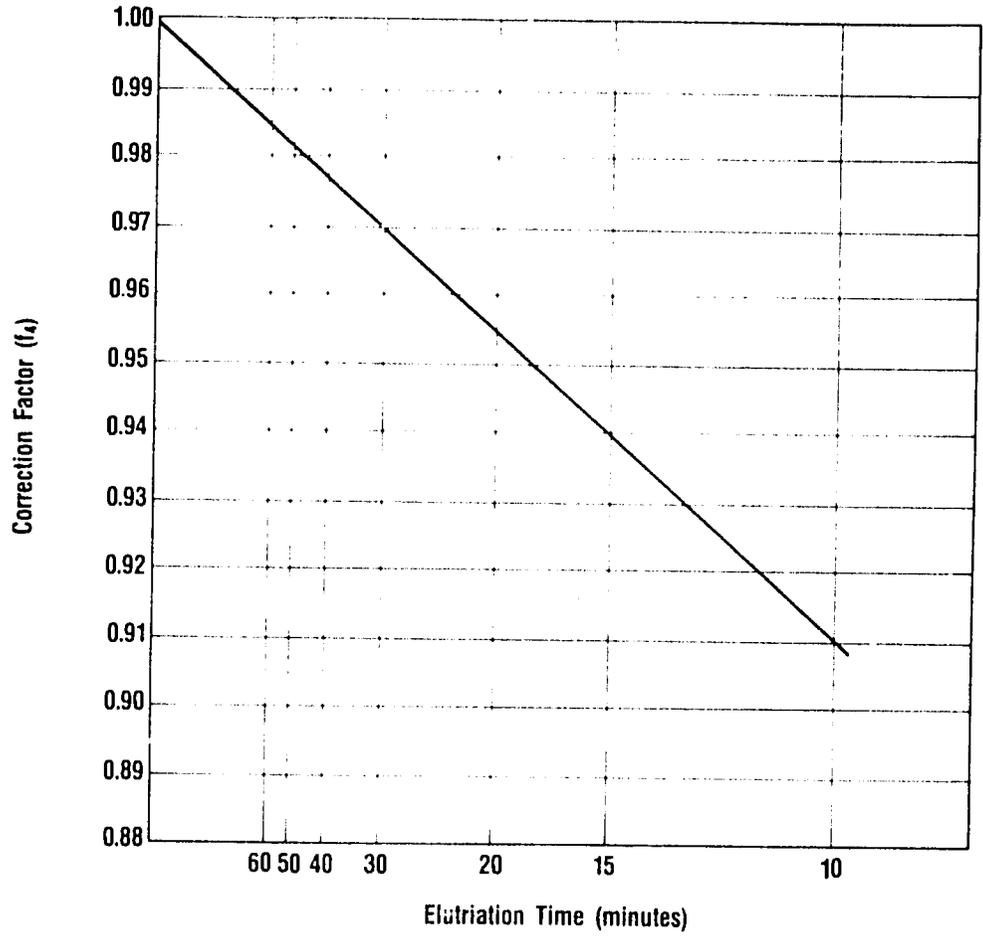


Figure 7. Time Correction Factor ( $f_4$ ) Chart.

WARMAN CYCLOSIZER RESULT SHEET		No.				
SAMPLE No		R231 77				
SAMPLE WT	gross	114.6				
	tare	50.2				
	net	64.4				
TEMPERATURE	C	10				
PARTICLE DENSITY		3.04				
FLOWRATE	mm	185				
FLOCCULATION TIME	min	60				
CORRECTION FACTOR	(temp)	1.14				
	(density)	0.90				
	(flow)	0.986				
	(time)	0.985				
No. 1 SAMPLE WT	gross	51.2				
	tare	51.1				
	net	0.1				
No. 2 SAMPLE WT	gross	51.0				
	tare	49.8				
	net	1.2				
No. 3 SAMPLE WT	gross	58.8				
	tare	50.0				
	net	8.8				
No. 4 SAMPLE WT	gross	63.1				
	tare	52.1				
	net	11.0				
No. 5 SAMPLE WT	gross	59.5				
	tare	51.6				
	net	7.9				
% RETAINED - No. 1 CYCLONE		0.2				
" " " No. 2 "		1.9				
" " " No. 3 "		13.7				
" " " No. 4 "		17.1				
" " " No. 5 "		12.3				
% PASSING - No. 1 CYCLONE		99.8				
" " " No. 2 "		97.9				
" " " No. 3 "		84.2				
" " " No. 4 "		67.1				
" " " No. 5 "		54.8				
OVERALL CORR. FACTOR		0.996				
d <sub>50</sub> No. 1 CYCLONE		42.3				
" " " No. 2 "		30.8				
" " " No. 3 "		21.5				
" " " No. 4 "		14.2				
" " " No. 5 "		10.8				
CALIBRATION DATA						
d <sub>50</sub> No. 1 CYCLONE		42.5				
" " " No. 2 "		30.9				
" " " No. 3 "		21.6				
" " " No. 4 "		14.3				
" " " No. 5 "		10.8				
REMARKS		USA/Central Florida Phosphate Rock				

SY83

Figure 8. Warman Cyclosizer® Result Sheet.

## Physical Compatibility in Blends

Physical compatibility in blends is the ability of two or more materials to remain thoroughly mixed during handling, storage, and application. Segregation of materials in a bulk blend is normally caused by mismatched particle sizes.

Determination of physical compatibility in bulk blends is carried out by performing size analyses of the individual materials to be blended. The size analyses are carried out according to the IFDC S-107 procedure.

After size distributions are determined, compatibility is determined by (1) comparing the cumulative particle-size distributions of each bulk-blend ingredient and (2) determining the median particle size of each bulk-blend ingredient.

### Procedure 1 (Cumulative Particle-Size Distribution)

Each cumulative size fraction is observed and compared with the equivalent cumulative size fraction of the other bulk-blend ingredients. The differences in percentage retained of these cumulative size fractions are noted, and the largest of these differences is used in the compatibility determination. The larger this difference the higher the segregation tendency. The following general guidelines can be used in determining compatibility:

Largest Cumulative Size Fraction Difference	Expected Compatibility
0%-10%	Good compatibility
11%-20%	Moderate compatibility (special handling precautions may reduce segregation tendencies)
> 20%	Incompatible

The attached size-distribution curves (Figure 1) illustrate how blends are evaluated for physical compatibility according to cumulative particle-size distribution.

### Procedure 2 (Median Particle Size)

The median particle size is determined by locating the 50% cumulative retained point from the size-distribution curve. The particle size (in millimeters) at the 50% cumulative retained point is multiplied by 100 and rounded to the nearest 5. This number is referred to as the Size Guide Number (SGN). The SGN for each bulk-blend ingredient is compared and evaluated by the following guidelines.

Size Guide Number Difference	Expected Compatibility
0-10	Good compatibility
11-20	Moderate compatibility (special handling precautions may reduce segregation tendencies)
> 20	Incompatible

The attached size distribution curves (Figure 2) illustrate how blends are evaluated for physical compatibility according to the Size Guide Number.

### Auxiliary Method

1. Size Analysis (Sieve Method)—IFDC S-107.

(Procedure 1-TVA Method; Procedure 2-Canadian Institute Method)

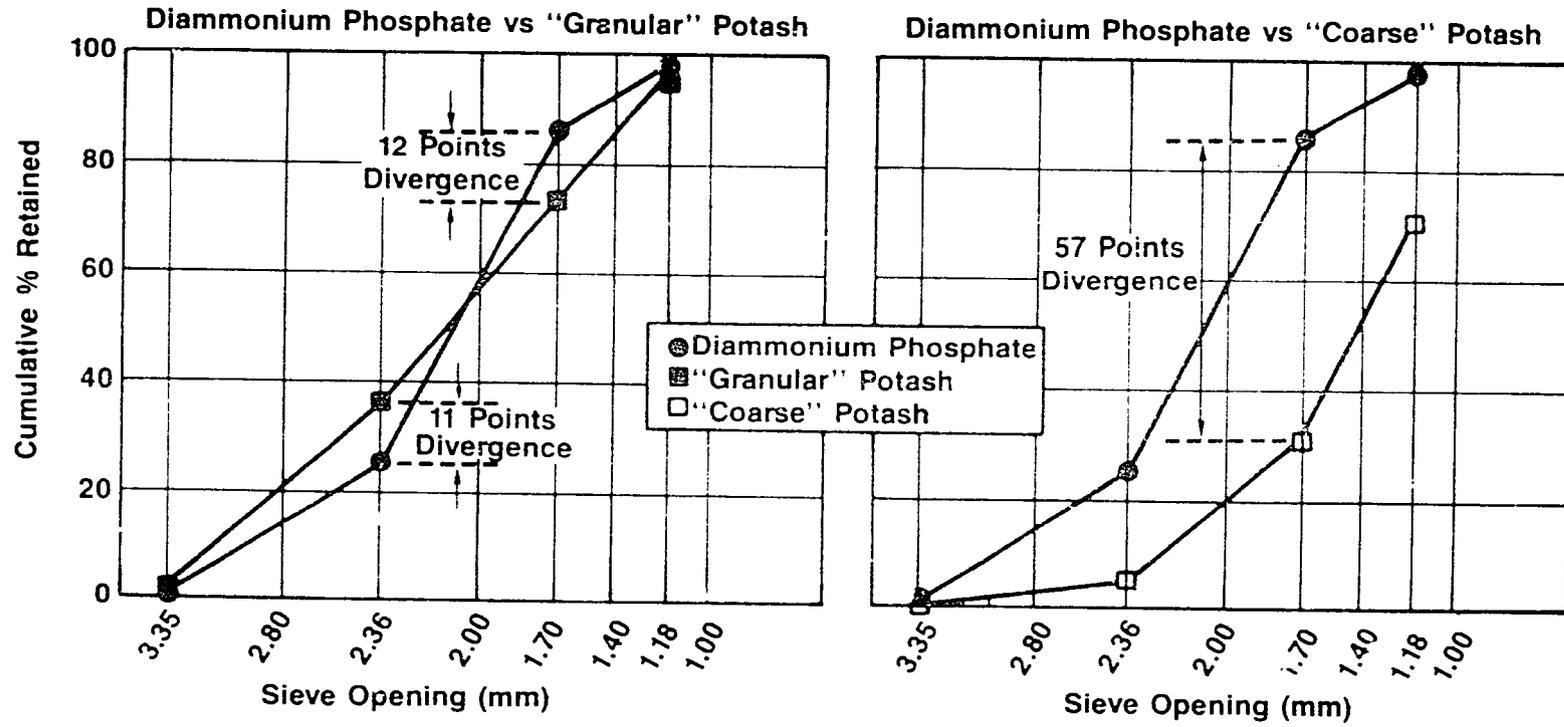


Figure 1. Physical Compatibility of Bulk Blend Ingredients According to Cumulative Particle-Size Distribution.

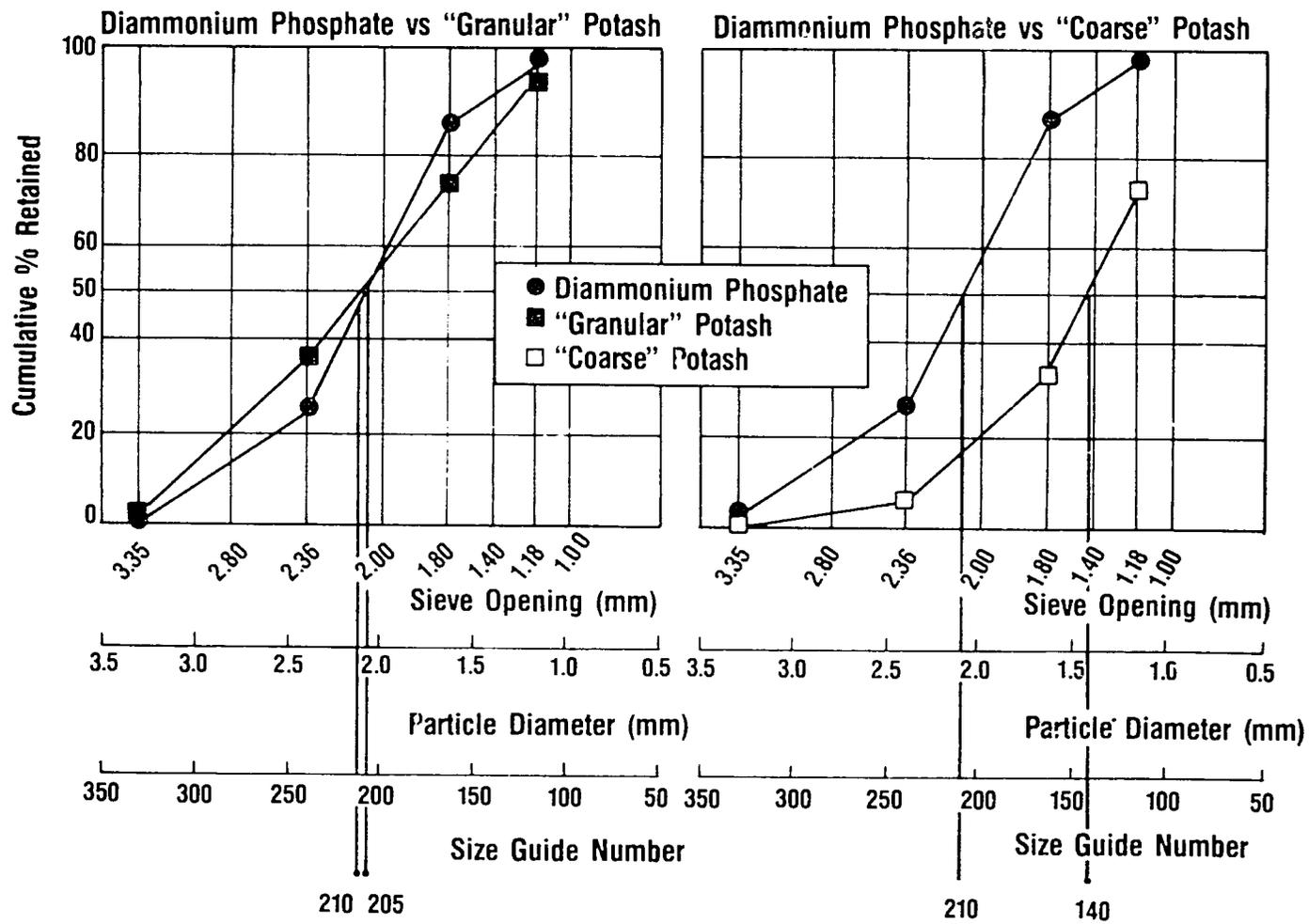


Figure 2. Physical Compatibility of Bulk Blend Ingredients According to Size Guide Number.

## Angle of Repose

The angle of repose is the angle at the base of the cone of fertilizer obtained by allowing a sample to fall onto a horizontal baseplate. It is of interest when considering storage capacity and the design of hoppers, chutes, conveyors, and sloped roofs of bulk-storage buildings.

### Apparatus

1. **Funnel**—Rigid plastics or stainless steel with a spout having an internal diameter of 25 mm.
2. **Square Baseplate (Figure 1)**—750 mm x 750 mm; rigid construction; smooth and made of wood, plastics, or corrosion-resistant metal. On the surface of the baseplate, four straight lines are engraved at angles of 45° to each other intersecting at the center of the plate.
3. **Funnel Support**—A frame of substantial construction to support the funnel so that the axis of the funnel passes through the center of the baseplate and the tip of the funnel spout is 120 mm above the surface of the baseplate.

