

**Manual
for
Determining Physical Properties of Fertilizer**

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Abstract

The International Fertilizer Development Center (IFDC) has prepared a 3rd edition 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 and Environmental Research Center (formerly National Fertilizer Development Center) and other organizations involved in the measurement of physical properties. IFDC does not endorse these methods as being standards.

This revised manual includes IFDC procedures related to the following physical properties of fertilizers: 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.

Additionally, IFDC has included six procedures that have been given the status of international standards by the International Organization for Standardization (ISO). These standards are reproduced with the permission of the ISO.

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. The 3rd edition of 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 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 and Environmental Research Center (NFERC) for valuable assistance in establishing a fertilizer physical properties laboratory. Special thanks go to Mr. George Hoffmeister and Mr. Frank Johnson, formerly with TVA, prior to their retirement. Many of the procedures included in the present manual were adopted from NFERC (formerly NFDC, National Fertilizer Development Center) and originally included in TVA's 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 has 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.

Additionally, in a new section of the manual, IFDC has included six procedures that have been given the status of international standards by the International Organization for Standardization (ISO). These ISO standards are reproduced with the permission of the ISO. Single copies of these standards can be obtained from any of its members or from ISO Central Secretariat, P.O. Box 56, 1211 Geneva 20, Switzerland.

The procedures in this manual may involve hazardous materials, operations, and equipment. These procedures do not purport to address the safety problems associated with their use. It is the responsibility of the user of these procedures to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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List of Abbreviations and Symbols

Weight

kg	kilogram
g	gram
mg	milligram
lb	pound
wt	weight

Linear Measurement

m	meter
mm	centimeter
cm	millimeter
μm	micron
ft	foot, feet
in	inch
dia	diameter

Volume

L	liter
mL	milliliter
m^3	cubic meter
cm^3	cubic centimeter
ft^3	cubic feet

Time

h	hour
min	minute
s	second

Area Measure

cm^2	square centimeter
r^2	radius squared
m^2	square meter

Other

rpm	revolutions per minute
%	percent
$^\circ$	degrees
\varnothing	diameter
cP	centipoise
mPa•s	millipascal•second
$^\circ\text{C}$	degrees centigrade
psig	pounds per square inch gauge
kg/cm^2	kilograms per square centimeter

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 differences in “moisture-holding capacity;” thus, determination of CRH is insufficient in itself to indicate the hygroscopicity of a fertilizer.

In this procedure, the hygroscopicities of fertilizers 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 pickup 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°C to +93°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°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.

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

where,

MA = moisture absorption, mg/cm²

FW = final weight of sample, g

OW = original weight of sample, g

ES = exposed surface, cm² = $\pi r^2 = 36.3 \text{ cm}^2$

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/cm³ = moisture-holding capacity, mg/cm³.

MA = moisture absorption, mg/cm².

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/cm³ = moisture-holding capacity, mg/cm³.

OW = original weight of sample, g.

V = sample cup volume, cm³.

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 (mg/cm ²)	Moisture Penetration (cm)	Moisture-Holding Capacity (mg/cm ³) (%)		
Type	Grade					
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	0.0	—	—	Excellent
Prilled Ammonium Nitrate	34-0-0	1,000 ^a	50 ^a	20	2.2	Poor
Granular Diammonium Phosphate	18-46-0	175	1.5	117	11.7	Excellent
Granular Monoammonium Phosphate	11-52-0	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)

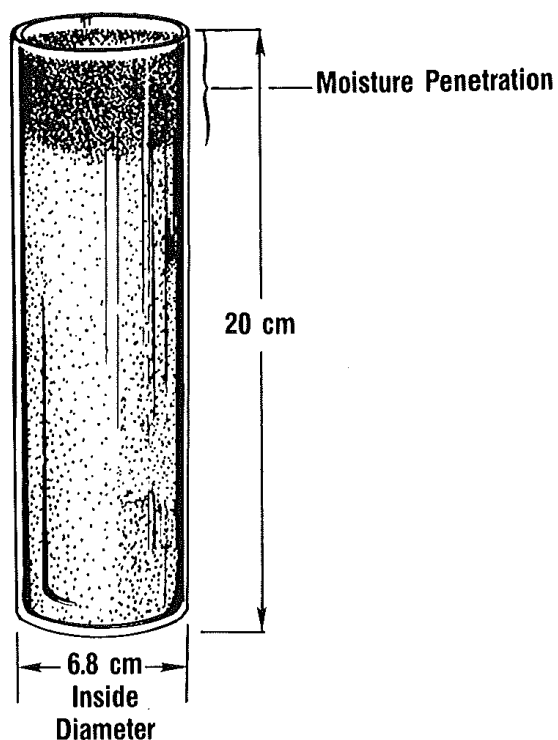


Figure 1. Glass Cylinder Used in Measuring Moisture Absorption and Penetration in a Fertilizer

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 pickup 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. Temperatures range from -18°C to $+93^{\circ}\text{C}$. 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 μm) 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 test temperature (usually 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 at least 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 a fresh sample is exposed 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 as described above.

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. Occasionally, samples may require exposures exceeding 3 h, even up to 1-3 days if moisture absorption is still progressively increasing.

In Figure 1 moisture absorption data obtained in tests of two products are plotted versus time of exposure. The data for the 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 CRH 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, Modified)

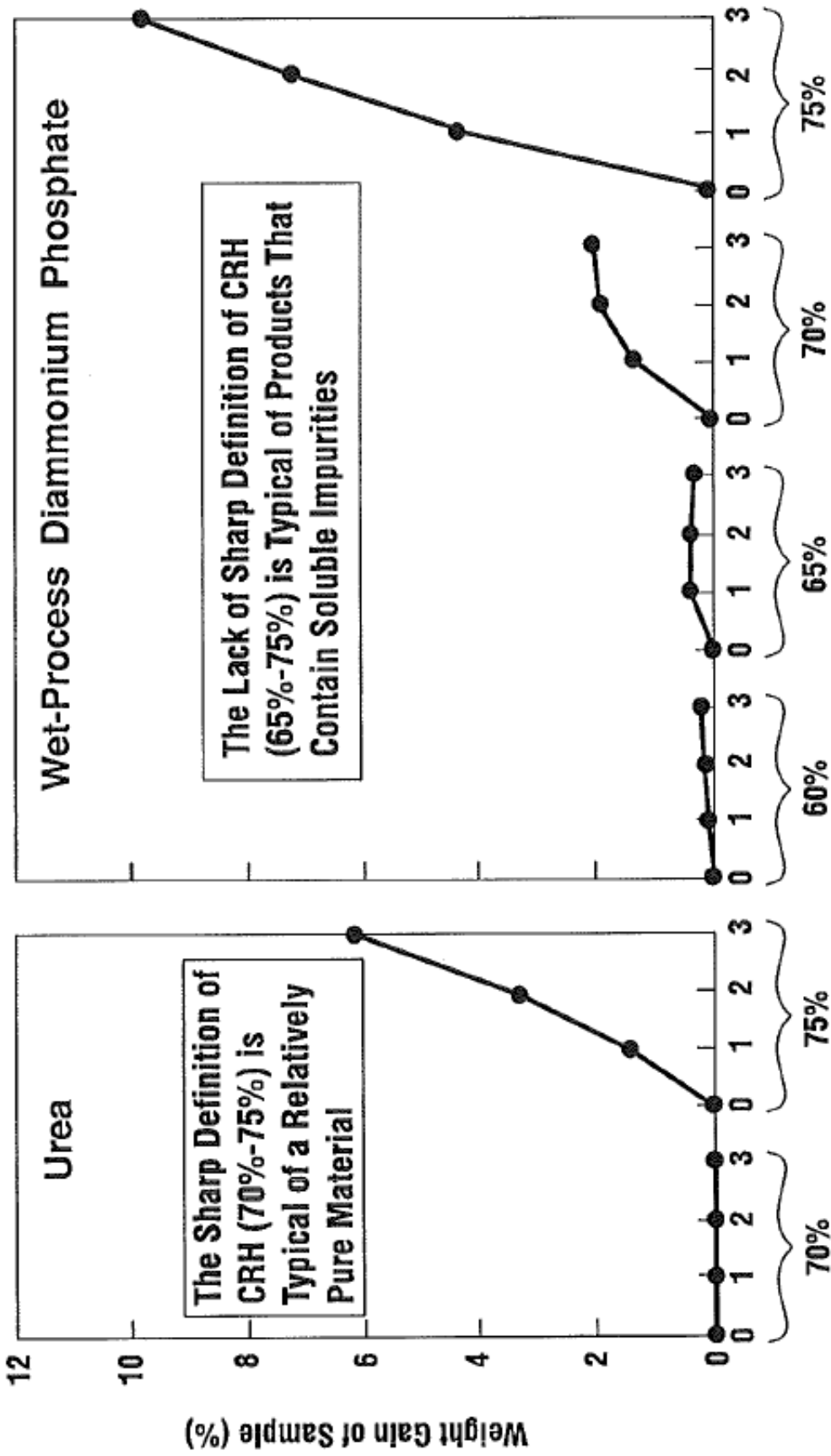


Figure 1. Hours of Exposure at Indicated Relative Humidity (30°C)

CRITICAL RELATIVE HUMIDITY (CRH)

(IFDC S-101)

Analyzed By: _____
 Dry Bulb Temperature: _____
 Wet Bulb Temperature: _____

Date Analyzed: _____
 Relative Humidity: _____

SAMPLE ID	TARE WEIGHT (g)	TARE + INITIAL SAMPLE WEIGHT (g)	SAMPLE WEIGHT (g)	WEIGHT GAIN AFTER					
				1 HOUR (g)	2 HOURS (g)	3 HOURS (g)	1 HOUR (%)	2 HOURS (%)	3 HOURS (%)

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^3 sample will assume. The other three circles should be etched at the level that 375-, 250-, and 125-cm^3 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 $+93^\circ\text{C}$. Relative humidity (RH) range of 20%-96%.

Sample Preparation

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

Procedure

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. A 500-cm^3 sample of the test fertilizer is placed in the inclined (20°) rotating drum. 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^3 (25% nonflowable), 250-cm^3 (50% nonflowable), and 125-cm^3 (75% nonflowable) marks become visible above the sample line.

Typical Values

Fertilizer		Flowability (Minutes at 30°C and 90% RH)		
		25% Nonflowable	50% Nonflowable	75% Nonflowable
Type	Grade			
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	135	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)

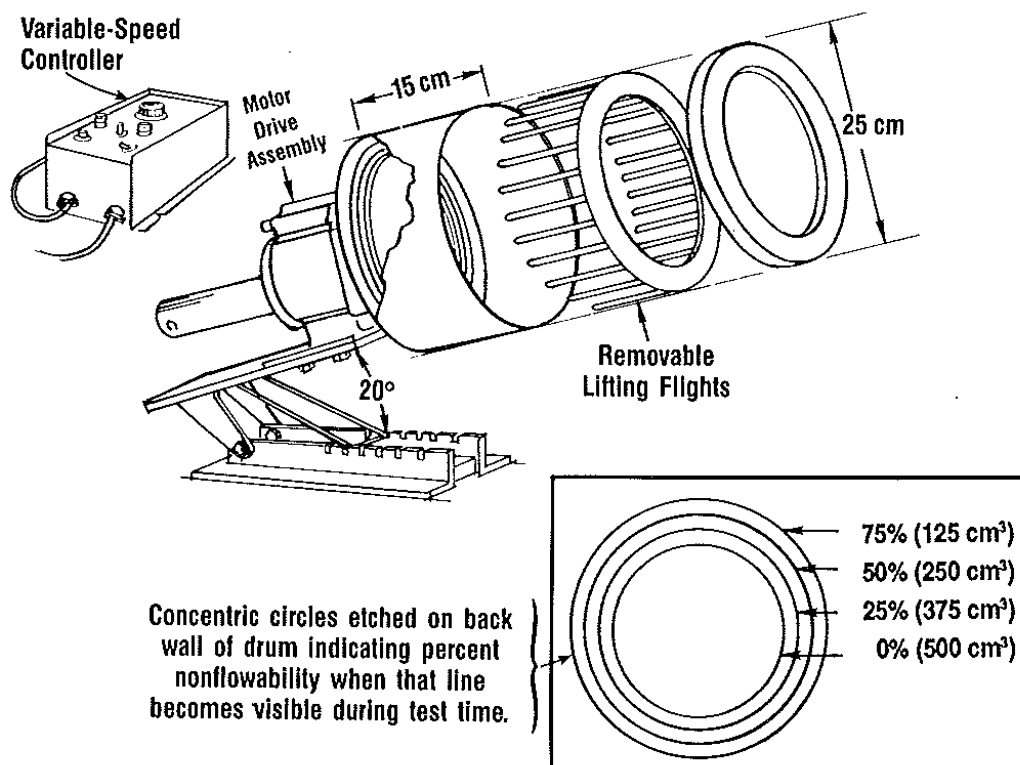


Figure 1. Rotary Drum for Measuring Flowability of Fertilizer When Exposed to Humid Conditions

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:

<u>Visual Observation</u>	<u>Rating</u>
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)

AMMONIUM NITRATE									
UREA									
AMMONIUM SULFATE									
TRIPLE SUPERPHOSPHATE									
SINGLE SUPERPHOSPHATE									
DIAMMONIUM PHOSPHATE									
MONOAMMONIUM PHOSPHATE									
POTASSIUM CHLORIDE									
POTASSIUM SULFATE									
X	OK	OK	OK	OK	OK	OK	OK	OK	OK
OK	L	OK	OK	OK	OK	OK	OK	OK	OK
OK	L	OK	OK	L	L	OK	OK	OK	OK
OK	OK	OK	OK	L	L	OK	OK	OK	OK
OK	OK	OK	OK	OK	OK	OK	OK	OK	OK
OK	OK	OK	OK	OK	OK	OK	OK	OK	OK
OK	OK	OK	OK	OK	OK	OK	OK	OK	OK

X = INCOMPATIBLE
 L = LIMITED COMPATIBILITY
 OK = COMPATIBLE

Figure 1. Chemical Compatibility of Blend Materials

CHEMICAL COMPATABILITY TEST

(IFDC S-104)

Analyzed By: _____
 Temperature, °C: _____
 Test Duration: _____

Date Analyzed: _____

VISUAL OBSERVATION PERIOD					
(Date/Day #)	SAMPLE ID	SAMPLE ID	SAMPLE ID	SAMPLE ID	SAMPLE ID

Caking Tendency (Large-Bag Method)

Caking is the formation of a coherent mass from individual particles 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-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 4°C. Relative humidity (RH) range of 45%-98%.
2. **Balance** – Platform, with a minimum 100-kg capacity and a resolution of 0.1 kg.
3. **Bags** – Woven polypropylene, with a 100 µm (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/cm² 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 the chamber.

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/cm² 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/cm². This pressure is equivalent to that placed on the bottom bag of a 20-bag stack of 22.7-kg (50-lb) bags.

The bags are stored for a specified period of time (usually 1, 3, and 6 months) at a specified temperature and RH. Alternatively, the bags can be stored in an open warehouse in uncontrolled, monitored conditions (temperature and RH). 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 each bag 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 is the formation of a coherent mass from individual particles 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-Humidity Chamber** – With horizontal airflow, internal dimensions of approximately 4.0 m wide, 5.5 m deep, and 4.0 m high. Temperatures range from 5°C to 40°C. Relative humidity (RH) range of 45%-98%.
2. **Graduated Cylinder** – With 2,000-mL capacity.
3. **Bags** – A 25- μm (1-mil) polyethylene inner liner, two 1-ply center paper bags, and a 100- μm (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/cm² 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 cm³ 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. A 10- to 15-mm thick plywood board is placed on top of the test bags. A deadweight pressure of 0.28 kg/cm² is applied by placing a “dummy” weight on top of the plywood board.

