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REGIONAL AIR POLLUTION STUDY

Sulfur Compounds and Particulate Size Distribution Inventory

by

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ABSTRACT

In conjunction with the Regional Air Pollution Study being conducted in the St. Louis Air Quality Control Region (AQCR), a methodology for estimating the amount of sulfur trioxide (SO_3) emitted by combustion sources was developed. It is based on SO_2/SO_3 ratios determined both experimentally and from literature surveys. The most likely value appears to be 1.85% of the SO_2 emissions. On this basis, about 22,000 tons of SO_3 are emitted yearly from combustion sources.

A fine particle size inventory for the area was also developed. The inventory gives a breakdown of particulate emissions in the range of 7 to .01 microns, based on production rates and collection efficiencies for point sources in the St. Louis AQCR. The information on the SO_2/SO_3 ratios and the particle size breakdown is stored in the RAPS Data Handling System.

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A methodology for estimating the amount of sulfur trioxide (SO_3) emitted by combustion sources in the St. Louis AQCR was developed. It is based on SO_2/SO_3 ratios determined both experimentally and from literature surveys. The most likely value appears to be 1.85% of the SO_2 emissions. On this basis, about 22,000 tons of SO_3 are emitted yearly from combustion sources.

An alternative method for SO_3 determination was evaluated and field tested. The "Shell" method, developed originally by Goksøyr and Ross, appears to give reliable results both in the laboratory and in the field.

A fine particle size inventory for the area was developed, based on earlier work by MRI. The inventory gives a breakdown of particulate emissions in the range of 7 to .01 microns, based on production rates and collection efficiencies for point sources in the St. Louis AQCR. The information can be stored in the RAPS Data Handling System.

Experimental data were obtained on particle size distribution of representative sources using an Andersen cascade impactor. The results indicated a bimodal distribution peaking at around 5 microns and at less than 7 microns.

2.0 INTRODUCTION

Within the framework of the Regional Air Pollution Study (RAPS) at St. Louis, MO., a high-resolution emission inventory has been assembled. Initially, this inventory was focused on one pollutant - sulfur dioxide - for which hourly, measured emission data were collected. This inventory was broadened to include all "criteria" pollutants. In addition, special inventories were also developed for trace pollutants, heat emissions and hydrocarbons.

This study is concerned with two classes of pollutants: sulfur compounds - primarily SO_3 (sulfur trioxide) since a detailed SO_2 (sulfur dioxide) inventory exists, and a particle size inventory, a refinement of the particle inventory available as part of the "criteria" pollutant inventory, which does not take particle size into consideration.

3.0 SULFUR COMPOUNDS

Hourly, measured emission data for all major point sources of SO_2 in the St. Louis AQCR have been gathered and are available in the RAPS emission inventory data base. This work is described in a series of reports [1-4].

In the St. Louis area, virtually all sulfur dioxide emissions (98+%) occur from point sources (stacks, vents, etc.). This is not to say that the remaining emissions are unimportant, since they originate essentially at street level (automotive emissions, residential and commercial heating etc.) and thus may contribute a disproportionate share to ambient concentrations.

3.1 SCOPE AND DEFINITIONS

This report deals with SO_3 emissions from stationary point sources. The term "sulfur trioxide" (SO_3) is used, though it is realized that in its particulate form, in which it is customarily collected, the compound is hydrated to sulfuric acid (H_2SO_4).

At stationary point sources, both SO_2 and SO_3 originate from the oxidation of sulfur or sulfur containing compounds. The bulk of the sulfur oxides originates from the combustion of fossil fuels, while the remainder comes from process operations such as the roasting of ores, the manufacture of sulfuric acid, etc.

The two oxides exist side by side in an equilibrium which is largely determined by operational conditions at the source. The amount of SO_3 present is usually expressed as a fraction of the SO_2 concentration.

3.2 DEVELOPMENT OF BASE DATA AND ALGORITHMS

3.2.1 Base Data

In conformity with the National Emission Data System (NEDS) [5], the RAPS Emission Inventory records basic fuel consumption and process data, rather than mass emissions of pollutants. The basic data are converted to mass flow of pollutants using emission factors, stored in a separate file. The advantage of this

arrangement is that it permits periodic updating of the relatively small emission factor file, without disturbing the large mass of base data.

NEDS is an annual system, based on yearly reports gathered by local or regional Air Pollution Control Agencies. By contrast, the RAPS emission inventory which covers the St. Louis Interstate Air Quality Control Region, is a collection of hourly values obtained directly for this purpose. Hourly data, based on a measured parameter such as fuel consumption, steam or power production are being obtained from all the major sources of pollutants in the AQCR. A major source for the purposes of this inventory, is one which individually emits more than 0.1% of the total of a given "criteria" pollutant in the area. "Criteria" pollutants for which national standards exist include SO_2, NO_x, CO, hydrocarbons and particulates.