### Sample Preparation

No special preparation is required other than obtaining a representative 10-kg sample.

### Procedure

Check that the baseplate is level and that the funnel is properly located in the support. Pour the sample into the funnel in a steady stream, taking care not to vibrate the apparatus, until the top of the cone that is formed on the baseplate reaches the tip of the funnel spout.

Mark the circumference of the base of the cone on the eight radii engraved on the baseplate. Remove the fertilizer from the baseplate and measure the four marked diameters. The angle of repose is calculated as follows:

$$\text{Angle of repose, degrees} = \text{Arctangent} \frac{2h}{\bar{d} - d_i}$$

where

h = height of the cone, i.e., the distance between the baseplate and the tip of the funnel spout, mm (usually 120 mm).

$\bar{d}$  = the arithmetic mean of the four diameters, mm.

$d_i$  = the internal diameter of the funnel spout, mm (usually 25 mm).

### Typical Values

Type	Fertilizer		Angle of Repose, Degrees
	Grade		
Prilled urea	46-0-0		28
Granular urea	46-0-0		38
Granular ammonium sulfate	21-0-0		38
Prilled ammonium nitrate	34-0-0		32
Granular diammonium phosphate	18-46-0		37
Granular monoammonium phosphate	11-55-0		37
Granular triple superphosphate	0-46-0		35
Granular potassium chloride	0-0-60		41

(Method is a Draft International Standard of the International Organization for Standardization, Unpublished)

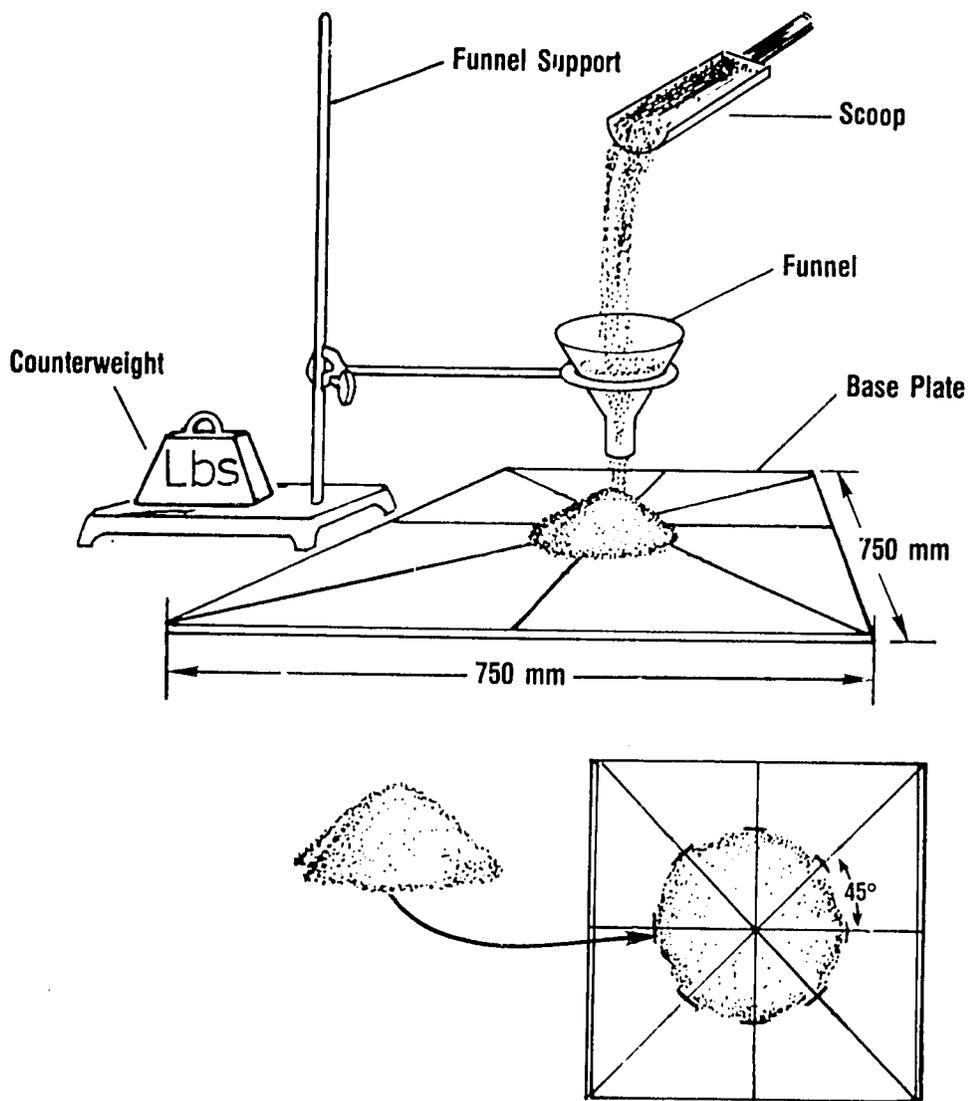


Figure 1. Apparatus for the Determination of Angle of Repose of Fertilizer.

## Bulk Density (Loose Pour)

Bulk density is the weight per unit volume of a material, including voids between particles. Loose-pour bulk density represents the minimum density (greatest volume occupancy) that would be expected from a given material. Bulk density is of interest in bag sizing, in calibration of volumetric feeders, and when considering capacity of storage bins and transport vehicles.

### Apparatus

1. **Metal Box (Figure 1)**—Open top, cube shape with a capacity of 0.0283 m<sup>3</sup> (1 ft<sup>3</sup>).
2. **Balance**—Top loading, with a minimum 100-kg capacity and a resolution of 1.0 kg.

### Sample Preparation

No special preparation is required other than obtaining a representative sample.

### Procedure

The test sample is poured gently into the metal box. The point of pouring is moved around to keep the surface approximately level during filling. An excess is allowed to pile on the filled box and then is removed by carefully passing a straightedge across the top of the box.

The box plus test sample then is weighed, and the weight of the empty box is subtracted to give the weight of the test sample. The bulk density is calculated as follows:

$$D_L = \frac{SW}{V}$$

where,

$D_L$  = loose-pour bulk density, kg/m<sup>3</sup>.

$SW$  = weight of test sample, kg.

$V$  = volume of metal box, 0.0283 m<sup>3</sup>.

### Typical Values

Type	Fertilizer	
	Grade	Bulk Density (Loose-Pour) (kg/m <sup>3</sup> )
Prilled urea	46-0-0	760
Granular urea	46-0-0	745
Granular ammonium sulfate	21-0-0	785
Prilled ammonium nitrate	34-0-0	830
Granular diammonium phosphate	18-46-0	1,000
Granular monoammonium phosphate	11-55-0	1,000
Granular triple superphosphate	0-46-0	1,120
Granular potassium chloride	0-0-60	1,040

(TVA Method)

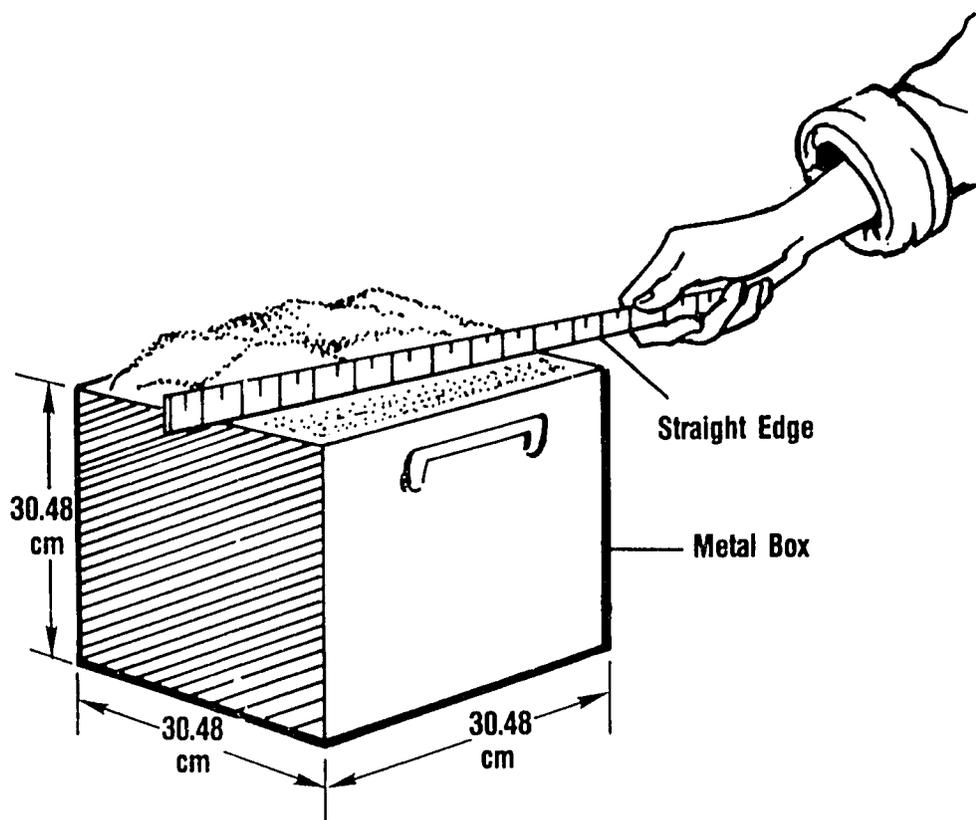


Figure 1. Metal Box (0.0283 m<sup>3</sup>) for Measuring Bulk Density (loose) of Fertilizers.

## Bulk-Density (Tapped)

Bulk density is the weight per unit volume of a material, including voids between particles. Tapped bulk density represents the maximum density to which a material might be reduced by vibration during processing or in transport. Bulk density is of interest in bag sizing, calibration of volumetric feeders, and when considering capacity of storage bins and transport vehicles.

- Apparatus**
1. **Metal Box (Figure 1)**—Open top, cube shape with a capacity of 0.0283 m<sup>3</sup> (1 ft<sup>3</sup>).
  2. **Balance**—Top loading, with a minimum 100-kg capacity and a resolution of 1.0 kg.

**Sample Preparation** No special preparation is required other than obtaining a representative sample.

**Procedure** The test sample is poured into the metal box. The point of pouring is moved around to keep the surface approximately level during filling. An excess is allowed to pile on the filled box and then is removed by carefully passing a straightedge across the top of the box. The box is lifted 15 cm off a concrete floor and dropped. Lifting and dropping are repeated until compaction of the sample is complete. The void created by dropping is refilled, and an excess is once again allowed to pile on the filled box. Without removing excess, the box is again lifted 15 cm off a concrete floor and dropped. Lifting and dropping are repeated until compaction of the sample is complete. The excess is then removed by carefully passing a straightedge across the top of the box.

The box plus the test sample then is weighed, and the weight of the empty box is subtracted to give the weight of the test sample. The bulk density is calculated as follows:

$$D_t = \frac{SW}{V}$$

where,

- $D_t$  = tapped bulk density, kg/m<sup>3</sup>.  
 $SW$  = weight of test sample, kg.  
 $V$  = volume of metal box, 0.0283 m<sup>3</sup>.

### Typical Values

Fertilizer		Bulk Density (Tapped) (kg/m <sup>3</sup> )
Type	Grade	
Prilled urea	46-0-0	835
Granular urea	46-0-0	820
Granular ammonium sulfate	21-0-0	865
Prilled ammonium nitrate	34-0-0	915
Granular diammonium phosphate	18-46-0	1,100
Granular monoammonium phosphate	11-55-0	1,100
Granular triple superphosphate	0-46-0	1,235
Granular potassium chloride	0-0-60	1,150

(TVA Method; Modified)

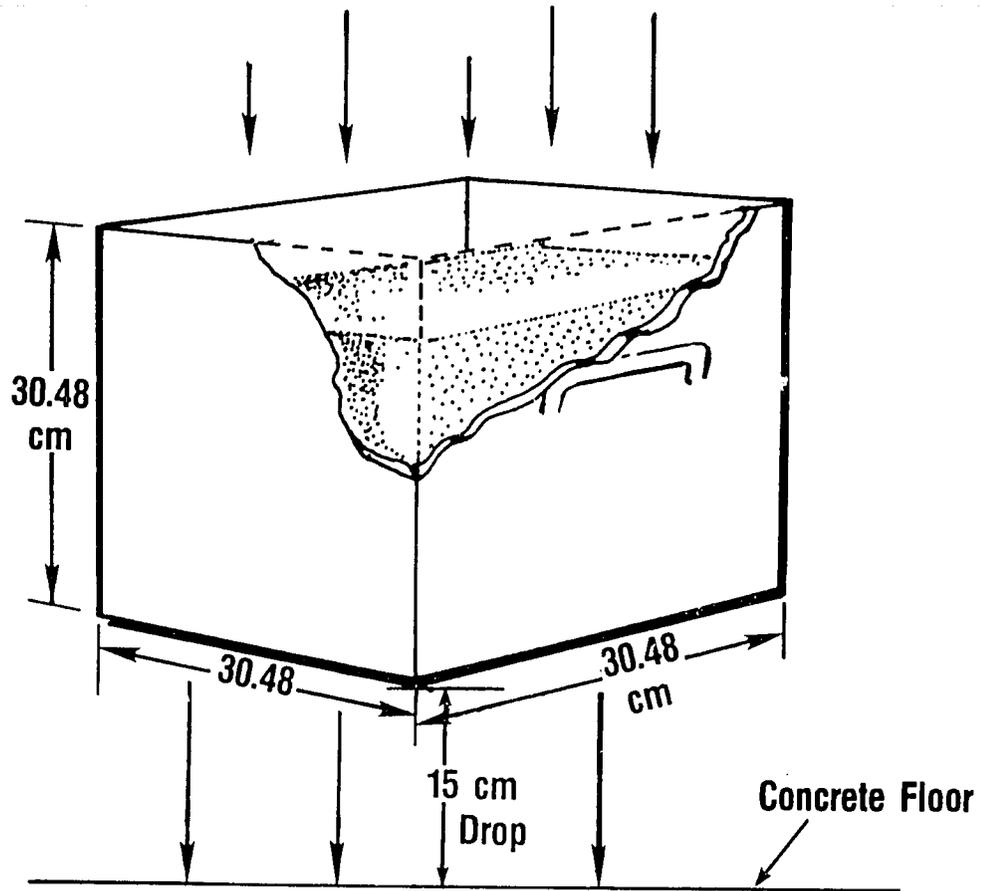


Figure 1. Metal Box (0.0283 m<sup>3</sup>) for Measuring Bulk Density (tapped) of Fertilizers, Illustrating 15-cm Drop.

## Apparent Density

Apparent density is the weight per unit volume of a material, excluding voids between particles. The apparent density of individual granules often is of interest in connection with development of new fertilizer processes; for a given product, variations in granule density can result in variations in hardness, moisture-holding capacity, and storage properties. Determinations of granule density are made in a special apparatus by measuring the volume of mercury that is displaced by submerging a known weight of granules.

- Apparatus**
- 1. Apparent Density Apparatus (Figure 1)**—This apparatus consists primarily of a tightly sealed glass sample chamber of about 10-mL capacity connected, through stopcocks, to (a) a source of vacuum for use in evacuating air from the chamber and (b) a reservoir of mercury for use in flooding of the chamber with mercury. A graduated (5-mL) buret is provided in the mercury supply system to measure the difference in volume of mercury required to flood the sample chamber with and without sample in the chamber. The sample chamber is equipped with a ground-glass cover to permit opening for introduction of a sample.
  - 2. Balance**—Analytical with a minimum 100-g capacity and a resolution of 0.0001 g.
  - 3. Test Sieve**—20 cm in diameter by 5 cm high with openings of 1.00 mm, a sieve cover, sieve pan, and a Ro-Tap<sup>®</sup> testing sieve shaker (Tyler Model B) or an equivalent shaker.