The bags are stored for a specified period of time (usually 1, 3, and 6 months) at a specified temperature and RH. Alternatively, the bags can be stored in an open warehouse in uncontrolled, monitored conditions (temperature and RH). 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 each bag 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)

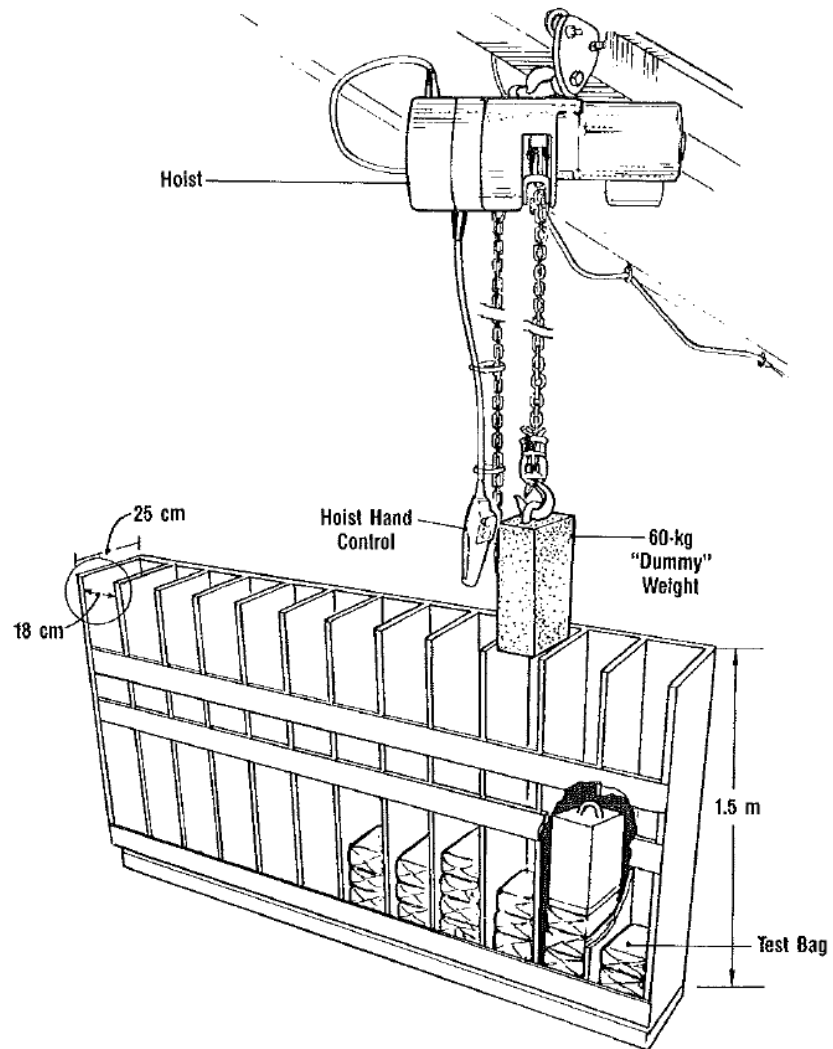


Figure 1. Storage Rack to Measure Caking Tendency of Fertilizer Using Small-Bag Method

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 22.4 mm to 38 μm . Unless a special procedure is set up, 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 22.4 mm to 38 μm (Table 1).
2. **Sieve Shakers** – Dry apparatus (Tyler Model B Ro-Tap® 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 μm)

Place sieve pan on the bottom of the nest. Split, 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 ± 0.1 g. Record weight from each sieve. Calculate results for each sieve as follows:

$$\% \text{ on sieve} = \frac{\text{Weight on sieve, g}}{\text{Weight of sample, g}} \times 100$$

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

Split, 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:

$$\% \text{ on sieve} = \frac{\text{Weight on sieve, g}}{\text{Weight of sample, g}} \times 100$$

Typical Values

Fertilizer		Cumulative % Retained on Indicated Sieve Openings, mm					
Type	Grade	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	11-52-0	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	0-0-60	0	5	38	75	93	99

(IFDC Method; this procedure basically conforms to the International Standard 8397 of the International Organization for Standardization, Case Postale 56, CH-1211, Geneva 20, Switzerland.)

Table 1. Fourth Root of Two Series of Sieve Openings^a

Size
22.4 mm
19.0 mm
16.0 mm
13.2 mm
12.5 mm ^b
11.2 mm
9.5 mm
8.0 mm
6.7 mm
6.3 mm ^b
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. Conforms to the R40/3 sieve series of the International Standard 565. International Organization for Standardization, Case Postale 56, CH-1211, Genève 20, Switzerland.

b. These sieves are not in the fourth root of two series, but they have been included because they are in common usage.

Table 2. Quantity of Material Required for Test Sieving^a

Screen Opening	Recommended Quantity (g)
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.

LABORATORY REPORT OF SIEVE ANALYSIS

(IFDC S-107)

Sample ID: _____

Analyzed By: _____

Weight_{initial}: _____

Date Analyzed: _____

STANDARD SIEVE SERIES			WEIGHT ON OR BETWEEN SIEVES		CUMULATIVE PERCENTAGE	
OPENING	TYLER No.	U.S. No.	(grams)	(%)	ON	PASSING
(mm)						
TOTALS						

Size Guide Number and Uniformity Index Determinations

The Size Guide Number (SGN) is the particle size which divides the mass of all particle in two equal halves, one having all the larger size particles and the other half having all smaller size particles. SGN is the median dimension expressed in millimeters to the second decimal then multiplied by 100. SGN is calculated by interpolation from size analyses data.

The Uniformity Index (UI) is the ratio of particle sizes, small to large in the product, expressed in percentage. UI is the ratio, times 100, of the size corresponding to the 95% level and the 10% in the cumulative distribution. As with SGN, UI is calculated from size analyses data using the same interpolation formula used for SGN.

The particle size analysis of a sample is determined by the size analysis, sieve method (IFDC S-107).

To determine SGN and UI, suppose a material has the following particle size analysis:

Fertilizer Particle Size Analysis	
Sieve, mm	Cumulative Percentage Retained, %
4.00	0.9
3.35	13.3
2.80	46.4
2.36	77.0
2.00	93.9
1.70	98.2

For SGN: The equation used for SGN is:

$$SGN = \frac{a (CRA - k)}{CRA - CRB} + b$$

where,

$$k = 50$$

$$a = \text{aperture difference} = 280 - 236 = 44$$

$$b = \text{aperture of the sieve retaining a proportion greater than } k = 236$$

$$CRA = \text{Cumulative Retained Above } k = 77.0$$

$$CRB = \text{Cumulative Retained Below } k = 46.4.$$

$$\text{Size Guide Number, } SGN = \frac{44 (77.0 - 50)}{77.0 - 46.4} + 236 = 275$$

For UI: The equations are:

$$\text{Large } (L) = \frac{a (CRA - k)}{CRA - CRB} + b \quad (1)$$

where,

$$k = 10$$

$$a = \text{aperture difference} = 400 - 335 = 65$$

$$b = \text{aperture of the sieve retaining a proportion greater than } k = 335$$

$$\text{CRA} = \text{Cumulative Retained Above } k = 13.3$$

$$\text{CRB} = \text{Cumulative Retained Below } k = 0.9.$$

$$\text{Small } (S) = \frac{a (\text{CRA} - k)}{\text{CRA} - \text{CRB}} + b \quad (2)$$

where,

$$k = 95$$

$$a = \text{aperture difference} = 200 - 170 = 30$$

$$b = \text{aperture of the sieve retaining a proportion greater than } k = 170$$

$$\text{CRA} = \text{Cumulative Retained Above } k = 98.2$$

$$\text{CRB} = \text{Cumulative Retained Below } k = 93.9.$$

$$UI = S/L * 100 \quad (3)$$

where,

S = Small Value

L = Large value

$$\text{Large } (L) = \frac{65 (13.3 - 10)}{13.3 - 0.9} + 335 = 353$$

$$\text{Small } (S) = \frac{30 (98.2 - 95)}{98.2 - 93.9} + 170 = 192$$

$$\text{Uniformity Index, } UI = \frac{192}{353} * 100 = 54$$

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/or (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 Fertilizer Institute Method)

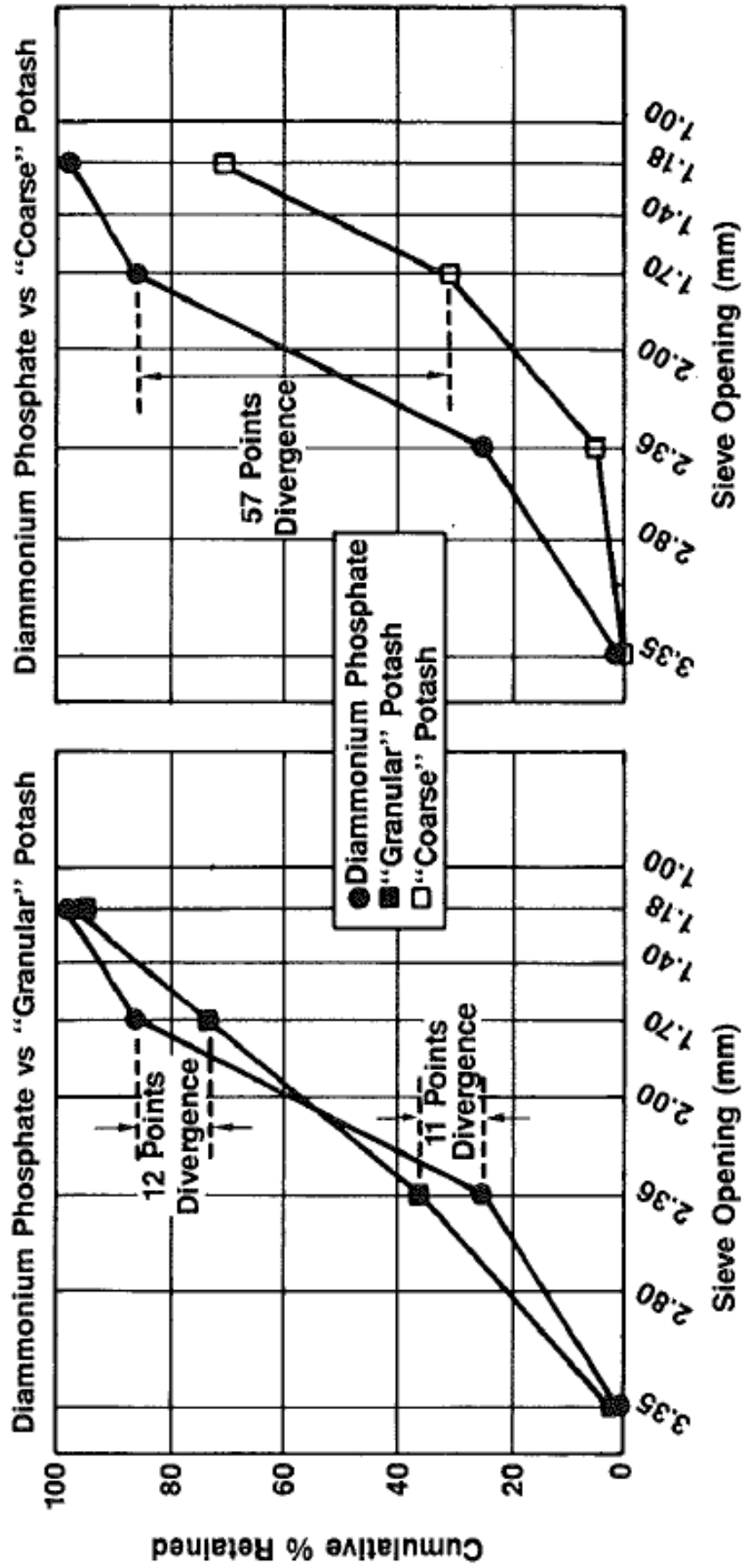


Figure 1. Physical Compatibility of Bulk Blend Ingredients According to Cumulative Particle-Size Distribution

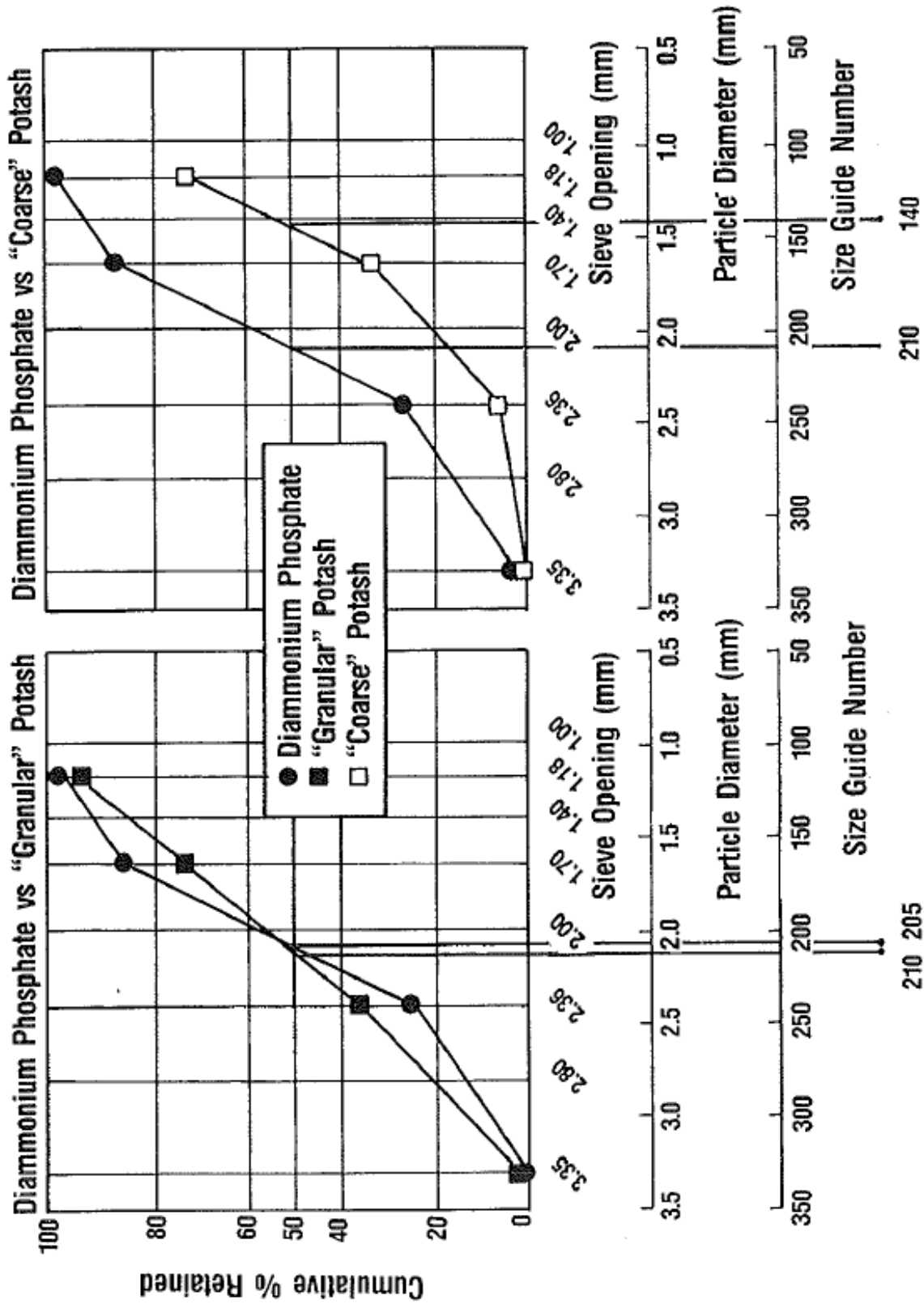


Figure 2. Physical Compatibility of Bulk Blend Ingredients According to Size Guide Number

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 plastic 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, plastic, 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 Range of Values

Fertilizer		Angle of Repose, Degrees
Type	Grade	
Prilled Urea	46-0-0	27-28
Granular Urea	46-0-0	34-38
Granular Ammonium Sulfate	21-0-0	36-38
Prilled Ammonium Nitrate	34-0-0	29-38
Granular Diammonium Phosphate	18-46-0	27-37
Granular Monoammonium Phosphate	11-52-0	28-37
Granular Triple Superphosphate	0-46-0	28-35
Granular Potassium Chloride	0-0-60	32-41

(This procedure basically conforms to the International Standard 8398 of the International Organization for Standardization, Case Postale 56, CH-1211, Genève 20, Switzerland.)