The RAPS Emission Inventory also contains data on smaller sources, emitting as little as 10 tons of SO_2 per year. Data on these sources are based on annual consumption or process figures, modified by an operating pattern peculiar to the source. The pattern, which is also stored in the RAPS Data Handling System, records the hours per day and days per week for normal operation, as well as any holiday or vacation periods. Using this information, average hourly SO_2 emission values can be obtained as an output. Since these sources make up less than 2% of all point source emissions, no significant errors are introduced by this method.

As a result of this effort, a detailed and relatively accurate record of SO_2 production exists, which can serve as a base for an SO_3 inventory.

3.2.2 Sulfur Dioxide - Sulfur Trioxide Ratios

In the presence of excess air in a combustion operation, a fraction of the sulfur dioxide is converted to sulfur trioxide (SO_3) according to

$$2\ SO_2 + O_2 \rightarrow 2\ SO_3 + 45.2\ Kcal$$

The reaction is exothermic; however, the reaction rates are negligible below 200°C (392°F), reach a maximum around 400°C (752°F) and taper off to zero at 1000°C (1832°F). Rapid conversion takes place only in the presence of a catalys

As would be expected from the reaction constant

$$K = \frac{(SO_2)^2 \times (O_2)}{(SO_3)^2}$$

the yield increases with excess oxygen.

The information of SO_3 in boiler stack gases has been investigated fairly extensively. Corbett [6] investigated the SO_3 formation in an oil-fired boiler. He found that 1 to 3% of the SO_2 was oxidized to SO_3. The amount of SO_3 found did not correlate with the percentage of sulfur in the oil or boiler conditions. Lee [7] used a wet-bottom, pulverized coal-fired research boiler, several types of coal, and varied the excess oxygen from 0.5 to 5%. He found a distinct relationship on excess oxygen (Fig. 1).

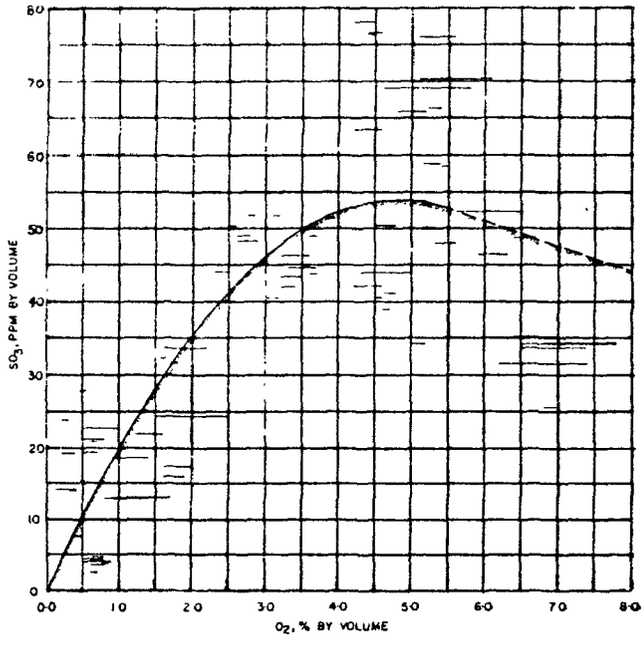


FIGURE 1: SO_2 CONCENTRATION PLOTTED AGAINST O_2 IN FLUE GAS FOR FOUR STAR COAL (Ref. 7)

In a later study [8] Lee obtained similar results in an oil-fired furnace. Gills [9] found a similar dependence on excess oxygen, but did not get a flattening of the curve up to 12% oxygen (Fig. 2). He also found a strong dependence on boiler size, with an 850 tons steam/hour boiler producing a 1% conversion to SO_3 at an oxygen level of 0.5%, while smaller boilers (up to 25 tons steam/hour) produce only a 0.25% conversion under similar conditions.

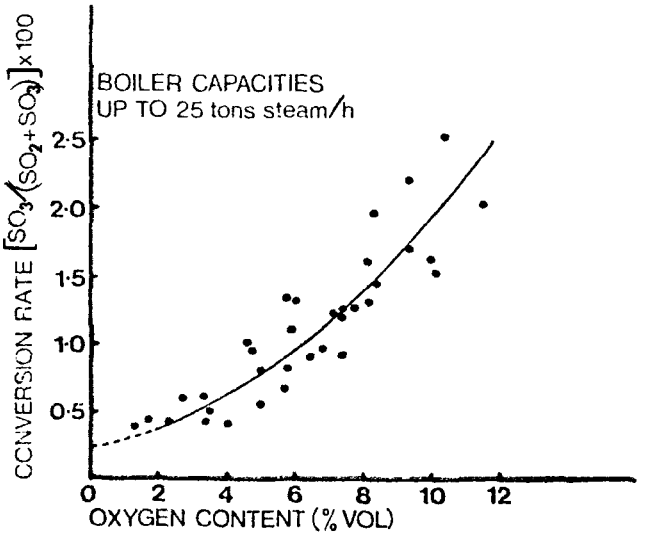


Figure 2: VARIATION OF SO_2 CONVERSION TO SO_3 WITH OXYGEN (Ref. 9)

The latter relationship was confirmed by Reese [10], who obtained the following results at 4% excess oxygen.

