

APPLICATION FOR FDER CONSTRUCTION PERMITS

AGRICO CHEMICAL COMPANY
SOUTH PIERCE CHEMICAL WORKS
POLK COUNTY, FLORIDA

EXISTING POLK COUNTY
AMBIENT SULFUR DIOXIDE MONITORING SUMMARY

AUGUST 29, 1980

SUMMARY OF POLK COUNTY AMBIENT SULFUR DIOXIDE MONITORING

During the period January 1977 through January 1978, Sholtes & Koogler Environmental Consultants conducted an ambient sulfur dioxide monitoring program for W.R.Grace in southwest Polk County, Florida. The network consisted of four (4) monitoring sites located as shown in the attached figures. The samples were collected by the EPA Reference Method (40 CFR 50, Appendix A). Each of the four samplers were temperature controlled.

The criteria used for establishing the monitoring site locations is detailed in the attached correspondence. Basically, the monitoring sites were selected using as criteria: (1) the results of an air quality modelling study conducted for W.R.Grace (See Attachments); and (2) the availability of electric power and security for the monitors.

The monitoring network was approved by the Florida Department of Environmental Regulation and was inspected and found satisfactory by representatives of that agency (see attached correspondence).

The results of the monitoring program which are detailed in the attached report, indicated that ambient 24-hour sulfur dioxide levels in southwest Polk County were well within the applicable Florida Air Quality Standard.

During the operation of the monitoring program, various quality assurance measures were incorporated to insure the validity of the monitoring data. The monitors were serviced during each six-day period by a field technician who recorded the necessary field data and inspected monitoring sites. Attached are sample copies of the field data sheets and a copy of the checklist the field technician followed. Although it is not specifically recorded on the field data sheet, the temperature of the container housing the sulfur dioxide bubbler tubes was checked each time the site was visited. The field technician had specific instructions to record on the data sheet any instance when the bubbler temperature exceeded 50° F.

The exposed absorbing solution was recovered from each monitoring site and stored for a period of three weeks at a temperature below 50° F. When three sets of samples were accumulated they were packed in a refrigerated container and shipped by United Parcel Service to the analytical laboratory in Jacksonville. A thermometer was packed with the samples.

Upon receipt at the laboratory the temperature of the samples was recorded and the samples transferred to refrigeration (4°C) until the samples were analyzed. Analysis took place within one week after receipt of the samples. This schedule was such that no one sample was held longer than 30 days before analysis.

During the analysis of the samples which was conducted on a Technicon Automatic Analyzer, approximately five percent (5%) blanks and five percent (5%) spike samples were run.

The laboratory data including the percent transmittance for each sample and the total micrograms of sulfur dioxide in each sample were forwarded to SKEC. The field technician likewise forwarded the field data sheets and orifices used for controlling the flow through the sampler to SKEC.

It should be noted that orifices were calibrated before and after each sample was run.

At SKEC the sulfur dioxide concentrations were calculated and orifices recalibrated and returned to the field technician.

Periodically spot checks were made on the sulfur dioxide concentration calculations by principals of SKEC.

The laboratory, Southern Analytical Laboratories, Inc., has participated for several years in the EPA Quality Assurance Program for Ambient Sulfur Dioxide Sample Analysis. The success of the laboratory in analyzing the unknown samples has been very good.

AMBIENT AIR SULFUR DIOXIDE
MONITORING NETWORK

JANUARY 1977 - JANUARY 1978

W.R. GRACE AND COMPANY
BARTOW, FLORIDA

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TABLE OF CONTENTS

	<u>Page</u>
I. INTRODUCTION	1
II. SULFUR DIOXIDE SAMPLING PROCEDURE	2
III. SAMPLING SITE DESCRIPTION	6
IV. DISCUSSION OF RESULTS	8
APPENDIX A	
APPENDIX B	

I. INTRODUCTION

Commencing January 19, 1977 and continuing through January 8, 1978, Sholtes & Koogler Environmental Consultants (SKEC) of Gainesville, Florida operated and maintained an ambient air sulfur dioxide monitoring network for W.R. Grace and Company in Polk County, Florida. The sampling was required of W.R. Grace by the Florida Department of Environmental Regulation (FDER) as a proviso on a construction permit for a sulfuric acid plant. The samples were collected by the EPA Reference Method (40 CFR 50, Appendix A). During this period a total of 239 out of a possible 240 samples were collected for data retrieval of 99.6 percent.

Of the 239 samples which were collected only two samples exceeded the minimum detectable limit for the sampling method of 25 micrograms per cubic meter. The maximum observed 24-hour concentration was on August 29, 1977 at Station No. 2. The concentration was 214.8 micrograms per cubic meter. The second highest concentration was also at Station 2 on April 19, 1977. The concentration was 92.0 micrograms per cubic meter. These compared to the Florida Department of Environmental Regulation (FDER) 24-hour SO_2 standard of 260 micrograms per cubic meter not to be exceeded more than once per year. It is suspected that the two high measured SO_2 levels resulted from a plant upset rather than from normal plant operation. The location of the upset was not investigated by SKEC.

The arithmetic average of the samples for each of the stations were all less than 10 micrograms per cubic meter. This compares with the annual FDER SO₂ standard of 60 micrograms per cubic meter. Table 1 is a summary of the sampling results observed at each site along with the arithmetic average for each station for the period in which these samples were collected. A tabulation of all sampling data is presented in the Appendix.

II. SULFUR DIOXIDE SAMPLING PROCEDURE

Ambient sulfur dioxide samplers were located at stations No. 1 through 4 shown in Figure 1. The samplers were operated on a six day schedule corresponding to the schedule adopted by FDER and the U.S. Environmental Protection Agency (EPA). Sulfur dioxide sampling was conducted using the EPA Reference Method (pararosaniline method) as outlined in the Federal Register (Volume 36, No. 21, Appendix A, January 30, 1971). The samplers were temperature controlled.

The method involves the placement of 50 mililiters of a potassium tetrachloromercurate (TCM) solution in a polypropylene absorber and sampling at a constant rate of approximately 0.2 liters per minute for a 24-hour period. The analysis for sulfur dioxide is a spectrophotometric method. A copy of the sampling and analytical procedures is included in the Appendix of this report.

Figure 2 is a schematic diagram of the sulfur dioxide sampling train used to collect the ambient samples. The components consist of a

TABLE 1

SUMMARY OF 24-HOUR SULFUR DIOXIDE
MONITORING DATA - W.R. GRACE NETWORK
POLK COUNTY, FLORIDA
JANUARY 1977 - JANUARY 1978

Site	No. of Samples	Sulfur Dioxide Data ($\mu\text{g}/\text{m}^3$)			
		24-Hour Concentrations			Annual Average
		High	2nd High	3rd High	
1	60	21.0	9.9	9.5	3.6
2	60	214.8	92.0	17.9	9.2
3	60	15.6	15.1	11.0	3.7
4	59	14.3	9.8	9.4	2.7

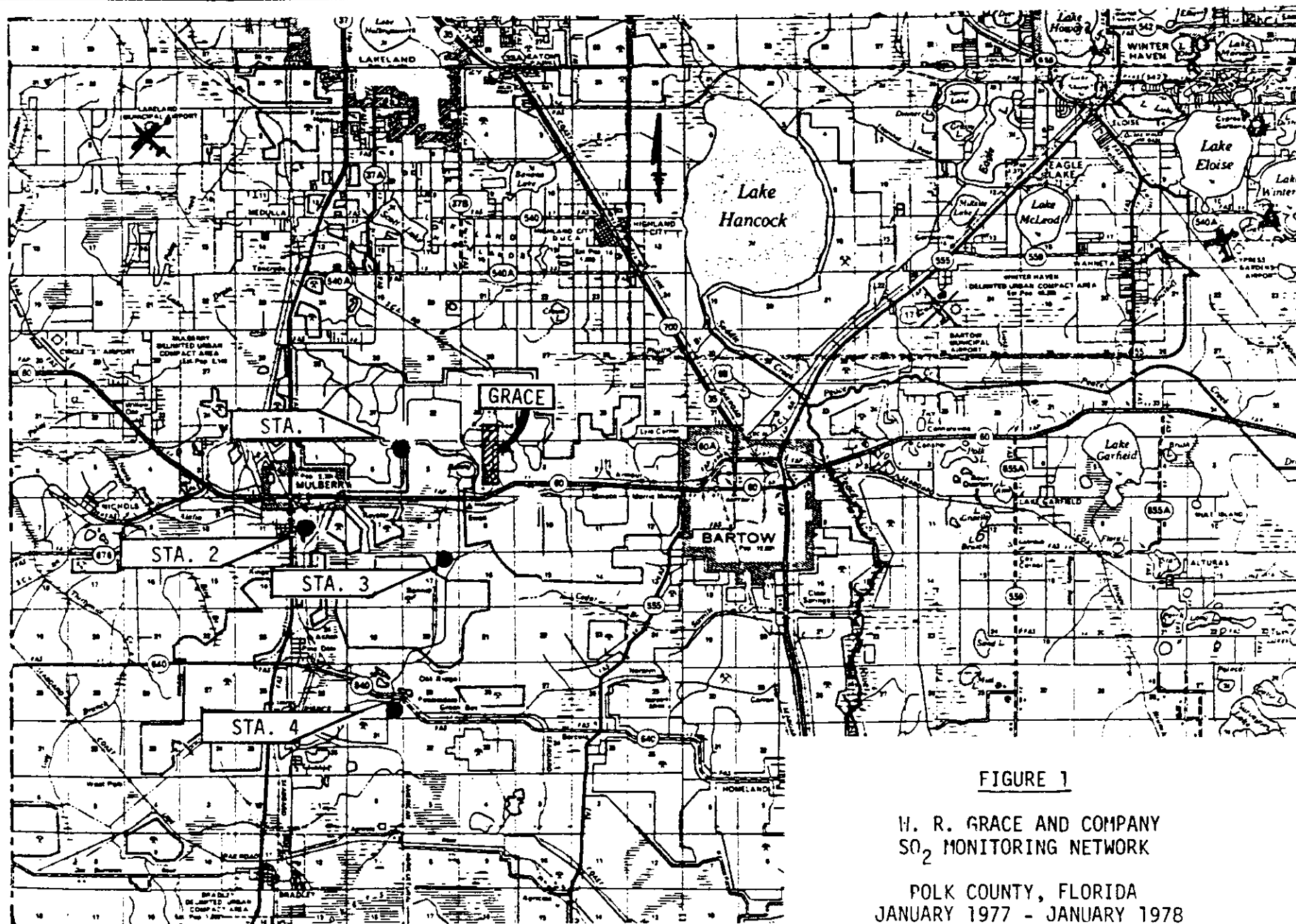
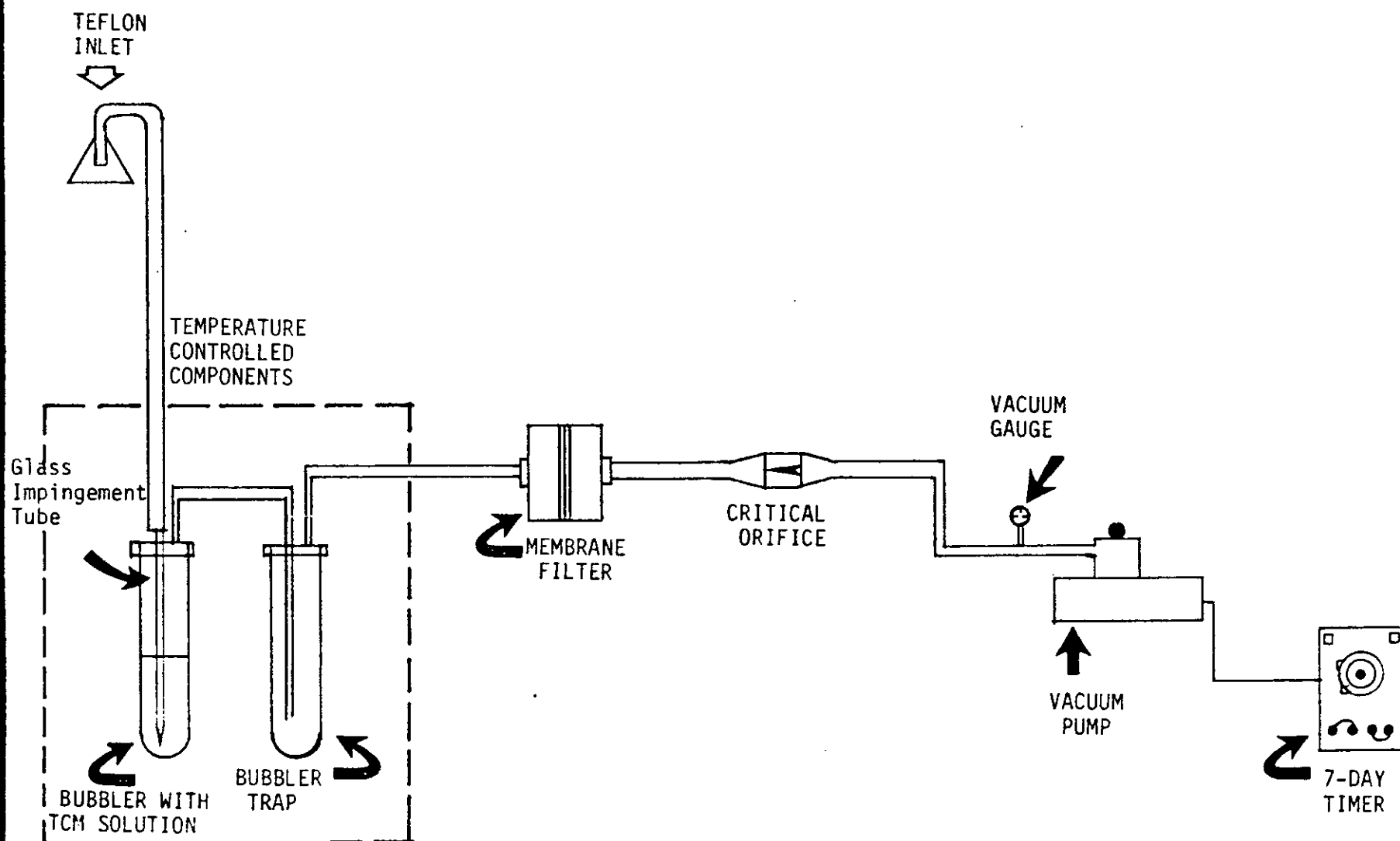