**Sample Preparation** The procedure may be carried out on granules of any desired size above 1.00 mm. The sample should be screened according to IFDC S-107 using the 1.00-mm sieve. The amount of sample required per determination is 4-7 g.

- Procedure**
1. With no sample in the chamber, stopcock "A," under the mercury buret, is opened and stopcock "B," above the sample chamber, is opened to "vent." The cover then is removed from the mercury reservoir above the buret and sufficient mercury is introduced to fill the system (including entire sample chamber) up to the level of the reference mark on the capillary tube above the sample chamber. (With this amount of mercury in the system, the level in the 5-mL graduated buret will be at the lower end of the calibrated scale). The cover on the mercury reservoir then is replaced, and stopcock "C," above the reservoir, is opened to vent.
  2. With the mercury level exactly at the reference mark on the capillary tube, a reading is taken of the mercury level in the buret. This reading represents "zero displacement" of mercury by sample since there is, as yet, no sample in the chamber.
  3. Stopcock "C" above the buret then is moved to the "vacuum" position and the vacuum is allowed to draw mercury out of the sample chamber and into the reservoir above the buret. When the sample chamber is almost emptied, the buret stopcock "A" is closed and stopcock "C" is returned to the "vent" position.
  4. The ground-glass cover on the sample chamber is removed and, by means of a powder funnel, an accurately weighed sample of 4-7 g is introduced into the chamber on top of the mercury in the chamber. The cover on the chamber then is replaced and sealed tightly.

5. Stopcock "B" is moved to the "vacuum" position, and a few minutes are allowed for evacuation of air from the sample in the chamber. Stopcock "A," under the mercury buret, then is opened, and mercury is allowed to flood the sample and rise somewhat above the reference mark on the capillary tube. (Since the top of the sample chamber is tapered down to only a capillary outlet, the granules of the sample are retained in the chamber while mercury flows freely through the capillary). Stopcock "A" then is closed, and stopcock "B" is reopened to "vent."
6. Finally, stopcock "C" again is moved to the vacuum position, and stopcock "A" is manipulated carefully until the mercury level in the capillary tube above the sample chamber drops exactly to the reference mark. A reading then is taken of the mercury level in the buret. The apparent density is calculated as follows:

$$\text{Apparent density, g/cm}^3 = \frac{\text{Weight of Sample, g}}{\text{Sample Volume, cm}^3}$$

where,

Sample volume (cm<sup>3</sup>) is taken to be equivalent to the final buret reading in mL minus the original buret reading in mL.

### Typical Values

Type	Fertilizer		Apparent Density (g/cm <sup>3</sup> )
	Type	Grade	
Prilled urea		46-0-0	1.32
Granular urea		46-0-0	1.23
Granular ammonium sulfate		21-0-0	1.59
Prilled ammonium nitrate		34-0-0	1.67
Granular diammonium phosphate		18-46-0	1.65
Granular monoammonium phosphate		11-55-0	1.72
Granular triple superphosphate		0-46-0	1.93
Granular potassium chloride		0-0-60	1.99

### Auxiliary Method

1. Size Analysis (Sieve Method)—IFDC S-107.

(TVA Method)

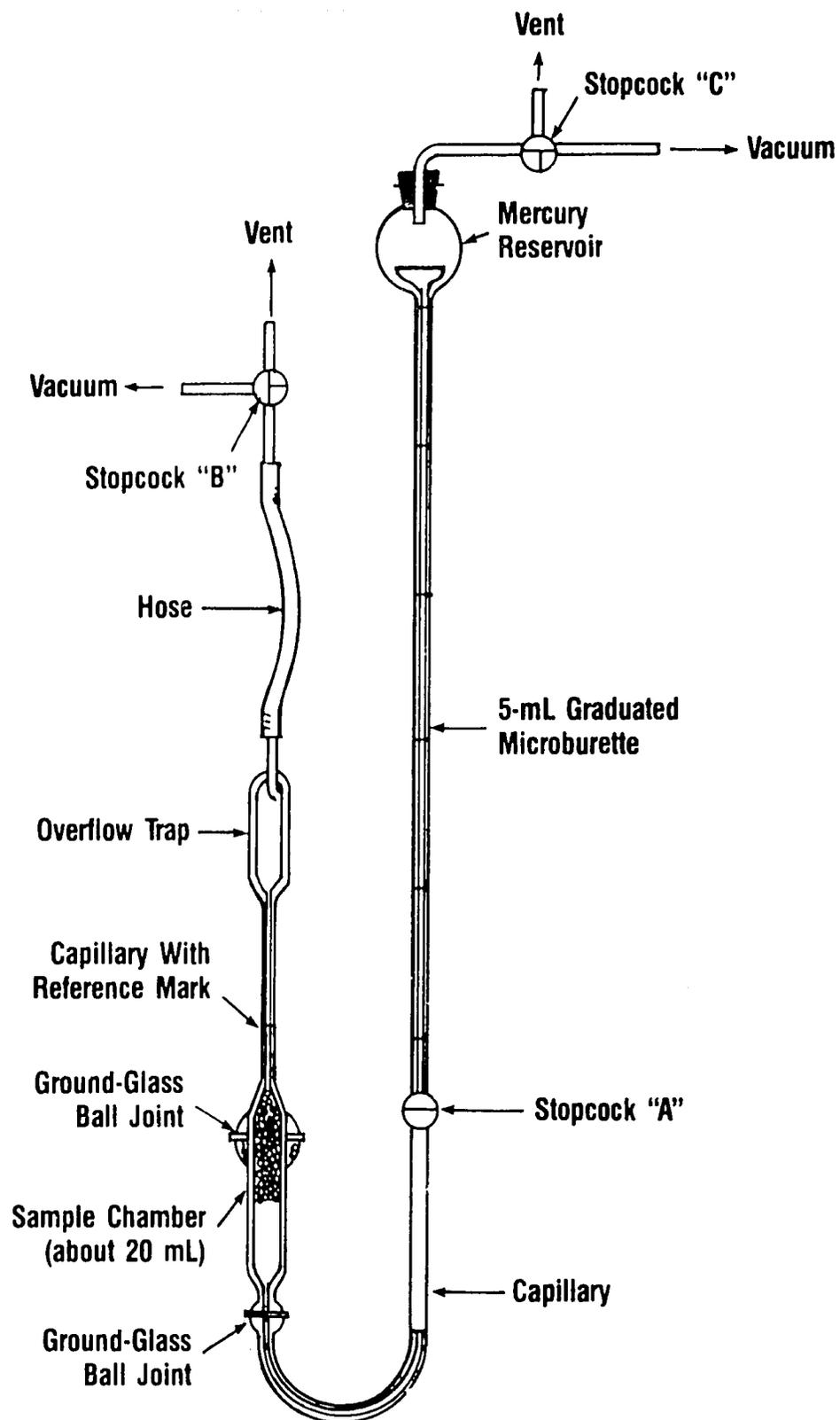


Figure 1. Apparatus for Determination of Apparent Density of Fertilizer Granules.

## True Density of Solids

True density is the weight per unit volume of a material, excluding voids between particles and all porous space. It is of interest in determining granulation and compaction efficiency.

- Apparatus**
1. **Beckman Air-Comparison Pycnometer (Figure 1)**—Model 930, including accessories such as sample cup, calibration balls, and sample holder clamp handle.
  2. **Balance**—Top loading with a minimum 100-g capacity and a resolution of 0.1 g.
  3. **Grinder**—Capable of reducing sample to 100% passing a 300- $\mu\text{m}$  opening.
  4. **Test Sieve**—20 cm in diameter and 5 cm high with openings of 300  $\mu\text{m}$ , a sieve cover, sieve pan, and a Ro-Tap<sup>®</sup> testing sieve shaker (Tyler Model B), or an equivalent shaker.

**Sample Preparation** A representative sample is reduced to 100% passing a sieve (according to IFDC S-107) with openings of 300  $\mu\text{m}$ .

**Procedure** Rotate both reference and measuring handwheels counterclockwise to rest against stops. Open coupling valve. Remove sample cup with sample holder clamp handle.

Turn measuring handwheel clockwise until starting number is set on counter. The starting number is stamped on a plate affixed to the side of the case above the measuring handwheel.

Insert the empty sample cup in sample compartment. Lock sample cup in place by pressing sample holder clamp handle down firmly.

Wait 15 s, then close coupling valve. Turn both handwheels clockwise simultaneously or alternately until reference handwheel rests against stop. Keep pointer on scale during this process.

Wait 10 s, then bring pointer to null with measuring handwheel. Open coupling valve. Read counter and record as zero offset.

Before removing sample cup, return both pistons to starting positions by rotating handwheels counterclockwise.

Repeat this procedure, but with a prepared and weighed sample (10-50 g) in the sample cup. Again read the counter and record the sample volume. The true density is calculated as follows:

$$\text{True density, g/cm}^3 = \frac{\text{Sample weight, g}}{(\text{Sample volume, cm}^3) - (\text{Zero offset, cm}^3)}$$

**Auxiliary Method** Size Analysis (Sieve Method)—IFDC S-107.

### Typical Values

Type	Fertilizer		True Density (g/cm <sup>3</sup> )
	Type	Grade	
Prilled urea		46-0-0	1.33
Granular urea		46-0-0	1.33
Ammonium sulfate		21-0-0	1.77
Ammonium nitrate		34-0-0	1.71
Diammonium phosphate		18-46-0	1.71
Monoammonium phosphate		11-55-0	1.86
Triple superphosphate		0-46-0	2.22
Potassium chloride		0-0-60	2.00

(Beckman Method; Modified)

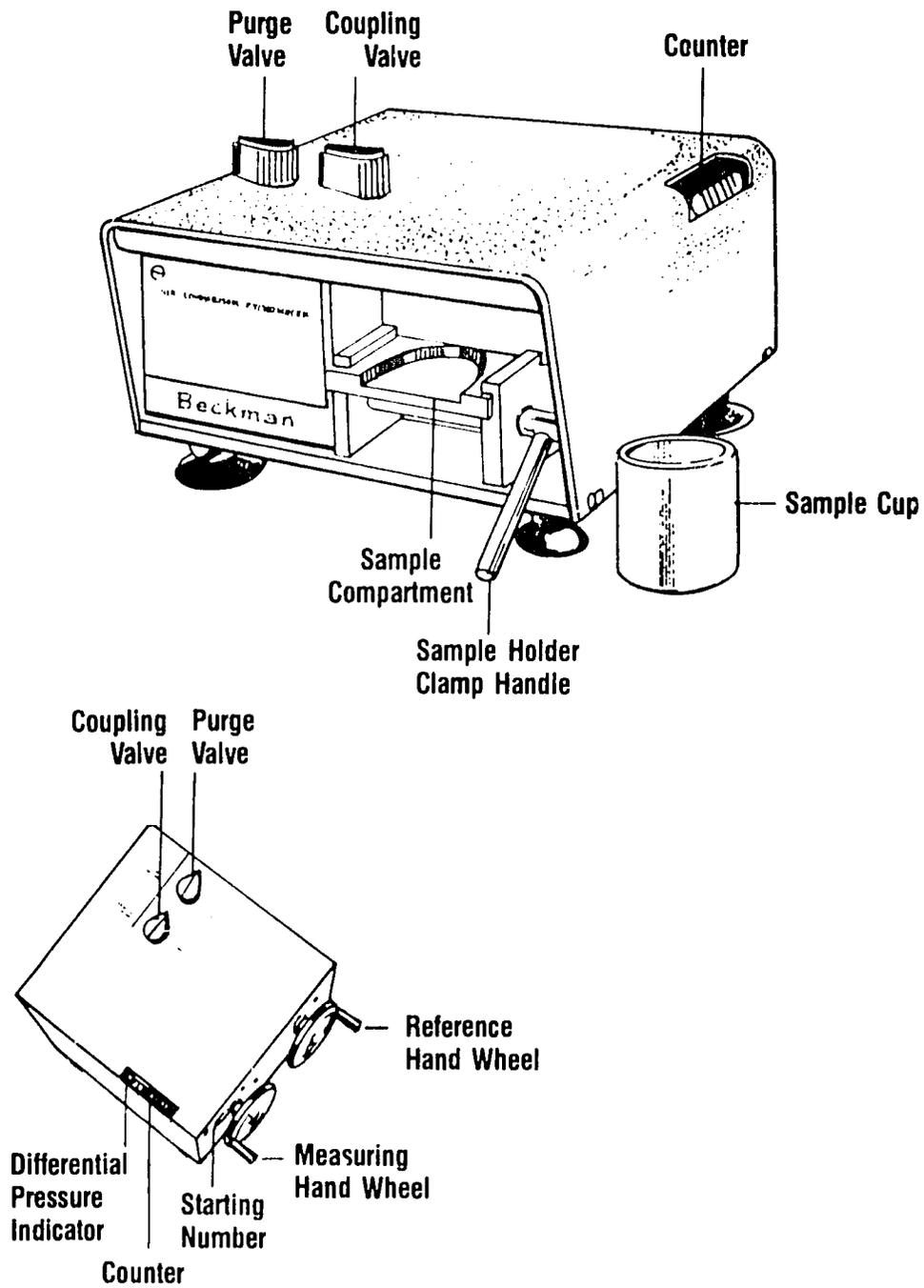


Figure 1. Beckman Air-Comparison Pycnometer for Measuring True Density of Solids.

## Granule Crushing Strength

Crushing strength is a measure of the resistance of granules to deformation or fracture under pressure. Crushing strength is of interest in estimating the expected handling and storage properties of a granular material and determining the pressure limits applied during bag and bulk storage.

- Apparatus**
1. **Test Sieves**—20 cm in diameter and 5 cm high with consecutive aperture sizes in the “fourth root of two” series, usually 2.80 mm and 2.36 mm, a sieve cover, a sieve pan, and a Ro-Tap® testing sieve shaker (Tyler Model B) or an equivalent shaker.
  2. **Compression Tester (Figure 1)**—Commercial hand-powered with detachable 5-kg and 25-kg capacity gauges, Chatillon or equivalent.

**Sample Preparation** A sample is screened (according to IFDC S-107) to obtain at least 25 granules of a closely sized fraction. The size fraction normally tested is minus 2.80 plus 2.36 mm.

