DEFINITION AND TEST PROCEDURES
FOR
AMMONIUM NITRATE FERTILIZER

The Fertilizer Institute
1015 18th Street, N.W.
Washington, D.C. 20036

August, 1984
DEFINITION AND TEST PROCEDURES FOR AMMONIUM NITRATE FERTILIZER

This booklet sets forth the definition and test procedures for ammonium nitrate fertilizer originally developed by the Agricultural Nitrogen Institute, a forerunner organization of The Fertilizer Institute (TFI). This booklet does not pertain to blasting agents or other ammonium nitrates. Previous revisions of this booklet by TFI are dated May 7, 1971, and January 16, 1973.

In May of 1984, TFI convened a Task Force of industry and government representatives, experts on the physical and chemical characteristics of ammonium nitrate fertilizer, to review this publication and to make any necessary changes as they deemed proper. Based on that review and the technical expertise and experience of the Task Force members, TFI published this revised edition dated August, 1984.

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### AMMONIUM NITRATE PUBLICATION REVIEW TASK FORCE

May 1984

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<td>Pensacola, FL</td>
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INTRODUCTION

It is felt that the following combination of definition and test procedures precludes the possibility of classifying as Ammonium Nitrate Fertilizer any ammonium nitrates which contain sufficient organics or other oxidizable material to present any hazard in transportation or storage. The definition alone would not be adequate nor would the test procedures alone be adequate to accomplish this end. However, the requirement that a material pass both the definition and the tests should accomplish the desired end. The combination of definition and tests is many times more severe than the currently used definition and test requirements.

The tests specified are in no way to be confused with or to be used to replace the industry standard test for distinguishing between explosives and blasting agents. The tests given herein are designed to define those materials which are properly considered to be Ammonium Nitrate Fertilizer.

The latest revision of this booklet is referenced in the U.S. Department of Transportation's "Hazardous Materials Regulations" (Code of Federal Regulations, Title 49, 173.182) as the criteria that must be met before a material can be shipped under the description "ammonium nitrate fertilizer, containing no more than 0.2% carbon UN 2067."
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SECTION 1.0 DEFINITION

Ammonium Nitrate Fertilizer is defined as solid ammonium nitrate containing a minimum of 33.0% nitrogen, having a minimum pH of 4.0 in a 10% aqueous solution, 0.20% maximum carbon, 0.010% maximum elemental sulfur, 0.150% maximum chloride as Cl, or particulated elemental metals sufficient to release 4.60 ml, maximum, of hydrogen from 50.0 gram sample and which will pass the detonation resistance test in Section 2.0 and the burning test in Section 4.0.

SECTION 2.0 DETONATION RESISTANCE TEST

2.1 Preparation of Sample for Testing

A representative sample of the material to be tested shall be used to fill a two-foot section (minimum length) of Schedule 40, three inch (Iron Pipe Size) black iron welded pipe, closed at one end with cardboard or equivalent closure. The material shall be compacted by dropping the filled pipe, one time, closed end down, from a height of six inches onto a board. If necessary, additional sample shall be added to bring the level of material to one inch below the lip of the pipe.

2.2 Priming

A Pentolite pellet weighing 4.7 grams is used for the primer in this test. The Pentolite used is 50% P.E.T.N. and 50% TNT. The pellet is one-inch in length, one-half inch in diameter, with a one-fourth inch hole in the top center one-fourth inch deep. One No. 6 blasting cap is taped into the Pentolite pellet. The pellet is buried in the test material, with the bottom of the pellet one-inch deep. Note: In order to standardize the test, all material shall be tested at 70°F.

2.3 Detonation

The primed pipe shall be placed in a vertical position behind a suitable barricade (to protect personnel from possible shrapnel). After personnel have taken cover, the primer shall be fired. Caution: This test shall be run only by qualified personnel and in facilities designed for explosive testing.

2.4 Interpretation of Results

Any material which passes the test will not propagate through the two-foot section of pipe and will leave some undamaged pipe. Any material which propagates through the two-foot length of pipe and which leaves no undamaged pipe cannot be classified as Ammonium Nitrate Fertilizer.