FIGURE 1

W. R. GRACE AND COMPANY
SO₂ MONITORING NETWORK

POLK COUNTY, FLORIDA
JANUARY 1977 - JANUARY 1978

FIGURE 2
AMBIENT SO₂ SAMPLING TRAIN
24-HOUR SAMPLING METHOD



teflon inlet tube connected to a polypropylene bubbler containing 50 mililiters of TCM solution. This is connected to a polypropylene trap, then to a membrane filter to clean the stream, and then to a critical orifice and a vacuum pump. The system is controlled with a 7-day timer.

The absorber solution is kept in a refrigerated container prior to, during, and after exposure to prevent sample and reagent deterioration. During the sampling period and during the subsequent storage prior to shipping the samples are maintained at 45-50°F. During shipping the samples are maintained at 50°F. Sampler and storage temperature is monitored by the field technicians and sample temperature is checked upon receipt at the laboratory.

III. SAMPLING SITE DESCRIPTIONS

Sampling Station No. 1 is located at UTM coordinates 3086.5 kilometers north and 407.5 kilometers east. This site is located on reclaimed land owned by W.R. Grace and Company. The station was located at a power sub-station which was the only obstruction within 1000 meters of the sampler. The site is approximately one mile north of Route 60 and slightly to the northwest of the W.R. Grace chemical complex. Between the site and the chemical complex is nothing but reclaimed land.

Station 2 is located at UTM coordinates 3084.7 kilometers north and 404.5 kilometers east. It is located in a residential area of south-

east Mulberry. This station is located in the back yard of a small housing development. To the north and east of the sampler there are no obstructions for approximately 100 meters. To the west and south there are residential homes which are no closer than 30 meters.

Station 3 is located at UTM coordinates 3084.0 kilometers north and 408.8 kilometers east. This sampler had no major obstructions within 500 meters. The sampler is located southwest of the W.R. Grace complex. There are no obstructions between W.R. Grace and the sampler.

Station 4 is located at UTM coordinates 3079.6 kilometers north and 407.2 kilometers east. This sampling site is located in a pasture almost due south of the W.R. Grace chemical complex. The sampler was located approximately 40 meters from a small mobile home with no other obstructions within 200 meters of the sampler. This sampler is located approximately one-half mile off Highway 640.

The SAROAD numbers for each of the sampling sites are:

Station No. 1	103680005
Station No. 2	103680006
Station No. 3	103680007
Station No. 4	103680008.

IV. DISCUSSION OF RESULTS

During the period January 19, 1977 - January 8, 1978 a total of 239 sulfur dioxide samples were collected in the vicinity of the W.R. Grace and Company phosphate fertilized complex and mining operations in Polk County.

Of the 239 sulfur dioxide samples collected, only two, or 0.8 percent, exceeded the 25 microgram per cubic meter minimum detectable level established for the monitoring method by EPA. The sampling and analytical procedures for the method will detect concentrations less than the minimum detectable but the absorption efficiency of sulfur dioxide is low and variable so that these values of less than 25 micrograms per cubic meter are not quantitative.

It can be concluded that the air quality measured by the W.R. Grace network is much better than that required to meet State and Federal air quality standards. The annual average SO_2 levels ranged from 3 to 9 micrograms per cubic meter, compared with the FDER standard of 60 micrograms per cubic meter.

Except for the two excursions measured at Site 2 (215 and 92 micrograms per cubic meter), the 24-hour SO_2 levels were less than one-tenth of the FDER standard of 260 micrograms per cubic meter.

APPENDIX A
SO₂ MONITORING DATA

W.R. GRACE AND COMPANY
AMBIENT SULFUR DIOXIDE MONITORING NETWORK
BARTOW, FLORIDA

24-Hour SO₂ Concentrations

($\mu\text{g}/\text{m}^3$)

Date	Station 1	Station 2	Station 3	Station 4
1/19/77	4.2	1.9	7.2	2.5
1/25/77	4.7	6.2	4.7	4.0
1/31/77	3.6	7.3	2.6	6.5
2/6/77	8.5	8.9	10.1	6.6
2/12/77	9.3	9.5	9.3	9.0
2/18/77	9.5	11.5	9.0	9.4
2/24/77	8.3	7.4	7.1	6.5
3/2/77	0.72	17.9	15.1	14.3
3/8/77	9.9	5.7	8.6	0.39
3/14/77	6.7	13.8	10.0	4.3
3/20/77	2.7	3.5	3.9	2.4
3/26/77	4.0	2.9	3.4	3.7
4/1/77	0.0	0.77	0.0	0.0
4/7/77	0.0	0.0	0.0	0.0
4/13/77	0.96	0.0	0.0	0.0
4/19/77	4.2	92.0	3.4	3.1
4/25/77	2.6	2.7	1.0	3.6
5/1/77	2.5	6.2	2.7	5.0
5/7/77	4.1	4.4	1.2	1.8
5/13/77	1.3	1.8	0.0	1.2
5/19/77	1.2	1.3	1.9	1.7

W.R. GRACE AND COMPANY
 AMBIENT SULFUR DIOXIDE MONITORING NETWORK
 BARTOW, FLORIDA
 24-Hour SO₂ Concentrations
 (μg/m³)

Date	Station 1	Station 2	Station 3	Station 4
5/25/77	1.0	3.5	0.3	0.0
5/31/77	0.3	0.3	0.0	0.6
6/6/77	1.0	1.4	0.3	0.6
6/12/77	3.3	7.1	6.0	4.2✓
6/18/77	1.0	3.4	5.0	1.8
6/24/77	0.0	15.3	15.6	6.6
6/30/77	2.5	10.5	11.0	0.0
7/6/77	3.8	8.5	9.3	0.0
7/12/77	21.0	13.4	10.9✓	3.9✓
7/18/77	6.2	9.0	3.0✓	5.2✓
7/24/77	5.1	4.4	4.4✓	6.7✓
7/30/77	2.8	7.9	5.7✓	7.1✓
8/5/77	2.1	2.2	4.4✓	9.8✓
8/11/77	7.2	9.4	6.6✓	---
8/17/77	1.8	3.0	5.6✓	0.4✓
8/23/77	0.03	0.8	0.6✓	0.5✓
8/29/77	3.3	214.8	1.6	1.0
9/4/77	2.4	0.8	0.4	0.0
9/10/77	3.9	5.5	1.6	0.0
9/16/77	1.1	2.5	4.5	4.1
9/22/77	4.3	7.8	6.2	5.5
9/28/77	1.2	2.8	5.1	7.3

W.R. GRACE AND COMPANY
 AMBIENT SULFUR DIOXIDE MONITORING NETWORK
 BARTOW, FLORIDA

24-Hour SO₂ Concentrations

(μg/m³)

Date	Station 1	Station 2	Station 3	Station 4
10/4/77	1.4	2.0	0.6	0.0
10/10/77	4.6	0.7	1.4	1.2
10/16/77	0.7	0.3	0.3	0.3✓
10/22/77	0.7	0.7	0.3	0.7✓
10/28/77	0.0	0.4	0.0	0.0
11/3/77	2.1	0.0	0.0	0.7✓
11/9/77	0.4	0.7	0.3	0.0
11/15/77	0.7	0.7	0.0	0.0
11/21/77	0.0	0.7	0.0	0.0
11/27/77	0.7	0.4	0.4	1.1✓
12/3/77	0.0	0.0	0.0	0.0
12/9/77	0.0	0.0	0.0	0.0
12/15/77	0.0	0.0	0.0	0.0 ^L
12/21/77	0.70	0.0	0.0	0.0
12/27/77	2.1	2.8	3.4	2.8✓
1/2/78	2.2	2.2	2.6	2.9✓
1/8/78	2.1	2.6	2.4	2.5✓
Arithmetic Mean	3.6	9.2	3.7	2.7✓
No. of Samples	60	60	60	59

APPENDIX B
FIELD AND ANALYTICAL PROCEDURES

$$C = \frac{P \times 10^6}{R_2 + R_1}$$

Where:

C = Concentration of SO_2 , $\mu\text{g./m.}^3$ at reference conditions.

P = Tube permeation rate, $\mu\text{g./minute}$.

R_2 = Flow rate of dilution air, liter/minute at reference conditions.

R_1 = Flow rate of inert gas, liter/minute at reference conditions.

8.2.2.3 Sampling and Preparation of Calibration Curve. Prepare a series (usually six) of standard atmospheres containing SO_2 levels from 25 to 300 $\mu\text{g./m.}^3$. Sample each atmosphere using similar apparatus and taking exactly the same air volume as will be done in atmospheric sampling. Determine absorbances as directed in 7.2. Plot the concentration of SO_2 in $\mu\text{g./m.}^3$ (x-axis) against $A - A_0$ values (y-axis), draw the straight line of best fit and determine the slope. Alternatively, regression analysis by the method of least squares may be used to calculate the slope. Calculate the reciprocal of the slope and denote as B_1 .

8.3 Sampling Efficiency. Collection efficiency is above 98 percent; efficiency may fall off, however, at concentrations below 25 $\mu\text{g./m.}^3$. (12, 13)

9. Calculations.

9.1 Conversion of Volume. Convert the volume of air sampled to the volume at reference conditions of 25° C. and 760 mm. Hg. (On 24-hour samples, this may not be possible.)

$$V_2 = V \times \frac{P}{760} \times \frac{298}{t + 273}$$

V_2 = Volume of air at 25° C. and 760 mm. Hg, liters.

V = Volume of air sampled, liters.

P = Barometric pressure, mm. Hg.

t = Temperature of air sample, °C.

9.2 Sulfur Dioxide Concentration.

9.2.1 When sulfite solutions are used to prepare calibration curves, compute the concentration of sulfur dioxide in the sample:

$$\mu\text{g. SO}_2/\text{m.}^3 = \frac{(A - A_0) (10^6) (B_1)}{V_2} \times D$$

A = Sample absorbance.

A_0 = Reagent blank absorbance.

10^6 = Conversion of liters to cubic meters.

V_2 = The sample corrected to 25° C. and 760 mm. Hg, liters.

B_1 = Calibration factor, $\mu\text{g./absorbance unit}$.

D = Dilution factor.

For 30-minute and 1-hour samples,
 $D = 1$.

For 24-hour samples, $D = 10$.