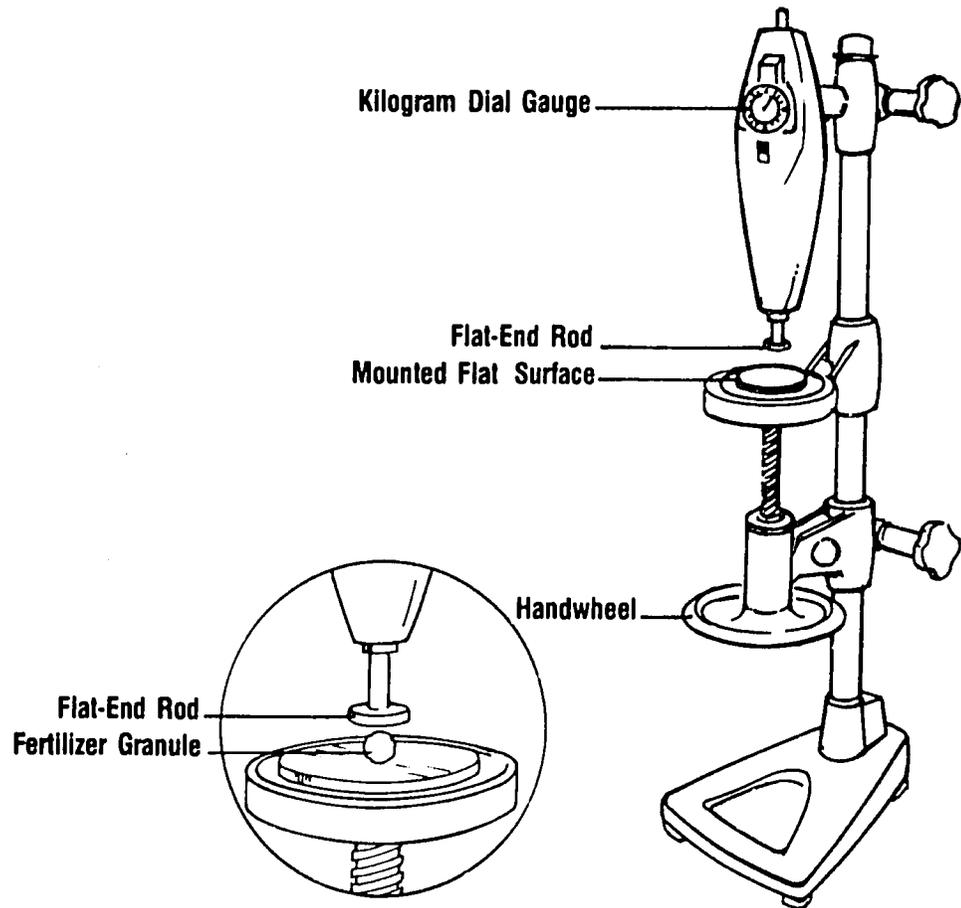
**Procedure** A commercial hand-powered compression tester is used for measuring the crushing strength. Individual granules are placed on a mounted flat surface, and pressure is applied by a flat-end rod attached to the compression tester. A gauge mounted in the compression tester measures the pressure in kilograms required to fracture the granule. At least 25 granules are tested, and the average of these measurements is taken as the crushing strength in kilograms of the minus 2.80-plus 2.36-mm fraction. Standard deviation and variance can also be calculated.

### Typical Values

Type	Fertilizer		Crushing Strength (kg/granule)
	Type	Grade	
Prilled urea		46-0-0	1.0
Granular urea		46-0-0	2.5
Granular ammonium sulfate		21-0-0	2.5
Prilled ammonium nitrate		34-0-0	1.7
Granular diammonium phosphate		18-46-0	3.9
Granular monoammonium phosphate		11-55-0	2.5
Granular triple superphosphate		0-46-0	2.9
Prilled potassium nitrate		13-0-44	1.9
Granular potassium chloride		0-0-60	4.0
Granular potassium sulfate		0-0-50	3.0

**Auxiliary Method** 1. **Size Analysis (Sieve Method)**—IFDC S-107.

(TVA Method)



**Figure 1. Chatillon Compression Tester for Measuring Granule Crushing Strength.**

## Abrasion Resistance (Rotary-Drum Method)

Abrasion resistance is the resistance to the formation of dust and fines as a result of granule-to-granule and granule-to-equipment contact. It is useful in determining material losses; handling, storage, and application properties; and pollution control equipment requirements.

- Apparatus**
1. **Rotary Drum (Figure 1)**—Stainless steel, with a rubber-covered, removable end plate. Length of the drum is 6.35 cm (2.5 in), and the diameter is 19.05 cm (7.5 in). Six flights are spaced equally around the inner circumference of the drum.
  2. **Motor-Drive Assembly**—Capable of a minimum 30 rpm.
  3. **Test Sieves**—20 cm in diameter and 5 cm high with aperture sizes of 1.00, 3.35, and 4.75 mm; a sieve cover, a sieve pan, and a Ro-Tap<sup>®</sup> testing sieve shaker (Tyler Model B) or an equivalent shaker.
  4. **Balls**—Fifty stainless steel, measuring 7.9 mm (5/16 in) in diameter with a total weight of 100 g ( $\pm 1.0$  g).
  5. **Balance**—Top loading with a minimum 500-g capacity and a resolution of 0.1 g.
  6. **Graduated Cylinder**—100-mL capacity.
  7. **Timer**—With starting, stopping, and reset mechanisms and a minimum resolution of 0.2 s.

**Sample Preparation** A sample is first screened (according to IFDC S-107) over 3.35- and 1.00-mm sieves. A 100-cm<sup>3</sup> portion of the minus 3.35- plus 1.00-mm fraction is used in the test. To avoid "blinding," the amount remaining on the 1.00-mm sieve should not exceed 140 cm<sup>3</sup>.

**Procedure** A 100-cm<sup>3</sup> portion of the screened fertilizer sample is weighed accurately and charged to the drum together with the 50 balls. The drum is closed and rotated at 30 rpm for 5 min. The contents are removed, hand screened over the 4.75-mm screen to remove the steel balls, and finally screened on the 1.00-mm screen for 5 min on the Ro-Tap<sup>®</sup> shaker. The material retained on the 1.00-mm screen then is weighed accurately, and "percent degradation" is calculated as follows:

$$\text{Degradation, \%} = 100 - 100 \times \frac{\text{Wt of -3.35 +1.00 mm fraction recovered}}{\text{Wt of -3.35 +1.00 mm fraction charged}}$$

### Typical Values

Type	Fertilizer		Abrasion Resistance (% degradation)
	Type	Grade	
Prilled urea		46-0-0	32.3
Granular urea		46-0-0	2.3
Granular ammonium sulfate		21-0-0	1.1
Prilled ammonium nitrate		34-0-0	5.6
Granular diammonium phosphate		18-46-0	0.6
Granular monoammonium phosphate		11-55-0	0.6
Granular triple superphosphate		0-46-0	0.6
Prilled potassium nitrate		13-0-44	20.8
Granular potassium chloride		0-0-60	3.8
Granular potassium sulfate		0-0-50	5.9

## Auxiliary Method

### 1. Size Analysis (Sieve Method)--IFDC S-107.

(TVA Method)

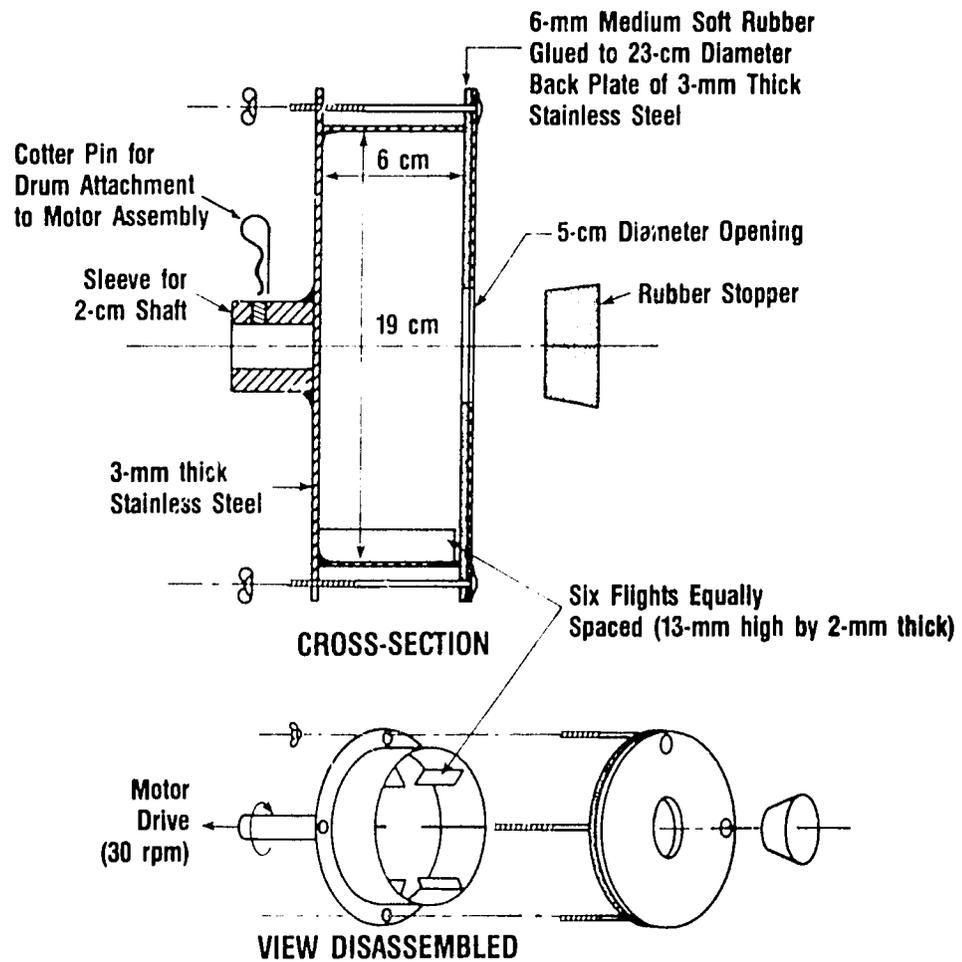


Figure 1. Rotary Drum Used in Abrasion Resistance Test.

## Abrasion Resistance (Sieve Method)<sup>1</sup>

Abrasion resistance is the resistance to the formation of dust and fines as a result of granule-to-granule and granule-to-equipment contact. It is useful in determining material losses; handling, storage, and application properties; and pollution control equipment requirements.

### Apparatus

1. **Test Sieves**—20 cm in diameter and 5 cm high (Table 1), a sieve cover, a sieve pan, and a Ro-Tap<sup>®</sup> testing sieve shaker (Tyler Model B) or an equivalent shaker.
2. **Balls**—50 stainless steel measuring 7.9 mm (5/16 in) in diameter with a total weight of 100 g ( $\pm 1.0$  g).
3. **Balance**—Top loading with a minimum 500-g capacity and a resolution of 0.1 g.
4. **Timer**—With starting, stopping, and reset mechanisms and a minimum resolution of 0.2 s.

### Sample Preparation<sup>2</sup>

Add approximately 200 g of a representative sample of fertilizer to be tested to a "fourth root of two" series of test sieves (Table 1) and screen according to IFDC S-107. The portion (subsample) of the total fertilizer sample used for the abrasion-resistance determination is obtained as follows.

1. Determine the sieve that retained the largest fraction of the total sample and save this fraction (Fraction 1).
2. From the two adjacent sieves (the next size up and down from Fraction 1), save the one that retains the next largest fraction (Fraction 2). The smaller fraction of these two is used in the next selection process.
3. From the two adjacent sieves (the next size up and down from Fractions 1 and 2), save the one that retains the next largest fraction (Fraction 3). Again, the smaller fraction of these two is used in the next selection process.
4. Repeat this procedure of selection until at least 85% of the total sample is collected.
5. Combine and thoroughly mix the fractions representing at least 85% of the sample. From this lot of material, take a 50-g ( $\pm 1$  g) subsample by using the standard riffing procedure.

For example, in the attached size analysis (Table 2) of granular triple superphosphate (GTSP), the sieve fractions selected to reach at least 85% retained are, in order of selection, 2.00 mm, 32.6%; 1.70 mm, 28.9%; 1.40 mm, 15.4%; and 2.36 mm, 14.9% for a total retention of 91.8%. Therefore, the size range selected for testing in this case would be minus 2.80 plus 1.40 mm.

### Procedure

Place the 50-g ( $\pm 1$  g) subsample of fertilizer on the sieve that retained the smallest particle-size fraction of the 85% lot of material described above. For the GTSP used in the example, the 50-g sample would be placed on a 1.40-mm sieve. Add 50 stainless steel balls (7.9 mm in diameter) to the screen, assemble cover and bottom pan, and shake in the sieve shaker for 10 min (operate unit without using tapper). Disassemble unit and weigh the material collected in the bottom pan.

1. Applicable to granular fertilizers in the size range of 500  $\mu\text{m}$  to 6.3 mm. Procedure must be modified for other size ranges.
2. Sample preparation procedure is designed to ensure that the particle-size distribution selected for testing is representative of the total sample.

## Calculation of Degradation Value

Calculate degradation value expressed in weight percent as follows:  
 Degradation, % =  $100 \times \frac{\text{Weight of material collected in bottom pan, g}}{\text{Weight of subsample, g}}$

(A high degradation value indicates a low resistance to abrasion).

## Typical Values

Type	Fertilizer		Abrasion Resistance (% degradation)
	Grade		
Prilled urea	46-0-0		21.0
Granular urea	46-0-0		0.4
Granular ammonium sulfate	21-0-0		1.0
Prilled ammonium nitrate	34-0-0		7.8
Granular diammonium phosphate	18-46-0		0.4
Granular monoammonium phosphate	11-55-0		0.4
Granular triple superphosphate	0-46-0		0.6
Prilled potassium nitrate	13-0-44		15.0
Granular potassium chloride	0-0-60		1.4
Granular potassium sulfate	0-0-50		4.8

## Auxiliary Method

1. Size Analysis (Sieve Method)—IFDC S-107.

(IFDC Method)

**Table 1. Fourth Root of Two Series of Sieve Openings<sup>a</sup>**

Size
6.30 mm
5.60 mm
4.75 mm
4.00 mm
3.35 mm
2.80 mm
2.36 mm
2.00 mm
1.70 mm
1.40 mm
1.18 mm
1.00 mm
850 μm
710 μm
600 μm
500 μm

a. Taken from Annual Book of American Society for Testing and Materials (ASTM), Section 14, General Methods and Instrumentation, *Standard Specification for Wire-Cloth Sieves for Testing Purposes*, Designation E11-81.

**Table 2. Laboratory Report of Sieve Analysis**


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**Material: Granular triple superphosphate**  
**Description: Steam granulated**

Sieve Openings	Retained but Passing Previous Screen (%)
3.35 mm	0.0
2.80 mm	2.4
2.36 mm	14.9
2.00 mm	32.6
1.70 mm	28.9
1.40 mm	15.4
1.18 mm	3.8
1.00 mm	1.3
850 $\mu\text{m}$	0.4
710 $\mu\text{m}$	0.1
600 $\mu\text{m}$	0.2
Total	100.0

---

## Impact Resistance

Impact resistance is an indication of the mechanical strength of fertilizer granules. Impact resistance is of interest when fan-type fertilizer spreaders are used, when a material is discharged from an overhead conveyor into a bulk pile, and when bags of material are dropped during handling.

- Apparatus**
1. **Test Sieves**—20 cm in diameter and 5 cm high (Table 1), a sieve cover, a sieve pan, and a Ro-Tap<sup>®</sup> testing sieve shaker (Tyler Model B) or an equivalent shaker.
  2. **Container**—With a 200-mL capacity.
  3. **Pipe**—Made of durable plastic or metal with a length of 10.7 m (35 ft) and an inside diameter of 15 cm.
  4. **Pipe Cap**—Made of durable plastic or metal sized to fit snugly over bottom of 10.7-m pipe. If cap is plastic, a 5-mm thick metal plate should be fabricated to fit snugly inside cap.
  5. **Balance**—Toploading with a minimum 250-g capacity and a resolution of 0.1 g.

**Sample Preparation**<sup>1</sup> Add approximately 200 g of a representative sample of fertilizer to be tested to a "fourth root of two" series of test sieves (Table 1) and screen according to IFDC S-107.

The portion (subsample) of the total fertilizer sample used for the abrasion-resistance determination is obtained as follows.