SECTION 3.0 ANALYTICAL METHODS

3.1 Nitrogen

3.1.1 Reference


3.1.2 Reagents

Formaldehyde, approximately 37%
Sodium hydroxide, 0.25 - 0.5 N. Standardize against primary standard.
3.1.3 Procedure

Rapidly weigh and transfer 1.5 g of unground sample to a 250-mL Erlenmeyer flask. Dissolve sample with about 50 mL distilled water and add 15 mL formaldehyde. Dilute to 150 - 200 mL and let stand five minutes. Titrate with 0.25 - 0.5 N sodium hydroxide using 5 drops of phenolphthalein as indicator or to pH of 8.6 if electroscopic end point is preferred. If colorimetric end point is used, titrate until there is no perceptible color change at point of contact. Determine blank on formaldehyde solution.

3.1.4 Calculation

\[
\% \text{ Nitrogen} = \frac{\text{net mL NaOH} \times N}{\text{wt sample}} \times 2.8014
\]

3.2 Carbon

3.2.1 Reference


3.2.2 Principle

Organic additives in ammonium nitrate fertilizer can be determined by measuring the carbon dioxide evolved upon combustion of the sample in oxygen. Water and the AN are added to freshly ignited magnesium oxide and the mixture heated to 700°C in a quartz combustion tube. The carbon dioxide is absorbed in barium hydroxide solution and determined by a gravimetric or turbidimetric method. Less than 0.1% carbon in AN can be determined. The total carbon obtained by combustion must be corrected if carbonates are present in the AN. The carbonate content is determined by a standard method, e.g., add acid to a sample of the AN in a closed system, aerate, and absorb CO₂ in Ba(OH)₂ as in the combustion test.

3.2.3 Reagents

- Magnesium oxide, reagent grade
- Copper oxide, reagent grade
- Silver gauze, reagent grade
- Barium hydroxide, 1M – Dissolve 315 g Ba(OH)₂·8H₂O, reagent grade, in CO₂-free distilled water and dilute to one liter. Allow to settle overnight.
- Oxygen, U.S.P.

3.2.4 Apparatus

- Quartz combustion tube, one-inch I.D.
- 2 Electric heating furnaces
- 1 Fused silica boat, 16 mm x 100 mm
- 1 Fritted glass washing bottle, 125 mL

3.2.5 Procedure

A stream of oxygen is passed through the combustion apparatus which consists of a one-inch diameter quartz tube connected to a 125-mL fritted disk gas washing bottle containing 75 mL barium hydroxide solution. The quartz combustion tube is packed with copper oxide wire to a length of 3 inches and with silver gauze to a length of 1 inch, and this section is heated with a stationary electric furnace. The sample is heated with a movable furnace or a Fisher burner.
Place 1.5 g magnesium oxide in the fused silica boat and heat to 700°C in the combustion tube. Disconnect the absorber bottle during this initial heating. After ten minutes, connect the absorber bottle and remove the boat from the combustion tube in a CO₂-free atmosphere. Freshly ignited magnesium oxide absorbs CO₂ very rapidly.

Add 4 g AN and 2 mL CO₂-free water to the boat. Carefully heat the sample until the ammonia and water have been removed; then continue to heat for 30 minutes at 700°C. The amount of barium carbonate in the absorber bottle can be determined (1) gravimetrically by filtering the precipitate through a medium sintered-glass filter crucible, washing with CO₂-free water, and drying to a constant weight at 110°C, (2) by visually comparing the turbidity in the absorber bottle with known amounts of sodium carbonate added to 75 mL of 1M barium hydroxide solution. A blank must be run on reagent grade AN. Oxygen must be used for blank and sample.

3.2.6 Calculation

\[
\%C = \frac{Wt \text{ Sample ppt} - Wt \text{ Blank ppt} \times 6.08}{Wt \text{ of Sample}}
\]

3.3 Elemental Metals

3.3.1 Reference

Procedure presented to The Fertilizer Institute by the Nitrogen Division, Allied Chemical Company Technical Service Group, Hopewell, Virginia.

3.3.2 Principle

Strong acid is reacted with elemental metals and hydrogen liberated is measured after any CO₂ liberated is absorbed with NaOH. In the event all the aluminum does not react with the acid, it will react with the caustic to liberate hydrogen under the conditions prescribed.

3.3.3 Apparatus

See Figure 1. If desired, the apparatus could be improved by having the flask and buret constructed with a ball or tapered joint.