9.2.2 When SO_2 gas standard atmospheres are used to prepare calibration curves, com-

pute the sulfur dioxide in the sample by the following formula:

$$\text{SO}_2, \mu\text{g./m.}^3 = (A - A_0) \times B_2$$

A = Sample absorbance.

A_0 = Reagent blank absorbance.

B_2 = (See 8.2.2.3).

9.2.3 Conversion of $\mu\text{g./m.}^3$ to p.p.m. If desired, the concentration of sulfur dioxide may be calculated as p.p.m. SO_2 at reference conditions as follows:

$$\text{p.p.m. SO}_2 = \mu\text{g. SO}_2/\text{m.}^3 \times 8.82 \times 10^{-4}$$

10. References.

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7.2.1.1 30-Minute and 1-Hour Samples. Transfer the sample quantitatively to a 25-ml. volumetric flask; use about 5 ml. distilled water for rinsing. Delay analyses for 20 minutes to allow any ozone to decompose.

7.2.1.2 24-Hour Sample. Dilute the entire sample to 50 ml. with absorbing solution. Pipet 5 ml. of the sample into a 25-ml. volumetric flask for chemical analyses. Bring volume to 10 ml. with absorbing reagent. Delay analyses for 20 minutes to allow any ozone to decompose.

7.2.2 Determination. For each set of determinations prepare a reagent blank by adding 10 ml. unexposed TCM solution to a 25-ml. volumetric flask. Prepare a control solution by adding 2 ml. of working sulfite-TCM solution and 8 ml. TCM solution to a 25-ml. volumetric flask. To each flask containing either sample, control solution, or reagent blank, add 1 ml. 0.6 percent sulfamic acid and allow to react 10 minutes to destroy the nitrite from oxides of nitrogen. Accurately pipet in 2 ml. 0.2 percent formaldehyde solution, then 5 ml. pararosaniline solution. Start a laboratory timer that has been set for 30 minutes. Bring all flasks to volume with freshly boiled and cooled distilled water and mix thoroughly. After 30 minutes and before 60 minutes, determine the absorbances of the sample (denote as A), reagent blank (denote as A_r) and the control solution at 648 nm. using 1-cm. optical path length cells. Use distilled water, not the reagent blank, as the reference. (Note: This is important because of the color sensitivity of the reagent blank to temperature changes which can be induced in the cell compartment of a spectrophotometer.) Do not allow the colored solution to stand in the absorbance cells, because a film of dye may be deposited. Clean cells with alcohol after use. If the temperature of the determinations does not differ by more than 2° C. from the calibration temperature (8.2), the reagent blank should be within 0.03 absorbance unit of the y-intercept of the calibration curve (8.2). If the reagent blank differs by more than 0.03 absorbance unit from that found in the calibration curve, prepare a new curve.

7.2.3 Absorbance Range. If the absorbance of the sample solution ranges between 1.0 and 2.0, the sample can be diluted 1:1 with a portion of the reagent blank and read within a few minutes. Solutions with higher absorbance can be diluted up to sixfold with the reagent blank in order to obtain onscale readings within 10 percent of the true absorbance value.

8. Calibration and Efficiency.

8.1 Flowmeters and Hypodermic Needle. Calibrate flowmeters and hypodermic needle (8) against a calibrated wet test meter.

8.2 Calibration Curves.

8.2.1 Procedure with Sulfite Solution. Accurately pipet graduated amounts of the working sulfite-TCM solution (6.2.9) (such as 0, 0.5, 1, 2, 3, and 4 ml.) into a series of 25-ml. volumetric flasks. Add sufficient TCM

solution to each flask to bring the volume to approximately 10 ml. Then add the remaining reagents as described in 7.2.2. For maximum precision use a constant-temperature bath. The temperature of calibration must be maintained within $\pm 1^\circ$ C. and in the range of 20° to 30° C. The temperature of calibration and the temperature of analysis must be within 2 degrees. Plot the absorbance against the total concentration in $\mu\text{g. SO}_2$ for the corresponding solution. The total $\mu\text{g. SO}_2$ in solution equals the concentration of the standard (Section 6.2.9) in $\mu\text{g. SO}_2/\text{ml.}$ times the ml. sulfite solution added ($\mu\text{g. SO}_2 = \mu\text{g./ml. SO}_2 \times \text{ml. added}$). A linear relationship should be obtained, and the y-intercept should be within 0.03 absorbance unit of the zero standard absorbance. For maximum precision determine the line of best fit using regression analysis by the method of least squares. Determine the slope of the line of best fit, calculate its reciprocal and denote as B. B. is the calibration factor. (See Section 6.2.10.1 for specifications on the slope of the calibration curve). This calibration factor can be used for calculating results provided there are no radical changes in temperature or pH. At least one control sample containing a known concentration of SO_2 for each series of determinations, is recommended to insure the reliability of this factor.

8.2.2 Procedure with SO_2 Permeation Tubes.

8.2.2.1 General Considerations. Atmospheres containing accurately known amounts of sulfur dioxide at levels of interest can be prepared using permeation tubes. In the systems for generating these atmospheres, the permeation tube emits SO_2 gas at a known, low, constant rate, provided the temperature of the tube is held constant ($\pm 0.1^\circ$ C.) and provided the tube has been accurately calibrated at the temperature of use. The SO_2 gas permeating from the tube is carried by a low flow of inert gas to a mixing chamber where it is accurately diluted with SO_2 -free air to the level of interest and the sample taken. These systems are shown schematically in Figures A2 and A3 and have been described in detail by O'Keefe and Ortman (9), Scaringelli, Frey, and Saltzman (10), and Scaringelli, O'Keefe, Rosenberg, and Bell (11).

8.2.2.2 Preparation of Standard Atmospheres. Permeation tubes may be prepared or purchased. Scaringelli, O'Keefe, Rosenberg, and Bell (11) give detailed, explicit directions for permeation tube calibration. Tubes with a certified permeation rate are available from the National Bureau of Standards. Tube permeation rates from 0.2 to 0.4 $\mu\text{g./minute}$, inert gas flows of about 60 ml./minute, and dilution air flow rates from 1.1 to 15 liters/minute conveniently give standard atmospheres containing desired levels of SO_2 (25 to 300 $\mu\text{g./m}^3$; 0.01 to 0.15 p.p.m. SO_2). The concentration of SO_2 in any standard atmosphere can be calculated as follows:

6.2.7 Sodium Thiosulfate Titrant (0.01 N). Dilute 100 ml. of the stock thiosulfate solution to 1,000 ml. with freshly boiled distilled water.

Normality = Normality of stock solution $\times 0.100$.

6.2.8 Standardized Sulfite Solution for Preparation of Working Sulfite-TCM Solution. Dissolve 0.3 g. sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) or 0.60 g. sodium sulfite (Na_2SO_3) in 500 ml. of recently boiled, cooled, distilled water. (Sulfite solution is unstable; it is therefore important to use water of the highest purity to minimize this instability.) This solution contains the equivalent of 320 to 400 $\mu\text{g./ml.}$ of SO_2 . The actual concentration of the solution is determined by adding excess iodine and back-titrating with standard sodium thiosulfate solution. To back-titrate, pipet 50 ml. of the 0.01 N iodine into each of two 500-ml. iodine flasks (A and B). To flask A (blank) add 25 ml. distilled water, and to flask B (sample) pipet 25 ml. sulfite solution. Stopper the flasks and allow to react for 5 minutes. Prepare the working sulfite-TCM solution (6.2.9) at the same time iodine solution is added to the flasks. By means of a buret containing standardized 0.01 N thiosulfate, titrate each flask in turn to a pale yellow. Then add 5 ml. starch solution and continue the titration until the blue color disappears.

6.2.9 Working Sulfite-TCM Solution. Pipet accurately 2 ml. of the standard solution into a 100 ml volumetric flask and bring to mark with 0.04 M TCM. Calculate the concentration of sulfur dioxide in the working solution:

$$\mu\text{g SO}_2/\text{ml.} = \frac{(A - B) (N) (32,000)}{25} \times 0.02$$

A = Volume thiosulfate for blank, ml.

B = Volume thiosulfate for sample, ml.

N = Normality of thiosulfate titrant.

32,000 = Milliequivalent wt. of SO_2 , $\mu\text{g.}$

25 = Volume standard sulfite solution, ml.

0.02 = Dilution factor.

This solution is stable for 30 days if kept at 5° C. (refrigerator). If not kept at 5° C., prepare daily.

6.2.10 Purified Pararosaniline Stock Solution (0.2 percent nominal).

6.2.10.1 Dye Specifications. The pararosaniline dye must meet the following performance specifications: (1) the dye must have a wavelength of maximum absorbance at 540 nm. when assayed in a buffered solution of 0.1 M sodium acetate-acetic acid; (2) the absorbance of the reagent blank, which is temperature-sensitive (0.015 absorbance unit/°C), should not exceed 0.170 absorbance unit at 22° C. with a 1-cm. optical path length, when the blank is prepared according to the prescribed analytical procedure and to the specified concentration of the dye; (3) the calibration curve (Section 8.2.1) should have a slope of 0.030 ± 0.002 absorb-

ance units/ $\mu\text{g. SO}_2$ at this path length when the dye is pure and the sulfite solution is properly standardized.

6.2.10.2 Preparation of Stock Solution. A specially purified (99–100 percent pure) solution of pararosaniline, which meets the above specifications, is commercially available in the required 0.20 percent concentration (Harleco®). Alternatively, the dye may be purified, a stock solution prepared and then assayed according to the procedure of Scaringelli, et al. (4).

6.2.11 Pararosaniline Reagent. To a 250-ml. volumetric flask, add 20 ml. stock pararosaniline solution. Add an additional 0.2 ml. stock solution for each percent the stock assays below 100 percent. Then add 25 ml. 3 M phosphoric acid and dilute to volume with distilled water. This reagent is stable for at least 9 months.

7. Procedure.

7.1 Sampling. Procedures are described for short-term (30 minutes and 1 hour) and for long-term (24 hours) sampling. One can select different combinations of sampling rate and time to meet special needs. Sample volumes should be adjusted, so that linearity is maintained between absorbance and concentration over the dynamic range.

7.1.1 30-Minute and 1-Hour Samplings. Insert a midjet impinger into the sampling system, Figure A1. Add 10 ml. TCM solution to the impinger. Collect sample at 1 liter/minute for 30 minutes, or at 0.5 liter/minute for 1 hour, using either a rotameter, as shown in Figure A1, or a critical orifice, as shown in Figure A1a, to control flow. Shield the absorbing reagent from direct sunlight during and after sampling by covering the impinger with aluminum foil, to prevent deterioration. Determine the volume of air sampled by multiplying the flow rate by the time in minutes and record the atmospheric pressure and temperature. Remove and stopper the impinger. If the sample must be stored for more than a day before analysis, keep it at 5° C. in a refrigerator (see 4.2).

7.1.2 24-Hour Sampling. Place 50 ml. TCM solution in a large absorber and collect the sample at 0.2 liter/minute for 24 hours from midnight to midnight. Make sure no entrainment of solution results with the impinger. During collection and storage protect from direct sunlight. Determine the total air volume by multiplying the air flow rate by the time in minutes. The correction of 24-hour measurements for temperature and pressure is extremely difficult and is not ordinarily done. However, the accuracy of the measurement will be improved if meaningful corrections can be applied. If storage is necessary, refrigerate at 5° C. (see 4.2).

7.2 Analysis.

7.2.1 Sample Preparation. After collection, if a precipitate is observed in the sample, remove it by centrifugation.

*Hartman-Leddon, 60th and Woodland Avenue, Philadelphia, PA 19143.

4.2 After sample collection the solutions are relatively stable. At 22° C. losses of sulfur dioxide occur at the rate of 1 percent per day. When samples are stored at 5° C. for 30 days, no detectable losses of sulfur dioxide occur. The presence of EDTA enhances the stability of SO₂ in solution, and the rate of decay is independent of the concentration of SO₂. (7)

5. Apparatus.

5.1 Sampling.

5.1.1 *Absorber.* Absorbers normally used in air pollution sampling are acceptable for concentrations above 25 µg./m.³ (0.01 p.p.m.). An all-glass midjet impinger, as shown in Figure A1, is recommended for 30-minute and 1-hour samples.

For 24-hour sampling, assemble an absorber from the following parts:

Polypropylene 2-port tube closures, special manufacture (available from Bel-Art Products, Pequannock, N.J.).