1. Determine the sieve that retained the largest fraction of the total sample and save this fraction (Fraction 1).
2. From the two adjacent sieves (the next size up and down from Fraction 1), save the one that retains the next largest fraction (Fraction 2). The smaller fraction of these two is used in the next selection process.
3. From the two adjacent sieves (the next size up and down from Fractions 1 and 2), save the one that retains the next largest fraction (Fraction 3). Again, the smaller fraction of these two is used in the next selection process.
4. Repeat this procedure of selection until at least 85% of the total sample is collected.
5. Combine and thoroughly mix the fractions representing at least 85% of the sample. From this lot of material, take a 100-g ( $\pm 1$  g) subsample by using the standard riffing procedure.

For example, in the attached size analysis (Table 2) of granular triple superphosphate (GTSP), the sieve fractions selected to reach at least 85% retained are, in order of selection, 2.00 mm, 32.6%; 1.70 mm, 28.9%; 1.40 mm, 15.4%; and 2.36 mm, 14.9% for a total retention of 91.8%. Therefore, the size range selected for testing in this case would be minus 2.80 plus 1.40 mm.

**Procedure** The 100-g ( $\pm 1$  g) subsample is weighed accurately and then poured through the 10.7-m pipe onto the metal plate within the pipe cap (Figure 1). The total drop distance should be 10.7 m (35 ft). The dropped sample is collected and screened over the previously selected bottom sieve (1.40-mm sieve for the GTSP example). The material retained on the previously selected bottom sieve then is weighed accurately, and the percentage of "shattered granules" is calculated as follows:

1. Sample preparation procedure is designed to ensure that the particle-size distribution selected for testing is representative of total sample.

$$\text{Shattered granules, \%} = 100 - 100 \times \frac{\text{material retained on bottom sieve after test, g}}{100 \text{ g } (\pm 1 \text{ g})}$$

## Typical Values

Fertilizer		Impact Resistance (% shattered granules)
Type	Grade	
Prilled urea	46-0-0	10.5
Granular urea	46-0-0	0.1
Granular ammonium sulfate	21-0-0	0.3
Prilled ammonium nitrate	34-0-0	4.5
Granular diammonium phosphate	18-46-0	0.8
Granular monoammonium phosphate	11-55-0	1.0
Granular triple superphosphate	0-46-0	0.6
Prilled potassium nitrate	13-0-44	8.6
Granular potassium chloride	0-0-60	3.6
Granular potassium sulfate	0-0-50	7.5

## Auxiliary Methods

### 1. Size Analysis (Sieve Method)—IFDC S-107.

(TVA Method; Modified)

**Table 1. Fourth Root of Two Series of Sieve Openings<sup>a</sup>**

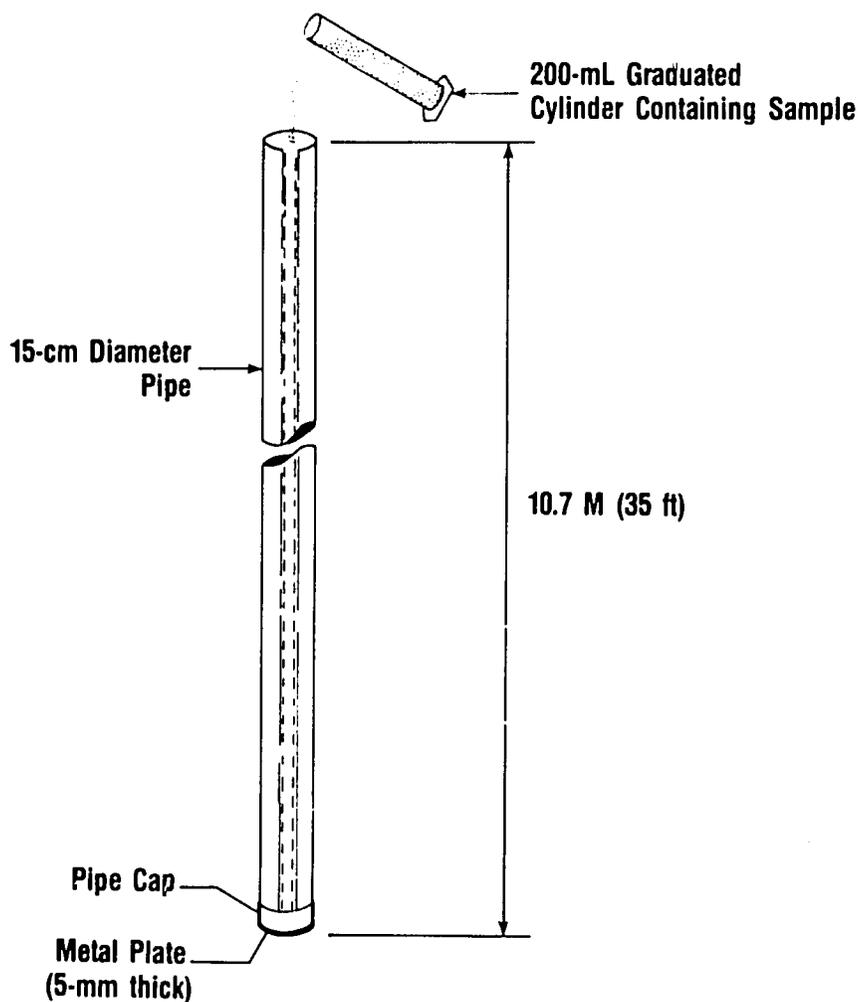
Size
25.0 mm
22.4 mm
19.0 mm
16.0 mm
13.2 mm
12.5 mm
11.2 mm
9.5 mm
8.0 mm
6.7 mm
6.3 mm
5.6 mm
4.75 mm
4.00 mm
3.35 mm
2.80 mm
2.36 mm
2.00 mm
1.70 mm
1.40 mm
1.19 mm
1.00 mm
850 μm
710 μm
600 μm
500 μm

a. Taken from Annual Book of American Society for Testing and Materials (ASTM), Section 14, General Methods and Instrumentation, *Standard Specification for Wire-Cloth Sieves for Testing Purposes*, Designation E11-81.

**Table 2. Laboratory Report of Sieve Analysis**

**Material:** Granular triple superphosphate.  
**Description:** Steam granulated.

Sieve Openings	Retained but Passing Previous Screen  (%)
3.35 mm	0.0
2.80 mm	2.4
2.36 mm	14.9
2.00 mm	32.6
1.70 mm	28.9
1.40 mm	15.4
1.18 mm	3.8
1.00 mm	1.3
850 $\mu\text{m}$	0.4
710 $\mu\text{m}$	0.1
600 $\mu\text{m}$	0.2
TOTAL	100.0

**Figure 1. Impact Resistance Test for Measuring Strength of Granules.**

## Determination of Shock Resistance of Prills

Shock resistance is the resistance to breakage of prills when impacted against a hard surface. It is of interest in the evaluation of handling and application properties of prills, particularly prilled urea.

- Apparatus**
1. **Apparatus (Figure 1)**—For determining the shock resistance of prills.
  2. **Compressed Air**—Capable of delivering a velocity of 21.2 m/s through the shock resistance apparatus.
  3. **Test Sieves**—20 cm in diameter and 5 cm high with aperture sizes of 1.00 mm and 3.35 mm, a sieve cover, a sieve pan, and a Ro-Tap™ testing sieve shaker (Tyler Model B) or an equivalent shaker.
  4. **Sphericity Apparatus**—For determining the particle roundness (see IFDC S-121, sphericity method).
  5. **Balance**—Top loading with a minimum 500-g capacity and a resolution of 0.1 g.

**Sample Preparation** A representative sample is screened (according to IFDC S-107) to minus 3.35 plus 1.00 mm. Enough material should be screened to retain a 250-g sample. Obtaining a 250-g sample may require screening 2 or 3 subsamples to avoid "blinding" of the screen.

**Procedure** Determine the percentage of round granules by subjecting a screened 250-g sample to the sphericity method (IFDC S-121). Recombine the 250-g sample.

Connect the shock resistance apparatus to the compressed air system and use the reducing valve to set the slant-gauge manometer pressure so that the air velocity in the shooting pipe is 21.2 m/s. The 250-g sample is then steadily fed through the funnel.

After all prills have been shot, remove the plastic cap and transfer the contents of the plastic receiver back to the hopper of the sphericity apparatus. Again, determine the percentage of round granules. The shock resistance is reported as the percentage of round prills remaining after the shock resistance test and is calculated as follows:

$$\text{SHOCK RESISTANCE, \% of unbroken prills} = \frac{A - (B - C)}{A}$$

where,

A = the weight, g, of original sample (250 g).

B = the weight, g, of round prills in the sieved product before the test.

C = the weight, g, of round prills in the sieved product after the test.

### Typical Values

Type	Fertilizer		Shock Resistance (% unbroken prills)
	Type	Grade	
Prilled urea "A"		46-0-0	69
Prilled urea "B"		46-0-0	57
Prilled urea "C"		46-0-0	79
Prilled urea "D"		46-0-0	93
Prilled urea "E"		46-0-0	88
Prilled urea "F"		46-0-0	75

**Auxiliary Method** 1. Sphericity—IFDC S-121.  
2. Size Analysis (Sieve Method)—IFDC S-107.

(Unie van Kunstmestfabrieken-DSM Method, Modified)

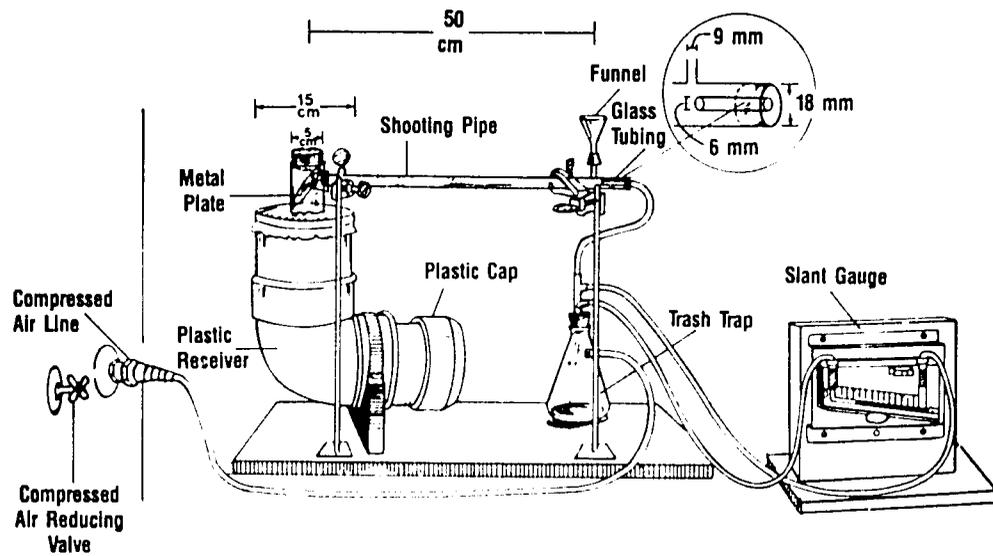


Figure 1. Apparatus for Determining the Shock Resistance of Prills.

## Handling Strength Test

The handling strength test determines the mechanical strength of granular fertilizers during maximum handling procedures. Breakage of granules can occur from the point of product belt discharge until the material is used by the farmer. This test considers the combination of granular crushing strength, abrasion resistance, and impact resistance of a fertilizer. It is of interest in determining handling and application properties.

### Apparatus

1. **Sieving Apparatus**—Large enough to quickly and efficiently screen a sample that will weigh at least 175 kg.
2. **Sieves**—With aperture sizes equivalent to the production screens used in processing the material.
3. **Front-End Loader**—With a minimum bucket lift height of 2 m and a minimum load capacity of approximately 0.5 t<sup>3</sup>.
4. **Forklift Truck**—With a minimum lifting capacity of 250 kg and a minimum lifting height of 1 m.
5. **Pallet**—At least 1.2 m in length and 1.0 m in width.
6. **Bags**—Woven polypropylene with a 0.1-mm (4 mil) polyethylene liner and a minimum capacity of 35.0 kg.
7. **Sewing Machine**—Commercial hand-held type.
8. **Balance**—Top loading with a minimum 5.0-kg capacity and a resolution of 1.0 g.
9. **Balance**—Platform with a minimum 600-kg capacity and a resolution of 1.0 kg.

### Sample Preparation

The material is first screened using sieves with openings equivalent in size to the production screens used in processing the material. This removes any over-size and undersize fractions in the sample. A minimum 175-kg screened sample should be used to perform the test. This sample is accurately weighed on the platform balance.

### Procedure

Test Step—Simulated Handling Procedure	Actual Handling Procedure Simulated
1. Pour material into a bulk pile from a 2-m height with front-end loader. The pouring area should have a concrete surface and retaining walls (so the entire sample can be recovered).	1. Product belt discharge.
2. Scoop up material with front-end loader and pour into a portable bin. Make sure all material is transferred.	2. Transfer of material from bulk pile to bagging hopper with front-end loader.
3. Charge each of five bags with one-fifth ( $\cong$ 35 kg) of sample in portable bin and sew. Any fines and shattered granules should be as equally distributed among the five bags as possible.	3. Bagging and sewing.
4. Stack charged bags on pallet.	4. Stacking of bags on pallet from bagging line (to be stored).
5. Raise loaded pallet with forklift truck to a height of 1 m.	5. Approximate height of fully loaded pallet.
6. Remove bags from pallet and drop on floor from this 1-m height.	6. Unloading from pallet onto a boxcar, truck, barge, or ship.
7. Restack charged bags on pallet.	7. Reloading on pallets from a boxcar, truck, barge, or ship.
8. Repeat steps 5, 6, and 7 five more times.	8. Maximum number of handling points after material is bagged.

Following completion of handling, the material is unbagged and again screened using the same sieves to separate the shattered and degraded material (as fines) from the intact granules. The shattered and degraded material is weighed on the top-loading balance. The handling strength is reported as the percentage of degradation/shattered granules and is calculated as follows:

$$\% \text{ degradation/shattered granules} = \frac{B}{A} \times 100$$

where,

A = amount of original sample, kg

B = amount of shattered and degraded granules, kg.

(IFDC Method)

# Sphericity

Sphericity is a measure of particle roundness of granular (or prilled) fertilizers. Sphericity is useful in determining the coating, handling, and storage properties of a fertilizer material.

- Apparatus**
1. **Sphericity Apparatus (Figure 1)**—Minimum 20-cm wide conveyor belt with side guards, adjustable inclination, variable-speed control, and a rake-type deflector mounted approximately 10 cm from belt discharge (top) point.
  2. **Feed Hopper (Figure 1)**—Mounted on a frame that can be situated over the conveyor belt so that the hopper discharge is at the conveyor belt midpoint. The hopper discharge has a gate that is opened and closed by a hand-controlled lever.
  3. **Dishpans (2)**—Minimum 20-cm width, capable of catching entire conveyor belt discharge.
  4. **Balance**—Top loading, with a minimum 300-g capacity and a resolution of 0.1 g.

**Sample Preparation** No special preparation is necessary other than obtaining a representative 250-g sample.

**Principle** To determine sphericity, a sample of granules is distributed over an inclined moving belt. The round granules (true) roll down the incline and are collected at the bottom of the belt. The distorted or broken granules (rejects) are carried upward and discharged at the top of the belt. The percentage weight of round granules is reported as the sphericity value.

**Procedure** The belt is inclined to 10° from horizontal and the belt speed is adjusted to 380 cm/min. These values can be adjusted to accommodate the sample material if it appears that the separation is not adequate.