3.3.4 Reagents

- Sodium carbonate
- 1:1 Hydrochloric acid
- 2N Sodium hydroxide

3.3.5 Procedure

Place 50 ± 0.1 g of ammonium nitrate in a 400 mL beaker. Add, 10 ± 1 g of sodium carbonate and about 200 mL of water. Filter through a Gooch crucible having a separable perforated bottom. Transfer all residue, which contains any metals present, from the beaker to the crucible and wash thoroughly.

Remove the perforated bottom along with the asbestos mat and residue from the crucible and place in a 125 mL filter flask. Attach an oblique bore stopcock to the sidearm of the flask and close the flask with a No. 5-1/2 rubber stopper through which is inserted a closed end 50 mL gas buret as shown in Figure 1.
Evacuate the flask and buret with a vacuum pump (10 minutes) and close the stopcock. Remove the vacuum pump line. Insert the outlet end of the stopcock in 1:1 hydrochloric acid and turn the stopcock to admit about 10 mL of the acid. Let stand for thirty minutes. The presence of metals or carbonates will be apparent by an evolution of gas from the acid. Insert the outlet end of the stopcock into a 250 mL beaker filled with sodium hydroxide solution of about 2 N strength. The addition of 2-3 drops of wetting agent (10% aerosol OT Solution) to the sodium hydroxide solution will aid in preventing adherence of gas bubbles to the wall of the apparatus, invert the apparatus two or three times to dislodge them.

Record the volume of gas (hydrogen), the temperature adjacent to the buret, and the barometric pressure. Run a blank in the same manner as the sample to account for desorbed gases from the reagents and for air present in the outlet end of the stopcock. Convert the volume of hydrogen to standard conditions as follows:

\[
\text{Corrected mL (STP)} = \frac{\text{Barometric Pressure} \times \text{mL H}_2 \times 273}{(273 + t^\circ \text{C}) \times 760}
\]

3.3.6 Calculation

\[
\% \text{Al} = \frac{\text{mL Hydrogen (STP)}}{\text{Wt. of Sample}} \times 0.0802
\]

Corrections for water vapor volume and hydrogen solubility are not applied in the calculation since their effect appears to be negligible.

3.3.7 Experimental Results

Test of this method with known amounts of aluminum and magnesium gave the following recovery value:

<table>
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<tr>
<th>Aluminum Added, g</th>
<th>Magnesium Added, g</th>
<th>mL H\textsubscript{2} evolved (STP)</th>
<th>Per Cent Added (50 g Sample Basis)</th>
<th>Per Cent Mg or Al found (50 g Sample Basis)</th>
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</table>
3.4 Elemental Sulfur - Method 1

3.4.1 Reference


3.4.2 Reagents

Bromine in carbon tetrachloride(a) - 10%. Add 10 g Ba to 90 g reagent grade
CCl₄. Stir until homogeneous. Store in glass-stoppered bottle.
Concentrated HNO₃
Concentrated HCl
Bromophenol Blue indicator - Grind 0.1 g of the powder with 1.5 mL 0.1N
NaOH solution and dilute to 25 mL.
Barium chloride solution - 10%. Dissolve 100 g BaCl₂.2H₂O in 900 mL H₂O
and filter through Whatman No. 42 paper. 1 mL = 14 mg S.

3.4.3 Procedure

Weigh 50 ± 0.1 g of sample into a 400 mL beaker and add about 200 mL of water.
Filter the solution through a Gooch crucible. Transfer all residue from beaker to
 crucible and wash thoroughly with distilled water.

Transfer residue to a 250 mL beaker. Evaporate the water to dryness then cool to
room temperature. Add 10 mL of bromine in carbon tetrachloride, cover and allow
to stand about thirty minutes, stirring several times. Add 15 mL concentrated
HNO₃, cover and allow to stand about thirty minutes, stirring several times.
Evaporate on hot plate to about 5 mL. Add 20 mL concentrated HCl and evaporate
to about 5 mL. Add about 50 mL water, filter, and wash with 2% HCl. Add two
drops bromophenol blue indicator and then ammonium hydroxide to first color
change. Add HCl dropwise until distinctly acid, then five drops in excess; dilute to
150 mL, heat to boiling, and add barium chloride solution slowly dropwise until
about 50% excess is present. Cover beaker and digest on steam bath at least one
hour. Cool to room temperature and filter on a Gooch crucible previously ignited at
800°C and weighed. Wash ten times with hot water. Ignite in muffle at 800°C for
at least twenty minutes. Cool in desiccator and weigh as BaSO₄.