Glass impingers, 6 mm. tubing, 6 inches long, one end drawn to small diameter such that No. 79 jewelers drill will pass through, but No. 78 jewelers drill will not. (Other end fire polished.)

Polypropylene tubes, 164 by 32 mm. (Nalgene or equal).

5.1.2 *Pump.* Capable of maintaining an air pressure differential greater than 0.7 atmosphere at the desired flow rate.

5.1.3 *Air Flowmeter or Critical Orifices.* A calibrated rotameter or critical orifice capable of measuring air flow within ±2 percent. For 30-minute sampling, a 22-gauge hypodermic needle 1 inch long may be used as a critical orifice to give a flow of about 1 liter/minute. For 1-hour sampling, a 23-gauge hypodermic needle five-eighths of an inch long may be used as a critical orifice to give a flow of about 0.5 liter/minute. For 24 hour sampling, a 27-gauge hypodermic needle three-eighths of an inch long may be used to give a flow of about 0.2 liter/minute. Use a membrane filter to protect the needle (Figure A1a).

5.2 Analysis.

5.2.1 *Spectrophotometer.* Suitable for measurement of absorbance at 548 nm. with an effective spectral band width of less than 16 nm. Reagent blank problems may occur with spectrophotometers having greater spectral band width. The wavelength calibration of the instrument should be verified. If transmittance is measured, this can be converted to absorbance:

$$A = \log_{10}(1/T)$$

6. Reagents.

6.1 Sampling

6.1.1 *Distilled water.* Must be free from oxidants.

6.1.2 *Absorbing Reagent (0.04 M Potassium Tetrachloromercurate (TOM)).* Dissolve 10.86 g. mercuric chloride, 0.064 g. EDTA (ethylenediaminetetraacetic acid, disodium salt), and 6.0 g. potassium chloride in water and bring to mark in a 1,000-ml. volumetric flask. (Caution: highly poisonous. If spilled on skin, flush off with water immediately). The pH of this reagent should be approximately 4.0, but it has been shown that there is no appreciable difference in collection efficiency over the range of pH 5 to pH 3. (7) The absorbing reagent is normally stable for 6 months. If a precipitate forms, discard the reagent.

6.2 Analysis.

6.2.1 *Sulfamic Acid (0.5 percent).* Dissolve 0.5 g. sulfamic acid in 100 ml. distilled water. Prepare fresh daily.

6.2.2 *Formaldehyde (0.2 percent).* Dilute 5 ml. formaldehyde solution (36-38 percent) to 1,000 ml. with distilled water. Prepare daily.

6.2.3 *Stock Iodine Solution (0.1 N).* Place 12.7 g. iodine in a 250-ml. beaker; add 40 g. potassium iodide and 25 ml. water. Stir until all is dissolved, then dilute to 1,000 ml. with distilled water.

6.2.4 *Iodine Solution (0.01 N).* Prepare approximately 0.01 N iodine solution by diluting 50 ml. of stock solution to 500 ml. with distilled water.

6.2.5 *Starch Indicator Solution.* Triturate 0.4 g. soluble starch and 0.002 g. mercuric iodide (preservative) with a little water, and add the paste slowly to 200 ml. boiling water. Continue boiling until the solution is clear; cool, and transfer to a glass-stoppered bottle.

6.2.6 *Stock Sodium Thiosulfate Solution (0.1 N).* Prepare a stock solution by dissolving 25 g. sodium thiosulfate (Na₂S₂O₃ · 5H₂O) in 1,000 ml. freshly boiled, cooled, distilled water and add 0.1 g. sodium carbonate to the solution. Allow the solution to stand 1 day before standardizing. To standardize, accurately weigh, to the nearest 0.1 mg., 1.5 g. primary standard potassium iodate dried at 180° C. and dilute to volume in a 500-ml. volumetric flask. To a 500-ml. iodine flask, pipet 50 ml. of iodate solution. Add 2 g. potassium iodide and 10 ml. of 1 N hydrochloric acid. Stopper the flask. After 5 minutes, titrate with stock thiosulfate solution to a pale yellow. Add 5 ml. starch indicator solution and continue the titration until the blue color disappears. Calculate the normality of the stock solution:

$$N = \frac{W}{M} \times 2.80$$

N = Normality of stock thiosulfate solution.

M = Volume of thiosulfate required, ml.

W = Weight of potassium iodate, grams.

$$2.80 = \frac{10^6 (\text{conversion of g. to mg.}) \times 0.1 (\text{fraction iodate used})}{35.67 (\text{equivalent weight of potassium iodate})}$$

APPENDIX A—REFERENCE METHOD FOR THE
DETERMINATION OF SULFUR DIOXIDE IN THE
ATMOSPHERE (PARAROSANILINE METHOD)

1. *Principle and Applicability.* 1.1 Sulfur dioxide is absorbed from air in a solution of potassium tetrachloromercurate (TCM). A dichlorosulfitomercurate complex, which resists oxidation by the oxygen in the air, is formed (1, 2). Once formed, this complex is stable to strong oxidants (e.g., ozone, oxides of nitrogen). The complex is reacted with pararosanine and formaldehyde to form intensely colored pararosanine methyl sulfonic acid (3). The absorbance of the solution is measured spectrophotometrically.

1.2 The method is applicable to the measurement of sulfur dioxide in ambient air using sampling periods up to 24 hours.

2. *Range and Sensitivity.* 2.1 Concentrations of sulfur dioxide in the range of 25 to 1,050 $\mu\text{g}/\text{m}^3$ (0.01 to 0.40 p.p.m.) can be measured under the conditions given. One can measure concentrations below 25 $\mu\text{g}/\text{m}^3$ by sampling larger volumes of air, but only if the absorption efficiency of the particular system is first determined. Higher concentrations can be analyzed by using smaller gas samples, a larger collection volume, or a suitable aliquot of the collected sample. Beer's Law is followed through the working range from 0.03 to 1.0 absorbance units (0.8 to 27 μg . of sulfite ion in 25 ml. final solution computed as SO_2).

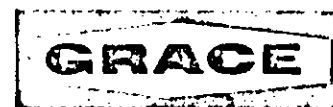
2.2 The lower limit of detection of sulfur dioxide in 10 ml. TCM is 0.75 μg . (based on twice the standard deviation) representing a concentration of 25 $\mu\text{g}/\text{m}^3 \text{SO}_2$ (0.01 p.p.m.) in an air sample of 30 liters.

3. *Interferences.* 3.1 The effects of the principal known interferences have been minimized or eliminated. Interferences by oxides of nitrogen are eliminated by sulfamic acid (4, 5), ozone by time-delay (6), and heavy metals by EDTA (ethylenediamine-tetraacetic acid, disodium salt) and phosphoric acid (4, 6). At least 60 μg . Fe (III), 10 μg . Mn(II), and 10 μg . Cr(III) in 10 ml. absorbing reagent can be tolerated in the procedure. No significant interference was found with 10 μg . Cu (II) and 22 μg . V(V).

4. *Precision, Accuracy, and Stability.* 4.1 Relative standard deviation at the 95 percent confidence level is 4.6 percent for the analytical procedure using standard samples. (5)

W. R. GRACE & CO.

AGRICULTURAL CHEMICALS GROUP



August 20, 1976

Mr. Joseph Tessitore
Department of Environmental Regulation
500 East Central Avenue - Suite 238
Winter Haven, Florida 33880

RE: #5 & #6 H₂SO₄ PLANTS

Dear Joe:

After consulting with John Koogler, of Sholtes & Koogler, about our proposed ambient air program, I like to offer the following:

- A. The isopleth map attached to the Construction Permits (Figure #7) is no longer applicable since #1 and #2 plants have been shut down and #4 plant has been brought up to design rate and accepted from the contractor. This means we have moved from Phase III to Phase IV - the operation of #3 and #4 plants only.
- B. Figures #8 and #10 represent the predicted isopleths of our operation during Phase IV and Phase V - after #5 and #6 plants come into operation.

After studying the enclosed maps, John suggested the following plan which is least costly but still gives you the information you want:

- 1. The operation (for 1 year) of the four indicated monitoring locations - three already existing and one new station on Grace property;
- 2. Note that in Phase IV (Figure #8) one station is within the predicted 15 and 20 isopleths and three cover the wider area between the 10 and 15 isopleths;
- 3. Note that in Phase V the new station will also fall between the predicted 15 and 20 isopleths;

Mr. Joseph Tessitore

-2-

August 20, 1976

4. John Koogler will personally supervise the operation of all four stations on the same schedule as the indicated two D.E.R. stations; this will increase the data available.
5. John will summarize and submit to Grace the data obtained each month; if you would like, John can include the information from the two D.E.R. stations. We will, in turn, forward the report to you.

After you have had a chance to study this proposal, I would like to get together with you and John Koogler to discuss the details. Since #5 and #6 plants are scheduled to be in operation by the first of next year, we should try to resolve this program as soon as possible so we can get as much data as possible before that time.

Sincerely,

W. R. GRACE & CO.
Agricultural Chemicals Group



M. J. Altenburger
Superintendent
Air & Water Quality Control

MJA:db

Enclosure

cc: J. R. Terry	(WO/Encl.)
M. P. McArthur	(WO/Encl.)
C. Peters	(W/Encl.)
John Koogler	(W/Encl.)

Table 1. Summary of the Proposed Three-Phase Expansion at W. R. Grace
Phosphate Fertilizer Complex Near Bartow, Florida.

Phase	Time Period	Plants in Operation	Operating Capacity (Tons H ₂ SO ₄ /Day)	Emission Rate (Lbs SO ₂ /Ton H ₂ SO ₄)
III	7-1-75	1	700	36
	to	2	700	36
	4-1-77 8-1-76	3	1000	10
IV	8-1-76 4-1-77	3	1000	10
	to	4	1600	4
	7-1-78 2-1-77			
V	7-1-78	3	1000	10
	2-1-77	4	1600	4
		5	1600	4
		6	1600	4

NOTE: Plants 4, 5 and 6 are proposed new plants.

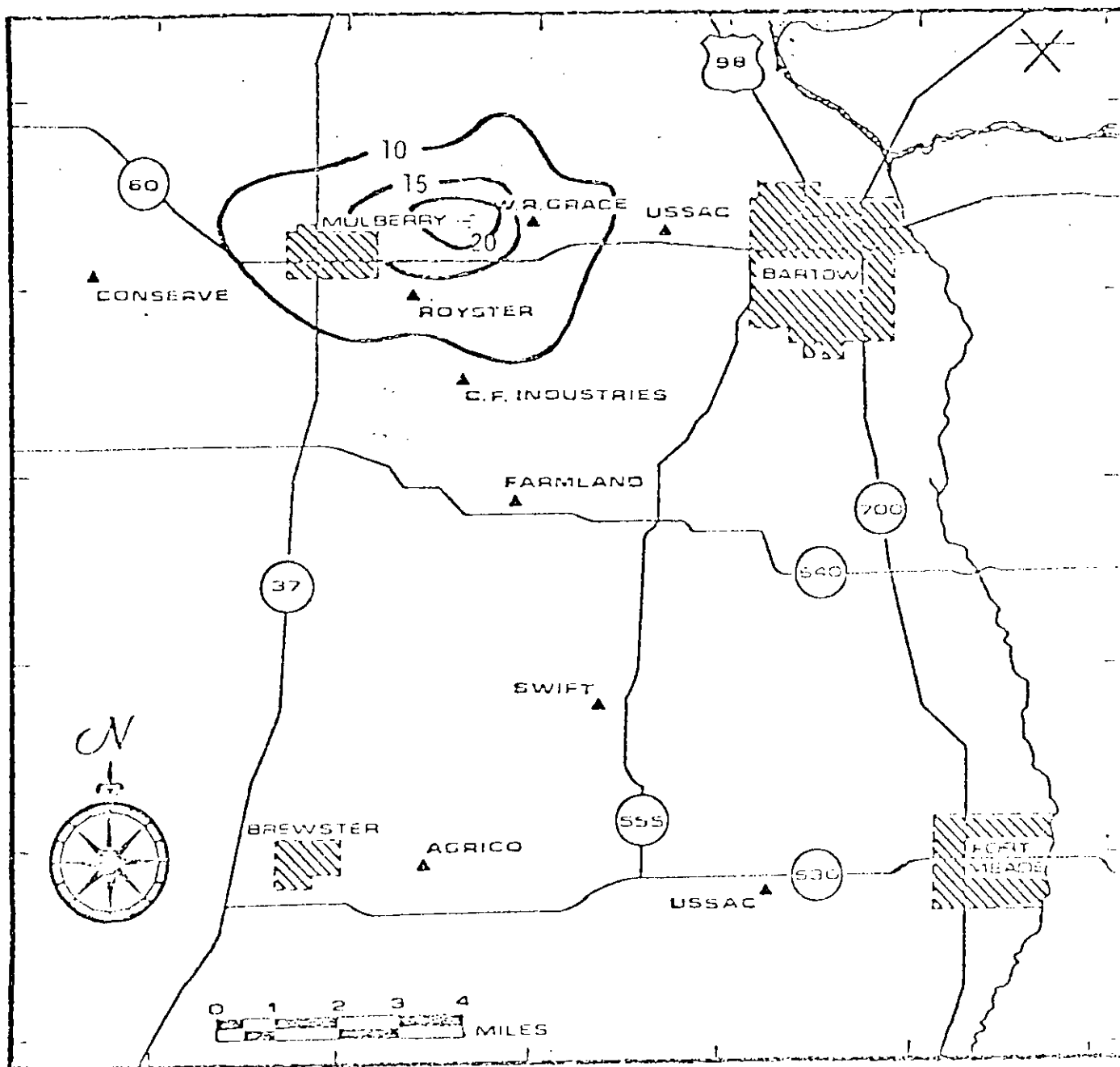


Figure 7. Isopleths of the Contribution to the Annual Average Ground-Level Sulfur Dioxide Concentrations from the W. R. Grace Site Under Emission Conditions Proposed During Phase III of the Grace Expansion Plan ($\mu\text{g}/\text{m}^3$).