TABLE 1

RELATIONSHIP BETWEEN BOILER SIZE AND SO_3 FORMATION

Installation size MW	% Conversion to SO_3
55	2.1
110	3.5
185	4.4

In general, the percentage conversion falls in the range of 0.5 to 5%, with absolute values below 50ppm of SO_3*.

Our results are shown in Table 2 and Figure 3.

The concentration of SO_2 varied from 120 ppm for a boiler operated on distillate oil to 2660 ppm SO_2 for a coal burning boiler. Average for coal burning boilers was about 1600 ppm. The SO_3 concentration ranged from 2.7 to 44.3 ppm, well within the range indicated by other investigators. As indicated in Figure 3, there appears to be a marked dependence on excess oxygen. The percentage of SO_3 increased with increasing oxygen up to about 9%, then dropped rapidly. This may be due to the cooling effect of large amounts of excess air. There did not seem to be any correlation with the sulfur content of the fuel nor did there appear to be any marked effect of boiler capacity on the amount or concentration of SO_3 produced.

The RMS average SO_3 emission appears to be about 1.85% of the SO_2 emission. This factor will be incorporated in the data handling system output program, which will report SO_3 emissions based on the corresponding SO_2 emissions. Using the current figures for SO_2, this amounts to an annual emission of 22,585 tons of SO_3 per year.

Analytical Methods for SO_3

The current standard method for SO_3 in stack gases is EPA Method 8 (CFR 40, 60.85, Appendix-Test Methods). In this method, the sample of stack gases is drawn through a series of impingers. The first impinger contains 100ml of 80% iso-propanol; the second and third 100ml of 3% hydrogen peroxide. There is a filter between the first and second impinger to retain entrained particulates. The contents of the impingers are analyzed for sulfate using the barium perchlorate-thorin method.

* An interesting exception was found by Gills [9] in brick kilns, where up to 28% of the sulfur oxides were in the form of SO_3.

TABLE 2

SULFUR OXIDE ANALYSIS AND RATIOS

	SO_2 lbs/SCF x 10^5	ppm	SO_3 lbs/SCF x 10^3	ppm	SO_3/SO_2 Wt.	Vol.	Excess O_2	Boiler Cap. lbs steam/hr	% Sulfur in Fuel
Wood River #1	2.15	120.5	6.07	2.7	2.82%	1.49%	8.9%	450,000	.29
#4	38.95	2183.5	34.50	15.4	1.13	.70	6.0	710,700	3.21
Highland	47.50	2662.8	32.85	14.7	.69	.55	4.8	60,000	3.25
Stag	11.90	667.1	16.76	7.5	1.41	1.12	11.2	50,000	3.25
GM	34.90	1956.5	98.90	44.3	2.83	2.26	8.9	80,000	3.46
Amoco	12.60	706.3	14.25	6.4	1.13	.91	10.5	200,000	*3.00
Average	24.60	1382.8	33.8	15.2	1.69	1.17	8.4		
RMS Average					1.86				