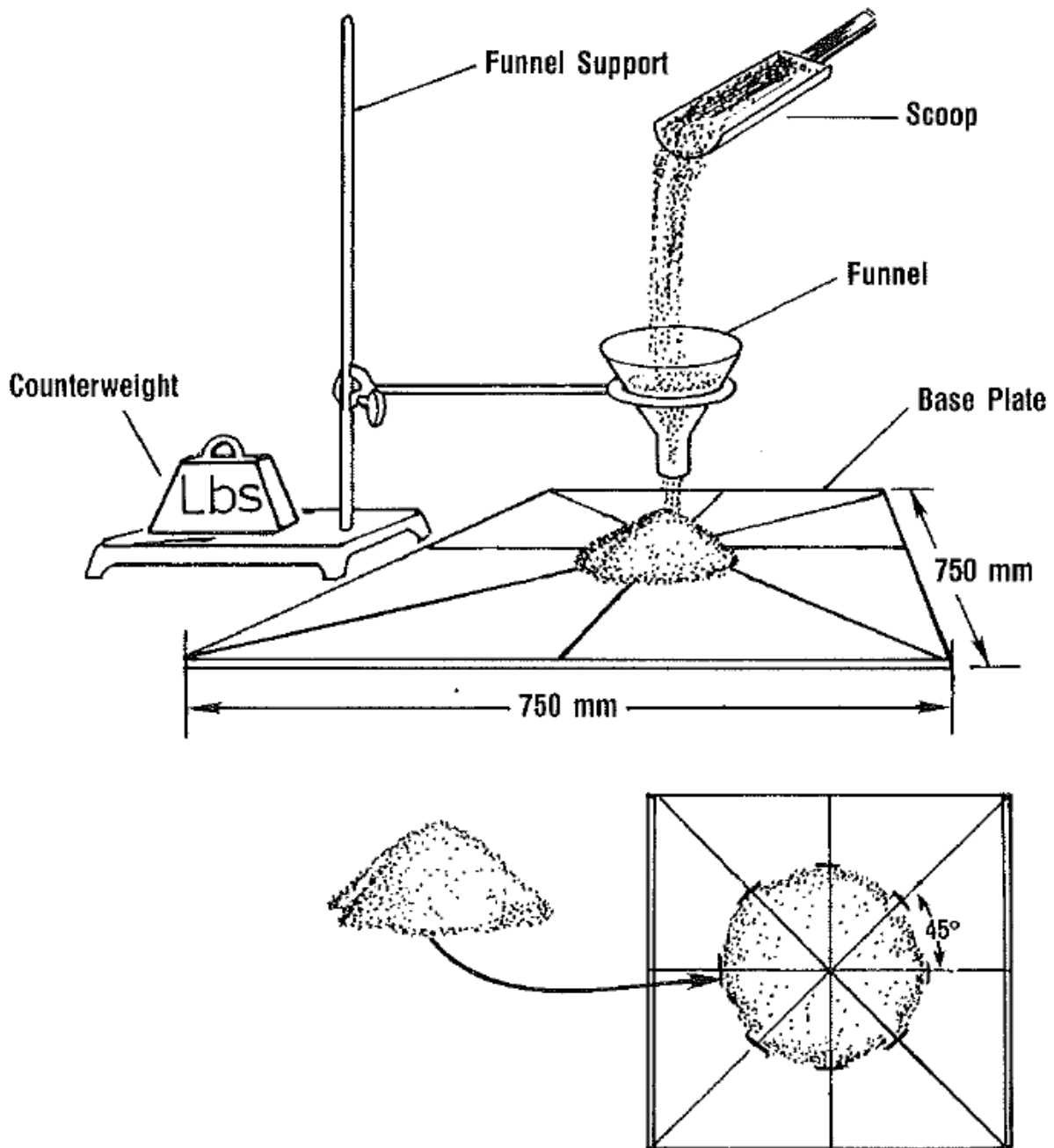


Figure 1. Apparatus for the Determination of Angle of Repose of Fertilizer

Bulk Density (Loose Pour)

Bulk density (loose) is the mass per unit volume of a material, after it has been poured freely into a container under clearly specified conditions. Bulk density is a measure of the material density, material porosity, and the voids between the particles of the material. 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 m³ (1 ft³).
2. **Balance** – Top loading, with a minimum 100-kg capacity and a resolution of 0.1 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 by 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/m³.

SW = weight of test sample, kg.

V = volume of metal box, 0.0283 m³.

Typical Range of Values

Fertilizer		Bulk Density (Loose-Pour)
Type	Grade	(kg/m ³)
Prilled Urea	46-0-0	720-820
Granular Urea	46-0-0	720-820
Granular Ammonium Sulfate	21-0-0	785-1,040
Prilled Ammonium Nitrate (High Density)	34-0-0	850-975
Granular Diammonium Phosphate	18-46-0	875-1,100
Granular Monoammonium Phosphate	11-52-0	900-1,100
Granular Triple Superphosphate	0-46-0	950-1,200
Granular Potassium Chloride	0-0-60	950-1,200

(TVA Method; This procedure basically conforms to the International Standard 3944 of the International Organization for Standardization, Case Postale 56, CH-1211 Geneva 20, Switzerland.)

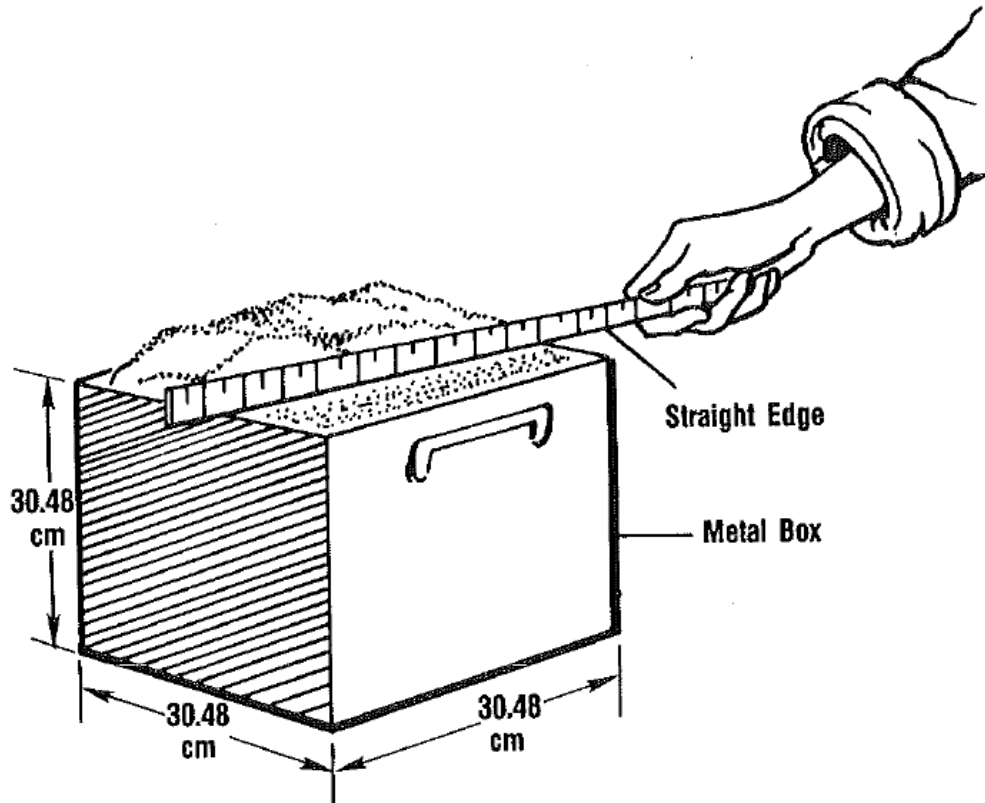


Figure 1. Metal Box (0.0283 m^3) for Measuring Bulk Density (Loose) of Fertilizers

Bulk Density (Tapped)

Bulk density (tapped) is the mass per unit volume of a material, poured into a container and then compacted under clearly specified conditions. Bulk density is a measure of the material density, material porosity, and the voids between the particles of the material. 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 m³ (1 ft³).
2. **Balance** – Top loading, with a minimum 100-kg capacity and a resolution of 0.1 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. Each side of the box is tapped several times. The box is lifted a few centimeters off a concrete floor and dropped. Lifting and dropping are repeated until compaction of the sample is complete. If a void in the box is created by the tapping and dropping action, an excess is once again poured into the box. The tapping, dropping, and filling procedure is repeated until no further compaction is evident. 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/m³.

SW = weight of test sample, kg.

V = volume of metal box, 0.0283 m³.

Typical Range of Values

Fertilizer		Bulk Density (Tapped) (kg/m ³)
Type	Grade	
Prilled Urea	46-0-0	820-900
Granular Urea	46-0-0	820-900
Granular Ammonium Sulfate	21-0-0	850-1,100
Prilled Ammonium Nitrate (High Density)	34-0-0	900-1,025
Granular Diammonium Phosphate	18-46-0	1,000-1,200
Granular Monoammonium Phosphate	11-52-0	1,000-1,200
Granular Triple Superphosphate	0-46-0	1,050-1,350
Granular Potassium Chloride	0-0-60	1,000-1,200

(TVA Method; Modified; This procedure basically conforms to the International Standard 5311 of the International Organization for Standardization, Case Postale 56 CH-1211, Geneva 20, Switzerland.)

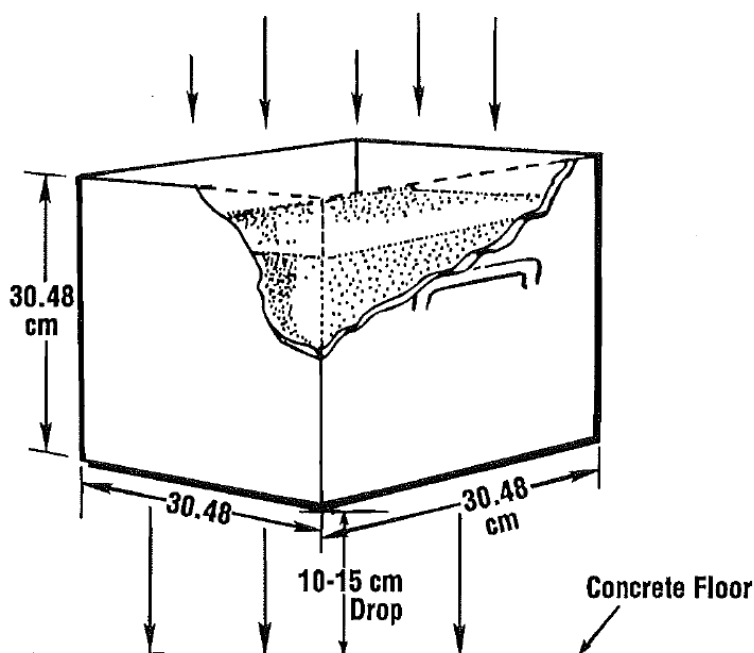


Figure 1. Metal Box (0.0283 m³) for Measuring Bulk Density (Tapped) of Fertilizers, Illustrating Drop

Apparent Density

Apparent density is the mass per unit volume of a material. Apparent density is a measure of the material density and material porosity and excludes the voids between the 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 20-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) burette 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® 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 and below 15.0 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 burette, is opened and stopcock “B,” above the sample chamber, is opened to “vent.” The cover then is removed from the mercury reservoir above the burette 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 burette 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 burette. This reading represents “zero displacement” of mercury by sample since there is, as yet, no sample in the chamber.
3. Stopcock “C” above the burette 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 burette. When the sample chamber is almost emptied, 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 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 burette, 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 burette. 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 (cm³) is taken to be equivalent to the final burette reading in mL minus the original burette reading in mL.

Typical Range of Values

Fertilizer		Apparent Density
Type	Grade	(g/cm ³)
Prilled Urea	46-0-0	1.30-1.32
Granular Urea	46-0-0	1.23-1.30
Granular Ammonium Sulfate	21-0-0	1.58-1.65
Prilled Ammonium Nitrate (High Density)	34-0-0	1.65-1.70
Granular Diammonium Phosphate	18-46-0	1.63-1.65
Granular Monoammonium Phosphate	11-52-0	1.67-1.78
Granular Triple Superphosphate	0-46-0	1.93-2.12
Granular Potassium Chloride	0-0-60	1.96-1.99

Auxiliary Method

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

(TVA Method)

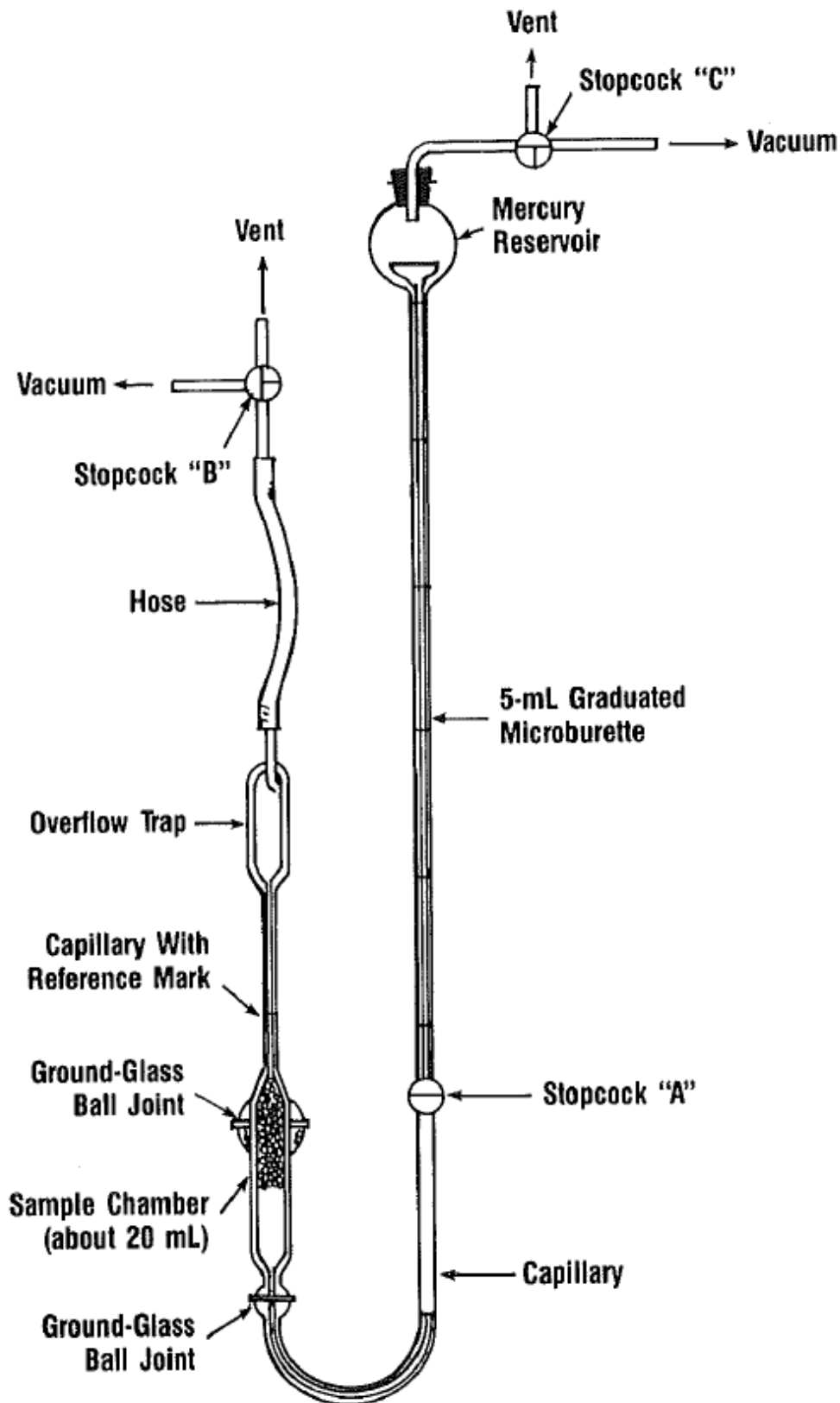


Figure 1. Apparatus for Determination of Apparent Density of Fertilizer Granules

True Density of Solids

True density is the mass 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. **Quantachrome MultiPycnometer (Figure 1)** – Model MVP-6DC, including accessories such as sample cells, calibration spheres, and fittings for gas tank connections.
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- μm opening.
4. **Test Sieve** – 20 cm in diameter and 5 cm high with openings of 300 μm , a sieve cover, sieve pan, and a Ro-Tap® 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 μm .

Purging of MultiPycnometer, taken from Quantachrome operating manual, if applicable: To purge contamination gas or vapor from the system, attach a length of hose to the “VENT” hose connection on the right side of the MultiPycnometer and immerse the other end of the hose into a beaker of water. Perform the following steps to purge:

1. Close the “GAS IN” toggle valve and open the “GAS OUT” toggle valve. Turn selector valve to “CELL.”
2. Open “GAS OUT RATE” control fully counterclockwise.
3. Close “GAS OUT RATE” needle valve fully clockwise. Do not force or overtighten and open the “GAS IN” toggle valve.
4. Adjust “GAS IN RATE” needle valve to give a slow rate of bubbling in the beaker of water.
5. Remove tubing from water.
6. After 10 to 20 minutes of flow, close the “GAS IN” toggle valve.

Procedure from Quantachrome Operating Manual

Power “ON” and allow 10 to 15 minutes for the pressure transducer to warm up and stabilize.

1. Weigh the small sample cell cup. Fill the small sample cell cup with material to be tested and weigh. Calculate the sample weight from the total weight of the sample minus the weight of the small sample cell.
2. Remove the cell cover, insert the sample cell cup into the cell holder, and replace cover.
3. Open the “GAS OUT” and “GAS OUT RATE” control. Wait for a stable zero reading.
4. Close the “GAS OUT” toggle valve and set the meter to zero.
5. Turn the selector valve to “REF.”
6. Open the “GAS IN” toggle valve and pressurize to approximately 17 psig or 1.195 kg/cm^2 using the “GAS IN RATE” needle valve to control the rate of pressurization. Stop the flow by closing the “GAS IN” toggle valve.
7. Record the display reading after it has stabilized. This value is the pressure reading after pressurizing the small reference volume or P_1 .
8. Turn the selector valve to “CELL.”

9. Record the display reading after it has stabilized. This value is the pressure reading after including the small sample cell volume or P_2 .
10. Vent the pressure slowly to prevent blowing powder out of the cell by opening the “GAS OUT” toggle valve with the “GAS OUT RATE” control slightly open.
11. Use equations (A) and (B) to calculate the true powder volume and true density of the material.

$$V_P = V_C - V_R \left(\left(\frac{P_2}{P_1} \right) - 1 \right) \quad A$$

where,

V_P = Volume of Powder (cm^3)

V_C = Volume of Small Sample Cell (cm^3) = 28.839 cm^3

V_R = Small Cell Reference Volume (cm^3) = 10.850 cm^3

P_1 = Pressure Reading After Pressurizing the Small Reference Volume

P_2 = Pressure Reading After Including V_C .

$$\text{True Density, } g/\text{cm}^3 = \frac{\text{Sample Weight, } g}{V_P, \text{cm}^3} \quad B$$

12. Repeat steps 3 through 11 two additional times. Once the readings have been taken three times, take the average the density values of the three runs.
13. If questions arise concerning procedure, please refer to the operating manual for the instrument.