#1 - #2 & #3

- ⊕ NEW STATIONS
- ⊕ EXISTING IND. MON. STATIONS
- D.E.R. STATIONS

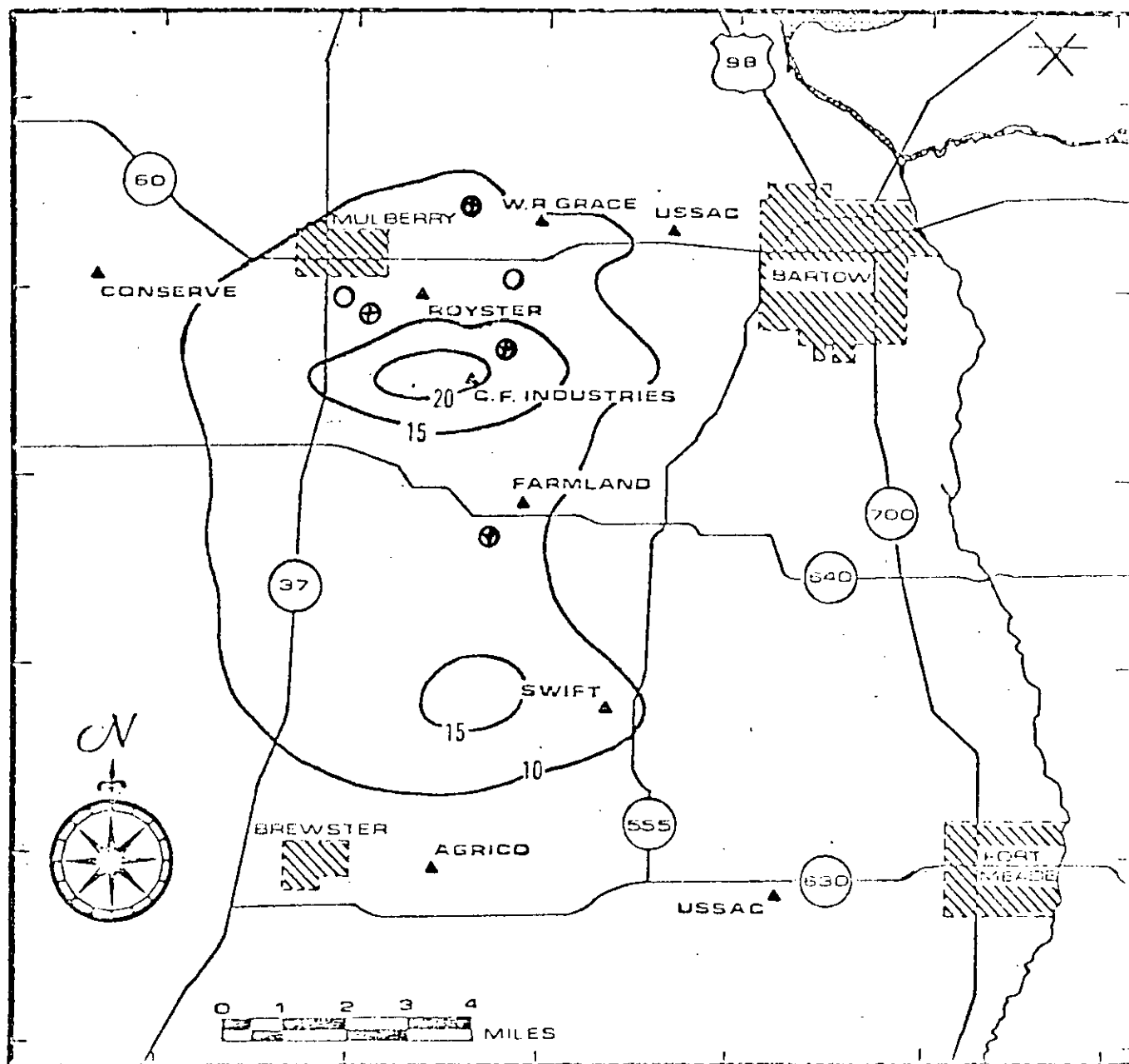


Figure 8. Isopleths of Predicted Annual Average Ground-Level Sulfur Dioxide Concentrations Near the W. R. Grace Plant Site, Polk County, Florida, Under Emission Conditions Proposed During Phase IV of the Grace Expansion Plan. ($\mu\text{g}/\text{m}^3$)

#34 #4

- ⊕ NEW STATION
- ⊕ EXISTING INDUSTRY STATION
- D.E.R. STATIONS

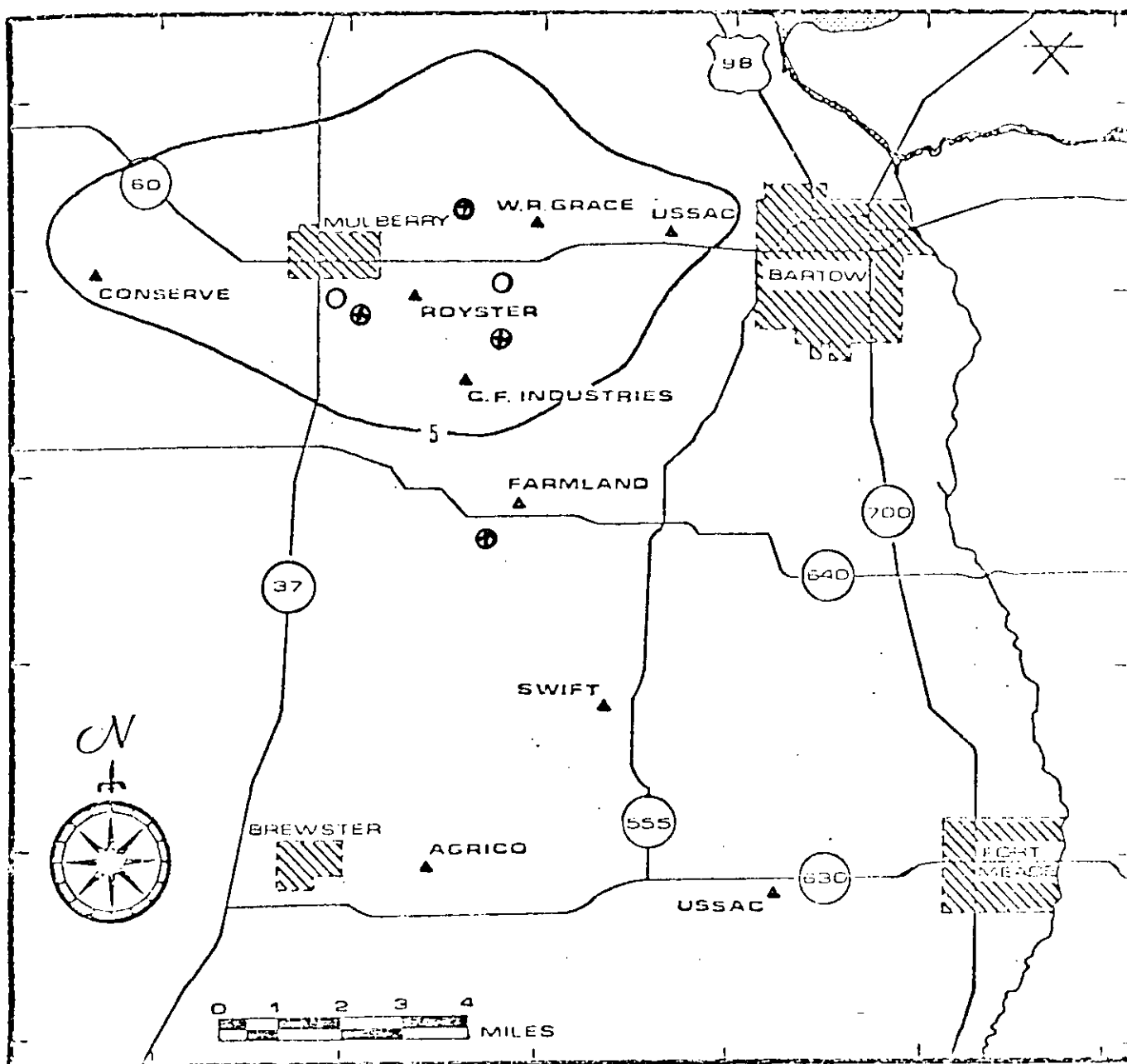


Figure 9. Isopleths of the Contribution to the Annual Average Ground-Level Sulfur Dioxide Concentrations from the W. R. Grace Site, Under Emission Conditions Proposed During Phase IV of the Grace Expansion Plan. ($\mu\text{g}/\text{m}^3$)