One dishpan is situated at each of the two discharge points. About 250 g of a representative sample is poured into the hopper, and the belt is started (up the incline). The granules are slowly discharged onto the belt midpoint by controlling the hand lever on the hopper. The discharge speed should be judged so that each granule has the opportunity to roll down the incline. Discharging the sample too rapidly will result in some round granules being carried into the reject container along with reject granules, even with the deflector in place. After all granules are collected in the dishpans, the true and reject granules are weighed and the sphericity value is calculated as follows:

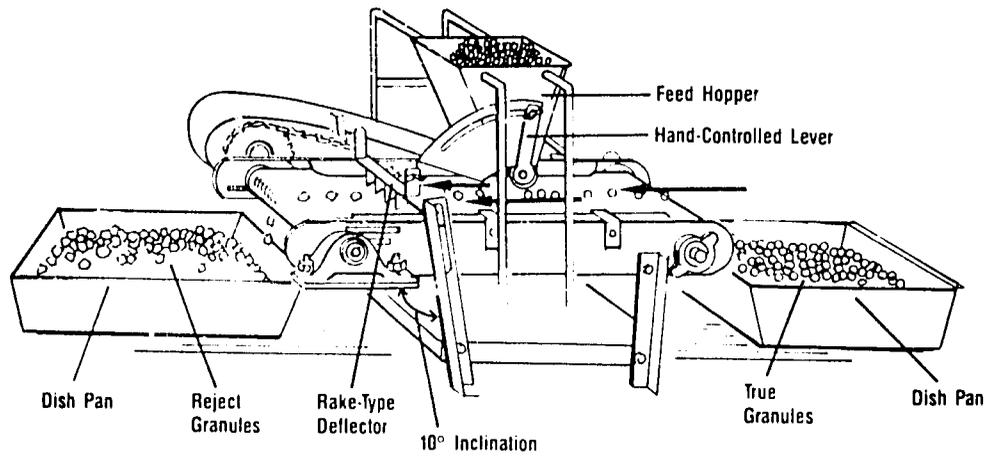
$$\text{Sphericity value, \%} = \frac{\text{True Granules, g}}{\text{True} + \text{Reject Granules, g}} \times 100$$

If the separation does not appear to be adequate, the efficiency of the test can be checked by separately passing the collection of true granules and the collection of reject granules over the belt and catching a true and reject sample from each of them. If the percentage of true granules in the reject sample is 2% or less and the percentage of reject granules in the true sample is 2% or less, the test is considered valid. If the value is greater than 2%, an adjustment should be made on the belt inclination and/or the belt speed and the test repeated.

## Typical Values

Fertilizer		Sphericity (% round granules)
Type	Grade	
Prilled urea "A"	46-0-0	93
Prilled urea "B"	46-0-0	89
Prilled urea "C"	46-0-0	47
Prilled urea "D"	46-0-0	34
Granular urea "E"	46-0-0	57
Granular urea "F"	46-0-0	2

(Fisons Fertilizers Limited; Modified)



**Figure 1. Sphericity Apparatus for Measuring Roundness of Granular Fertilizer.**

## Conditioner Adherence

A conditioner is a substance added to a fertilizer to maintain good physical quality during storage and handling.

Granular fertilizers vary in surface smoothness and other characteristics that affect the adherence of conditioner coatings. Also, conditioners themselves vary in adherence characteristics. To study these variations, coating tests and measurement of adherence are carried out by a procedure that involves the following two steps: (1) coating a 250-cm<sup>3</sup> sample of fertilizer with conditioner under closely controlled conditions in a small coating drum and (2) measurement of dust loss during passage of the coated sample through a specially designed "dedusting tower." Dustiness of fertilizer can also be determined by this method; simply bypass the first step (coating) of the procedure.

### Apparatus

1. **Rotary Drum (Figure 1)**—Stainless steel, nonflighted, with a rubber-covered, removable end plate. Length of the drum is 6.35 cm (2.5 in), and the diameter is 19.05 cm (7.5 in).
2. **Motor-Drive Assembly**—Capable of a minimum of 30 rpm.
3. **Graduated Cylinder**—250-mL capacity.
4. **Test Sieve**—20 cm in diameter and 5 cm high with aperture size of 1.00 mm and a sieve pan.
5. **Timer**—With starting, stopping, and reset mechanisms and a minimum resolution of 0.2 s.
6. **Balance**—Top loading with a minimum 100-g capacity and a resolution of 0.1 g.
7. **Dedusting Tower (Figure 2)**—The tower consists of a 64-cm (25 in) long section of an 8.6-cm (3-3/8 in) diameter glass tubing fitted inside with a series of seven baffles. These baffles are made of a 1.3-cm (1/2 in) mesh hardware cloth soldered to three vertical support rods. Each baffle is circular, of a diameter slightly less than the inside diameter of the tower, and mounted in a horizontal position. The entire baffle assembly fits loosely in the tower and is easily removable for cleaning. Air is metered into the tower through a side entry just above the bottom reservoir at a rate (about 18 m<sup>3</sup>/h) calculated to give a superficial velocity of 0.9 m/s upward through the tower.
8. **Pouring Jar**—Funnel top with a 10- to 15-mm diameter opening to facilitate pouring without spillage.
9. **Weighing Pan**—Large enough to hold at least 500 cm<sup>3</sup> of fertilizer.

### Sample Preparation

An uncoated sample is first hand screened over a 1.00-mm sieve to remove any dust or fines. A 250-cm<sup>3</sup> portion of the plus 1.00-mm fraction is used in the test. Obtaining a 250-cm<sup>3</sup> sample may require screening two or three subsamples to avoid "blinding" of the screen.

### Procedure

**Coating**—It has been found that the degree of adherence of conditioners is dependent on the amount of rolling provided during the coating operation. It is important, therefore, that a standardized coating procedure be used when comparing various conditioners and that the amount of rolling be within the range that can be provided in plant conditioning drums. The following procedure was designed to meet these requirements.

In conducting a test, a 250-cm<sup>3</sup> charge of dust-free, uncoated fertilizer is first weighed and placed in the drum; this provides a 14% loading of drum by volume. Rotation is then adjusted to 30 rpm, which corresponds to 30% of the "critical

speed"<sup>1</sup> for a drum of this size. As rotation continues, the desired weight of the conditioner is charged quickly through the 5-cm diameter opening, which is then closed with the rubber stopper. Rotation is continued at 30 rpm for 15 min following addition of conditioner. Calculations that take into account the drum speed, diameter, and loading indicate that with a retention time of 15 min the average distance through which a granule rolls in the drum is about 215 m.<sup>2</sup> This amount of rolling has been found to give near-maximum adherence with most conditioners tested and is within the range attainable in properly designed plant-scale coating drums. At the end of the 15-min coating period, the drum is stopped and removed from the motor-drive assembly. The removable end then is turned upward, unbolted, and lifted off. The contents, including all loose dust, are then transferred quantitatively to the weighing pan and weighed on the balance.

**Adherence Test**—The accurately weighed coated sample from the coating operation is charged to the pouring jar and poured downward through the dedusting tower countercurrent to the stream of air. Pouring time should be approximately 10 s. In the passage of the sample down the tower, granules are deflected and distributed by contact with the wire grids, which promotes the entry of loose conditioner into the air stream. The loose conditioner is ejected with the air stream through the open top of the tower. When all fertilizer granules reach the bottom reservoir, the airflow is stopped. The baffle assembly then is shaken and tapped to free any fertilizer granules or conditioner particles that may have become lodged in the tower. When it is certain that all the fertilizer has fallen into the reservoir, the bottom stopper is removed and the contents are drained into the weighing pan. Although the fertilizer can be reweighed at this point to determine conditioner loss during the single pass down the tower, the usual practice in our work has been to re-pass the fertilizer down the tower for a total of six passes before weighing. With six passes, essentially all of the nonadhered and loosely adhered conditioner is removed. Conditioners that show less than about 85% adherence after six passes have been found to present serious dust problems.

With well-screened fertilizer of reasonable hardness, only an insignificant amount of attrition has been found to result from the laboratory coating and dedusting operations; therefore, weight loss during passage through the tower can be assumed to result entirely from loss of conditioner. The percentage adherence can be calculated as follows:

$$\% \text{ adherence} = 100 \cdot \frac{100(a-b)}{c}$$

where,

- a = weight, g, of conditioned sample before passage through tower.
- b = weight, g, of sample after six passes through tower.
- c = weight, g, of conditioner applied in coating drum.

(TVA Method)

1. Brook, A. T. "Granular Compound Fertilizers." Paper No. LE/761. Joint Technical-Agronomic Conference of ISMA, Lausanne, Switzerland. September 1956. This author found that at speeds above 30% of the critical, the rolling action of the bed is disrupted. Critical speed (rpm) is defined as

$$76.5/\sqrt{\text{drum dia., ft}}$$

2. The method for calculating roll distance in drums is described in TVA Special Report S-430.

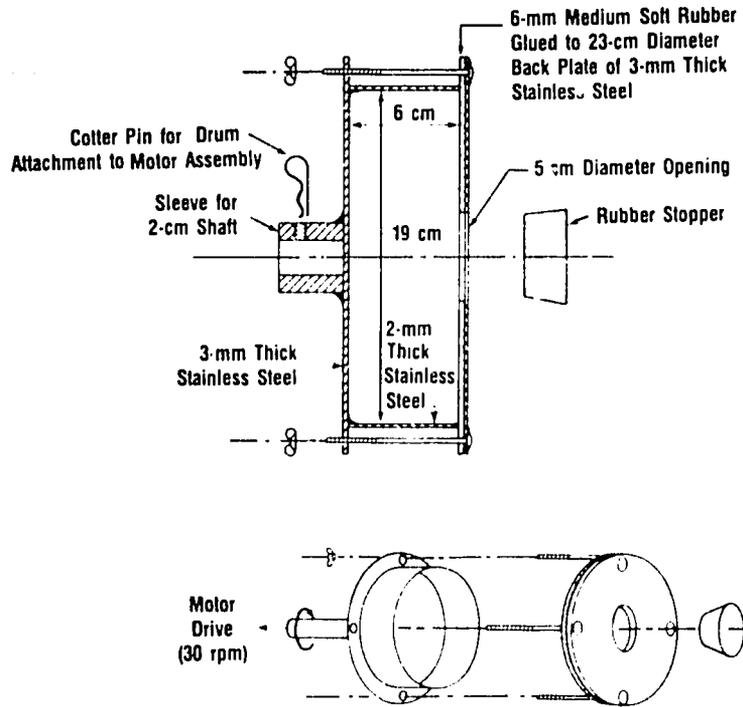


Figure 1. Laboratory-Scale Rotary Conditioning Drum.

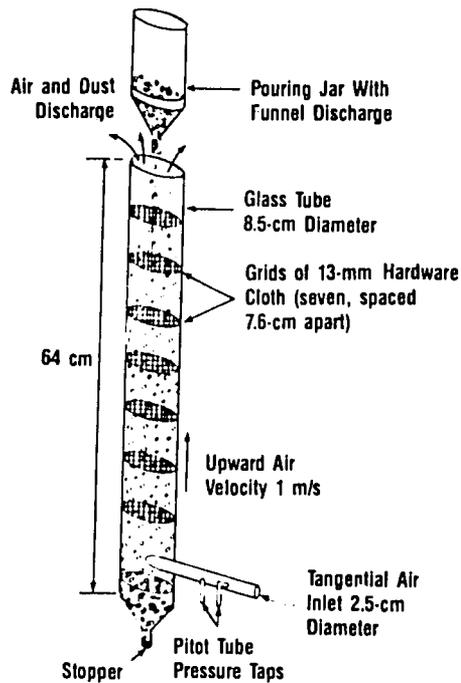


Figure 2. Dedusting Tower for Measurement of Conditioner Adherence.

## Specific Surface Area

Specific surface area of a solid material is the surface area per unit weight and gives an indication of the fineness of a material. It is particularly useful in determining the fineness of phosphogypsum created during phosphoric acid production but also can be used for other fine powders. These data can be used to evaluate reaction efficiencies and filtration rates.

### Apparatus

1. **Blaine Air Permeability Apparatus (Figure 1)**—Consisting of permeability cell, perforated disk, plunger, filter paper, manometer, and manometer liquid.
2. **Timer**—With starting, stopping, and reset mechanisms, and a resolution of at least 0.2 s.
3. **Balance**—Analytical with a minimum 160-g capacity and a resolution of 0.001 g.
4. **Rod**—At least 10 cm long and  $1.1 \pm 0.1$  cm in diameter.
5. **Jar**—Approximately 150 mL capacity.
6. **Beckman Air Comparison Pycnometer**—Model 930 or equivalent for determination of true density of solids (see IFDC S-114).
7. **Glass Microscope Slide**

### Principle

The Blaine air permeability apparatus consists essentially of a means of drawing a definite quantity of air through a prepared bed of the test sample of definite porosity. It should be stated that porosity in this method refers to interparticle spaces. The number and size of the pores in a prepared bed of definite porosity are a function of the size of particles and determine the rate of airflow through the bed.

### Sample Preparation

Enclose approximately 20 g of the sample in a 150-mL jar and shake vigorously for 2 min to fluff the sample and break up lumps or agglomerates. Allow the jar to stand unopened for 2 min more, then remove the lid and stir gently to distribute the fine fraction that has settled on the surface after fluffing. The sample should be at room temperature when tested.

### Calibration of Apparatus<sup>1</sup>

**Sample**—Calibrate the air permeability apparatus by using the current lot of National Bureau of Standards standard reference material No. 114.<sup>2</sup> The standard reference material should be at room temperature when tested and prepared as previously described.

**Bulk Volume and Bed Porosity of Compacted Bed of Sample**—Determine the bulk volume of the standard reference material by the mercury displacement method as follows.

Seat the perforated disk on the ledge in the permeability cell with inscribed side face down.

Place two filter paper disks in the permeability cell, pressing down the edges, using a rod having a diameter slightly smaller than that of the cell, until the filter disks are flat on the perforated metal disk; then fill the cell with mercury (American Chemical Society [ACS] reagent grade or better) removing any air bubbles adhering to the wall of the cell. Use tongs when handling the cell.

1. The apparatus should be calibrated periodically to correct for possible wear on the plunger or permeability cell or to reflect any changes made in the equipment such as a new type of filter paper or new manometer fluid.
2. Available from National Bureau of Standards, Office of Standard Reference Materials, Chemistry Building, Washington, D.C. 20234.

Level the mercury with the top of the cell by lightly pressing a glass microscope slide against the mercury surface until the glass is flush to the surface of the mercury and rim of the cell, being sure that no bubble or void exists between the mercury surface and the glass plate. Remove the mercury from the cell, weigh, and record the weight of the mercury ( $M_a$ ). Remove one of the filter disks from the cell.

Fill the cell with a prepared trial quantity ( $W_s$ ) of 2.950 g of standard reference material. Tap the side of the cell lightly in order to level the bed of material.

Place a filter paper disk on top of the material and compress with the plunger until the plunger collar is in contact with the top of the cell. Slowly withdraw the plunger a short distance, rotate about 90°, repress, and then slowly withdraw. The prepared bed of material should be firm.

If the material is too loose or cannot be compressed to the desired volume (plunger collar in contact with the top of the cell), adjust the trial quantity ( $W_s$ ) of material used. Record new weight used. In no case should more than thumb pressure be used to secure the proper bed, nor should thumb pressure be such that the plunger "rebounds" from the top of cell when pressure is removed.

Into the unfilled space at the top of the cell, add mercury, remove entrapped air and level off the top as before. Remove the mercury from the cell, weigh, and record the weight of the mercury ( $M_b$ ).