*Weighted average

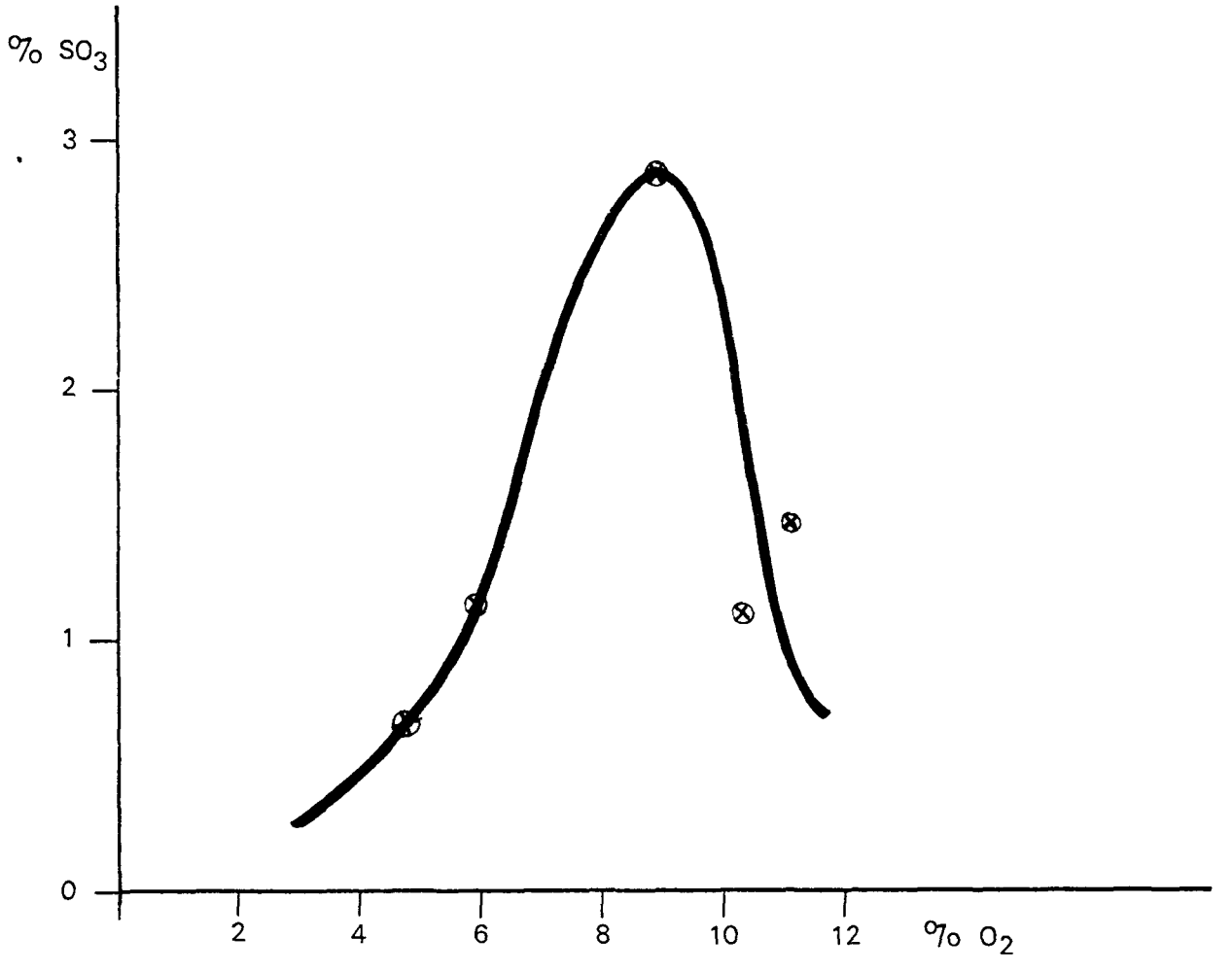


FIGURE 3

PERCENTAGE CONVERSION OF SO_2 TO SO_3

Recent work cast doubts on both accuracy and reproducibility of Method 8[11]. The method assumes that only SO_3 (sulfuric acid mist) will be retained in the first impinger and the filter (both of which are analyzed together). However, Hillenbrand[12] found that substantial amounts of SO_2 are retained in the first impinger, some of which is subsequently oxidized to SO_3, thus contributing to high results. For this reason a different technique was used, which was first described by Goksøyr and Ross[13] and subsequently verified by Lisle and Sensenbaugh [14]. The method is generally referred to as the "Shell" method, as it was developed in their laboratories. The method is based on the condensation of sulfuric acid mist at temperatures below its dew point (but above the dew point of water) in a condenser backed up by a fritted glass filter (Fig. 4). The condensate is washed out and titrated.

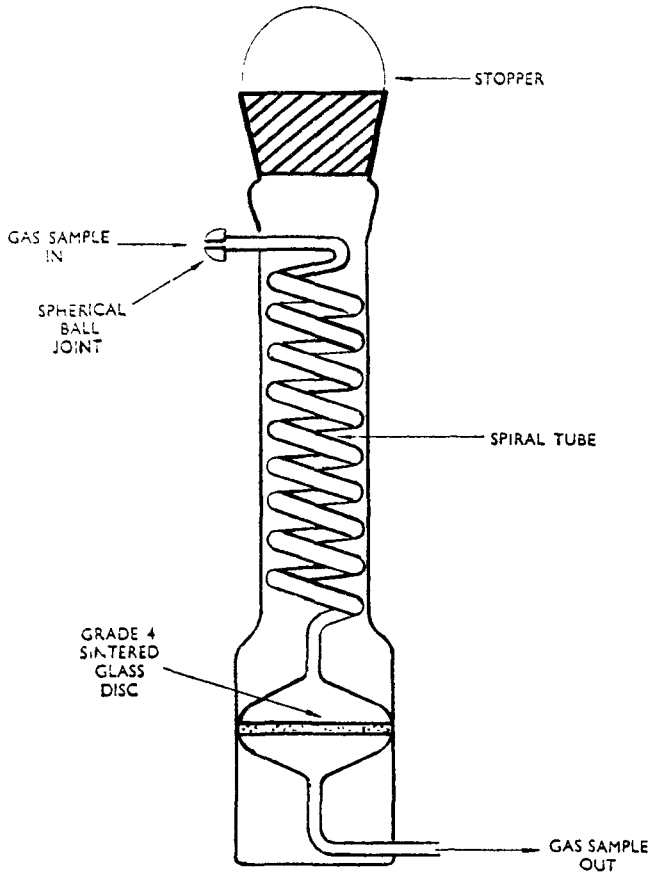


FIGURE 4: SULPHUR TRIOXIDE COLLECTOR (Ref. 12)

Data presented in references 12 and 13 indicate that adsorption of SO_3 is essentially complete, repeatability is excellent, SO_2 in concentrations up to 2000ppm does not interfere and a precision of $\pm$ 0.3ppm of SO_3 can be readily attained.