#3 #4

- ⊕ NEW PLANT
- ⊕ EXISTING PLANT
- POLLUTION STATION

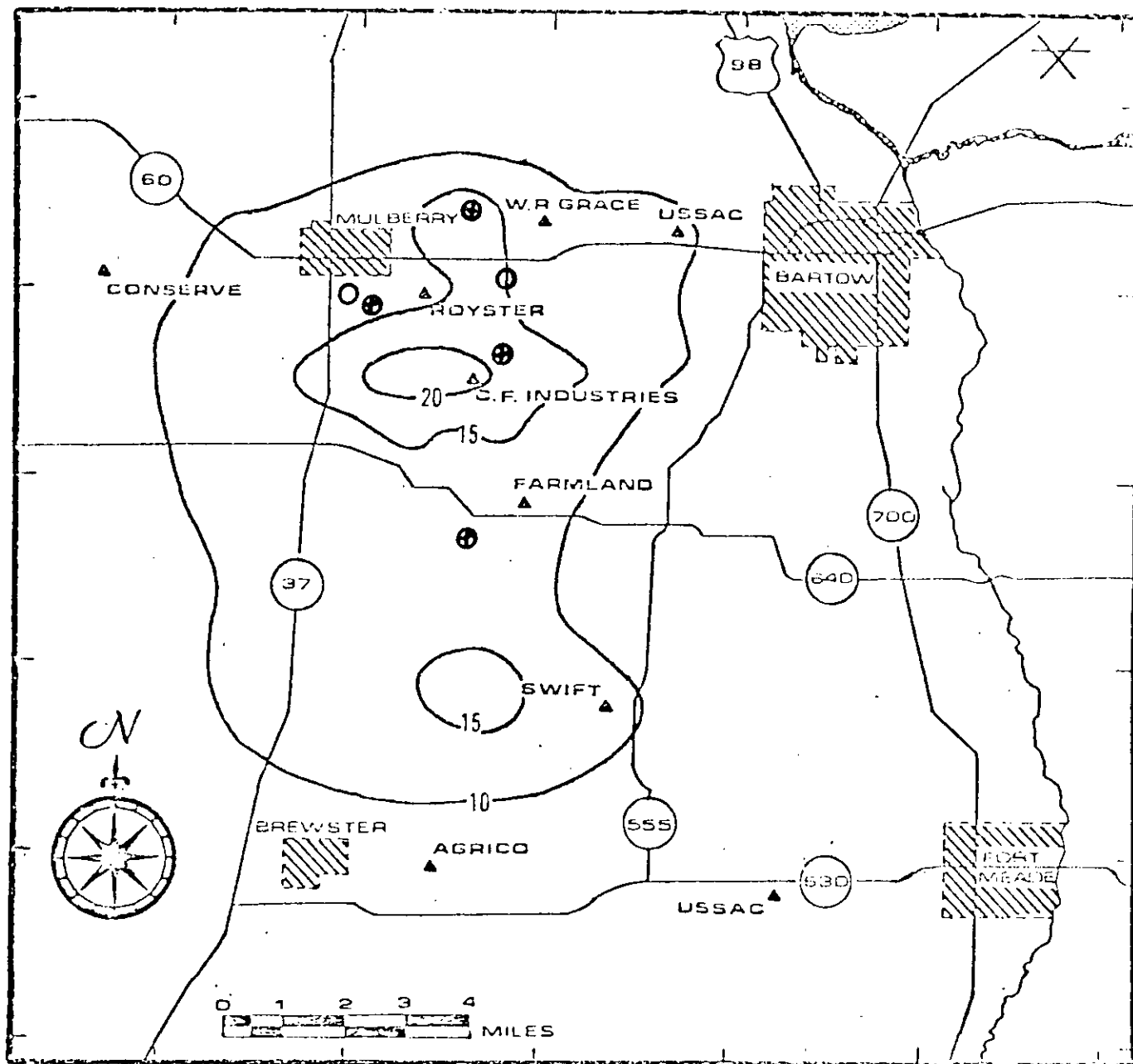


Figure 10. Isopleths of Predicted Annual Average Ground-level Sulfur Dioxide Concentrations Near the W. R. Grace Plant Site, Polk County, Florida, Under Emission Conditions Proposed During Phase V of the Grace Expansion Plan. ($\mu\text{g}/\text{m}^3$)

#3-#4-#5 & #6

- ① NEW STATION
- ② EXISTING INDUSTRIAL STATION
- D.E.R. STATIONS

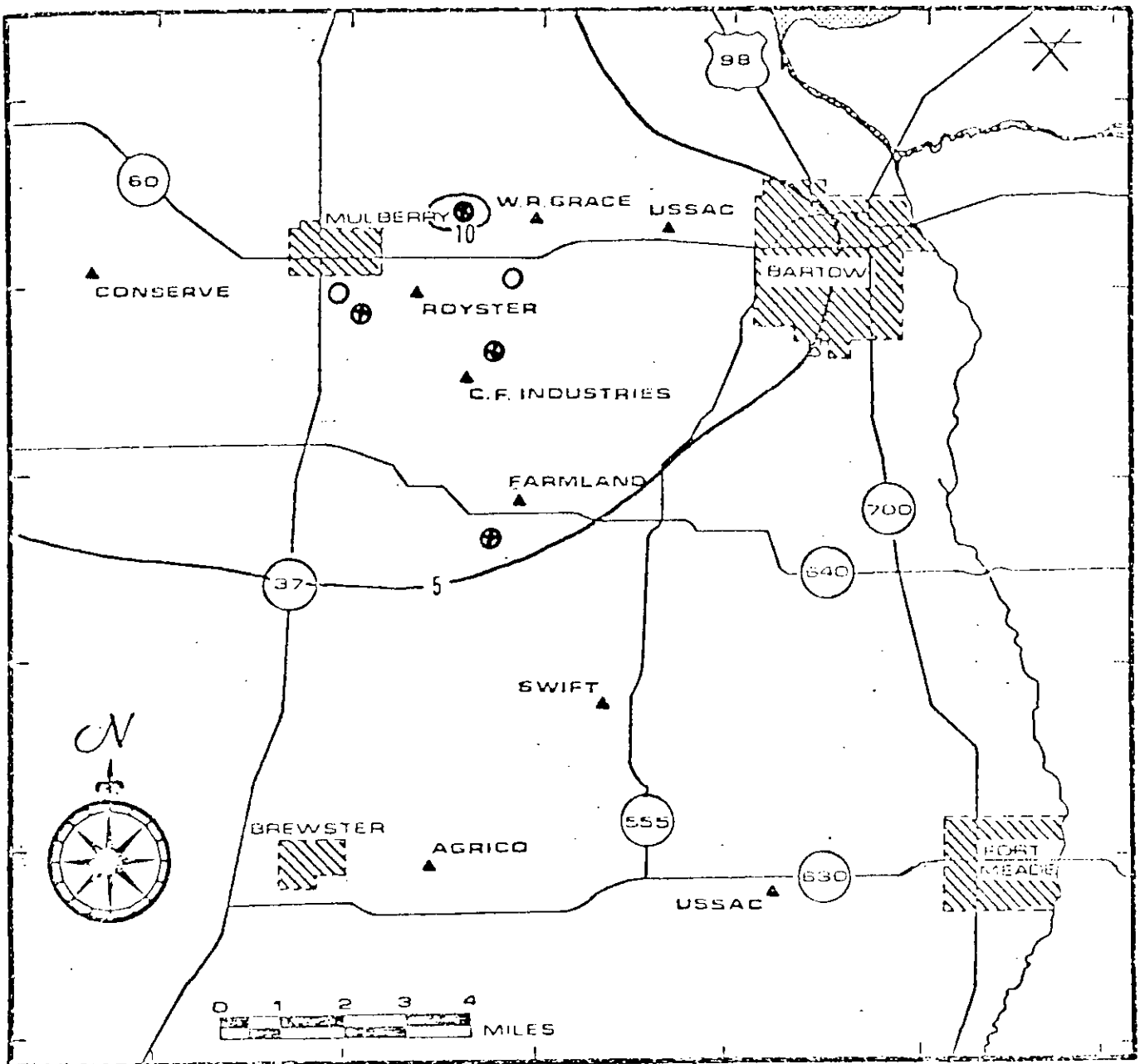


Figure 11. Isopleths of the Contribution to the Annual Average Ground-Level Sulfur Dioxide Concentrations from the W. R. Grace Site Under Emission Conditions Proposed During Phase V of the Grace Expansion Plan. ($\mu\text{g}/\text{m}^3$)

#3 - #4 - #5 + #6

W. R. GRACE & CO.

AGRICULTURAL CHEMICALS GROUP



November 3, 1976

Mr. Joseph Tessitore
Department of Environmental Regulation
500 East Central Avenue - Suite 238
Winter Haven, Florida 33880

RE: AMBIENT AIR MONITORING SO₂ PROGRAM FOR COMPLIANCE WITH
PROVISOS OF CONSTRUCTION PERMITS FOR #5 AND #6 SULFURIC
ACID PLANTS.

Dear Joe:

This letter is to confirm our agreement reached during this morning's meeting with John Koogler in your office.

It was agreed that:

1. W. R. Grace would activate, as soon as possible, the four station network described and located in the letter and enclosures mailed to you August 20, 1976;
2. This network will be under the supervision of Sholtes & Koogler; John Koogler will give you the exact sampling locations, sampling method, analytical methods, etc. to document the entire procedure;
3. This monitoring program will continue for a period of one year in an effort to identify the effects of activating #5 and #6 plants in addition to the existing #3 and #4 plants;
4. This program will be terminated after one year if #5 and #6 plants are demonstrated to be meeting the emission regulations of the Department of Environmental Regulation;

Mr. Joseph Tessitore

-2-

November 3, 1976

Thank you for your co-operation in this matter.

Sincerely,

W. R. GRACE & CO.
Agricultural Chemicals Group



M. J. Altenburger
Superintendent
Air & Water Quality Control

MJA:db

cc: J. R. Terry
M. P. McArthur
C. F. Peters
John Koogler ✓



SHOLTES & KOOGLER, ENVIRONMENTAL CONSULTANTS

1213 N.W. 6th Street Gainesville, Florida 32601 (904) 377-5822

November 8, 1976

Mr. Joe Tessitore
Florida Department of
Environmental Regulation
Post Office Box 9205
Winter Haven, Florida 33880

Dear Joe:

This letter will confirm the information that Mike Altenburger and I discussed with you during our meeting in your office on November 3, 1976 regarding the W. R. Grace ambient sulfur dioxide monitoring program.

As we discussed, we will place four monitors at the locations shown on the attached map. The UTM coordinates of these stations are:

<u>Station</u>	<u>UTM North (km)</u>	<u>UTM East (km)</u>
1	3086.5	407.5
2	3084.7	404.5
3	3084.0	408.8
4	3079.6	407.2

These sites were selected because they are in locations which will reasonably represent existing air quality and will monitor the impact of W. R. Grace sulfuric acid plants 5 and 6 when these plants are put on-line.

The frequency of sampling at each of the four sites will be once every sixth day using the monitoring schedule adopted by the FDER and EPA. Each of the samplers will run for a 24-hour period from midnight through midnight on the scheduled sampling date.

The monitoring method used at each of these four stations will be the 24-hour reference method for the determination of ambient concentrations of SO₂ as developed by EPA and published in the Federal Register of April 30, 1971. Recommendations published in the document, "The Effect of Temperature on Stability of Sulfur Dioxide Samples Collected by the Federal Reference Method," (EPA report no. EPA-600/4-76-024 by R. G. Fuerst, F. P. Scaringelli and J. H. Margeson, EPA Office of Research and Development, Research Triangle Park, North Carolina, May 1976) will be incorporated into the

Mr. Joe Tessitore

-2-

November 8, 1976


monitoring program to overcome the temperature instability of the monitoring method. The recommendations adopted include the refrigeration of the bubbler sampler and the refrigeration of the exposed samples during storage and shipping prior to analysis.

If you have any questions regarding the sampler locations or the field and analytical procedures, please feel free to contact me.

One matter which we did not discuss, but which deserves some clarification, is the method of reporting the monitoring data. I would appreciate it if you will let me know the frequency of reporting which you desire and whether or not you would like SAROAD numbers assigned to each of the monitoring sites and the data reported on SAROAD forms. I will look for your response on this matter.

Very truly yours,

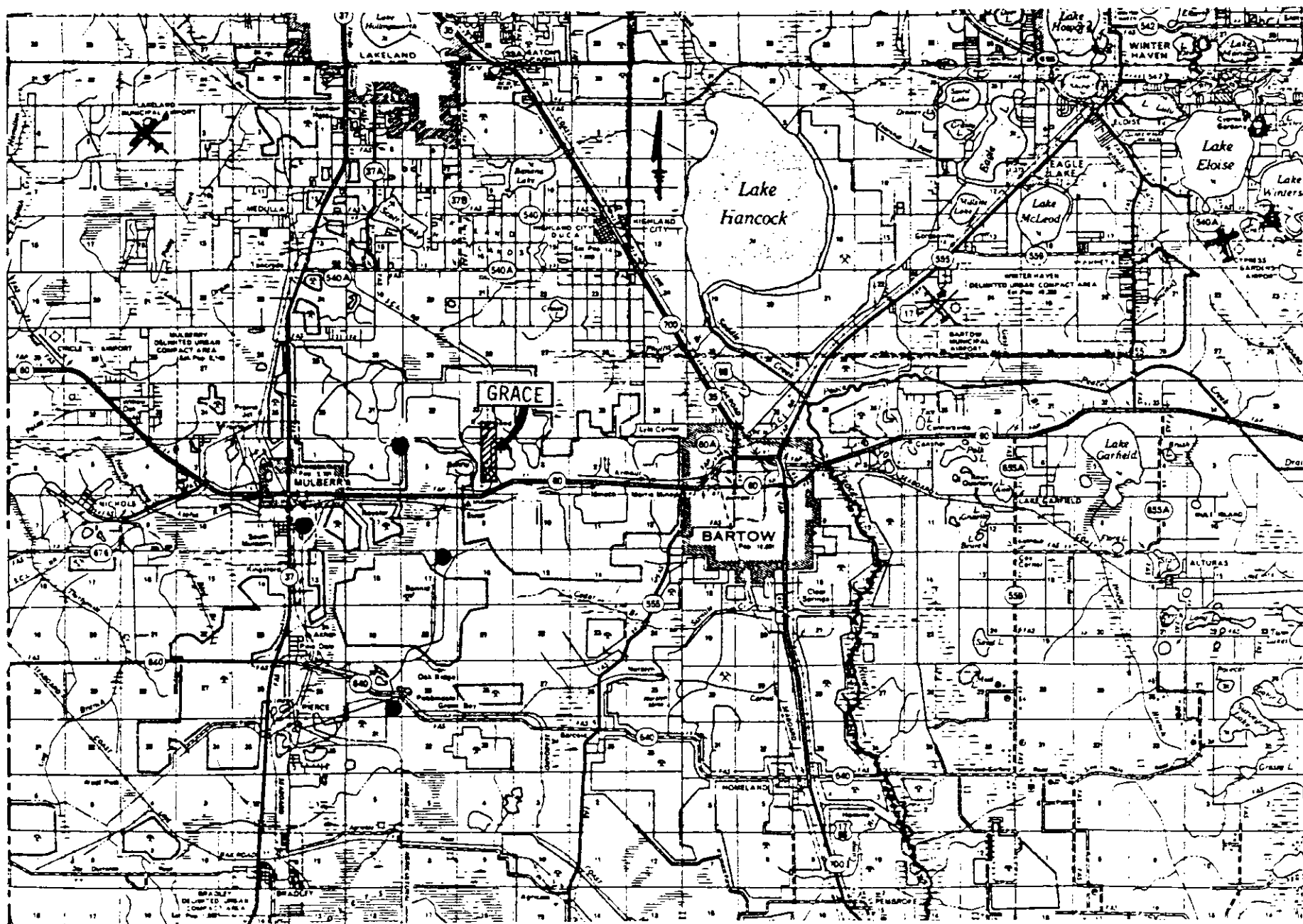
SHOLTES & KOOGLER
ENVIRONMENTAL CONSULTANTS



John B. Koogler, Ph.D., P.E.