Auxiliary Method

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

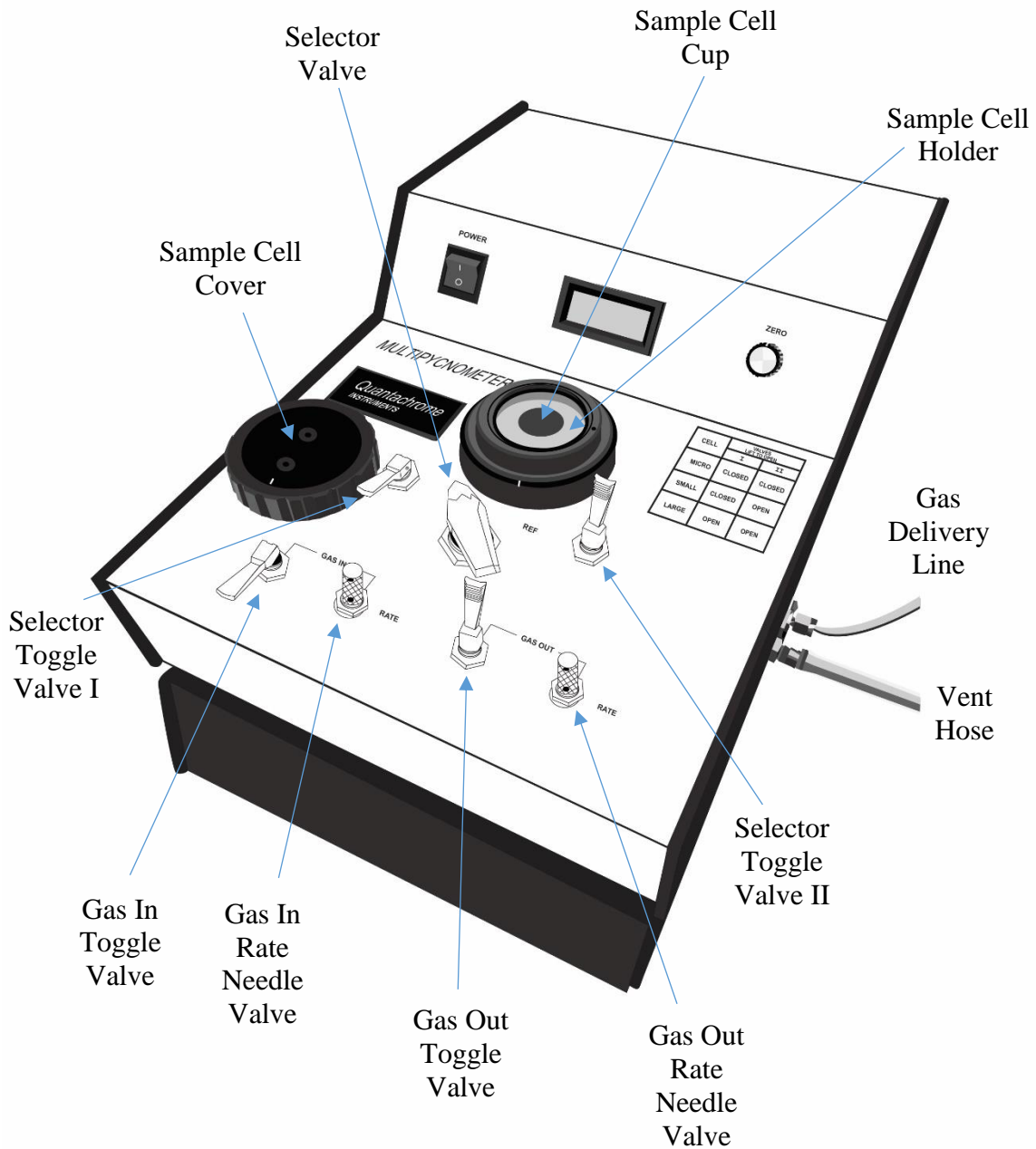


Figure 1. Quantachrome Instruments MultiPycnometer for Measuring True Density of Solids

TRUE DENSITY OF SOLIDS

(Quantachrome Instruments Model MVP-6DC)

(IFDC S-114)

Sample ID: _____

Analyzed By: _____

Date Analyzed: _____

Tare Weight: _____ g

Total Weight: _____ g

Sample Weight: _____ g

Small Cell Reference Volume (V_R): _____ cm^3 Small Sample Cell Volume (V_C): _____ cm^3

Operational Equation

$$V_P = V_C - V_R \left(\left(\frac{P_2}{P_1} \right) - 1 \right)$$

 V_P = Volume of Powder (cm^3) V_C = Volume of Small Sample Cell (cm^3) V_R = Small Cell Reference Volume (cm^3) P_1 = Pressure Reading After Pressurizing the Reference Volume P_2 = Pressure Reading After Including V_C

	DATA			
	Run 1	Run 2	Run 3	Average of 3 Runs
P_1	_____	_____	_____	_____
P_2	_____	_____	_____	_____
V_P	_____	_____	_____	_____
Density	_____	_____	_____	_____

Granule Crushing Strength

Crushing strength is the minimum force required to crush individual particles. 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 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 Range of Values

Fertilizer		Crushing Strength
Type	Grade	(kg/granule)
Prilled Urea	46-0-0	0.8-1.2
Granular Urea	46-0-0	1.5-3.5
Granular Ammonium Sulfate	21-0-0	1.5-2.5
Prilled Ammonium Nitrate	34-0-0	1.2-1.7
Granular Diammonium Phosphate	18-46-0	3.0-5.0
Granular Monoammonium Phosphate	11-52-0	2.0-3.0
Granular Triple Superphosphate (Accretion Process)	0-46-0	4.5-8.0
Granular Triple Superphosphate (Steam Granulation)	0-46-0	1.5-3.0
Prilled Potassium Nitrate	13-0-44	1.5-2.0
Granular Potassium Chloride	0-0-60	3.0-5.0
Granular Potassium Sulfate	0-0-50	3.0-4.0

Auxiliary Method

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

(TVA Method)

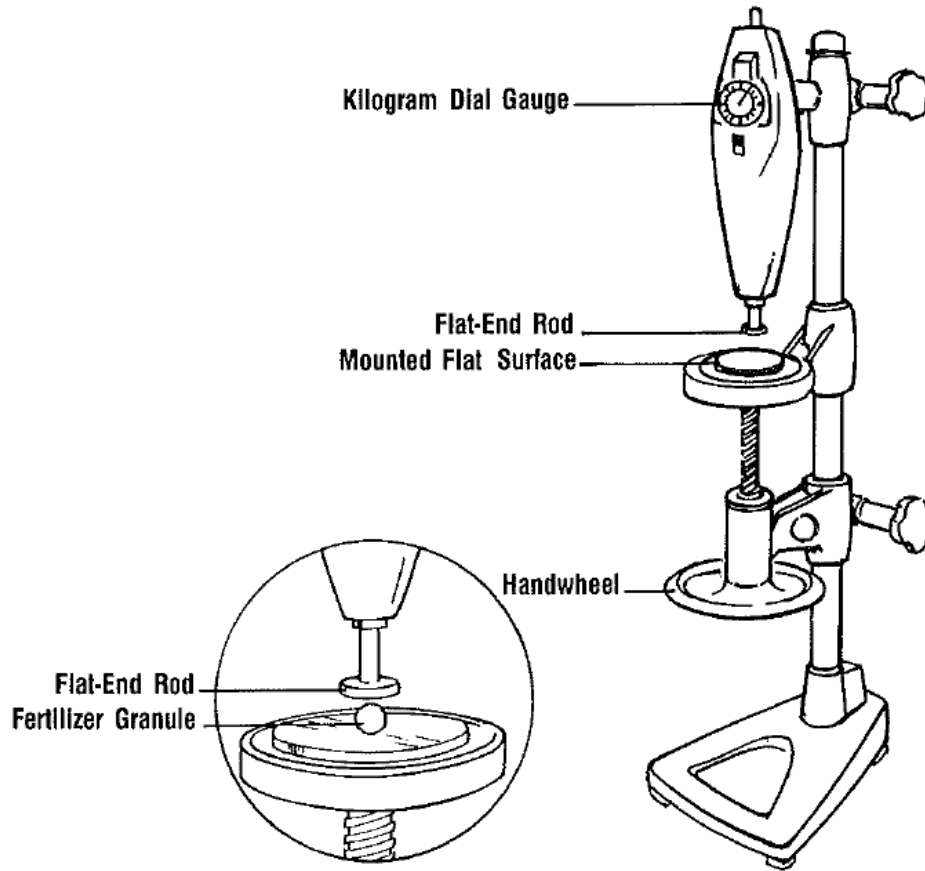


Figure 1. Chatillon Compression Tester for Measuring Granule Crushing Strength

GRANULE CRUSHING STRENGTH

(IFDC S-115)

Analyzed By: _____

Size Fraction: _____

Sample ID:					
Date of Test:					
Date of Crushing Test:					
No.	kg/granule	kg/granule	kg/granule	kg/granule	kg/granule
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
Average					
Range					

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® 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 (± 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 representative 100-cm³ portion of the minus 3.35- plus 1.00-mm fraction is used in the test. A representative portion is obtained by riffing the sample to an amount as close as possible to 100 cm³. To avoid “blinding,” the amount remaining on the 1.00-mm sieve should not exceed 140 cm³.

Procedure

A 100-cm³ 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® shaker. The material retained on the 1.00-mm screen then is weighed accurately, and “percent degradation” is calculated as follows:

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

Typical Range of Values

Fertilizer		Abrasion Resistance
Type	Grade	(% Degradation)
Prilled Urea	46-0-0	10.0-30.0
Granular Urea	46-0-0	0.2-3.0
Granular Ammonium Sulfate	21-0-0	0.2-1.0
Prilled Ammonium Nitrate	34-0-0	1.5-27.0
Granular Diammonium Phosphate	18-46-0	0.5-2.0
Granular Monoammonium Phosphate	11-52-0	0.5-2.0
Granular Triple Superphosphate (Accretion Process)	0-46-0	0.1-1.0
Granular Triple Superphosphate (Steam Granulation)	0-46-0	1.0-5.0
Prilled Potassium Nitrate	13-0-44	10.0-20.0
Granular Potassium Chloride	0-0-60	2.0-5.0
Granular Potassium Sulfate	0-0-50	5.0-7.0

Auxiliary Method

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

(TVA Method)

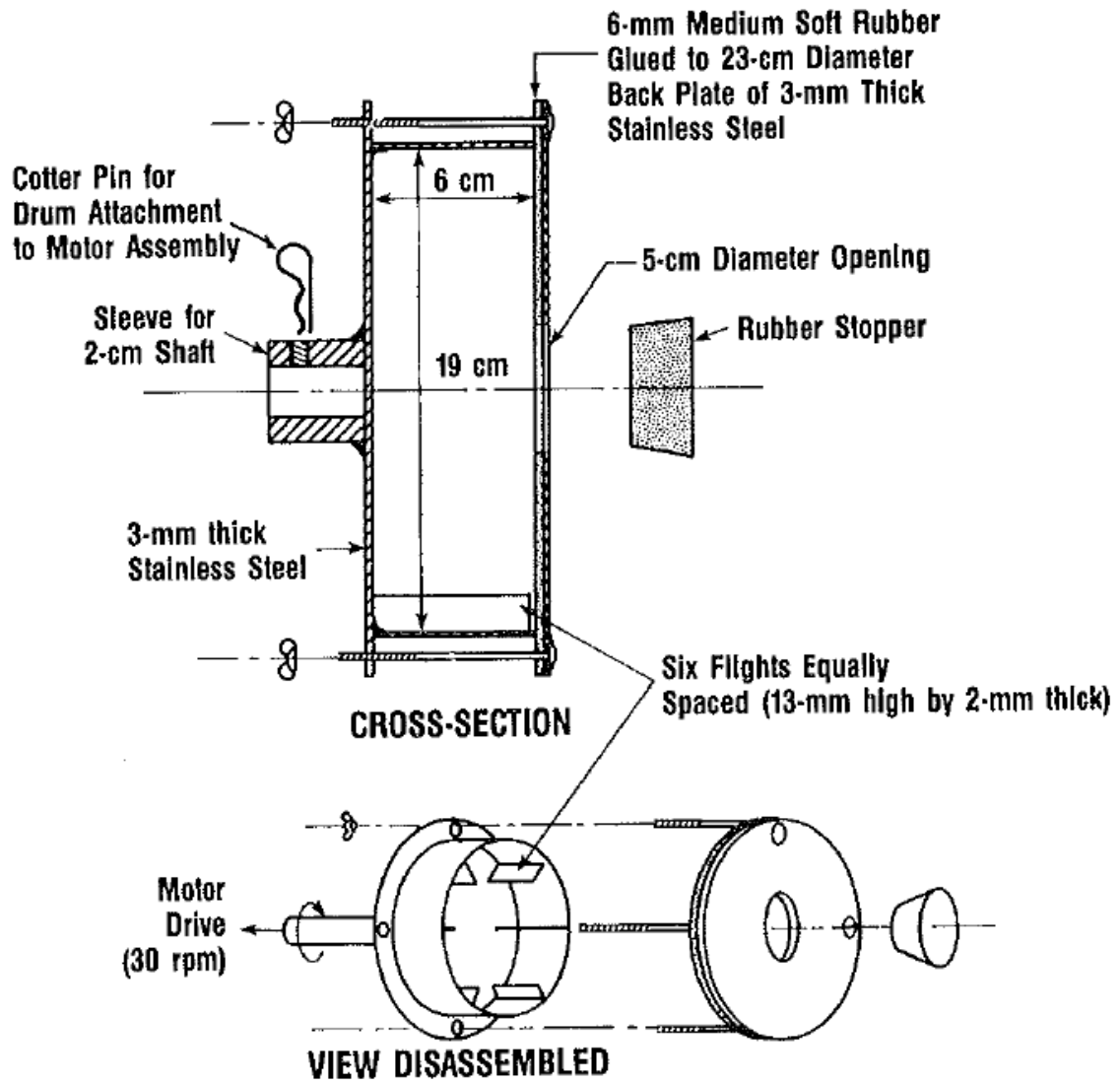


Figure 1. Rotary Drum Used in Abrasion Resistance Test

Abrasion Resistance (Sieve Method)¹

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® 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 (± 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 Preparation²

Add 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 (± 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 (± 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 μ m to 6.7 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:

$$\text{Degradation, \%} = 100 \times \frac{\text{Weight of material collected in bottom pan, g}}{\text{Weight of subsample, g}}$$

Typical Range of Values

Fertilizer		Abrasion Resistance
Type	Grade	(% Degradation)
Prilled Urea	46-0-0	10.0-30.0
Granular Urea	46-0-0	0.4-2.0
Granular Ammonium Sulfate	21-0-0	0.5-1.5
Prilled Ammonium Nitrate	34-0-0	5.0-20.0
Granular Diammonium Phosphate	18-46-0	0.4-1.0
Granular Monoammonium Phosphate	11-52-0	0.4-1.0
Granular Triple Superphosphate	0-46-0	0.6-1.0
Prilled Potassium Nitrate	13-0-44	10.0-20.0
Granular Potassium Chloride	0-0-60	1.0-3.0
Granular Potassium Sulfate	0-0-50	4.0-6.0

Auxiliary Method

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

(IFDC Method)

Table 1. Fourth Root of Two Series of Sieve Openings^a

Size
6.70 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. Conforms to the R40/3 sieve series of the International Standard 565. International Organization for Standardization, Case Postale 56, CH-1211, Genève 20, Switzerland.

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 μm	0.4
710 μm	0.1
600 μm	0.2
Total	100.0

Impact Resistance

Impact resistance is the resistance of granules to breakage upon impact against a hard surface. Impact resistance is of interest when fan-type fertilizer spreaders are used, when a material is discharged from an overhead point into a bulk pile (such as loading into a shiphold), 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® 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** – Top loading with a minimum 250-g capacity and a resolution of 0.1 g.

Sample Preparation¹

Add 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 (± 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.