3.4.4 Calculation

% Sulfur as S = \( \frac{\text{Wt BaSO}_4 \times 32.06 \times 100}{\text{Wt Sample} \times 233.42} \) or \( \frac{\text{Wt BaSO}_4 \times 13.735}{\text{Wt Sample}} \)

(a) NOTE: Bromine is an irritant and should be handled with caution. Carbon tetra-
chloride is a carcinogen and should be used with extreme caution.

3.5 Sulfur - Method 2

3.5.1 Reference - ASTM Method D1266-80

3.5.2 Apparatus

Photometer - Preferably a spectrophotometer having effective band width of
about 50 nm and equipped with a blue-sensitive phototube for use at 450 nm,
or alternatively a filter photometer equipped with a color filter having a
maximum transmission at approximately 425 nm.
Absorption Cells - Cells having optical path lengths of 5 cm are preferred. With use, the cells may become coated with a film. To remove this film, wash the cells with a detergent using a soft brush. Rinse thoroughly with deionized water following cleaning.

NOTE: The procedure as written assumes an absorbance change of about 0.100 for each 0.1 mg of sulfur in 50 mL of solution measured in a 5 cm cell. Photometers employing cells of shorter optical paths give proportionately poorer precision.

Scoop - Capable of dispensing $0.30 \pm 0.01$ g of 20 to 30 mesh barium chloride dihydrate.

Magnetic Stirrer - Equipped with 1-1/4 inch tetrafluoroethylene (Teflon) covered stirring bars.

3.5.3 Reagents

Alcohol - Glycerin Mixture - Mix 2 volumes of 95% ethyl alcohol with 1 volume of anhydrous glycerin. To this solution in a 1-liter volumetric flask, add 10 mL of H$_2$SO$_4$ (1 mL = 1 mg S) and make up to volume with alcohol-glycerin solution. Each milliliter of the resulting solution is then equivalent to about 0.01 mg of sulfur.

Barium Chloride Dihydrate - (BaCl$_2$.2H$_2$O), 20 to 30 mesh crystals.

NOTE: The crystal size of the BaCl$_2$.2H$_2$O is an important variable that affects the development of turbidity.

Hydrochloric Acid (1 + 12) - Add 77 mL of concentrated hydrochloric acid (HCl, sp. gr. 1.19) to a liter volumetric flask and dilute to the mark with deionized distilled water.

Sulfuric Acid (1 mL = 1.0 mg S) - Dilute 62.4 $\pm$ 0.1 mL of 1 N sulfuric acid (H$_2$SO$_4$) to exactly 1 liter with deionized distilled water. Check the dilution by titration against standard sodium hydroxide (NaOH) solution of about the same normality and adjust the concentration, if necessary, so that each milliliter of this solution is equivalent to 1.0 mg of sulfur.

Sulfuric Acid (1 mL = 0.100 mg S) - Dilute 6.24 $\pm$ 0.01 mL of 1 N sulfuric acid (H$_2$SO$_4$) to exactly 1 liter with deionized distilled water. Check the dilution by titration against standard NaOH solution of about the same normality and adjust the concentration, if necessary, so that each milliliter of this solution is equivalent to 0.100 mg of S.

Water, Deionized Distilled - Percolate distilled water through a column of mixed anion and cation exchange resins.

NOTE: A means for determining when to replace the exchange resins should be supplied. Use of a simple electrical conductivity meter has been found satisfactory for this purpose.

3.5.4 Calibration

Into 50 mL volumetric flasks introduce, by means of a buret, 0.25, 0.50, 0.75, 1.00, 1.50, 2.00, 3.00 and 5.00 mL of H$_2$SO$_4$ (1 mL = 0.100 mg S). Add 3.0 mL of HCl (1 + 12) to each flask, dilute to volume with water, and mix thoroughly. Prepare a reagent blank standard in a similar way, omitting the H$_2$SO$_4$. 