The method was then evaluated in our laboratories. The results of the evaluation are shown in Appendix I; they indicate an average 100.1 $\pm$ 6.5% recovery with no significant interference from any of the variables tested.

4.0 PARTICULATE SIZE INVENTORY

Emissions of particulate materials constitute a more complex problem than gaseous emissions, since the properties of particles are determined not only by their composition, but also by their size and shape. In fact, the most important properties of particles, their effect on visibility, their life-time as suspended materials, and to a large extent, their effect on health, are all determined by particle size. On all of these counts small particles, 5 microns or less in diameter, are responsible for most of the observed effects.

The common methods of collection and reporting of particulate emissions do not distinguish particle size. Total particulate matter is reported on a weight basis, which biases the results in favor of large particles. Since large particles are only of local importance - they settle out rapidly - and are generally not involved in health effects because they are readily retained by the body's screening mechanisms, there are good reasons why particulate emission data should be reported in such a way as to provide maximum information on small particles.

4.1 DEFINITIONS AND SCOPE OF INVENTORY

There is no universally accepted definition of "fine particles", but most authors agree on a range of 3 to 5 microns as the upper limit. Particles smaller than approximately 5 microns have settling velocities in still air of the order of 0.01 cm/sec and tend to stay aloft almost indefinitely. Particles can be either solid or liquid.

The most up-to-date study of fine particulate emissions is contained in EPA Technical Report entitled "Fine Particulate Emission Inventory and Control Survey" [15]. The methodology contained in that report was applied to the St. Louis AQCR. In addition, samples were taken at representative emission sources using an Andersen cascade impactor. Data developed from this study are also included.

4.2 DEVELOPMENT OF INVENTORY FOR AQCR-70

In order to prepare a particle size inventory within the scope of RAPS, compatible with the NEDS and RAPS Data Handling Systems, the procedure outlined below was used. No effort was devoted to the inventory of area sources, mobile sources, chemical and physical characterization of these particulates.

4.2.1 Method

The method put forth in the "Fine Particulate Emission Inventory and Control Survey" uses the following equation for the calculation of particulate emissions:

$$E_{d_1-d_2} = \frac{Pe_f C_t}{2000} * f_1(d) * f_2(d) \qquad (1)$$

where

$E_{d_1-d_2}$ = emission rate for particles with diameter between d_1 and d_2

P = production rate

e_f = emission factor (uncontrolled)

C_t = percentage of production capacity on which control equipment is installed (for that device)

$f_1(d)$ = emitted particle size distribution

$f_2(d)$ = penetration = (1-fractional efficiency of control system)

The size ranges are (in microns):

.01 - .05, .05 - .1, .1 - .5, .5 - 1, 1 - 3, 3 - 7.

The data sources are:

(1) RAPS coding forms (or NEDS computer listing)

(2) "NEDS Source Classification Codes and Emission Factor Listing" (SCC listing), July 1974.

(3) "Fine Particulate Emission Inventory and Control Survey" (EPA-450/3-74-040), January 1974.

The equations used for calculating the total particulate emissions are:

$$E_{d_1-d_2}(\text{total}) = E_{d_1-d_2}(\text{controlled}) + E_{d_1-d_2}(\text{uncontrolled}) \qquad (2)$$

where $E_{d_1-d_2}$ (controlled) is expressed in equation 1, and

$$E_{d_1-d_2}\ (\text{uncontrolled}) = \frac{Pe_f(1-C_t)}{2000} * f_1\ (d) \qquad (3)$$

The assumption used here is that f_2 (d) applies to that fraction of the emission which is specified by C_t and $(1-C_t)$ has no control and therefore $f_2(d) = 1$.

The algorithm for a computer program may be something like the following:

I For every point source

Ia Look up from RAPS coding form, the SCC code — Card 4

Ib Look up P (annual data) — Card 5

Ic Look up C_t (control efficiency) — Card 3

Id Look up CID (control device ID code) — Card 3

II From EFACTR file. Look up e_f (uncontrolled emission factor) for the corresponding SCC number.

III From SIZE file. Look up size distribution in fractional values for each size range for the corresponding SCC number.

IV From the EFCNCY file. Look up the fractional efficiency of each size for the corresponding control device as identified by the code number CID.

V Calculate the emissions using equations 1, 2 and 3.

The following are examples of the three computer input data files:

File Name: SIZE

SCC Code	.01-.05	.05-.1	.1-.5	.5-7	1-3	3-7
			Fractional Values			

File Name: EFACTR

SCC Code	Emission Factor
	Pounds per Ton

File Name: EFCNCY

CID	.01-.05	.05-.1	.1-.5	.5-.1	1-3	3-7
		Efficiency				

4.2.2 Particulate Size Inventory Data Files

Of the three files required for calculations of the particulate size inventory, one, the emission factor file EFACTR, is already contained in the RAPS inventory. The other two were developed on this Task and are given in Appendix II.

