

Jeb Bush  
Governor

# Department of Environmental Protection

Northeast District  
7825 Baymeadows Way, Suite B200  
Jacksonville, Florida 32256-7590

David B. Struhs  
Secretary

## CERTIFIED - RETURN RECEIPT

February 22, 2000

Mr. P. Rodney Wilson, Executive Vice President  
PCS Sales (USA), Inc.  
5750 Old Orchard Road, Suite 440  
Skokie, IL 60077

Dear Mr. Wilson:

Columbia County - AP  
PCS Sales (USA), Inc.  
Truck/Rail Transfer Terminal

The Bureau of Air Regulation has completed its review of the proposed Post Construction Monitoring Plan for the referenced facility dated May 11, 1999. After conducting further refined modeling, the Department concurs with the recommendation that one nipher gauge be used to satisfy the requirements of Rule 62-212.600(2)(c), F.A.C. Furthermore, the Department is in agreement that the location of the nipher gauge be on the property boundary, immediately north of the sulfur system at the location of maximum predicted off-property sulfur deposition, i.e. as shown in Attachment 2 of the May 11, 1999 submittal.

In order to assure proper quality assurance, the particulate deposition monitoring shall be conducted in accordance with the attached DEP Reference Method for Monitoring the Deposition of Sulfur Particulate.

As stated in Rule 62-212.600(2)(c), the postconstruction air quality and deposition monitoring shall be conducted for two years from the date of issuance of the initial air operation permit for the facility, and through the permitting process, may continue for an additional time period if so determined.

Should you have any questions concerning this matter, please contact either Cleve Holladay, Bureau of Air Regulation, at (850) 488-1344 or Rita Felton-Smith, Northeast District Office, at (904) 448-4310, extension 237.

Sincerely,

Christopher L. Kirts, P.E.  
District Air Program Administrator

RFs  
cc: Charlie Pultz, P.E., PCS Phosphate  
Pradeep Raval, Koogler and Associates  
Al Linero, NSR  
Cleve Holladay, BAR

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# DEP Reference Method for Monitoring the Deposition of Sulfur Particulate.

## Principle

A modified Nipher Gauge is placed at the monitoring site. The amount of deposited elemental sulfur is determined monthly using a standard turbidimetric or colormetric method.

## Apparatus

1. A modified Nipher Gauge constructed to the dimensions shown in the attached figure and equipped with a bird ring.
2. For the turbidimetric method, an instrument to measure turbidity.
3. For the colormetric method, the apparatus specified in EPA Method 375.2 and AOAC Method 2.162.

## Reagents

1. Reagents for the turbidimetric method include reagent grade acetone; reagent grade phosphorus pentoxide; reagent grade potassium hydroxide; reagent grade recrystallized sulfur; and reagent grade pyrogallol.
2. Reagents for the colormetric method include reagent grade phosphorus pentoxide; reagent grade potassium hydroxide; and reagent grade pyrogallol.

## Sampling

1. Thoroughly rinse the modified Nipher Gauge. Place distilled water in the modified Nipher Gauge jar so that the level stands at one-half the cylinder depth when the test is started. In cold weather, mix a sufficient volume of antifreeze with the water to prevent freezing. Modified Nipher Gauge liquid should be kept at a reasonable level during the testing period (at least one inch of water at all times).

2. A sampling period shall be one calendar month corrected to 30 days. Allowance of +2 days is permissible for setting out or collecting sampling jars or both.

3. No attempt shall be made to remove collected particulate sample from the modified Nipher Gauge jar at the field site. The modified Nipher Gauge deposition collection jar shall be covered and taken to the laboratory for analysis of the contents.

4. Selection of Sampling Site. The following specific recommendations shall be used as a guide in the selection of a site. If conditions do not permit application of these recommendations, note shall be made of this.

a. The sampling station shall have a free exposure so that the sample is collected by gravity settling only. It must be free from undue local sources of pollution and interference from buildings or other higher objects or structures. Accessibility and security (freedom from tampering) are major considerations in the selection of a site.

b. The top of the modified Nipher Gauge shall be located a minimum of two meters and a maximum of 10 meters above the ground. It shall be at least one meter above any other nearby surface, such as a roof. Higher objects such as parapets, signs, penthouses, and the like shall not be more than 30 degrees from the horizontal, as measured in section d., below.

c. Locate the gauge at least 100 meters away from any known source of sulfur particulate.

d. When high buildings in the immediate vicinity of a monitor cannot be avoided, the top of any building shall be not more than 30 degrees above a sampling point.

5. Number of Sampling Stations. For each area or zone to be tested, a minimum of one sampling station shall be provided. When several adjacent areas or zones are to be sampled, orderly spacing of the stations shall be made so that they are approximately equally distant from each

other and from boundaries of the area. Record the vertical distance from the top of the modified Nipher Gauge to ground level for each sampling station.

#### Laboratory Procedure

Filtration of the sample and determination of the weight of insolubles, sample volume, and pH may be done in accordance with common laboratory practice.