6
Pour the entire contents of each flask into a 100 mL beaker, add by means of a pipet 10 ± 0.1 mL of alcohol - glycerin mixture, and mix for 3 minutes on the magnetic stirrer. Select a stirring speed just below that which might cause loss of sample through splashing. Maintain this speed throughout the entire procedure.

Allow the solution to stand undisturbed for 4 minutes. Transfer to an absorption cell and measure the initial absorbance, using water as reference.

Return the solution to the beaker and add 0.30 ± 0.01 g of BaCl₂·2H₂O crystals, either by weighing this amount or by use of the scoop. Stir with the magnetic stirrer for exactly 3 minutes. Allow to stand for an additional 4 minutes, transfer to the cell, and again measure the absorbance relative to water.

Following steps described in 2nd, 3rd, 4th paragraphs above, obtain a reagent blank reading by subtracting the initial absorbance of the reagent blank standard from that obtained after addition of BaCl₂·2H₂O. This reading should not exceed 0.105 (includes influence of added sulfur).

Obtain the net absorbance for each standard by subtracting the initial absorbance and reagent blank reading from the absorbance obtained in accordance with the 4th paragraph above. Plot the net absorbance of each standard against milligrams of sulfur contained in 50 mL of solution, and draw a smooth curve through the points.

Check the calibration curve daily by making single determinations to detect possible shifts.

3.5.5 Procedure

Weigh 2 to 4 g of sample into a 400 mL beaker and add about 200 mL of water. Filter the solution through a Gooch crucible. Transfer all residue from beaker to crucible and wash thoroughly with distilled water.

Transfer residue to a 250 mL beaker. Evaporate the water to dryness then cool to room temperature. Add 10 mL bromine in carbon tetrachloride, cover and allow to stand about thirty minutes, stirring several times. Add 15 mL concentrated HNO₃, cover and allow to stand about thirty minutes, stirring several times. Evaporate on hot plate to about 5 mL. Add 20 mL concentrated HCl and evaporate to about 5 mL, transfer to a 50 mL volumetric flask with analytical rinsing practice and dilute to volume.

Pour the entire contents of the 50 mL volumetric flask containing the solution to be analyzed into a 100 mL beaker and proceed as directed in the 2nd, 3rd, and 4th paragraphs of the calibration procedure. Treat a blank in the same way and obtain reagent blank reading by subtracting its initial absorbance from that obtained after addition of BaCl₂·2H₂O.

NOTE: Should the blank reading exceed 0.120, the precision obtainable will be impaired. In this event, make an analysis of the reagents alone to determine which reagents are at fault.

Obtain the net absorbance of the analysis solution by subtracting the initial absorbance and reagent blank reading from that obtained after addition of BaCl₂·2H₂O.

Convert net absorbance to milligrams of sulfur by using the calibration curve.
3.5.6 Calculation

Calculate the amount of sulfur in the sample as follows:

\[
\text{Sulfur content, ppm} = \frac{A}{WF} \times 1000
\]

where:

- \( A \) = milligrams of sulfur read from the calibration curve.
- \( W \) = grams of sample.
- \( F \) = aliquot fraction of the sample solution used for analysis.

3.6 Chlorides

3.6.1 Reference


3.6.2 Reagents

Silver Nitrate Solution - Dissolve 5 g AgNO₃ in water and dilute to 1 L. Standardize against pure, dry NaCl. Potassium Chromate Indicator - Dissolve 5 g K₂CrO₄ in water and dilute to 100 mL.

3.6.3 Procedure

Weigh 10.0 g of sample, transfer to 250 mL Erlenmeyer flask and dissolve in about 40 mL distilled water. Dilute to 50 mL, add 1 mL potassium chromate indicator and titrate with standardized silver nitrate to a persistent reddish-brown color. During the titration dropwise addition of the silver nitrate while constantly swirling the flask will yield the best end point.

3.6.4 Calculation

\[
\% \text{ Cl} = \frac{\text{mL AgNO}_3 \times F \times 100}{\text{wt sample}}
\]

Where \( F = \frac{g \text{ Cl}}{mL \text{ AgNO}_3} \)

SECTION 4.0 BURNING TEST

4.1 Introduction

The following information sets forth the findings of our Technical Committee with respect to the burning test.