The SIZE-file, in a matrix form, gives the particle-size distribution of emitted particulates for any one of the forty-four SCC-codes listed in column 2. Each column between columns 3 through 8 lists the fractional value of the total particulate effluent that falls within the corresponding particle-size range.

All values to the right of the double line (columns 2-8) are keypunched for computer input with the READ format: (I8, 6F4.0), blanks = 0. The fractional values in the F-format are left justified with no decimal points.

The EFCNCY-file lists the fractional efficiency of the effluent control device for each particle-size range. The control device is identified by the CID number under column 2 and the particle-size ranges have the same diameter groupings as that in the SIZE-file.

All values to the right of the double line (columns 2-8) are keypunched for computer input with the READ format:

(I3, 6F4.0), blanks = 0

The fractional values in the F-format are left justified with no decimal points.

Both files were keypunched. The cards are available for input into the RAPS Data Handling System.

4.3 EXPERIMENTAL PARTICLE SIZE DISTRIBUTION DATA

In connection with the emission factor verification program carried out as part of the RAPS study, data were gathered on particles size distribution of a number of representative sources.

4.3.1 Method and Equipment

Particle size testing was performed with an Andersen Stack Sampling head coupled with the apparatus used for standard EPA method for particulates. The Andersen is a fractionating inertial impactor which separates particles according to aerodynamic characteristics.

The Mark II sampling head consists of a stainless case, plate holder and nine jet plates. The plates have a pattern of precision-drilled orifices. The nine plates, separated by 2.5 millimeter stainless steel spacers, divide the sample into eight fractions or particle size ranges. The jets on each plate are arranged in concentric circles which are offset on each succeeding plate. The size of the orifices is the same on a given plate, but is smaller for each succeeding downstream plate. Therefore, as the sample is drawn through the sampler at a constant flow rate, the jets of air flowing through any particular plate direct the particulates toward the collection area on the downstream plate directly below the circles of jets on the plate above. Since the jet diameters decrease from plate to plate, the velocities increase such that whenever the velocity imparted to a particle is sufficiently great, its inertia will overcome the aerodynamic drag of the turning airstream and the particle will be impacted on the collection surface.

The Mark III is identical to the Mark II except the location of the orifices in the plates have been modified to permit the use of a special collection substrate (glass fiber in our tests). This permits lighter tare of weights for gravimetric analyses and a collection of material for chemical analysis. Figure 5 illustrates the Andersen sampling head and an exploded view of the plate holder and plates.

4.3.2 Measurements of Particle Size

Particle Size Distribution measurements have been conducted at five of the seven test sites sampled in 1975. Initially only the Andersen Mark II plates were available. Because of this the only results available at the first test site are the weight distribution. On subsequent tests, runs were made with both the Mark II plates and Mark III plates with glass fiber filters for comparison. Sites that have been tested for particle size are shown in Table 3. Some of the filter samples were inspected microscopically and a few of these were also