JBK/jvp

cc: Mike Altenburger





REUBIN O.D. ASKEW
GOVERNOR

STATE OF FLORIDA
DEPARTMENT OF ENVIRONMENTAL REGULATION

CENTRAL SUBDISTRICT
500 EAST CENTRAL AVENUE
P.O. BOX 9205
WINTER HAVEN, FLORIDA 33880

JOSEPH W. LANDERS, JR.
SECRETARY

December 20, 1976

M. J. Altenburger, Superintendent
Air & Water Quality Control
W. R. Grace & Company
Agricultural Chemicals Group
Post Office Box 471
Bartow, Florida 33830

Re: Ambient Air Monitoring SO₂ Program for Compliance with
Provisions of Construction Permits AC53-2655 & AC53-2656
Sulfuric Acid Plants # 5 & 6

Dear Mr. Altenburger:

The ambient SO₂ monitoring plan for the subject sulfuric acid plants which was submitted on 11-30-76 and further detailed in Sholtes & Koogler letter dated 11-8-76 was reviewed and found acceptable in meeting the provisions for the subject construction permits. Although the construction permit provisos require continuous automatic monitoring of ambient SO₂, the subject program will be acceptable if the temperature instability of SO₂ is eliminated by refrigeration of the bubbler sampler and refrigeration of the samples during storage and shipping prior to analysis.

If you have any questions, do not hesitate to call on me.

Sincerely,

John C. Barnett, P.E.
Branch Office Manager

JCB:JLT:bat

cc: John Koogler ✓

Subject: Saroad Site Codes

From: William J. Snider
Air Operations Branch
Region IV Saroad Contact

To: State: *Florida*

The attached Saroad Identification Forms have been updated at NADS. The assigned Saroad code is as indicated in pencil at the top right hand corner (Car 1 A).

If further information is needed, please contact me at 404/526-2864.

Date:

TO: SAROAD Contact:
(Air Quality Chief)

FL

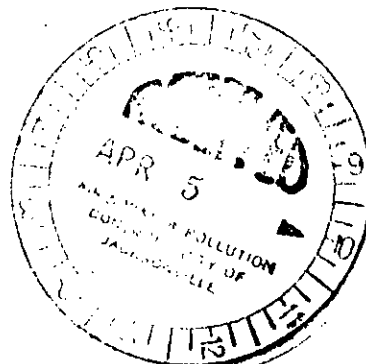
Enclosed you will find the following air quality (SAROAD) report(s):

Report	# copies	period
<input type="checkbox"/> NA 219 (1 hour data)	_____	_____
<input type="checkbox"/> NA 209 (24 hour data)	_____	_____
<input type="checkbox"/> NA 211 (Year Frequency)	_____	_____
<input type="checkbox"/> NA 212 (Year by Quarters)	_____	_____
<input type="checkbox"/> NA 240 (Quarterly Frequency with 2nd max)	_____	_____
<input type="checkbox"/> NA 202S (Inventory by Site)	_____	_____
<input type="checkbox"/> NA 202P (Inventory by Pollutant)	_____	_____
<input type="checkbox"/> NA 217Y (Yearly Frequency - No Site Desc.)	_____	_____
<input type="checkbox"/> NA 217Q (Quarterly Frequency - No Site Desc.)	_____	_____
<input type="checkbox"/> (Particulate Trends - # Violations)	_____	_____
<input type="checkbox"/> (SO ₂ Trends - # Violations)	_____	_____
<input type="checkbox"/> (O _x Trends - # Violations)	_____	_____
<input type="checkbox"/> (NO _x Trends)	_____	_____
<input type="checkbox"/> (CO Trends - # Violations)	_____	_____
<input type="checkbox"/> NA 252 (Active Sites Reporting)	_____	_____
<input type="checkbox"/> SAROAD Editor Parameter File	_____	_____
<input type="checkbox"/> NA 026 (Pre-edit or Receipt)	_____	_____
<input type="checkbox"/> NA 027 (Edit)	_____	_____
<input type="checkbox"/> Duplicate Data Edit	_____	_____
<input type="checkbox"/> Table #1 (Monitoring Sites not Reporting during quarter)	_____	_____
<input type="checkbox"/> SAROAD-PL (Sorted raw data listing prior to edit)	_____	_____

Other:

SAROAD SITE
CODES

Comments:



If you have any questions please contact Barry Gilbert or myself at 404/526-2864.

ENVIRONMENTAL PROTECTION AGENCY

National Aerometric Data Bank

Research Triangle Park, N. C. 27711

REVISIONS:

() CHANGE

() ADD

() DELETE

SAROAD Site Identification Form

LE 70076

Form Completed By SHOLTES & KOOGLER
JB KOOGLER 904/377-5822 Date 3/15/77New ☒

TO BE COMPLETED BY THE REPORTING AGENCY

DO NOT WRITE HERE

A FLORIDA

State

SOURCE ORIENTED

Project

POLK COUNTY FL

City Name (23 characters)

POLK COUNTY FL

County Name (15 characters)

City Population (right justified)

						0	0	0
52	53	54	55	56	57	58	59	

Longitude

Deg. Min. Sec.

0								
62	63	64	65	66	67	68	69	

Easting Coord., meters

Latitude

Deg. Min. Sec.

N								
70	71	72	73	74	75	76		

Northing Coord., meters

TM Zone

7								
62	63	64	65	66	67	68	69	

3	0	8	7	4	0	0		
70	71	72	73	74	75	76		

State

Area

Site

A	1	0	3	6	8	0	0	0	5
1	2	3	4	5	6	7	8	9	10

Agency

Project

J
11

0	2
12	13

Region

Action

4
77

0
78

7
79

1
80

B SHOLTES & KOOGLER ENV. CONSULTANTS

Supporting Agency (61 characters)

1213 NW 6TH ST

Supporting Agency, continued

GAINESVILLE FL

State

Area

Site

B									
1	2	3	4	5	6	7	8	9	10

Agency Project

SMSA

Action

11

12	13

0	0	0	0
14	15	16	17

1
80

State

Area

Site

C									
1	2	3	4	5	6	7	8	9	10

Agency

Project

Action

11

12	13

80

State

Area

Site

D									
1	2	3	4	5	6	7	8	9	10

Agency

Project

Action

11

12	13

80

State

Area

Site

E									
1	2	3	4	5	6	7	8	9	10

Agency

Project

Action

11

12	13

80

C) WR GRACE NETWORK STA 1

Optional: Comments that will help identify

BARTOW FL

the sampling site (132 characters)

SO2 MONITOR PER 40CFR50; TEMP

CONTROLLED

1.8 KM NORTH SR 60

Abbreviated Site Address (25 characters)

NAB No. 158-R0012

Approval expires 6/30/76

(over)

ENVIRONMENTAL PROTECTION AGENCY

National Aerometric Data Bank

Research Triangle Park, N. C. 27711

SAROAD Site Identification Form

SHULTES & KOOGLER

Form Completed By JP KOOGLER 904/377-5822 Date 3/15/77

New



() DELETE

DEVIATIONS:

() CHANGE

() ADD

TO BE COMPLETED BY THE REPORTING AGENCY

DO NOT WRITE HERE

FLORIDA

State

SOURCE ORIENTED

Project

MULBERRY FL

City Name (23 characters)

POLK COUNTY

County Name (15 characters)

City Population (right justified)

				3	1	5	0
52	53	54	55	56	57	58	59

Longitude

Deg. Min. Sec.

Latitude

Deg. Min. Sec.

0

W

N

Easting Coord., meters

Northing Coord., meters

7

		4	0	4	5	0	0
62	63	64	65	66	67	68	69

3	0	8	4	7	0	0
70	71	72	73	74	75	76

SHULTES & KOOGLER ENV CONSULTANTS

Supporting Agency (61 characters)

1213 NW 6TH ST

Supporting Agency, continued

GAINESVILLE FL

WR GRACE NETWORK STA 2

Optional: Comments that will help identify

BARTOW FL

the sampling site (132 characters)

SO2 MONITOR PER 40CFR50; TEMP

CONTROLLED

KINGSFORD RD MULBERRY FL

Abbreviated Site Address (25 characters)

WB No. 158-R0012

Valid expires 6/30/76

(over)

State

Area

Site

A	1	0	3	6	8	0	0	0	6
1	2	3	4	5	6	7	8	9	10

Agency

Project

J

02

Region

Action

4

07

1

State

Area

Site

B									
1	2	3	4	5	6	7	8	9	10

Agency Project

SMSA

Action

0000

1

State

Area

Site

C									
1	2	3	4	5	6	7	8	9	10

Agency

Project

Action

State

Area

Site

D									
1	2	3	4	5	6	7	8	9	10

Agency

Project

Action

State

Area

Site

E									
1	2	3	4	5	6	7	8	9	10

Agency

Project

Action

ENVIRONMENTAL PROTECTION AGENCY
National Aerometric Data Bank
Research Triangle Park, N. C. 27711

SAROAD Site Identification Form

REVISIONS:

() CHANGE

() ADD

() DELETE

7076

Completed By SHOLTES & KOOGLER JBK DOGLER 904/377-5822 Date 3/15/77 New ☒

TO BE COMPLETED BY THE REPORTING AGENCY

DO NOT WRITE HERE

FLORIDA

State

SOURCE ORIENTED

Project

POLK COUNTY FL

City Name (23 characters)

POLK COUNTY FL

County Name (15 characters)

City Population (right justified)

000

Longitude

Deg. Min. Sec.

W 62 63 64 65 66 67 68 69

Easting Coord., meters

408800

Latitude

Deg. Min. Sec.

N 70 71 72 73 74 75 76

Northing Coord., meters

3084000

State Area Site
A 1 0 3 6 3 0 0 0 7

Agency

J

Project

02

Region

4

Action

1

SHOLTES & KOOGLER ENV CONSULTANTS

Supporting Agency (61 characters)

1213 NW 6TH ST

Supporting Agency, continued

GAINESVILLE FL

WR GRACE NETWORK STA 3

Optional: Comments that will help identify

BARTOW FL

the sampling site (132 characters)

SO2 MONITOR PER 40CFR 50 ; TEMP.