1. Use a filter paper for filtration. The filter paper sample is dried under vacuum over phosphorus pentoxide in the presence of potassium hydroxide and pyrogallol to prevent oxidation of sulfur before the determination.
2. When dry, the sample is weighed and the total amount of filtered material is recorded.
3. A sub-sample of the particulate material is obtained and finely ground in a mortar and a portion of this sub-sample is accurately weighed.
4. Free sulfur determination by the turbidimetric method.
  - a. Extract the sample by shaking for five minutes with acetone in a centrifuge tube. A clear supernatant liquid is obtained by centrifugation at approximately 200 revolutions per minute and 22 centimeters radius for at least 15 minutes.
  - b. A suitable aliquot of this solution is mixed with about 80 milliliters of distilled water in a 100 milliliter calibrated flask, with the flask being gently agitated during the sample addition. Colloidal sulfur begins to form immediately after the solvent has been exchanged.
  - c. The liquid in the flask is then diluted to the mark with distilled water, mixed, and set aside for three hours, after which the optical density of the solution is measured at 420 millimicrons.
  - d. The sulfur content of the original sample is calculated by reference to a calibration graph plotted

from the results obtained when a standard solution of sulfur in acetone is used.

e. If the concentration of sulfur in the extract exceeds 200 grams per milliliter, the acetone dissolution is to be repeated at a lower ratio of sample to acetone to ensure complete removal of sulfur from the acetone.

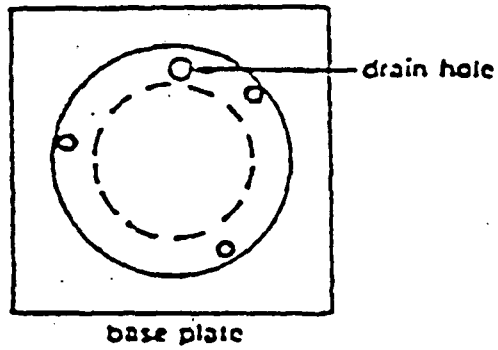
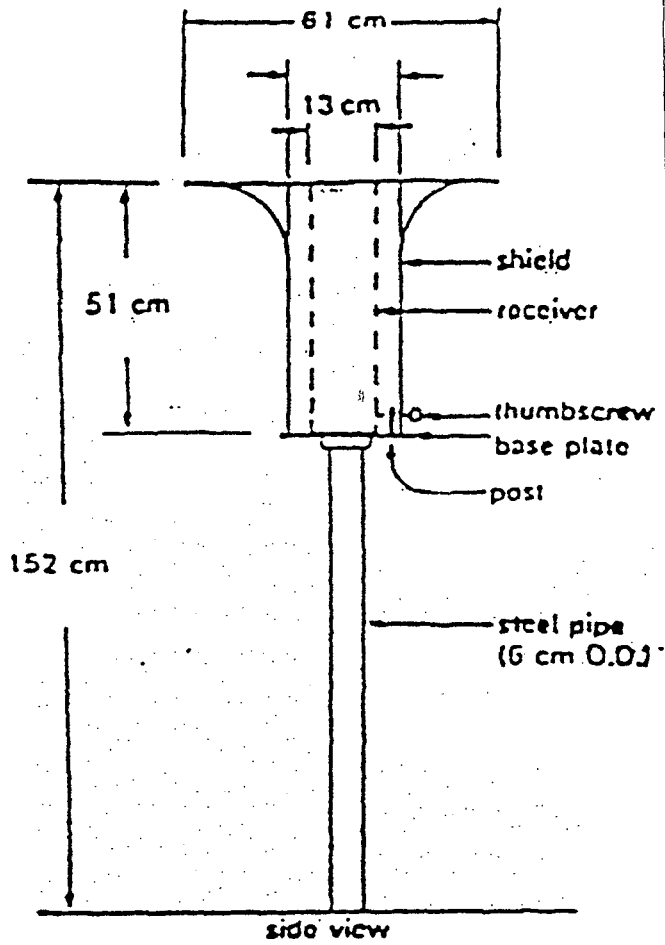
5. Sulfur determination by colormetric method

a. Extract the elemental sulfur from the sample and oxidize to a sulfate using AOAC Method 2.162, Determination of Free Sulfur.

b. Determine the quantity of sulfur in the sample using EPA Method 375.2, Sulfate Determination by Methylthymol Blue Method. The total free sulfur present in the original sample shall be reported as milligrams of sulfur as sulfur.

Calculations and Record Keeping

Divide the weight of the total insolubles and the weight of the sulfur fraction, determined to the nearest 0.1 milligram, by the cross-sectional area of the inside of the top opening of the modified Nipher Gauge sample collection jar times the time period over which the sample was collected. Determine the cross-sectional area to the nearest square millimeter. Determine the sample exposure time to the nearest hour. Measure the bulk pH of the sample water before the sulfur extraction. Record the total (insoluble) and the sulfur deposition rates in units of kilogram per hectare per month. Record the sample volume in milliliters, its bulk pH, sampling period and site identification information along with any notes or comments that might be helpful in later evaluating the sampling results.



225