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

Procedure

The 100-g (± 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:

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

Typical Range of Values

Fertilizer		Impact Resistance
Type	Grade	(% Shattered Granules)
Prilled Urea	46-0-0	5.0-15.0
Granular Urea	46-0-0	0.1-1.0
Granular Ammonium Sulfate	21-0-0	0.1-0.5
Prilled Ammonium Nitrate	34-0-0	3.0-6.0
Granular Diammonium Phosphate	18-46-0	0.5-1.5
Granular Monoammonium Phosphate	11-52-0	0.5-1.5
Granular Triple Superphosphate	0-46-0	0.0-1.0
Prilled Potassium Nitrate	13-0-44	5.0-10.0
Granular Potassium Chloride	0-0-60	2.0-5.0
Granular Potassium Sulfate	0-0-50	5.0-10.0

Auxiliary Methods

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

(TVA Method; Modified)

Table 1. Fourth Root of Two Series of Sieve Openings^a

Size
22.4 mm
19.0 mm
16.0 mm
13.2 mm
12.5 mm ^b
11.2 mm
9.5 mm
8.0 mm
6.7 mm
6.3 mm ^b
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

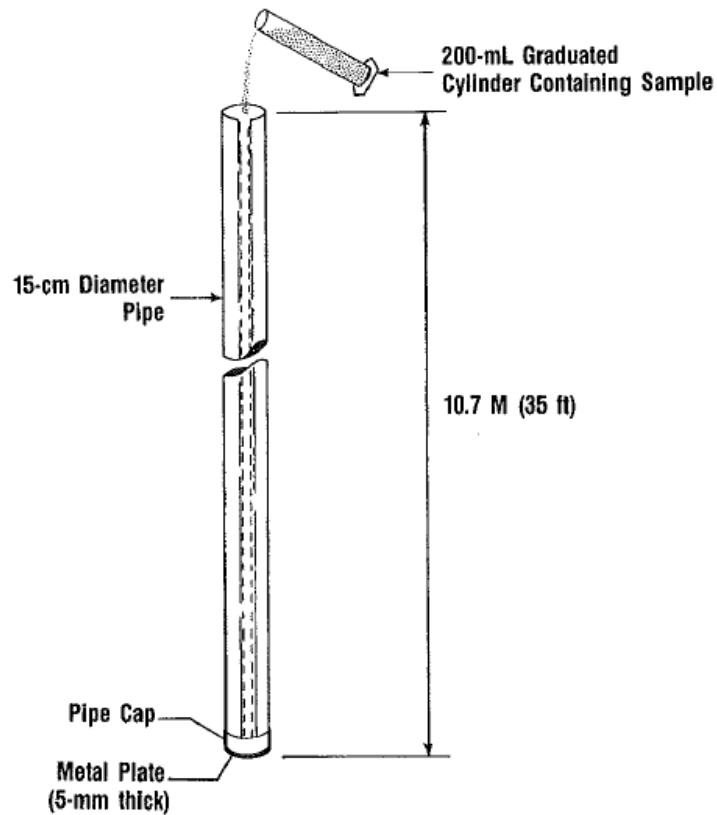
- a. Conforms to the R40/3 sieve series of the International Standard 565. International Organization for Standardization, Case Postale 56, CH-1211, Genève 20, Switzerland.
- b. These sieves are not in the fourth root of two sieves, but they have been included because they are in common usage.

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 μm	0.4
710 μm	0.1
600 μm	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:

$$\text{SHOCK RESISTANCE, \% of unbroken prills} = \frac{B}{A} \times 100\%$$

where,

A = the percentage of round prills in the sieved product before the test.

B = the percentage of round prills in the sieved product after the test.

Typical Values

Fertilizer		Shock Resistance
Type	Grade	(% unbroken prills)
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)

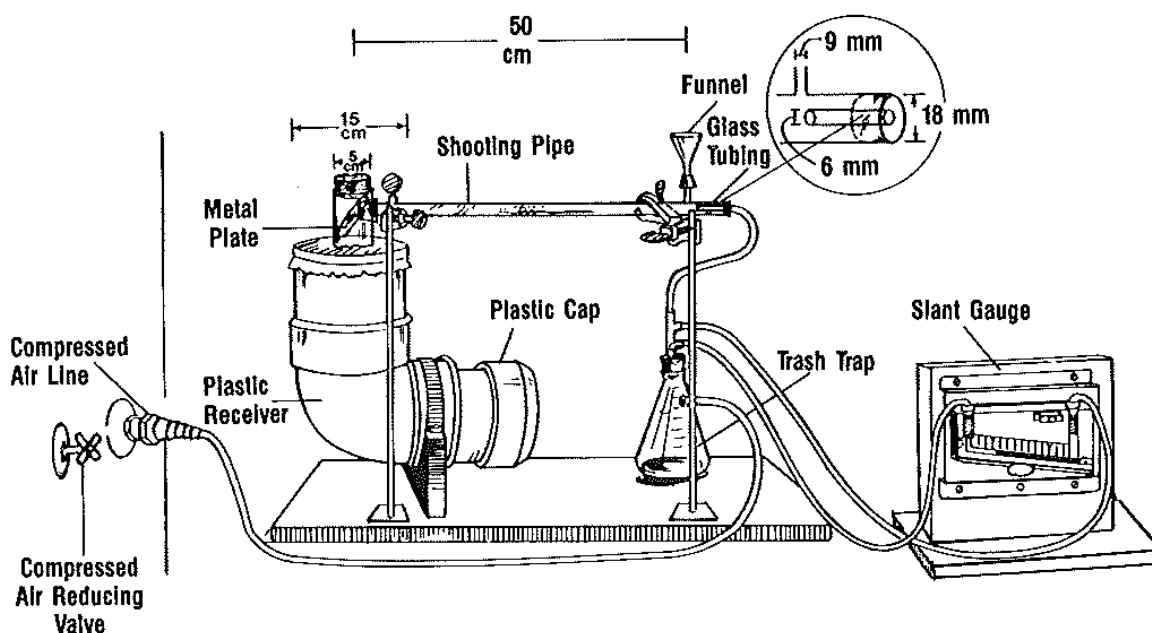


Figure 1. Apparatus for Determining the Shock Resistance of Prills

Handling Strength Test

The handling strength test determines the mechanical strength of granular fertilizers during maximum handling procedures. Breakage of product 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 m³.
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 100 µm (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 oversize 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

<u>Test Step – Simulated Handling Procedure</u>	<u>Actual Handling Procedure Simulated</u>
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:

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

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)

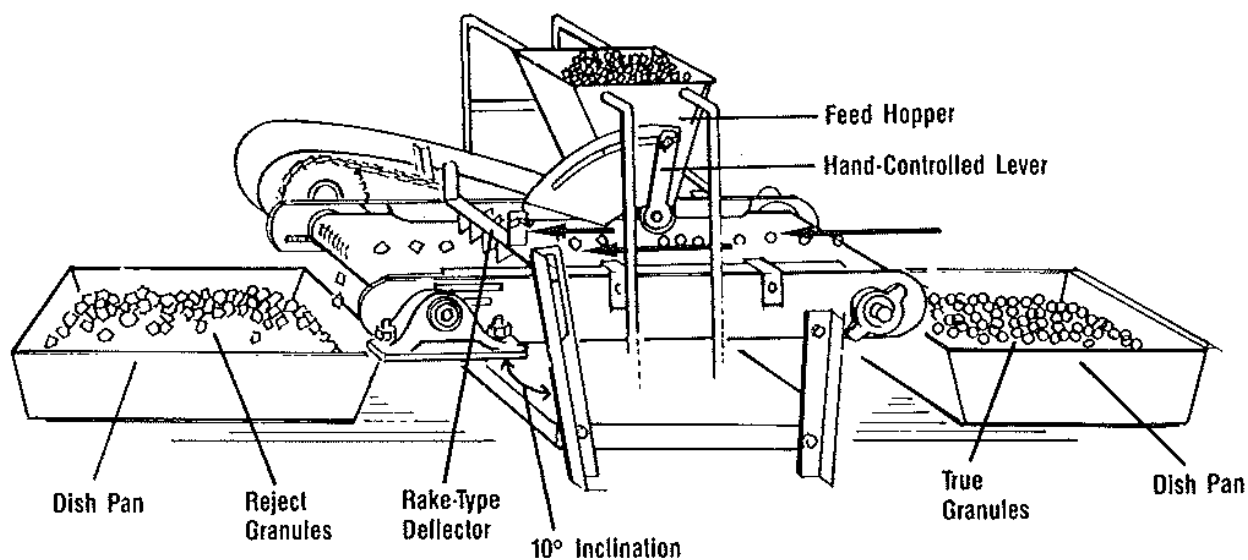


Figure 1. Sphericity Apparatus for Measuring Roundness of Granular Fertilizer

Conditioner Adherence/Dustiness

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-cm³ 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 granular fertilizers can also be determined by this method (see dustiness section of this 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 m³/h) calculated to give a superficial velocity of 0.9 m/s for conditioner adherence and 1.0 m/s for dustiness of granular fertilizers.
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 cm³ 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-cm³ portion of the plus 1.00-mm fraction is used in the test. Obtaining a 250-cm³ sample may require screening two or three subsamples to avoid “blinding” of the screen. For measuring dustiness, the sample should be used “as-received,” without any screening.

Procedure

Coating – It has been found that the degree of adherence of conditioners partially 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 provided in plant conditioning drums. The following procedure was designed to meet these requirements.

In conducting a test, a 250-cm³ 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”¹ 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.² 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 repass 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:

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

where,

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.

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)

Dustiness – For measuring dustiness of granular fertilizers, an unscreened representative sample (approximately 250 cm³) should be accurately weighed and passed through the dedusting tower (as described in adherence test) only once at a velocity of 1.0 m/s. The amount of dust is calculated as follows:

$$\text{Dustiness, mg/kg of product} = \frac{D(1,000)}{OW} \times 1,000$$

Where,

D = weight, g, of dust loss after one pass through dedusting tower at 1.0 m/s velocity.

OW = weight, g, of original sample before pass through dedusting tower.

Samples can be rated according to the following values.

Dust Content (mg/kg of product)	Observed Dustiness in Handling
<200	Little dust emission
200-500	Some dust emission
500-1,000	High dust emission
1,000-2,000	Very high dust emission
>2,000	Extremely high dust emission

(IFDC method)

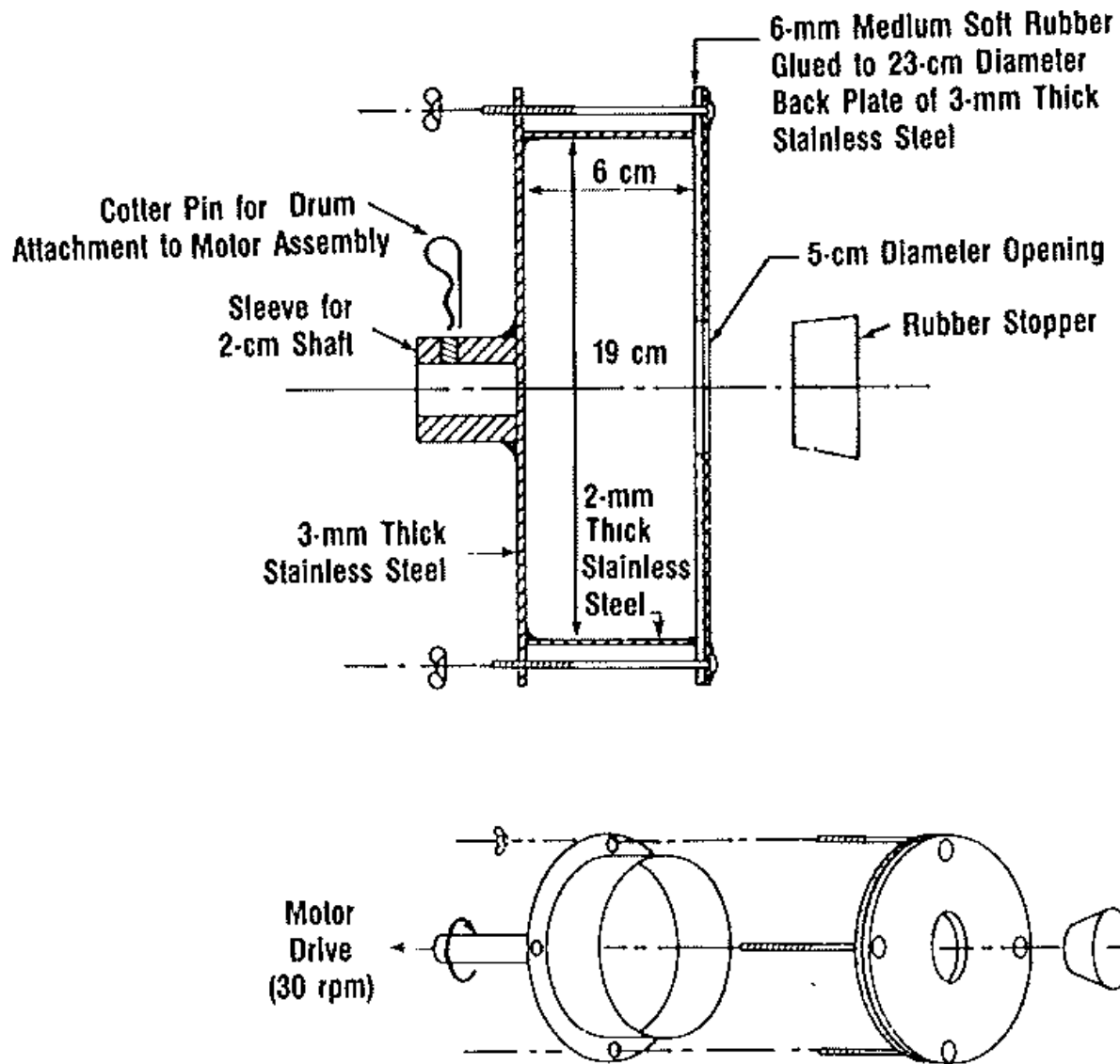


Figure 1. Laboratory-Scale Rotary Conditioning Drum

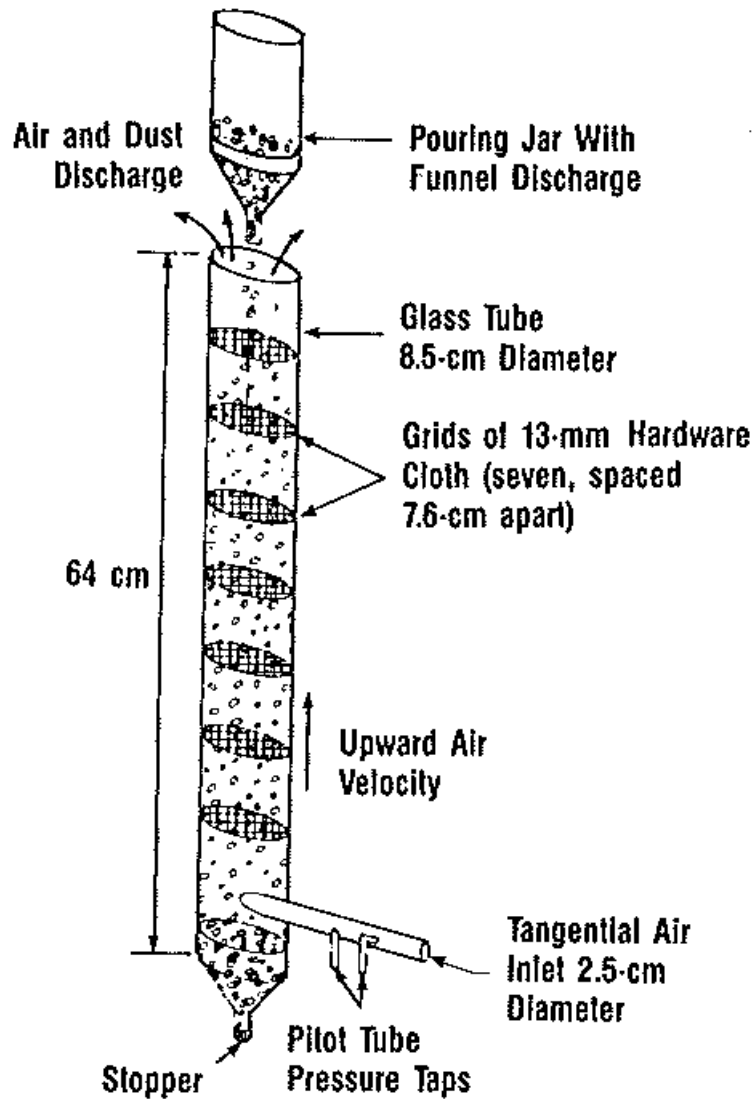


Figure 2. Dedusting Tower for Measurement of Conditioner Adherence or Dustiness

CONDITIONER ADHERENCE

(IFDC S-122)

Analyzed By: _____

Date Analyzed: _____

Sample Volume: _____

Drum

Dedusting Tower

RPMs: _____

Air Velocity: 1 meter / second

Retention time: _____

H₂O Displacement: 0.55 inches of water

SAMPLE ID	ORIGINAL SAMPLE WEIGHT	WEIGHT LOSS OF DUST AFTER EACH OF SIX SUCCESSIVE PASSES						FINAL SAMPLE WEIGHT	CONDITIONER WEIGHT	CONDITIONER ADHERENCE
	(A)	(g)						(B)	(C)	100 - $\frac{100(A-B)}{C}$
	(g)	1	2	3	4	5	6	(g)	(g)	(%)

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® 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 Preparation¹

Add 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 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

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

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, so the sieve submerged in the water attached to the wrist-action shaker would have openings of 850 µm. It is important to make 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 so 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		Disintegration Rate, %	
Type	Grade	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	11-52-0	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^a

Size
6.70 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
425 μm
355 μm
300 μm

a. Conforms to the R40/3 sieve series of the International Standard 565. International Organization for Standardization, Case Postale 56, CH-1211, Genève, Switzerland.