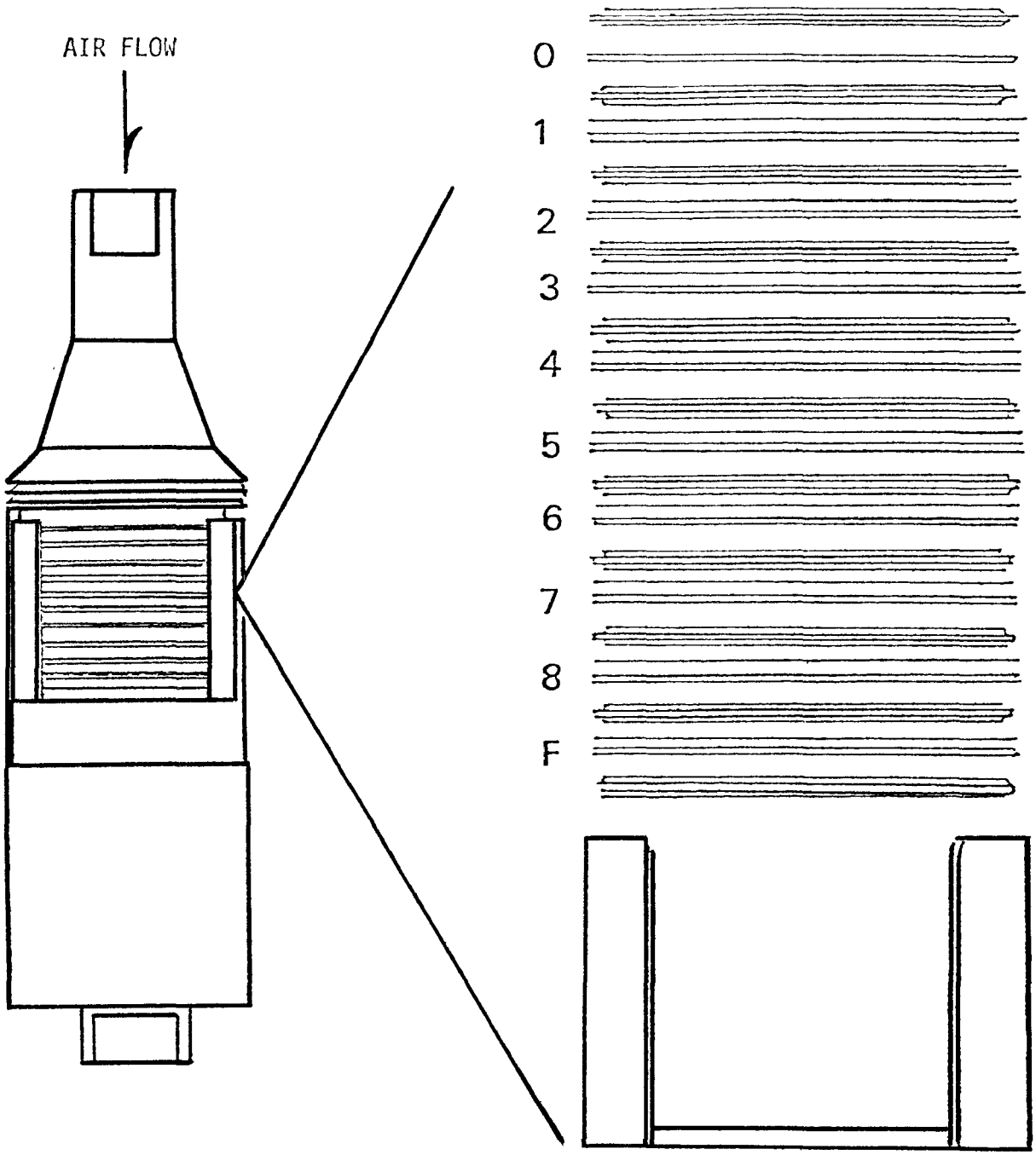


FIGURE 5

ANDERSEN STACK SAMPLER

analyzed by x-ray fluorescence. A summary of the results of the testing is given in Table 3. Particle size is given as aerodynamic size for spherical particles with unit density.

TABLE 3

PARTICLE SIZE DISTRIBUTION RESULTS

Source	Scc Code	% vs Particle Size				
		>7μ	3-7μ	1-3μ	0.5-1μ	< 0.5μ
Ill. Power - Wood River	1-01-002-02	22.5	22.8	18.5	8.3	27.9
Highland Electric	1-01-002-08	26.6	18.9	10.0	12.7	31.8
Stag Brewery	1-02-002-05	37.4	16.0	7.6	18.3	20.7
General Motors	1-02-002-09	14.3	24.4	18.5	9.2	33.6
Amoco	3-06-001-02	13.9	8.9	22.0	18.8	36.4
	3-06-001-03					

At General Motors, fourteen tests were performed to evaluate variations of testing methods consisting of placing the Andersen in-stack, out of stack (in oven) using Mark II plates and Mark III plates with filters. Each of these methods has its advantages which may make it desirable for any one individual test. The main objective of these tests was to arrive at a testing arrangement to be used on all subsequent tests. As it turned out there was no clearcut single method which proved better than the others.

Sampling in the stack avoids any problems with extracting a sample and having some of it deposited in the probe. Also the sample head is at the same temperature as the stack gases which avoids any problems of condensation. In-stack sampling, however, means the impaction surface is vertical and is subject to having the sample dislodged in handling. When sampling must be done vertically in a duct, from the top down, this method cannot be used.

Sampling with the Andersen sampler in the sample oven at the end of a heated probe affords much better handling. The sample head can be kept vertically with the plates horizontal at all times. The sample head is also clamped in place and doesn't have to be threaded on to the probe, which avoids more handling.

Isokinetic sampling rates can be determined more readily when the Andersen is in the oven since the probe has a pitot attached and the probe remains in the stack (for in-stack sampling a pitot measurement is made, the pitot is removed

and the sampler is inserted to approximately the same position). There are two problems with sampling this way: the oven can be heated only to 350°F, which may not be as high as the temperature in the stack, and larger particles tend to be deposited in the probe, which lowers the weight of the deposit on the first two plates.

Parallel sampling with both the Mark II plates above and the Mark III plates with filters indicates that there isn't any significant difference in the weight of catch and the size distribution between these two methods. If the Mark II model is used, the number of tests is limited by how many sets of plates are available. With the Mark III plates and filters more runs can be performed by changing the filters between runs with the available time being the only constraint on the number of runs. More care must be taken in assembling the Mark III, since the filters are pre-cut to match the plates and must be properly aligned to avoid blocking any holes.

As a result of these comparison tests, it was decided that testing would be performed with the Mark III plates and filters and that the Andersen sample head would be placed in the oven for ease in handling and subsequent analysis.

Photomicrographs have been made by Illinois Institute of Technology Research Institute (IITRI) of samples collected on each stage from three Andersen runs. These pictures confirm that the Andersen does in fact separate by particle size as the instructions would indicate. Evidence of this is shown in Figures 6, 7 and 8 from General Motors and Figures 9, 10, 11 and 12 from the Stag Brewery.