Calculate the bulk volume occupied by the standard reference material to the nearest 0.005 cm<sup>3</sup> as follows:

$$V = (M_a - M_b)/\rho_m \quad [1]$$

where:

$V$  = bulk volume of standard reference material, cm<sup>3</sup>.

$M_a$  = grams of mercury required to fill the cell, no sample being in the cell.

$M_b$  = grams of mercury required to fill the portion of the cell not occupied by the prepared bed of standard reference material in the cell.

$\rho_m$  = density of mercury (13.54 g/cm<sup>3</sup>).

Calculate the bed porosity of the standard reference material as follows:

$$\epsilon_s = 1 - \left( \frac{W_s}{\rho_s V} \right) \quad [2]$$

where:

$\epsilon_s$  = bed porosity of standard reference material.

$W_s$  = grams of standard reference material required (as determined in bulk volume measurement).

$\rho_s$  = true density of standard reference material, g/cm<sup>3</sup> (see certificate of analysis accompanying standard reference material).

$V$  = bulk volume of standard reference material, cm<sup>3</sup>.

**Permeability Test of Standard Reference Material**—Insert the permeability cell containing the standard reference material (after mercury is removed) in the standard-taper joint of the manometer tube. Make certain that an airtight connection is obtained by using a little stopcock grease.

Using the vacuum bulb, slowly evacuate the air in the one arm of the manometer U-tube until the liquid reaches the top mark, and then close the valve tightly. Start the timer when the bottom of the meniscus of the manometer liquid reaches

the second (next to the top) mark and stop the timer when the bottom of the meniscus of liquid reaches the third (next to the bottom) mark. Note the time interval measured and record in seconds.

## Test Procedure

**Size of Test Sample**—The weight of the test sample shall be determined in the same manner as that used in determining the weight of the standard reference material in the calibration procedures.

**Preparation of Bed of Test Sample**—The bed of test sample is prepared in the same manner as that used in preparing a bed of the standard reference material in the calibration procedure.

**Permeability Test of Test Sample**—The permeability test for the test sample is performed using the same method as that used for the standard reference material in the calibration procedure.

## Calculation

Calculate the specific surface value of the test sample in accordance with the following equation:

$$S_t = \frac{S_s \rho_s (1 - \epsilon_s) \sqrt{\epsilon_t^3} \sqrt{T_t}}{\rho_T (1 - \epsilon_T) \sqrt{\epsilon_s^3} \sqrt{T_s}}$$

where:

- $S_t$  = specific surface of the test sample,  $\text{cm}^2/\text{g}$ .
- $S_s$  = specific surface of the standard reference material,  $\text{cm}^2/\text{g}$  (see certificate of analysis accompanying standard reference material).
- $\rho_T$  = true density of test sample (determined according to IFDC S-114).
- $\rho_s$  = true density of standard reference material,  $\text{g}/\text{cm}^3$  (see certificate of analysis accompanying standard reference material).
- $\epsilon_t$  = porosity of prepared bed of test sample (calculated as described previously in the calibration procedure, except that the density and weight of the **test sample** are used).
- $\epsilon_s$  = porosity of prepared bed of standard reference material (calculated previously).
- $T_t$  = measured time interval, seconds, of manometer drop for test sample.
- $T_s$  = measured time interval, seconds, of manometer drop for standard reference material during calibration procedures.

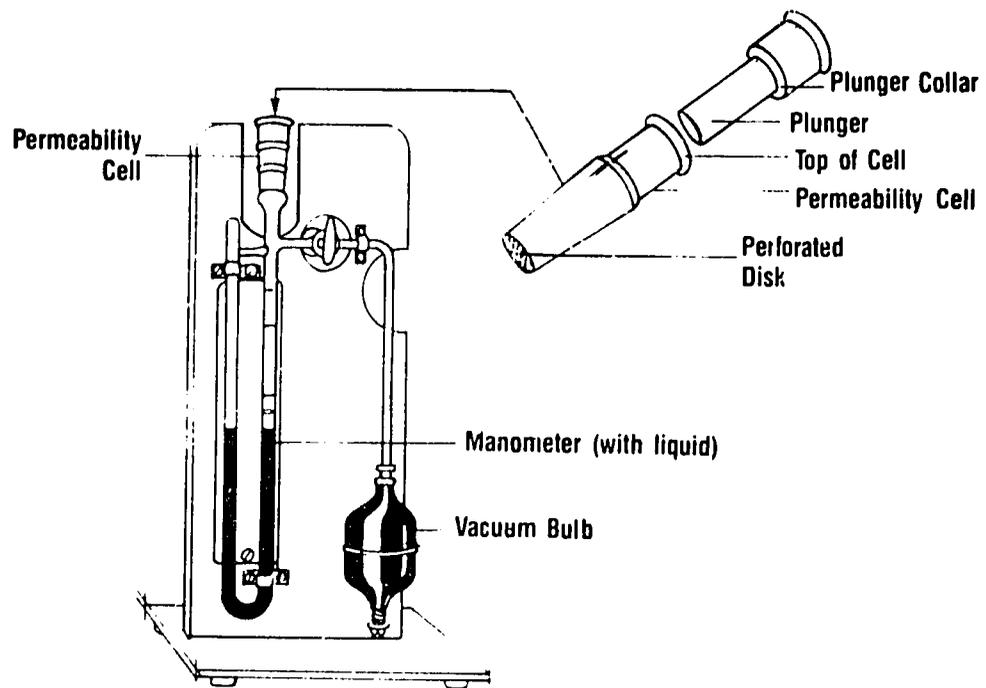
## Typical Values

Phosphogypsum Rock Source	Specific Surface ( $\text{cm}^2/\text{g}$ )
United States/Central Florida	1,500
United States/North Carolina	1,950
Morocco/Khouribga	2,775
Jordan/El Hassa	3,050
Philippines/Bantigue	2,175
South Africa/Phalaborwa	1,875
Uganda/Sukulu Hills	2,125

## Auxiliary Test Procedures Used

1. True Density of Solids—IFDC S-114.

(American Society for Testing and Materials Method, Modified)



**Figure 1. Blaine Air Permeability Apparatus for Measuring the Specific Surface Area of Fine Powders.**

## Disintegration Rate in Water

The following procedure is performed on screened fertilizer to determine its disintegration rate in water. Disintegration rate represents the percentage of granules that will disintegrate in mildly agitated water during a standard exposure time. This test gives a relative indication of the disintegration of a material once it is applied to the soil.

### Apparatus

1. **Test Sieves**—20 cm in diameter and 5 cm high (Table 1), a sieve cover, a sieve pan, and a Ro-Tap<sup>®</sup> testing sieve shaker (Tyler Model B) or an equivalent shaker.
2. **Dishpan**—12-L capacity (approximately 35 cm long, 30 cm wide, 15 cm deep).
3. **Hotplate**
4. **Shaker**—Burrell wrist-action or equivalent.
5. **Oven**—Capable of attaining a temperature of 105°C.
6. **Balance**—Top loading, with a minimum 250-g capacity and a resolution of 0.1 g.

### Sample Preparation<sup>1</sup>

Add approximately 200 g of a representative sample of fertilizer to be tested to a "fourth root of two" series of test sieves (Table 1) and screen according to IFDC S-107.

The portion (subsample) of the total fertilizer sample used for the disintegration rate determination is obtained as follows.

1. Determine the sieve that retained the largest fraction of the total sample and save this fraction (Fraction 1).
2. From the two adjacent sieves (the next size up and down from Fraction 1), save the one that retains the next largest fraction (Fraction 2). The smaller fraction of these two is used in the next selection process.
3. From the two adjacent sieves (the next size up and down from Fractions 1 and 2), save the one that retains the next largest fraction (Fraction 3). Again the smaller fraction of these two is used in the next selection process.
4. Repeat this procedure of selection until at least 85% of the total sample is collected.
5. Combine and thoroughly mix the fractions representing at least 85% of the sample. From this lot of material, take two 20-g subsamples by using the standard riffing procedure.

For example, in the attached size analysis (Table 2) of granular triple superphosphate (GTSP), the Tyler sieve fractions selected to reach at least 85% retained are, in order of selection, 2.00 mm, 32.6%; 1.70 mm, 28.9%; 1.40 mm, 15.4%; and 2.36-mm, 14.9% for a total retention of 91.8%. Therefore, the size range selected for testing in this case would be minus 2.80 plus 1.40 mm.

### Procedure

A 12-L capacity dishpan is filled with water to approximately a 10-cm depth. The water is maintained at 30°C. A sieve to screen out the disintegrated particles of the sample is attached to a wrist-action shaker and submerged in the pan to a level where 1.5 cm of water covers the sieve face. The selection of the sieve is done by finding, in the "fourth root of two" series of test sieves, the third sieve below the bottom one in the sieve range selected for testing (see Sample Preparation). In the GTSP example given, the bottom sieve in the selected sieve range has an opening of 1.40 mm; thus, the sieve submerged in the water attached to the wrist-action shaker would have openings of 850  $\mu$ m. It is important to make

1. Sample preparation procedure is designed to ensure that the particle-size distribution selected for testing is representative of total sample.

sure that no air bubbles are trapped beneath the sieve face. The shaker is adjusted to a setting (usually 2.5 for the Burrell model) at which the water is agitated just enough to cause constant movement of the sample over the screen face and at the same time not allow splashing of water over the screen frame.

A 20-g subsample of the selected size is then placed on the sieve face for a 60-s period. The sieve is then removed and dried in an oven for 1 h at 105°C. The dried sample is then removed from the sieve and weighed; its weight is recorded.

The procedure is repeated with a second 20-g subsample of the selected size but agitated for 300 s.

**Results** Disintegration rates (Dr) for both time periods are calculated as follows:

$$Dr = \frac{Sq - A}{Sq} \times 100$$

where,

Sq = original subsample quantity (usually 20 g).

A = amount of sample retained on screen, g.

### Typical Values

Fertilizer Type	Grade	Disintegration Rate, %	
		60 Seconds	300 Seconds
Prilled urea	46-0-0	100	100
Granular urea	46-0-0	100	100
Prilled ammonium nitrate	34-0-0	100	100
Granular diammonium phosphate	18-46-0	33	69
Granular monoammonium phosphate	11-55-0	20	49
Granular triple superphosphate	0-46-0	17	50
Prilled potassium nitrate	13-0-44	91	100
Granular potassium chloride	0-0-60	97	99
Granular potassium sulfate	0-0-50	35	93

### Auxiliary Method 1. Size Analysis (Sieve Method)—IFDC S-107.

(IFDC Method)

**Table 1. Fourth Root of Two Series of Sieve Openings<sup>a</sup>**

Size
6.30 mm
5.60 mm
4.75 mm
4.00 mm
3.35 mm
2.80 mm
2.36 mm
2.00 mm
1.70 mm
1.40 mm
1.18 mm
1.00 mm
850 $\mu\text{m}$
710 $\mu\text{m}$
600 $\mu\text{m}$
500 $\mu\text{m}$
425 $\mu\text{m}$
355 $\mu\text{m}$
300 $\mu\text{m}$

a. Taken from Annual Book of American Society for Testing and Materials (ASTM), Section 14, General Methods and Instrumentation, *Standard Specification for Wire-Cloth Sieves for Testing Purposes*, E-11-81.

**Table 2. Laboratory Report of Sieve Analysis**

**Material: Granular triple superphosphate**  
**Description: Steam granulated**

Sieve Openings	Retained but Passing Previous Screen (%)
3.35 mm	0.0
2.80 mm	2.4
2.36 mm	14.9
2.00 mm	32.6
1.70 mm	28.9
1.40 mm	15.4
1.18 mm	3.8
1.00 mm	1.3
850 $\mu\text{m}$	0.4
710 $\mu\text{m}$	0.1
600 $\mu\text{m}$	0.2
TOTAL	100.0

## Porosity

Porosity is a measurement of the pore space within fertilizer granules. External and, to some extent, internal pores can be detected. Pores that have openings to the granule surface are considered external pores; pores that are completely enclosed within a granule are considered internal pores. Excessive porosity is often the reason for weak granules. Also, in fertilizer compacting and briquetting systems, porosity is an indication of the degree of compaction.

### Apparatus

1. **Apparent Density Apparatus**—For determining apparent density (see IFDC S-113).
2. **Beckman Air-Comparison Pycnometer**—For determining true density of solids (see IFDC S-114).
3. **Balance**—With a minimum 100-g capacity and a resolution of 0.1 g.
4. **Test Sieves**—20 cm in diameter and 5 cm high, one with openings of 300  $\mu\text{m}$  and one with openings of 1.00 mm, a sieve cover, a sieve pan, and a Ro-Tap<sup>®</sup> testing sieve shaker (Tyler Model B) or an equivalent shaker.
5. **Bottom Pan**—For inclusion under test sieve.
6. **Grinder**—Capable of reducing sample to 100% passing a 300- $\mu\text{m}$  opening.

### Sample Preparation

A representative granular sample is reduced by grinding to 100% passing a sieve (according to IFDC S-107) with openings of 300  $\mu\text{m}$ . This sample is used to determine the true density in the air-comparison pycnometer.

The granular sample used in the apparent density apparatus to determine apparent density is screened over a 1.00-mm sieve to remove any fines or dust.

### Procedure

The true density of the ground granular sample is determined by the Beckman air-comparison pycnometer (IFDC S-114).

The apparent density of the unground granular sample is determined by the apparent density apparatus (IFDC S-113).

The porosity value is calculated as a percentage of the difference in true density and apparent density as follows:

$$\text{Porosity, \%} = \frac{T_e - A_e}{T_e} \times 100$$

where,

$T_e$  = true density, g/cm<sup>3</sup>.

$A_e$  = apparent density, g/cm<sup>3</sup>.

### Auxiliary Methods

1. **Apparent Density**—IFDC S-113.
2. **True Density of Solids**—IFDC S-114.
3. **Size Analysis (Sieve Method)**—IFDC S-107.

**Typical Values**

	Fertilizer		Porosity % of Pores
	Type	Grade	
Prilled urea		46-0-0	1
Granular urea		46-0-0	8
Granular ammonium sulfate		21-0-0	10
Prilled ammonium nitrate		34-0-0	2
Granular diammonium phosphate		18-46-0	4
Granular monoammonium phosphate		11-55-0	8
Granular triple superphosphate		0-46-0	13
Granular potassium chloride		0-0-60	1

(IFDC Method)

## True Density of Liquids

True density is the weight per unit volume at a specified temperature. It is of interest in the calibration of volumetric meters; as a quality control method in determining nutrient concentrations of liquid feeds and scrubber liquors; or in evaluating the storage, handling, and transportation characteristics of a fluid.

### Apparatus

1. **Pycnometers (Figure 1)**<sup>1</sup>—Of the Gay-Lussac type or equivalent; approximately 10-mL capacity.
2. **Water bath**—Capable of controlling temperature to 0.1°C with a minimum range from 0° to 100°C.
3. **Balance**—Analytical with a minimum 160-g capacity and a resolution of 0.0001 g.

### Sample Preparation

No special preparation is required other than obtaining a representative sample.

### Procedure

Weigh the empty, dry pycnometer to the nearest 0.0001 g after letting it stand for at least 30 min at room temperature. Fill the dry pycnometer until the neck is about half full. Insert the glass pycnometer stopper with a rotary motion to secure a firm seat, making sure no air is entrapped. Place the filled pycnometer in the water bath (so that the top of the stopper is slightly above the water level) at a specified temperature (T) for at least 30 min. Carefully wipe off with soft absorbent paper any liquid from the top flat surface of the stopper while the pycnometer is still in the water bath. Then remove the pycnometer, allow to stand at room temperature for 30 min, dry, and weigh to the nearest 0.0001 g. The true density is calculated as follows:

$$\text{True density at temperature T, g/mL} = \frac{S-P}{V}$$

where

S = weight of pycnometer full of liquid, g.