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 μm	0.4
710 μm	0.1
600 μm	0.2
Total	100.0

Porosity

Porosity is a measurement of the pore space within fertilizer granules. External and, for the most part, 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. **Quantachrome Instruments MultiPycnometer** – 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 μm and one with openings of 1.00 mm, a sieve cover, a sieve pan, and a Ro-Tap® testing sieve shaker (Tyler Model B) or an equivalent shaker.
5. **Grinder** – Capable of reducing sample to 100% passing a 300- μm 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 μm . This sample is used to determine the true density in the multipycnometer.

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 Quantachrome Instruments MultiPycnometer (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:

$$\text{Porosity, \%} = \frac{T_q - A_q}{T_q} \times 100$$

Where,

T_q = true density, g/cm^3 .

A_q = apparent density, g/cm^3 .

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 Range of Values

Fertilizer		Porosity
Type	Grade	% of Pores
Prilled Urea	46-0-0	1-3
Granular Urea	46-0-0	5-8
Prilled Ammonium Nitrate	34-0-0	2-4
Granular Ammonium Sulfate	21-0-0	8-10
Granular Diammonium Phosphate	18-46-0	2-6
Granular Monoammonium Phosphate	11-52-0	5-10
Granular Triple Superphosphate	0-46-0	10-15
Granular Potassium Chloride	0-0-60	1-2

(IFDC Method)

POROSITY

(IFDC S-125)

Analyzed By: _____

Date Analyzed: _____

SAMPLE ID	TRUE DENSITY MEASUREMENT	APPARENT DENSITY MEASUREMENT	POROSITY
	$T\rho$	$A\rho$	$\frac{(T\rho - A\rho)}{T\rho} \times 100$
	(g/cm ³)	(g/cm ³)	(%)

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)**¹ – 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.
4. **Glass Thermometers** – With ranges capable of measuring the desired temperature with a resolution of at least 0.1°C.

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.

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

Typical Values

Phosphoric Acid Rock Source	Acid P ₂ O ₅ (%)	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, Modified)

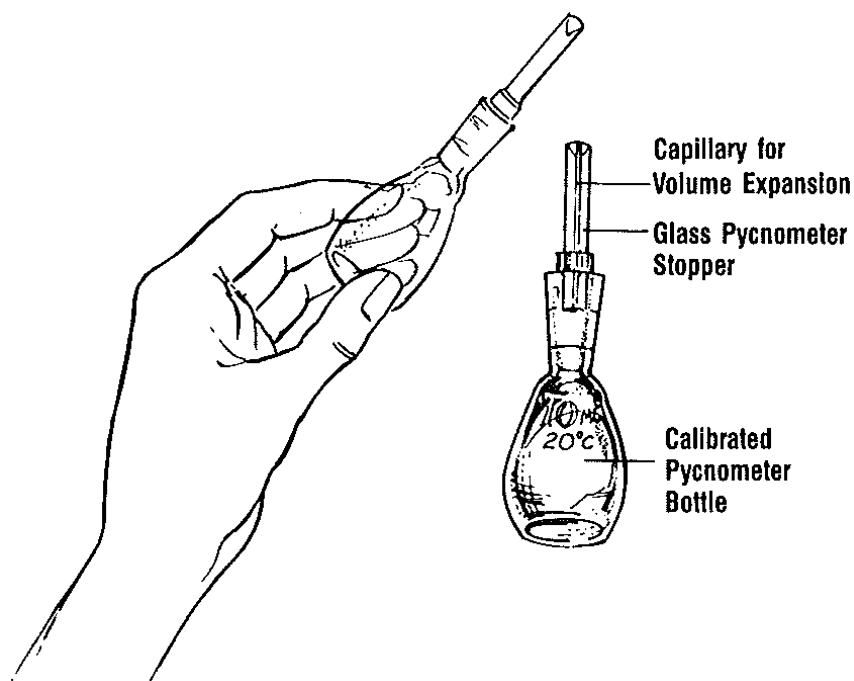


Figure 1. Gay-Lussac Type Pycnometer for Measuring True Density of Liquids

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)**¹ – 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 clean² 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:

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

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.

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.

ρ = density, g/mL, at the same temperature used for measuring the efflux time (see IFDC L-200).

v = kinematic viscosity, centistokes.

Typical Values

	Acid P ₂ O ₅ (%)	Dynamic Viscosity, cP	
		30°C	75°C
Phosphoric Acid Rock Source			
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)

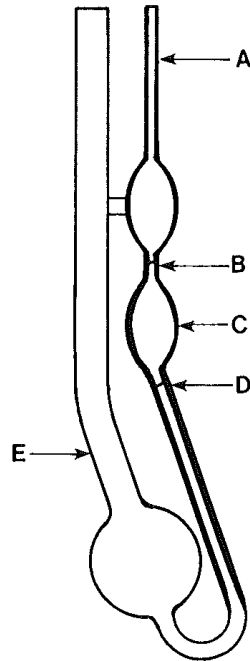


Figure 1. Cannon-Fenske Capillary Viscometer for Liquids

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

Type	Fluid	Grade	Dynamic Viscosity, cP		
			24°C	30°C	75°C
United States/Central Florida merchant-grade					
	phosphoric acid	54% P ₂ O ₅	–	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)

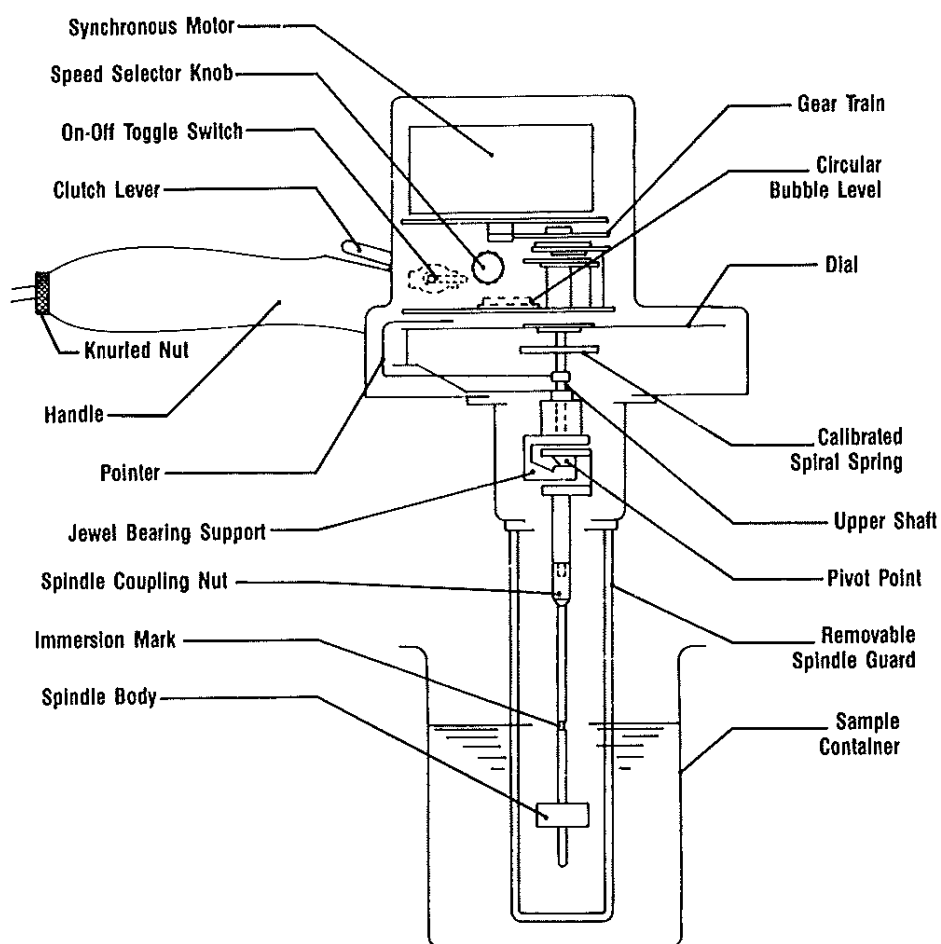


Figure 1. Brookfield Synchro-Lectric Rotating Spindle Viscometer

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

International Organization for Standardization (ISO) International Standard Procedures for Measuring Physical Properties of Solid Fertilizers

ISO 3944/Fertilizers – Determination of Bulk Density (Loose)

ISO 5311/Fertilizers – Determination of Bulk Density (Tapped)

**ISO 5313/High Nitrogen Content, Straight Ammonium Nitrate Fertilizers – Determination
of Oil Retention**

ISO 7837/Fertilizers – Determination of Bulk Density (Loose) of Fine-Grained Fertilizers

ISO 8397/Solid Fertilizers and Soil Conditioners – Test Sieving

ISO 8398/Solid Fertilizers – Measurement of Static Angle of Repose

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Fertilizers – Determination of Bulk Density (Loose)

0 Introduction

The bulk densities (loose and tapped) of a fertilizer provide information relative to the required size of packaging materials, store-houses, stock-rooms, etc. Generally, the bulk density (tapped) is up to 10% greater than the bulk density (loose), and sometimes it may exceed this value. Both bulk densities depend on the actual density, surface form and particle size of the fertilizers.

The bulk density (loose) can be used to calculate the maximum volume of a given weight of fertilizer which may be expected in practice. The actual volume occupied by a given weight of fertilizer will normally be within the range calculated from the bulk density (loose) and the bulk density (tapped).

1 Scope

This International Standard specifies a method for the determination of the bulk density (loose) of solid fertilizers, except powder fertilizers.

The method is applicable to dry fertilizers only. If the fertilizer has absorbed moisture during transport or storage, it is necessary to dry it in an environmental chamber, with constant low humidity, prior to the determination.

The method is not suitable for materials which contain a large proportion of particles exceeding 5 mm in diameter.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7742:1988, *Solid fertilizers – Reduction of samples*.

ISO 8358:1991, *Solid fertilizers – Preparation of samples for chemical and physical analysis*.

3 Definition

For the purposes of this International Standard, the following definition applies,

3.1 Bulk density (loose) of a fertilizer: The mass per volume of a fertilizer after it has been tipped freely into a container under specified conditions.

The bulk density (loose) is expressed in grams per cubic centimetre (g/cm^3).

4 Principle

Pouring of the fertilizer from a specified tunnel into a specified measuring cylinder of known volume and weighing of the contents of the cylinder.

5 Apparatus

5.1 Balance, capable of weighing to the nearest 1 g.

5.2 Apparatus for determination of bulk density (loose), having the approximate dimensions given in figure 1 and consisting of the following:

5.2.1 Removable measuring cylinder, without a spout. The capacity to the brim shall be known to the nearest cubic centimetre.

CAUTION – It is important that those parts of the apparatus which are in contact with the fertilizer are made of corrosion-resistant material (glass, plastics, etc.).

5.2.2 Firmly mounted funnel.

5.3 Spatula, approximately 120 mm x 20 mm, or other suitable scraper.

6 Preparation of test sample

Prepare the test sample by the methods given in ISO 7742 and ISO 8358, ensuring that the sample is sufficient to carry out two separate determinations.

7 Procedure

Pour into the closed funnel (5.2.2) a quantity of the fertilizer greater than that needed to fill the measuring cylinder (5.2.1). Fully open the slide of the funnel so that the contents discharge into the measuring cylinder in 6 s to 12 s.

If the fertilizer does not flow freely, keep the outlet clear by inserting a rod of 3 mm to 4 mm diameter into the opening.

When the cylinder overflows, close the slide of the funnel and scrape away the surplus fertilizer using the spatula or other suitable tool (5.3). Avoid vibration of the filled measuring cylinder.

Remove the measuring cylinder from below the funnel and weigh its contents to the nearest 1 g of the total mass.

Carry out two determinations, in rapid succession, on separate test portions taken from the same test sample.

8 Expression of results

8.1 Method of calculation and formula

The bulk density (loose) ρ of the fertilizer, in grams per cubic centimetre, is given by the equation

$$\rho = \frac{m}{V}$$

where

- m is the mass, in grams, of the test portion;
 V is the capacity up to the brim, in cubic centimetres, of the measuring cylinder.

Take as the result the arithmetic mean of the two determinations provided that the requirement for repeatability (see 8.2) is satisfied.

8.2 Repeatability

The difference between the results of two determinations, carried out in rapid succession by the same operator, using the same apparatus, shall not exceed 0,01 g/cm³.

9 Test report

The test report shall include the following particulars:

- a) identification of the sample;
- b) reference to the method used;
- c) the result and the method of expression;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard or regarded as optional.

Dimensions (approximate) in millimetres

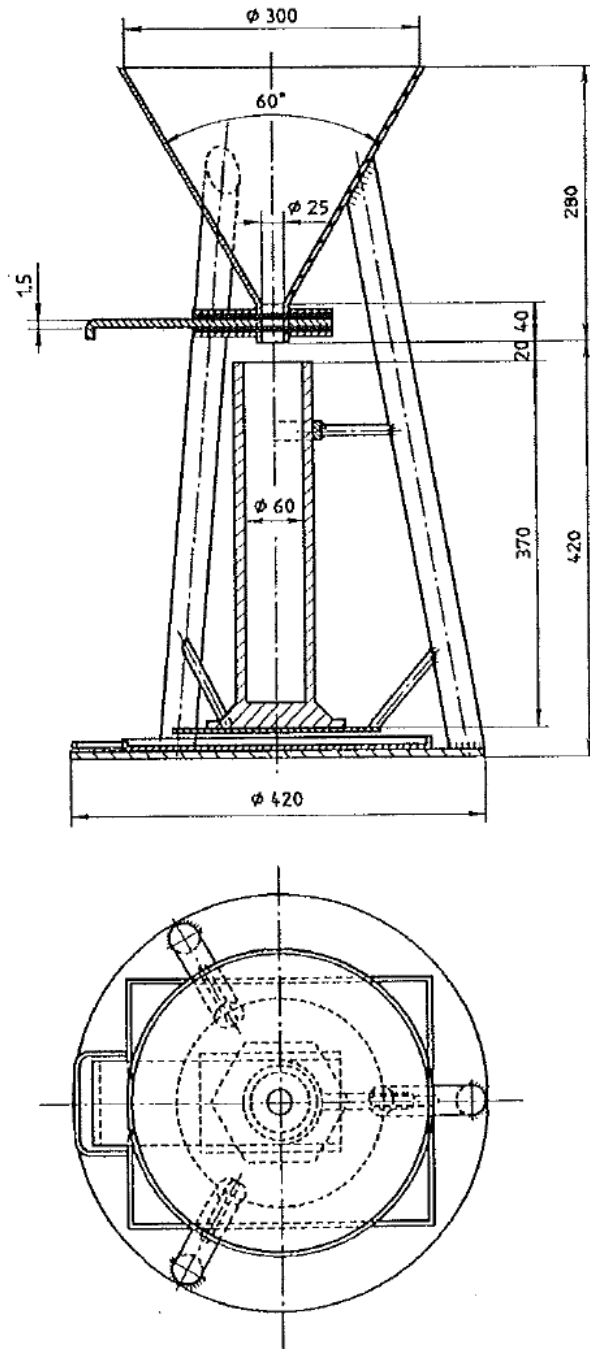


Figure 1. Apparatus for the Determination of Bulk Density (Loose) of Fertilizers

Fertilizers – Determination of Bulk Density (Tapped)

0 Introduction

The bulk densities (loose and tapped) of a fertilizer provide information relative to the required size of packaging materials, store-houses, stock-rooms, etc. Generally, the bulk density (tapped) is up to 10% greater than the bulk density (loose), and sometimes it may exceed this value. Both bulk densities depend on the actual density, surface form and particle size of the fertilizers.

The bulk density (loose) can be used to calculate the maximum volume of a given weight of fertilizer which may be expected in practice. The actual volume occupied by a given weight of fertilizer will normally be within the range calculated from the bulk density (loose) and the bulk density (tapped).

1 Scope

This International Standard specifies two methods for the determination of the bulk density (tapped) of solid fertilizers:

- the machine tapping method (method 1);
- the hand tapping method (method 2).

These methods are applicable to dry fertilizers only. If the fertilizer has absorbed moisture during transport or storage, it is necessary to dry it in an environmental chamber, with constant low humidity, prior to the determination.

Neither method is suitable for materials which contain a large proportion of particles exceeding 5 mm in diameter.

Method 2 is applicable only to spherical granules and to prills. It is not applicable to sharp-edged grains.

NOTE 1 Because of the differences in tapping technique, the two methods will not necessarily give the same value for the bulk density (tapped).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International

Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3944:1992, Fertilizers – Determination of bulk density (loose).

ISO 7742:1988, Solid fertilizers – Reduction of samples.

ISO 8358:1991, Solid fertilizers – Preparation of samples for chemical and physical analysis.

3 Definition

For the purposes of this International Standard, the following definition applies,

3.1 Bulk density (tapped) of a fertilizer: The mass per unit volume of a fertilizer tipped into a container and then compacted under clearly specified conditions.