CONTROLLED

BONNIE MINE RD

Abbreviated Site Address (25 characters)

State Area Site
B 1 2 3 4 5 6 7 8 9 10

Agency Project

11

12 13

SMSA

0000

Action

1

State Area Site
C 1 2 3 4 5 6 7 8 9 10

Agency Project

11

12 13

Action

80

State Area Site
D 1 2 3 4 5 6 7 8 9 10

Agency Project

11

12 13

Action

80

State Area Site
E 1 2 3 4 5 6 7 8 9 10

Agency Project

11

12 13

Action

80

Mo. No. 158-R0012

approval expires 6/30/76

(over)

ENVIRONMENTAL PROTECTION AGENCY

National Aerometric Data Bank

Research Triangle Park, N. C. 27711

REVISIONS:

() CHANGE

() ADD

() DELETE

70076

SAROAD Site Identification Form

Form Completed By SHOLTES & KOOGLER
SR KOOGLER 904/377-5822 Date 3/15/77 New ☒

TO BE COMPLETED BY THE REPORTING AGENCY

DO NOT WRITE HERE

FLORIDA

State

SOURCE ORIENTED

Project

POLK COUNTY FL

City Name (23 characters)

POLK COUNTY FL

County Name (15 characters)

City Population (right justified)

52	53	54	55	56	57	58	59	0	0

Longitude

Deg. Min. Sec.

Latitude

Deg. Min. Sec.

0

W

N

Zone

Easting Coord., meters

Northing Coord., meters

7

62	63	64	65	66	67	68	69	4	0

70	71	72	73	74	75	76	3	0	7

SHOLTES & KOOGLER ENV CONSULTANTS

Supporting Agency (61 characters)

1213 NW 6TH ST

Supporting Agency, continued

GAINESVILLE FL

WR GRACE NETWORK STA 4

Optional: Comments that will help identify

BARTOW FL

the sampling site (132 characters)

SO2 MONITOR PER 40CFR50; TEMP

CONTROLLED

0.2 KM SOUTH SR 640

Abbreviated Site Address (25 characters)

MB No. 158-R0012

Approval expires 6/30/76

State			Area			Site			
A	1	0	3	6	8	0	0	0	8
1	2	3	4	5	6	7	8	9	10

Agency

Project

J

02

Region

Action

4

07

1

77

80

State			Area			Site			
B									
1	2	3	4	5	6	7	8	9	10

Agency Project

SMSA

Action

0000

11

12 13

14 15 16 17

80

State			Area			Site			
C									
1	2	3	4	5	6	7	8	9	10

Agency Project

Action

11

12 13

80

State			Area			Site			
D									
1	2	3	4	5	6	7	8	9	10

Agency Project

Action

11

12 13

80

State			Area			Site			
E									
1	2	3	4	5	6	7	8	9	10

Agency Project

Action

11

12 13

80

(over)

ENVIRONMENTAL PROTECTION AGENCY

NATIONAL AEROMETRIC DATA BANK

RESEARCH TRIANGLE PARK, N.C. 27711

SAROAD MULTIPLE-STATION DATA FORM.

OMB No 158 R0012
Approval Expires 6/30/71

<div style="border: 1px solid black; padding: 2px; display: inline-block;">3</div> <div style="border-bottom: 1px solid black; width: 100%;"></div>	<div style="border-bottom: 1px solid black; width: 100%;"></div> <div style="text-align: center; font-weight: bold;">AGENCY</div>	<div style="border-bottom: 1px solid black; width: 100%;"></div> <div style="text-align: center; font-weight: bold;">STATE</div>	<div style="border-bottom: 1px solid black; width: 100%;"></div> <div style="text-align: center; font-weight: bold;">AGENCY</div>	<div style="border-bottom: 1px solid black; width: 100%;"></div> <div style="text-align: center; font-weight: bold;">PROJECT</div>	<div style="border-bottom: 1px solid black; width: 100%;"></div> <div style="text-align: center; font-weight: bold;">TIME</div>								
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SITE ADDRESS C					SITE ADDRESS D								
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SITE ADDRESS G					SITE ADDRESS H								
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SITE ADDRESS K					SITE ADDRESS L								
<div style="text-align: center; font-weight: bold;">DAY</div> <div style="display: flex; justify-content: space-around;"> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> </div> <div style="display: flex; justify-content: space-around; font-size: 8px;"> 22 23 </div>		<div style="text-align: center; font-weight: bold;">START HOUR</div> <div style="display: flex; justify-content: space-around;"> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> </div>		<div style="text-align: center; font-weight: bold;">AREA</div> <div style="display: flex; justify-content: space-around;"> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> </div>		<div style="text-align: center; font-weight: bold;">SITE</div> <div style="display: flex; justify-content: space-around;"> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> </div>		<div style="text-align: center; font-weight: bold;">VALUE</div> <div style="display: flex; justify-content: space-around;"> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> <div style="border: 1px solid black; width: 20px; height: 20px;"></div> </div>					
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SHOLTES & KOOGLER, ENVIRONMENTAL CONSULTANTS
1213 N.W. 6th Street Gainesville, Florida 32601 (904) 377-5822

August 22, 1977

Mr. Robert Stephens
Florida Department of
Environmental Regulation
500 E. Central Avenue
Suite 238
Winter Haven, FL 33880

Re: W.R. Grace Company--Operating Permits for No. 5 and 6
Sulfuric Acid Plants

Dear Bob:

I would like to confirm the meetings we had on Monday, August 15, 1977 in the Bartow area to review the W.R. Grace monitoring network. The visit included a review of the four monitoring sites and an inspection of the interior of the samplers at stations 2, 3 and 4. The No. 1 sampler was not inspected due to the fact that I did not have a key for this sampler. In addition to inspecting the sites, I outlined the procedures followed by our field technician, Mr. George Vallejo, and I gave you a copy of the checklist George follows each time he services one of the samplers. You also had an opportunity to meet George when he met us at the No. 2 sampling site.

In discussing our program with you following the tour you informed me that you had no objection to the sampling procedures that we employed. You did ask however that in the future we correct calculated sulfur dioxide concentrations to standard temperature, pressure and dry conditions. I confirmed that we would do this and further volunteered that we would go back and calculate a representative number of previously recorded sulfur dioxide data to standard temperature, pressure and dry conditions.

I would also like to report to you that the pump on the No. 4 sampler has been replaced. This is the pump that did not operate during our tour.

Mr. Robert Stephens

August 22, 1977

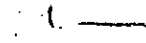
Page Two

I would also like to confirm that during our site visit I transmitted to you ambient sulfur dioxide data collected at the four monitoring sites through July 12, 1977. Included in the material transmitted to you was a tabular summary of all data collected at each of the four sampling sites and SAROAD forms with the ambient data appropriately entered.

I enjoyed our visit and look forward to seeing you again.

Very truly yours,

SHOLTES & KOGLER
ENVIRONMENTAL CONSULTANTS


John B. Koogler, Ph.D., P.E.

JBK:vw

cc: Mr. Mike Altenburger

Take With You

Old data sheet

New set of orifices and data sheet

TCM

4 shipping tubes with labels

Labels to include: W. R. Grace
Site No. (1, 2, 3 or 4)
Sample date

Wash bottle with distilled water

At Each Site

Check refrigerator temperature

Check timer time (make note if time not correct and set to correct time
and date)

Change timer trips to next sample date

Turn on pump and record final vacuum

While pump is running, check bubble rate in sample tube

Turn pump off

Put TCM in shipping tube

Rinse sample tube with distilled water

Put fresh TCM in sample tube

Change orifice

Turn pump on

Record initial vacuum

Check bubble rate

Turn pump off

Check sample line

Keep area around sampler clean

General

Store samples, TCM, and distilled water in refrigerator

Ship samples every third week to Jacksonville

Ship orifices and data sheets every third week to Gainesville

SAMPLING NETWORK W. R. Grace Pollutant SO₂
 Date Absorbant Prepared _____ Sampling Date 1/2/78 Sample Start Time 0001
 Date Absorbers Placed in Field _____ Sample Pick-up Date _____

Station No.	Orifice No.	Initial Vacuum (in. Hg)	Final Vacuum (in. Hg)	Sample Time (min)	Initial Calibration (L/M)	Final Calibration (L/M)	Average Flow Rate (L/M)
<u>1</u>	<u>S-48</u>	<u>25"</u>	<u>20"</u>	<u>1440</u>	<u>.194</u>	<u>.193</u>	<u>.1935</u>
<u>2</u>	<u>S-49</u>	<u>20"</u>	<u>20"</u>	<u>1440</u>	<u>.194</u>	<u>.191</u>	<u>.1925</u>
<u>3</u>	<u>S-50</u>	<u>20"</u>	<u>20"</u>	<u>1440</u>	<u>.200</u>	<u>.188</u>	<u>.189</u>
<u>4</u>	<u>S-51</u>	<u>20"</u>	<u>20"</u>	<u>1440</u>	<u>.194</u>	<u>.194</u>	<u>.194</u>

Wind Speed (mph) 0 - 3 _____
 4 - 8 _____
 > 8 _____

Relative Humidity (%) _____
 Average Temp. (°F) _____
 Average Pressure (mmHg) _____

REMARKS: _____

Initial calibration 8 Dec 77
Final Final " 25 Jan 78

Signature _____

FIELD DATA SHEET
 (1/2/78)

Final

SAMPLING NETWORK W. R. Grace Pollutant SO₂
Date Absorbant Prepared _____ Sampling Date 12/27/77 Sample Start Time 0001
Date Absorbers Placed in Field _____ Sample Pick-up Date _____

Station No.	Orifice No.	Initial Vacuum (in. Hg)	Final Vacuum (in. Hg)	Sample Time (min)	Initial Calibration (L/M)	Final Calibration (L/M)	Average Flow Rate (L/M)
<u>1</u>	<u>S-18</u>	<u>26"</u>	<u>20"</u>	<u>1440</u>	<u>.200</u>	<u>.197</u>	<u>.1985</u>
<u>2</u>	<u>S-44</u>	<u>20"</u>	<u>20"</u>	<u>1440</u>	<u>.202</u>	<u>.202</u>	<u>.202</u>
<u>3</u>	<u>S-45</u>	<u>20"</u>	<u>20"</u>	<u>1440</u>	<u>.204</u>	<u>.206</u>	<u>.205</u>
<u>4</u>	<u>S-46</u>	<u>20"</u>	<u>20"</u>	<u>1440</u>	<u>.203</u>	<u>.202</u>	<u>.2025</u>

Wind Speed (mph) 0 - 3 _____ Relative Humidity (%) _____
4 - 8 _____ Average Temp. (°F) _____
> 8 _____ Average Pressure (mmHg) _____

REMARKS: _____

Initial Calibration 8 Dec - 77 Newman

Final Calibration 25 Jan - 78

Signature W. R. Grace

FIELD DATA SHEET
(12/27/77)

AMBIENT SAMPLING DATA SHEET

Project Name Sholtest Koor lens
Project No. 77-088-001
Month/Year 78

Sample Type SO₂
Sample Duration 24 hour
Field Sampler SK-EC

SAMPLE NO.	DATE	STN & RUN #s	% T	Total ug/sample	FLOW RATE	ug/m ³	Comments
250	12-27-77	WR Grace 1	92.5	0.6		2.1	
251	12-27-77	" 2	91.9	0.8		2.2	
252	12-27-77	" 3	91.4	1.0		3.4	
253	12-27-77	" 4	91.9	0.8		2.8	
254	1-2-78	" 1	92.5	0.6		2.2	
255	1-2-78	" 2	92.5	0.6		2.2	
256	1-2-78	" 3	92.1	0.7		2.6	
257	1-12-78	" 4	92.0	0.8		2.9	
258	1-8-78	" 1	92.4	0.6		2.1	
259	1-8-78	" 2	92.1	0.7		2.6	
260	1-8-78	" 3	92.1	0.7		2.4	
261	1-8-78	" 4	92.1	0.7		2.5	
262	1-14-78	" 2	93.6	0.3			
263	" " "	" 3	93.8	0.2			
264	" " "	" 4	94.0	0.2			
265	" " "	" 5	92.5	11.9			
266	" " "	" 6	84.9	3.1			
267	" " "	" 7	87.0	2.4			
268	" " "	" 8	34.0	29.3			
269	" " "	" 9	88.9	1.8			
270	" " "	" Blank	94.5	0.0			
271	" " "	" 11	93.3	0.4			
272	" " "	" 12	92.6	0.6			
273	1-14-78	" 13	90.5	1.3			
274	1-20-78	" 2	94.3	0.1			
275	"	" 3	61.5	12.4			
276	"	" 4	62.0	12.1			
277	"	" 5	77.4	5.8			
278	"	" 6	58.9	13.6			
279	"	" 7	60.0	13.1			
280	"	" 8	38.6	25.7			
281	"	" 9	66.5	10.1			
282	"	" Blank	94.6	0.0			
283	"	" 11	78.9	5.2			
284	"	" 12	63.8	11.3			
285	1-20-78	" 13	77.0	5.9			

Sample temperature when received at Laboratory