P = weight of empty pycnometer, g.

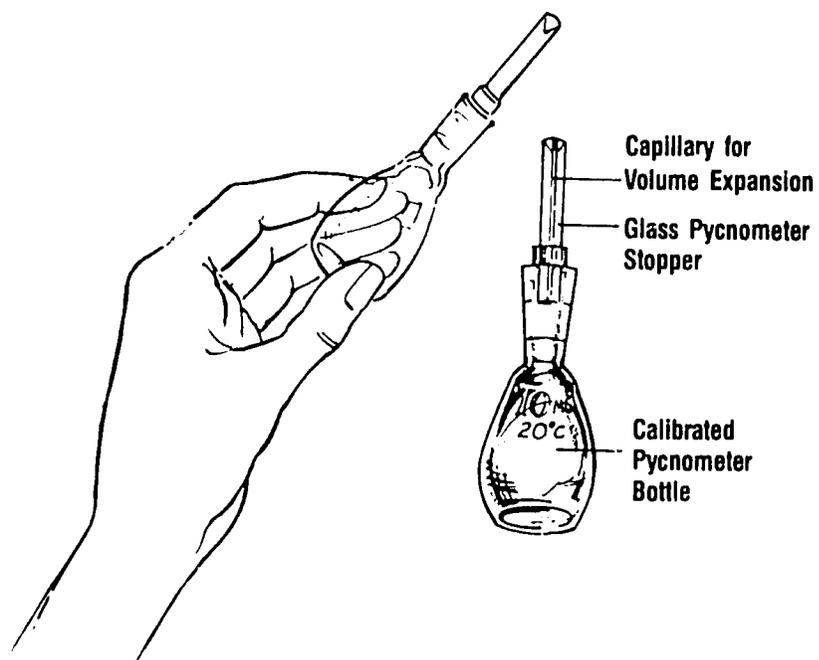
V = volume of pycnometer, mL.

### Typical Values

Phosphoric Acid Rock Source	Acid P <sub>2</sub> O <sub>5</sub> (%)	True Density, g/mL	
		30°C	75°C
United States/Central Florida	29	1.35	1.33
United States/Central Florida	51	1.67	1.65
United States/Central Florida	53	1.69	1.67
United States/Central Florida	54	1.75	1.72
United States/North Carolina	28	1.30	1.28
United States/North Carolina	56	1.71	1.69
Morocco/Khouribga	30	1.28	1.25
Morocco/Khouribga	53	1.64	1.61
Uganda/Sukulu Hills	30	1.32	1.30
Uganda/Sukulu Hills	52	1.68	1.66
Philippines/Bantigue	29	1.27	1.25
Philippines/Bantigue	52	1.62	1.60
South Africa/Phalaborwa	27	1.27	1.25
South Africa/Phalaborwa	52	1.62	1.60

(American Society for Testing and Materials Method, Modified)

1. Pycnometer should be calibrated regularly by determining the exact volume with reagent-grade mercury.



**Figure 1. Gay-Lussac Type Pycnometer for Measuring True Density of Liquids.**

## Viscosity (Capillary Method)

Viscosity is the internal resistance to flow exhibited by a fluid (opposite of fluidity). Viscosity is a useful measurement in evaluating the storage, handling, and transportation characteristics of a fluid. This method is not suitable for measuring liquids containing solids in suspension (see IFDC L-202).

- Apparatus**
1. **Glass Capillary Kinematic Viscometers (Figure 1)**<sup>1</sup>—Cannon-Fenske type or equivalent (see Table 1).
  2. **Viscosity Water Bath**—Glass jar type, capable of controlling temperature to 0.1°C with a minimum range from 0° to 100°C.
  3. **Viscometer Holder**—Capable of holding viscometer such that the liquid sample is level and completely submerged in the water bath.
  4. **Glass Thermometers**—With ranges capable of measuring the desired temperatures with a resolution of at least 0.1°C.
  5. **Timer**—With starting, stopping, and reset mechanisms and a minimum resolution of 0.2 s.
  6. **Vacuum**—House or portable vacuum pump capable of pulling sample into viscometer.

**Sample Preparation** No special preparation is necessary other than obtaining a representative sample.

**Procedure** A clean<sup>2</sup> viscometer having a range covering the estimated viscosity (Table 1) is filled by inverting and immersing tube "A" (Figure 1) into the liquid sample and applying suction to "E." When the sample rises to etched line "D," turn the viscometer upright and wipe tube "A" clean.

Place the viscometer into viscometer holder and place in water bath at the desired temperature (T). The viscometer should be positioned so that the entire sample is beneath the water level. Allow the sample to reach temperature equilibrium, usually 30 min.

Apply suction to tube "A" and bring sample into bulb "C," a short distance above mark "B."

The efflux time is measured as the time interval from the moment the meniscus passes by mark "B" to the moment when the meniscus passes by mark "D" when allowed to flow freely. Repeat the determination of efflux time until consecutive measurements are within one second of each other. If the efflux time is less than 200 s or greater than 1,000 s, a viscometer with a smaller or larger capillary, as appropriate, should be selected and the efflux time rechecked.

Calculation of kinematic viscosity is determined and reported as follows:

1. Viscometers should be calibrated regularly with a known standard fluid.
2. The viscometers should be cleaned between each determination with deionized water, followed by acetone. Clean dry air should then be blown through the viscometer to remove any remaining solvent. The viscometers should be periodically cleaned with chromic acid.

$$v \text{ (at specified temperature } T) = Ct$$

where,

- v = kinematic viscosity, centistokes.
- C = calibration constant of the viscometer, centistokes/second.
- t = efflux time, seconds.

Calculate the dynamic viscosity as follows:

$$n \text{ (at specified temperature)} = \rho v$$

where,

- n = dynamic viscosity, cP.
- $\rho$  = density, g/mL, at the same temperature used for measuring the efflux time (see IFDC L-200).
- v = kinematic viscosity, centistokes.

### Typical Values

Phosphoric Acid Rock Source	Acid P <sub>2</sub> O <sub>5</sub> (%)	Dynamic Viscosity, cP	
		30°C	75°C
United States/Central Florida	29	1.7	0.8
United States/Central Florida	51	41.0	12.7
United States/Central Florida	53	51.6	14.7
United States/Central Florida	54	84.6	18.3
United States/North Carolina	28	3.0	1.4
United States/North Carolina	56	63.5	19.2
Morocco/Khouribga	30	3.7	1.6
Morocco/Khouribga	53	26.6	8.3
Uganda/Sukulu Hills	30	4.2	1.7
Uganda/Sukulu Hills	52	43.2	14.2
Philippines/Bantigue	29	2.7	1.1
Philippines/Bantigue	52	92.0	18.0
South Africa/Phalaborwa	27	1.8	0.8
South Africa/Phalaborwa	52	26.4	8.3

### Auxiliary Method

#### 1. True Density of Liquids—IFDC L-200.

(Cannon-Fenske Method; Modified)

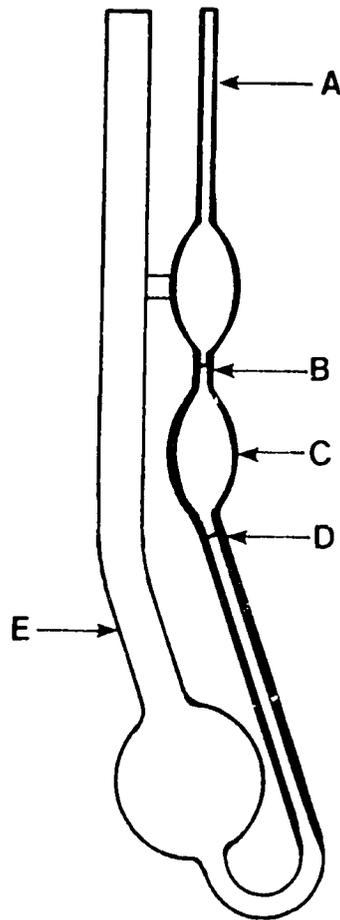


Figure 1. Cannon-Fenske Capillary Viscometer for Liquids.

Table 1. Recommended Viscosity Ranges for the Cannon-Fenske Routine Viscometers

Size	Centistokes/Second Approximate Constant	Centistokes Range
25	0.002	0.5 to 2
50	0.004	0.8 to 4
75	0.008	1.6 to 8
100	0.015	3 to 15
150	0.035	7 to 35
200	0.1	20 to 100
300	0.25	50 to 250
350	0.5	100 to 500
400	1.2	240 to 1,200
450	2.5	500 to 2,500
500	8	1,600 to 8,000
600	20	4,000 to 20,000

## Viscosity (Rotating Spindle Method)

Viscosity is the internal resistance to flow exhibited by a fluid (opposite of fluidity). Viscosity is a valuable measurement in evaluating the storage, handling, and transportation characteristics of a fluid. This method is suitable for measuring liquids with or without suspended solids.

### Apparatus

1. **Brookfield Viscometer (Figure 1)**—LV model or equivalent, with four spindles covering a viscosity range from 0 to 100,000 cP, and a spindle guard.
2. **Beaker**—Noncorrosive, with a capacity of 600 mL and a minimum internal diameter of 8.0 cm.
3. **Glass Thermometers**—With ranges capable of measuring the desired temperatures with a resolution of at least 0.1°C.
4. **Water Bath**—Capable of controlling temperature to 0.1°C with a minimum range from 0° to 100°C.

### Sample Preparation

No special preparation is necessary other than obtaining a representative sample. If liquids containing solids are measured, special precaution should be taken to prevent solids from settling to bottom of beaker. This can be accomplished by shaking, agitating, or stirring sample immediately before taking measurement.

### Procedure

Fill the beaker to the 600-mL mark with a representative sample. Place the beaker in the water bath at the desired temperature. Position the beaker so that the entire sample is beneath the water level. Allow the sample to reach temperature equilibrium, usually 30 minutes.

Select the proper spindle and rpm setting for the viscometer on the basis of the estimated sample viscosity (Table 1). The rpm setting should be adjusted only while the viscometer is on and the spindle is turning. Attach the selected spindle (left-handed thread) and spindle guard to the viscometer. Submerge the viscometer in the sample so that the liquid level is at the spindle immersion mark (Figure 1). Level the viscometer by using the circular bubble level.

Depress the clutch and turn on the viscometer motor. Release the clutch and allow the dial to rotate until the pointer stabilizes at a fixed position on the dial, normally 30 s. Again, depress the clutch and stop the viscometer motor so that the pointer position on the dial can be read. If the pointer reads 0 or 100, increase or decrease the rpm setting accordingly. If the pointer still reads 0 or 100, replace the spindle with a larger or smaller size accordingly. Calculate and report the dynamic viscosity as follows:

$$n \text{ (at specified temperature)} = df$$

where,

n = dynamic viscosity, cP.

d = dial reading.

f = dial adjustment factor (Table 1).

### Typical Values

Fluid		Dynamic Viscosity, cP		
Type	Grade	24°C	30°C	75°C
United States/Central Florida				
merchant-grade phosphoric acid	54% P <sub>2</sub> O <sub>5</sub>	-	85	18
Solution fertilizer	10-34-0	50	-	-
Solution fertilizer	7-21-7	50	-	-
Solution fertilizer	32-0-0	50	-	-
Suspension fertilizer	10-30-0	250	-	-
Suspension fertilizer	4-12-24	400	-	-

(Brookfield Manual; Modified)

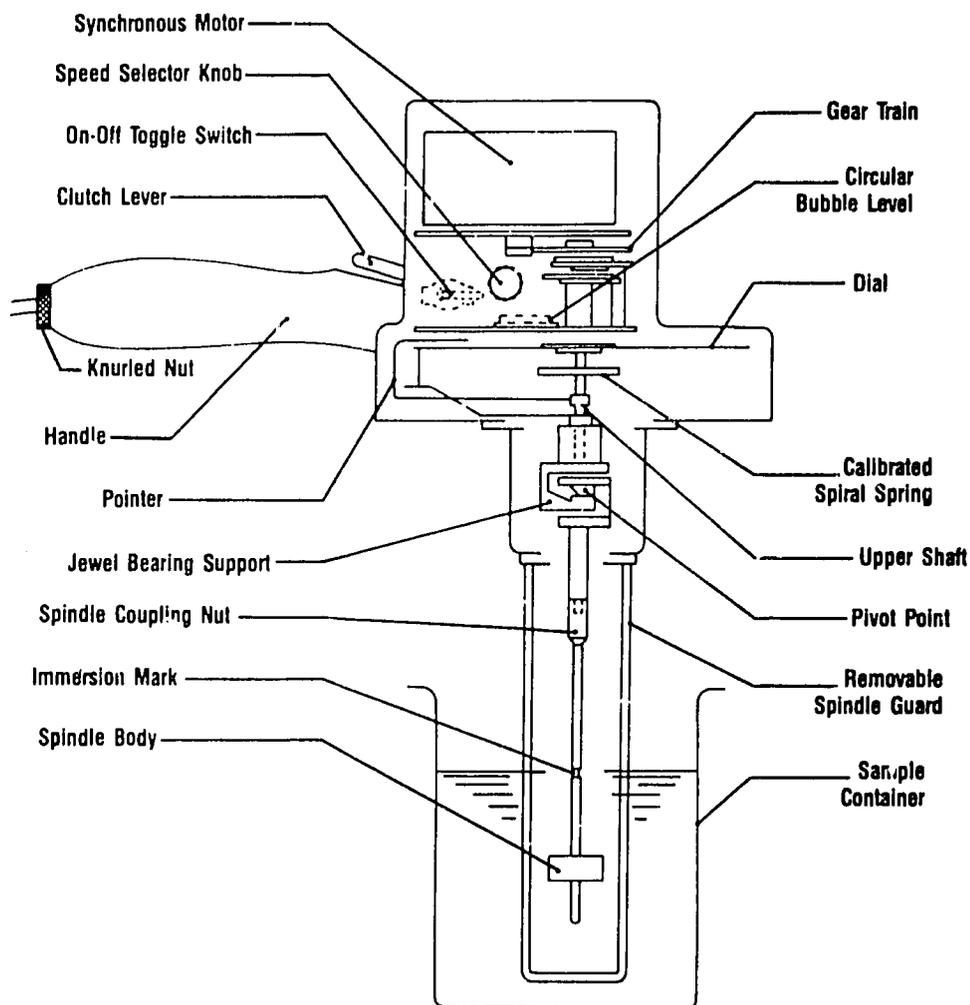


Figure 1. Brookfield Synchro-Lectric Rotating Spindle Viscometer.

**Table 1. Spindle and Rpm Selection Based on Viscosity Ranges in Centipoises**

Spindle Number	Rpm Setting	Dial Adjustment Factor	Viscosity Range (cP)
1	60	1	0-100
1	30	2	0-200
1	12	5	0-500
1	6	10	0-1,000
2	60	5	0-500
2	30	10	0-1,000
2	12	25	0-2,500
2	6	50	0-5,000
3	60	20	0-2,000
3	30	40	0-4,000
3	12	100	0-10,000
3	6	200	0-20,000
4	60	100	0-10,000
4	30	200	0-20,000
4	12	500	0-50,000
4	6	1,000	0-100,000