The bulk density (tapped) is expressed in grams per cubic centimetre (g/cm^3).

4 Preparation of test sample

Prepare the test sample by the methods given in ISO 7742 and ISO 8358, ensuring that the sample is sufficient to carry out two separate determinations.

5 Method 1 – Machine tapping method

5.1 Principle

Pouring of the fertilizer from a specified funnel into a specified measuring cylinder of known volume, tapping by means of a tapping machine, and weighing of the contents of the cylinder.

5.2 Apparatus

5.2.1 Balance, capable of weighing to the nearest 1 g.

5.2.2 Apparatus for determination of bulk density (loose), according to ISO 3944, with a collar of transparent plastic and a measuring-cylinder holder with guide clamp (see figure 1).

5.2.3 Tapping machine, having a camshaft the cams of which lift the guide clamp, measuring-cylinder holder, and measuring cylinder once per revolution. The rotational frequency of the camshaft shall be $250 \text{ min}^{-1} \pm 15 \text{ min}^{-1}$. (See figure 1.)

5.2.4 Spatula, approximately 120 mm x 20 mm, or other suitable scraper.

5.3 Procedure

Pour into the closed funnel of the apparatus (5.2.2) a quantity of the fertilizer greater than that needed to fill the measuring cylinder.

Fully open the slide of the funnel so that the contents discharge into the measuring cylinder in 6 s to 12 s.

If the fertilizer does not flow freely, keep the outlet clear by inserting a rod of 3 mm to 4 mm diameter into the opening.

Remove the measuring cylinder from its holder, slip on the plastic collar and add by hand a quantity of fertilizer such that, after tapping, the fertilizer still remains several centimetres above the top of the measuring cylinder.

Place the measuring cylinder firmly in its holder in the tapping machine, and tap 2 500 times.

Remove the measuring cylinder from the tapping machine, remove the collar, and scrape away the surplus fertilizer heaped on the measuring cylinder by means of the spatula (5.2.4).

Weigh the contents of the measuring cylinder to the nearest 1 g of the total mass.

Carry out two determinations, in rapid succession, on separate test portions taken from the same test sample.

6. Method 2 – Hand tapping method

6.1 Principle

Pouring of the fertilizer from a specified funnel into a specified measuring cylinder of known volume, tapping the walls of the cylinder by hand, and weighing of the contents of the cylinder.

6.2 Apparatus

6.2.1 Balance, capable of weighing to the nearest 1 g.

6.2.2 Apparatus for determination of bulk density (loose), according to ISO 3944.

6.2.3 Spatula, approximately 120 mm x 20 mm, or other suitable scraper.

6.2.4 Rod, made of wood, plastic, or similar material, about 200 mm long and about 10 mm in diameter.

6.3 Procedure

Pour into the closed funnel of the apparatus (6.2.2) a quantity of the fertilizer greater than that needed to fill the measuring cylinder.

Open the slide of the funnel so that the contents discharge into the measuring cylinder in 20 s to 25 s. During the discharge, tap the sides of the measuring cylinder gently two to three times per second with the rod (6.2.4) to ensure compaction of the material.

If the fertilizer does not flow freely, keep the outlet clear by inserting a rod of 3 mm to 4 mm diameter into the opening.

Close the slide of the funnel, then twice raise the measuring cylinder 2 mm to 3 mm and drop it to complete the compaction. Scrape away the surplus fertilizer heaped on the measuring cylinder by means of the spatula (6.2.3).

Remove the measuring cylinder from below the funnel and weigh its contents to the nearest 1 g of the total mass.

Carry out two determinations, in rapid succession, on separate test portions taken from the same test sample.

7. Expression of results

7.1 Method of calculation and formula

The bulk density (tapped) ρ_t of the fertilizer is given, in grams per cubic centimetre, by the formula

$$\rho_t = \frac{m}{V}$$

where

- m is the mass, in grams, of the test portion;
 V is the volume up to the brim, in cubic centimetres, of the measuring cylinder.

Take as the result the arithmetic mean of the two determinations if the requirement concerning repeatability (see 7.2) is satisfied.

7.2 Repeatability

The difference between the results of two determinations, carried out in rapid succession by the same operator using the same apparatus, shall not exceed 0,01 g/cm³.

8. Test report

The test report shall include the following particulars:

- a) identification of the sample;
- b) the reference to the method used;
- c) the result and the method of expression;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard or regarded as optional.

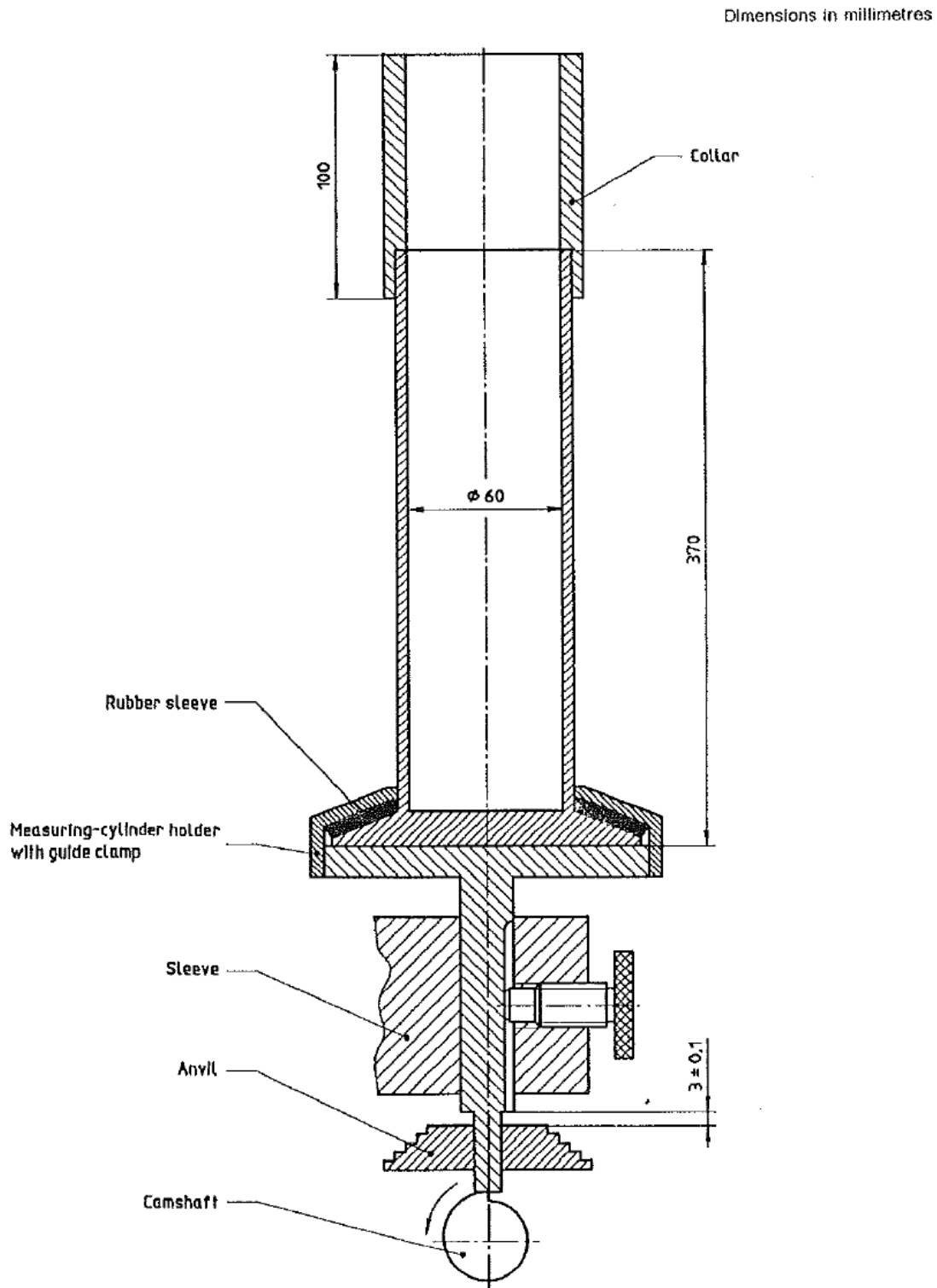


Figure 1. Apparatus for the Determination of Bulk Density (Tapped) by Machine Tapping

High Nitrogen Content, Straight Ammonium Nitrate Fertilizers – Determination of Oil Retention

0 Introduction

The porosity of a high nitrogen content, straight ammonium nitrate fertilizer can be measured by means of the determination of the gas oil retention, called by convention oil retention.

The method specified in this International Standard is an empirical method, requiring the minimum of apparatus, and gives results with an acceptable level of reproducibility.

1 Scope and field of application

This International Standard specifies a method for the determination of the gas oil retention of solid, high nitrogen content, straight ammonium nitrate fertilizers.

The method is applicable to fertilizers which do not contain materials soluble in gas oil and which are prilled or granular.

2 References

ISO 3310/1, *Test sieves – technical requirements and testing – Part 1: Test sieves of metal wire cloth.*

ISO 8358, *Fertilizers – Preparation of samples for analysis.*¹

3 Definition

Oil retention of a high nitrogen content, straight ammonium nitrate fertilizer: The quantity of gas oil retained by the fertilizer determined under the conditions specified.

It is expressed as a percentage by mass.

4 Principle

Total immersion of a test portion in gas oil for a specified period, followed by the draining away and the removal of surplus gas oil under specified conditions. Measurement of the increase in mass of the test portion.

1) At present at the stage of draft.

5 Reagent

Gas Oil

Viscosity: 1,3 to 5,0 mPa·s (1,6 to 6,0 cSt) at 40°C.

Density: 0,82 to 0,86 g/mL at 15°C.

Sulfur Content < 1,0% (*m/m*)

Ash < 0,1% (*m/m*)

The density of the gas oil shall be mentioned in the test report.

6 Apparatus

Ordinary laboratory apparatus, and

6.1 Balance, capable of weighing to the nearest 0,01 g.

6.2 Beaker, of capacity 500 mL.

6.3 Funnel, of plastic material, preferably with a cylindrical wall at the upper end, diameter approximately 200 mm.

6.4 Test sieve, complying with ISO 3310/1, aperture size 0,5 mm, fitting into the funnel (6.3).

6.5 Filter paper, rapid filtering grade, crêped, soft, of surface density 150 g/m².

7 Preparation of test sample

Prepare the test sample in accordance with ISO 8358.

8 Procedure

8.1 Sieve the test sample using the test sieve (6.4) to remove particles less than 0,5 mm. Weigh, to the nearest 0,01 g, approximately 50 g of the sieved test sample into the beaker (6.2).

Add sufficient of the gas oil (clause 5) to cover the test portion completely and agitate very gently to ensure that all surfaces are covered. Allow the beaker to stand at a temperature of $25 \pm 2^\circ\text{C}$ for 1 h.

8.2 Fit the test sieve (6.4) into the funnel (6.3). Transfer the contents of the beaker quantitatively onto the test sieve. Distribute the fertilizer on the surface of the sieve. Incline the funnel with the test sieve and allow to drain for 1 h to remove the majority of the excess gas oil.

8.3 Spread the test portion quantitatively on a double sheet of filter paper (6.5) and cover with a similar double sheet. Remove any excess surface gas oil by gently rolling flat the test portion between the sheets of filter paper. Repeat this operation with fresh sheets of filter paper until there are no visible traces of gas oil on them.

NOTE – The treatment with filter paper should be performed carefully in order to prevent crushing of granules.

8.4 Weigh the test portion and the gas oil retained by the granules to the nearest 0,01 g immediately after rolling.

8.5 Carry out two determinations in rapid succession on separate test portions taken from the same test sample.

9 Expression of results

9.1 Method of Calculation and Formula

The oil retention, expressed as a percentage by mass of fertilizer, is given by the formula

$$\frac{m_2 - m_1}{m_1} \times 100$$

where

m_1 is the mass, in grams, of the sieved test portion (8.1);

m_2 is the mass, in grams, of the test portion and the retained gas oil (8.4).

Take as the result the arithmetic mean of the two determinations if the requirement concerning repeatability (see 9.2) is satisfied. Otherwise, carry out two further determinations.

9.2 Repeatability

The difference between the results of two determinations, obtained simultaneously or in rapid succession by the same analyst, using the same apparatus, on identical test material, under the same operating conditions, should not exceed $0,3 \sqrt{\bar{\chi}}$, where $\bar{\chi}$ is the arithmetic means of two determinations, at a confidence level of 95%.

9.3 Reproducibility

The difference between the results of two determinations, obtained by different analysts in different laboratories, on identical test material, should not exceed $0,8 \sqrt{\bar{\chi}}$, where $\bar{\chi}$ is the arithmetic mean of two determinations, at a confidence level of 95%.

10. Test report

The test report shall include the following particulars:

- a) the reference to the method used;
- b) the identification of the test sample;
- c) the density of the gas oil;
- d) the result and the method of expression;
- e) any unusual features noted during the determination;
- f) any operation not included in this International Standard or regarded as optional.

Fertilizers – Determination of Bulk Density (Loose) of Fine-Grained Fertilizers

0 Introduction

A method for the determination of the bulk density (loose) of solid fertilizers having particle sizes within the frequently encountered range from about 0,5 mm to 5 mm is specified in ISO 3944:1992, *Fertilizers – Determination of bulk density (loose)*. This method is not, however, suitable for fine-grained fertilizers having a large proportion of particles of diameters less than 0,5 mm. Such fertilizers pass with difficulty, in most cases, from the specified funnel into the measuring cylinder, generally cause considerable dust nuisance, and are inclined to form hollow spaces (air cavities) within their bulk volume. The bulk density values obtained are, thus, too low.

In the case of fine-grained fertilizers, therefore, it is necessary to use a dust-tight, non-clogging apparatus, with a relatively wide measuring cylinder.

1 Scope

This International Standard specifies a method for the determination of the bulk density (loose) of solid fine-grained fertilizers.

The method is applicable to fertilizers which contain a large proportion of particles of diameters less than 0,5 mm.

NOTE 1 For fertilizers which contain a large proportion of particles of diameters within the range from 0,5 mm to 5 mm, a method is specified in ISO 3944.

The method is applicable to dry fertilizers only. If the fertilizer has absorbed moisture during transport or storage, it is necessary to dry it in an environmental chamber, with constant low humidity, prior to the determination.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7742:1988, *Solid fertilizers – Reduction of samples*.

ISO 8358:1991, *Solid fertilizers – Preparation of samples for chemical and physical analysis*.

3 Definition

For the purposes of this International Standard, the following definition applies.

Bulk density (loose) of a fertilizer: The mass per volume of a material after it has been tipped freely into a container under specified conditions.

The bulk density (loose) is expressed in grams per cubic centimetre (g/cm³).

4 Principle

Pouring of the fertilizer from the specified filling device into a specified measuring cylinder of known volume and weighing of the contents of the cylinder.

5 Apparatus

5.1 Balance, capable of weighing to the nearest 1 g.

5.2 Apparatus for determination of bulk density (loose), having the approximate dimensions given in figure 1 and consisting of the following:

5.2.1 Filling device (4) with spring-suspended locking lever (5), for holding or loosening the hinged cover. The hinged cover is opened by manipulating the lever so that the contents of the filling device discharge into the measuring cylinder.

CAUTION – It is important that those parts of the apparatus which are in contact with the fertilizer are made of corrosion-resistant material (glass, plastics, etc.).

5.2.2 Measuring cylinder (1), of capacity $1\ 000\ \text{cm}^3 \pm 5\ \text{cm}^3$.

5.2.3 Intermediate piece (2) with hinged cover (3).

5.3 Spatula, approximately 200 mm x 20 mm, or other suitable scraper.

6 Preparation of test sample

Prepare the test sample by the methods given in ISO 7742 and ISO 8358, ensuring that the sample is sufficient to carry out two separate determinations.

7 Procedure

Pour the fertilizer into the filling device (5.2.1) up to the brim. Open the hinged cover by manipulating the locking lever.

After 2 min, remove the empty filling device and the intermediate piece (5.2.3) from the measuring cylinder (5.2.2). Scrape away the surplus fertilizer from the measuring cylinder using the spatula or other suitable tool (5.3).

Weigh the contents of the measuring cylinder to the nearest 1 g.

Carry out two determinations in rapid succession, on separate test portions taken from the same test sample.

8 Expression of results

8.1 Method of calculation

The bulk density (loose), ρ , of the fertilizer, in grams per cubic centimetre, is given by the equation

$$\rho = \frac{m}{V}$$

where

- m is the mass, in grams, of the test portion;
- V is the capacity up to the brim, in cubic centimetres, of the measuring cylinder.

Take as the result the arithmetic mean of the two determinations provided that the requirement for repeatability (see 8.2) is satisfied.