Efforts to make this burning procedure sufficiently sensitive and quantitative to allow distinction between Ammonium Nitrate Fertilizer and Ammonium Nitrate Fertilizer to which as little as 0.5 wt % fuel oil was added were unsuccessful. Distinction between Ammonium Nitrate Fertilizer and Ammonium Nitrate Fertilizer plus 6 wt % fuel oil can easily be distinguished.
In the tests conducted to develop this definition of Ammonium Nitrate Fertilizer, the analytical procedures and/or the detonation test disqualified materials that would not have been disqualified by this burning procedure. However, there is the possibility that new materials may contain substances which will catalytically increase the decomposition rate of ammonium nitrate but are not specified in the Definition. The burning test is designed to screen such materials.

4.2 **Equipment**  
(see Figure 2)

- **Burning tower** - Two 55-gallon 18 gauge steel drum frames welded together form the outer wall and two 30-gallon 18 gauge steel drum frames welded together form the inner wall. The inner wall can also be fabricated from 16 gauge carbon steel sheeting with the same dimensions as the 30-gallon drums. The space between the inner and outer walls is filled with rock wool insulation. Steel rings are welded between the two walls at the top and bottom to provide a closed insulation chamber. A 15” x 15” tight lock fire door is located at the bottom of the tower.

- **Fire grate** - A circular triple grate-screen of diameter slightly less than the diameter of the inner tower with 1/8” x 1/2” openings; the screens placed on top of each other with opening positions staggered 45 degrees. A stand or grate holder of appropriate design and dimensions and 12” high is fabricated from 1” angle iron.

- **Catch pan** - Stainless steel pan of a diameter slightly less than the diameter of the inner tower and 6” high.

- **Tower (top) cover** - 1/8” thickness by 26” diameter carbon steel plate.

4.3 **Procedure**

The burning tower is laid horizontal on the ground. The fire grate and stand resting inside the catch pan are slipped into the inner chamber. The equipment assembly is then raised to a vertical position. Top of the fire grate is approximately 16” from the bottom of the tower. Dirt is piled around the bottom circumference of the tower to a height of 12-15” except at the fire door.

Two 80-pound Kraft paper bags of the material to be tested are placed in the tower. One extra paper bag, cut or torn into pieces approximately 2” on a side, is mixed as uniformly as possible with the material in each bag.

Ten pounds of soft pine wood, 1/2” (T) x 2” (W) x 8” (L) pieces, are piled loosely in place in the space beneath the fire grate.

A standard railroad fuse is ignited and placed at the center beneath the wood pile.

When the fire has reached such an intensity that molten ammonium nitrate starts dropping into the fire, the fire door is closed, dirt is piled around it, and the top cover is put in place. Personnel should then retire to a safe position for observation of the test.

The burning is observed for rate of gas evolution visually and aurally, flames, oxides of nitrogen and other means by which rate of burning can be estimated. In general, compositions which contain appreciable quantities of oxidizable material will liberate less nitrogen dioxide, i.e., the evolved gases will be less highly colored.

The test is considered complete whenever the gas evolution becomes extremely slow or stops.
4.4 Evaluation of Results

Ammonium nitrate formulations in which the ammonium nitrate is completely decomposed in one hour or less after the apparatus is closed off shall not be described as Ammonium Nitrate Fertilizer for purposes of transportation.

4.5 Notes

a) The burning test should not be attempted until the substance undergoing testing has met chemical specifications in the Definition and passed the detonation resistance test.

b) The burning test should only be conducted by fully qualified personnel in specially designed equipment, and under carefully controlled conditions.

c) Unknown substances considered potentially hazardous should first be screened by conducting the test with the fire door and top partially open. When such systems are being tested, personnel should leave the immediate vicinity of the test apparatus as soon as the fire is started.

d) If the results are marginal (60 minutes ± 10 minutes) it is recommended that the burn test be repeated at least two times to determine similar results at least two out of three times.
Apparatus For
Determination of Elemental
Metals in Ammonium Nitrate Fertilizer

Figure 1.—A 25 ML closed end buret with ground ball joint and a thick walled flask with matching joint and stopcock side arm can be made by most laboratory glassware manufacturers.
Figure 2
Tower: Two 55 gallon steel (18 gauge) drums welded together for outer wall and two 30-gallon steel drums welded together for inner wall.