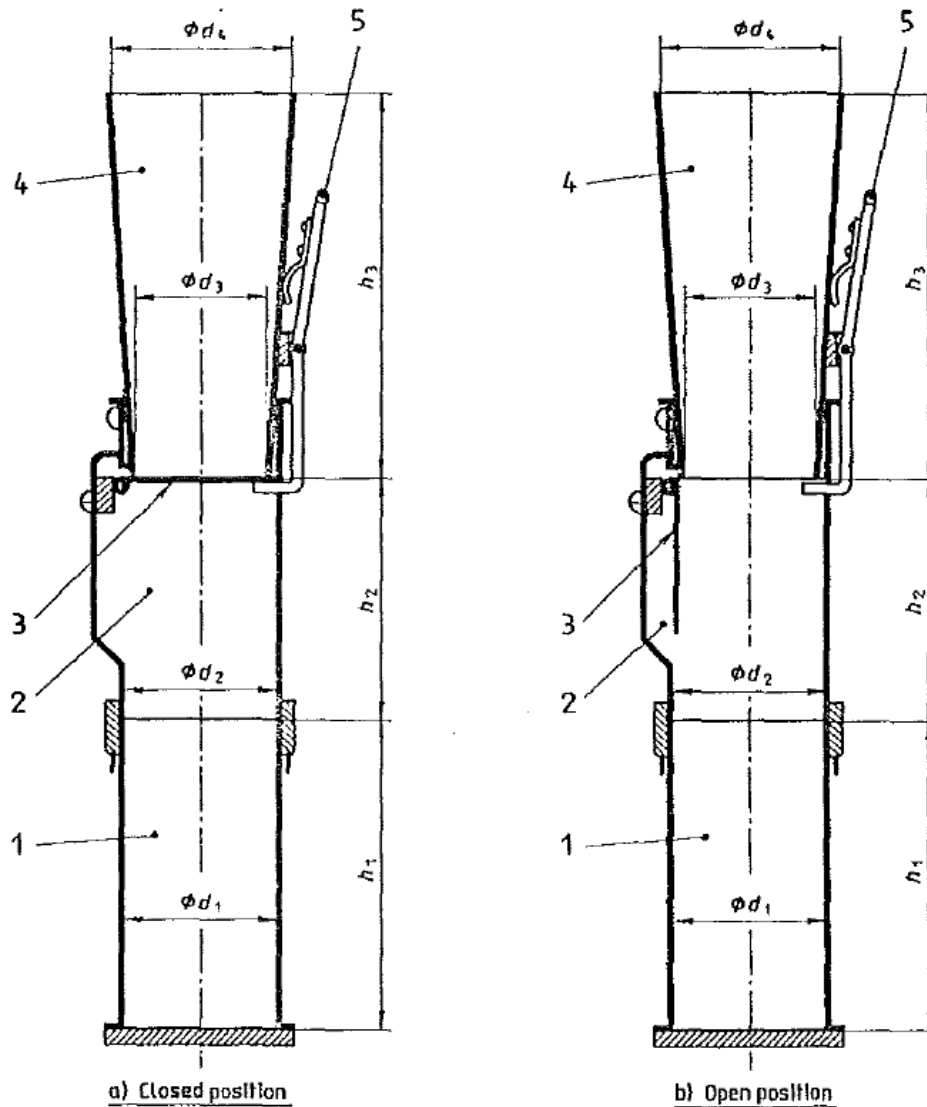
8.2 Repeatability

The difference between the results of two determinations, carried out in rapid succession by the same operator using the same apparatus, shall not exceed 0,02 g/cm³.

9 Test report

The test report shall include the following particulars:

- a) identification of the sample;
- b) reference of the method used;
- c) the result and the method of expression;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard or regarded as optional.



- 1 Measuring cylinder
Internal diameter $d_1 = 87 \text{ mm} \pm 1 \text{ mm}$
Internal height h_1 corresponding to a capacity of $1000 \text{ cm}^3 \pm 5 \text{ cm}^3$
- 2 Intermediate piece
Internal diameter $d_2 = 87 \text{ mm} \pm 1 \text{ mm}$
height $h_2 = 135 \text{ mm} \pm 1 \text{ mm}$
- 3 Hinged cover
- 4 Filling device
Lower internal diameter $d_3 = 79 \text{ mm} \pm 1 \text{ mm}$
upper internal diameter $d_4 = 99 \text{ mm} \pm 1 \text{ mm}$
height $h_3 = 199 \text{ mm} \pm 1 \text{ mm}$
- 5 Spring-suspended locking lever

Figure 1. Apparatus for Determination of Bulk Density (Loose) of Fine-Grained Fertilizers

Solid Fertilizers and Soil Conditioners – Test Sieving

1 Scope and field of application

This International Standard specifies a method for the determination of the particle size distribution of solid fertilizers and soil conditioners by test sieving.

NOTE – The applicability of the method has been tested with sieves of nominal sizes of openings between 100 µm and 5,60 mm.

2 References

ISO 565, *Test sieves – Woven metal wire cloth, perforated plate, and electroformed sheet – Nominal sizes of openings.*

ISO 2395, *Test sieves and test sieving – Vocabulary.*

ISO 2591, *Test sieving.*

ISO 3310-1, *Test sieves – Technical requirements and testing – Part 1: Test sieves of metal wire cloth.*

ISO 3944, *Fertilizers – Determination of bulk density (loose).*

ISO 3963, *Fertilizers – Sampling from a conveyor by stopping the belt.*

ISO 7410, *Fertilizers and soil conditioners – Final samples – Practical arrangements.*

3 Definitions

For the purpose of this International Standard, the definitions of ISO 2395 apply.

4 Principle

Dry sieving of a fertilizer sample with one or more test sieves using a mechanical sieving machine.

5 Apparatus

5.1 Balance, capable of weighing to the nearest 0,1 g.

5.2 Stainless steel woven wire test sieves, 200 mm diameter, complying with ISO 3310-1, with a lid and receiver for the sieves.

5.3 Mechanical shaker (sieving machine), capable of imparting both horizontal and vertical motion to material inside a nest of sieves.

5.4 Stopwatch.**5.5 Soft brush.****6 Sampling**

See ISO 3963 and ISO 7410.

7 Preparation of the test portion

Reduce the sample (a method will form the subject of a future International Standard) to the quantity required for the sieving test. This quantity should be approximately that indicated in column 2 of Table 1 for the sieve corresponding to the dominant size fraction of the sample, provided that the size distribution does not cause excess volume on any of the sieves in the set as indicated in column 3 of Table 1.

Table 1. Recommended Charges for Test Sieving

Nominal aperture size	Bulk volume of material*	
	Recommended volume of charge	Maximum volume of residue permitted on the sieve at the completion of sieving
	cm ³	cm ³
8,00 mm	500	250
5,60 mm	400	200
4,00 mm	350	175
2,80 mm	240	120
2,00 mm	200	100
1,40 mm	160	80
1,00 mm	140	70
710 µm	120	60
500 µm	100	50
355 µm	80	40
250 µm	70	35
180 µm	60	30
For non-friable and non-adhesive materials only		
125 µm	50	25
90 µm	40	20

* The mass of material can be calculated by multiplying the volume by the bulk density, determined by the method described in ISO 3944, of the material to be sieved.

8 Procedure

8.1 Select a maximum of seven test sieves from the range of principal sizes listed in ISO 565 to cover the range of particle size expected. Assemble the sieves in ascending order of aperture size on top of the receiver.

NOTE – Normally the principal sizes (R20/3) should be used. In special cases, however, it may be necessary to use supplementary sizes (R 20).

8.2 Weigh the test portion to the nearest 0,1 g, place it on the top sieve, and fit the cover.

8.3 Place the assembled nest of sieves on the mechanical shaker and shake for 10 min.

8.4 Remove the sieves from the nest and weigh the quantity retained on each sieve and in the receiver to the nearest 0,1 g. Particles caught in the mesh of the sieve may be removed by brushing the reverse side of the sieve.

8.5 Carry out at least two tests on separate test portions prepared from the same laboratory sample.

9 Expression of results

9.1 Sum the masses of the fractions retained on the sieves and in the receiver.

NOTE – The sum of these masses should not differ by more than 1% from the original mass of the test portion.

9.2 Calculate each fraction mass as a percentage of the sum of these masses.

10. Precision

The statistical information given here is only intended as a guideline to what can be expected. The values are based on the evaluation of collaborative studies which have been carried out using sieves of nominal sizes of openings between 100 µm and 5,60 mm.

10.1 Repeatability, r

The difference $[X_1 - X_2]$ between two single results (X_1 and X_2) found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value r , expressed as a percentage, given by equation (1) and with a maximum value of 2,5%, on average not more than once in 20 cases in the normal and correct operation of the method. Both results should be considered suspect if the repeatability value r is exceeded.

$$r = 0,5 \sqrt{\bar{\chi}} \quad \dots (1)$$

where $\bar{\chi}$ is the mean value of the two results, expressed as a percentage by mass, of the fractions.

10.2 Reproducibility, R

Single results (X_1 and X_2) on identical test material reported by two laboratories will differ by more than the reproducibility value R , expressed as a percentage, given by equation (2) and with a maximum value of 12,5%, on average not more than once in 20 cases in the normal and correct operation of the method. Both results should be considered suspect if the reproducibility value R is exceeded.

$$R = 2,5 \sqrt{\bar{\chi}} \quad \dots (2)$$

where $\bar{\chi}$ is as defined in 10.1.

10.3 Examples (see table 2)

Table 2. Examples of precision data

$\bar{\chi}$	r	R	For r		For R	
			X_{\min}^*	X_{\max}^{**}	X_{\min}^*	X_{\max}^{**}
1	0,5	(2,5)	0,8	1,3	0	2
4	1	5	3,5	4,5	1,5	6,5
9	1,5	7,5	8,3	9,8	5,3	12,8
16	2	10	15	17	11	21
25	2,5	12,5	23,8	26,3	18,8	31,3
36	2,5	12,5	34,8	37,3	29,8	42,3

* X_{\min} is the smaller of the two corresponding fractions X , expressed as a percentage by mass.

** X_{\max} is the larger of the two corresponding fractions X , expressed as a percentage by mass.

11. Test report

The test report shall include the following Information:

- the reference of the method used, i.e., ISO 8397;
- all information necessary for the complete identification of the sample;
- the nominal sizes of openings of the test sieves used;
- the mean values of the percentage by mass of material retained on each sieve;
- any operations not specified in this International Standard or in the International Standards to which reference is made, or regarded as optional, together with any circumstances likely to have affected the results.

NOTE – Methods of recording results of test sieving in tabular form are shown in ISO 2591.

Solid Fertilizers – Measurement of Static Angle of Repose

1 Scope

This International Standard specifies a method for the measurement of the static angle of repose of solid fertilizers. The method is applicable to free flowing fertilizers and is suitable for measuring static angles of repose greater than 20°; the method is not suitable for materials which contain a large proportion of particles exceeding 5 mm in diameter.

NOTE – The measurement of the static angle of repose is of importance for the calculation of storage capacities. The angle measured according to this method is normally the maximum value resulting in practice. For the determination of the dynamic angle of repose (angle of slide) other methods (e.g., a tilting box method) are available.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3944:1980, *Fertilizers – Determination of bulk density (loose)*.

ISO 5725:1986, *Precision of test method – Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

3 Definition

For the purpose of this International Standard, the following definition applies.

Static angle of repose: The angle at the base of the cone of fertilizer obtained by allowing a sample to fall onto a horizontal base-plate under specified conditions.

4 Principle

Measurement of the height and diameter of the base of the cone of fertilizer obtained by allowing a sample to fall through a fixed distance from a defined funnel onto a horizontal baseplate. Calculation of the angle of repose.

5 Apparatus

Only the dimensions given in the text are mandatory.

The apparatus (see Figure 1) shall be composed of the following parts:

5.1 Funnel, of rigid plastic or stainless steel, having a spout of internal diameter 25 mm and fitted with a slide.

NOTE – The funnel illustrated in Figure 1 is identical to that specified ISO 3944.

5.2 Squarebase-plate, 750 mm x 750 mm, of rigid construction, made of metal, plastic, or wood. The base-plate shall be plain, undeformable, and damp-proof. Its surface shall not be polished but shall be matt; if necessary, this condition can be achieved by fixing a suitable filter paper on the surface of the base-plate. Four straight lines shall be drawn at angles of 45° to each other, intersecting at the center of the plate.

5.3 Funnel Support, comprising a frame of substantial construction to support the funnel (5.1) so that the axis of the funnel passes through the center of the base-plate and the tip of the funnel spout is 120 mm above the surface of the base-plate.

6 Procedure

6.1 Test Sample

Take a test sample of appropriate size.

NOTE – Usually 10 kg will be sufficient.

6.2 Determination

6.2.1 Carry out the determination at ambient temperature.

6.2.2 Check that the base-plate (5.2) is level and that the funnel (5.1) is properly located and fixed in the support (5.3).

6.2.3 Pour the test sample (6.1) into the funnel (5.1) with the slide closed. Then open the slide and allow the fertilizer to discharge in a steady stream, taking care not to vibrate the apparatus. If the amount of fertilizer is not sufficient, i.e., if the top of the cone which is formed on the base-plate does not reach the tip of the funnel spout, pour an additional amount of the test sample into the funnel in a steady stream with the slide still open until the top of the cone reaches the tip of the funnel spout.

Mark the circumference of the base of the cone on the eight radii drawn on the base-plate, ignoring any grains which form only a single layer around the base of the cone. Remove the fertilizer from the base-plate and measure the four marked diameters.

7 Expression of results

The angle of repose, α , expressed in degrees, is given by the equation

$$\alpha = \arctan \left(\frac{240}{\bar{d} - 25} \right)$$

or

$$\alpha = \arctan \left(\frac{2h}{\bar{d} - d_i} \right)$$

where

h is the height of the cone, in millimetres.

\bar{d} is the arithmetic mean of the four diameters measured as specified in 6.2.3, in millimetres.

d_i is the internal diameter of the funnel spout, in millimetres.

8 Precision

The precision of the method was established by an inter-laboratory test carried out in accordance with ISO 5725. For the obtained repeatability limit and reproducibility limit a probability level of 95% holds.

8.1 Repeatability

The absolute difference between two single test results, obtained under repeatability conditions, will exceed 1° on average not more than once in 20 cases. Both results should be considered suspect if this limit is exceeded.

NOTE – repeatability conditions: Conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment under the same conditions within short intervals of time.

8.2 Reproducibility

The absolute difference between two single test results, obtained under reproducibility conditions, will exceed by more than 2° on average not more than once in 20 cases. Both results should be considered suspect if this limit is exceeded.

NOTE – reproducibility conditions: Conditions where test results are obtained with the same method on identical test material in different laboratories with different operators using different equipment under different conditions.

9 Test report

The test report shall include the following particulars:

- a) The reference of the method used, i.e., ISO 8398;
- b) The results and the method of expression used;
- c) Details of any unusual features noted during the determination;
- d) Details of any operation not specified in this International Standard, or in the International Standard to which reference is made, or regarded as optional.

Dimensions in millimetres

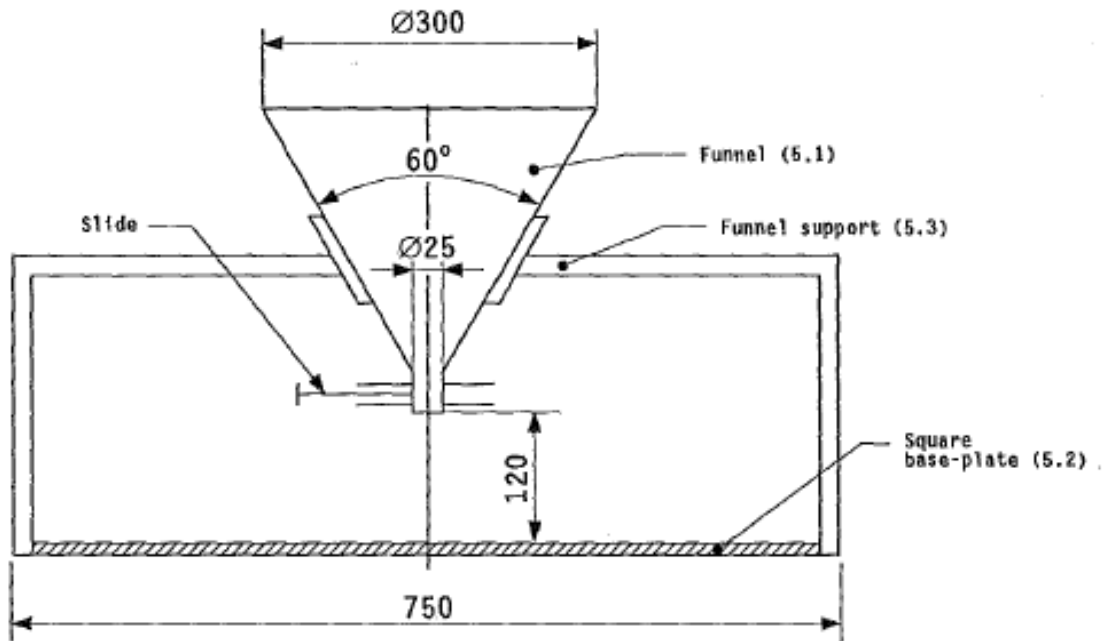


Figure 1. Apparatus for the Determination of Static Angle of Repose