Figure 6 is from stage 2 taken at 163x. This shows a high percentage of fly ash and partially fused clays and minerals, average particle size is approximately 6 microns. Figure 7 is from stage 4 taken at 163x. This shows much smaller particles, a high percentage of fly ash and more Fe_2O_3, and an average particle size of approximately 2 microns. Figure 8 is from stage 6 taken at 163x. This shows mostly submicron partially burned coal, fly ash and Fe_2O_3.

For spherical particles with unit density stage 2 should have separated from 10.9 to 17 microns, stage 4 from 5.0 to 7.3 microns, and stage 6 from 1.7 to 3.2 microns. Since fly ash has a density between 2 and 3, these stages will actually separate smaller particles.

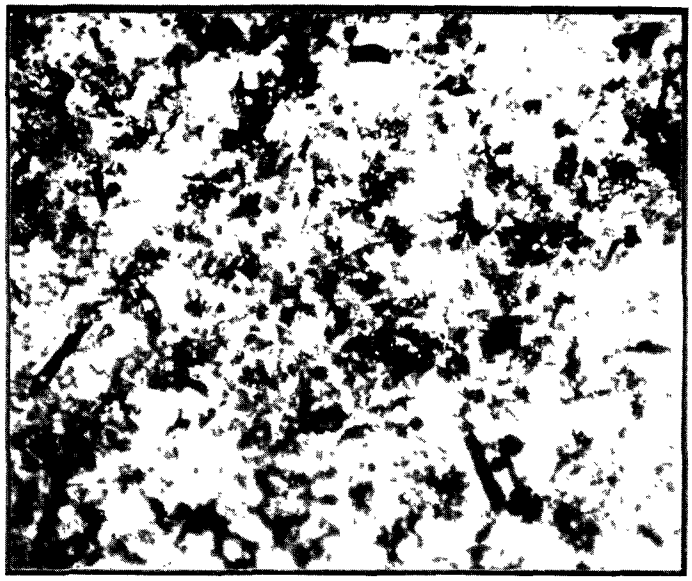
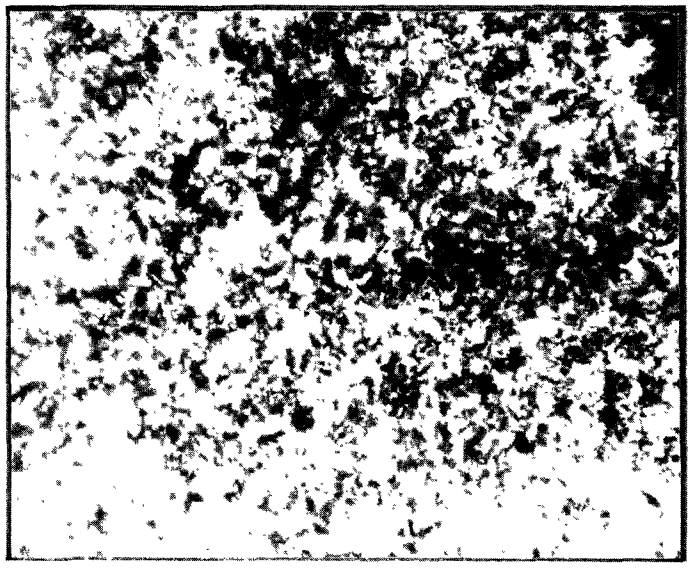


FIGURE 6



DEPOSITS ON STAGES
2,4 AND 6
GENERAL MOTORS

FIGURE 7

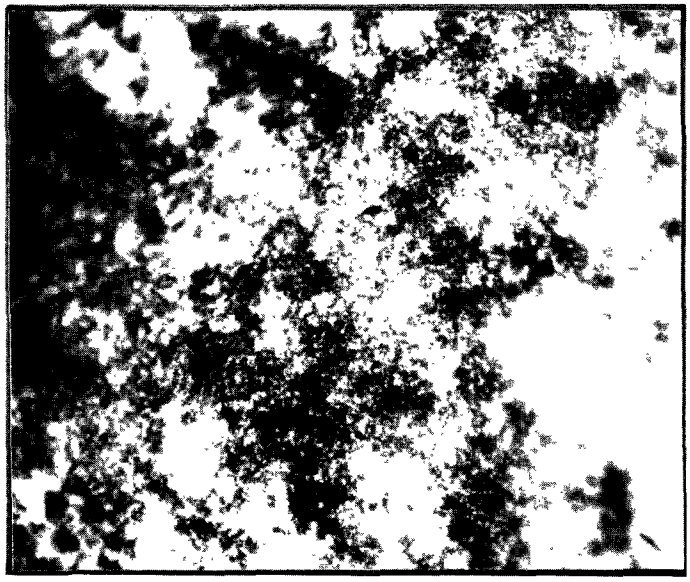


FIGURE 8

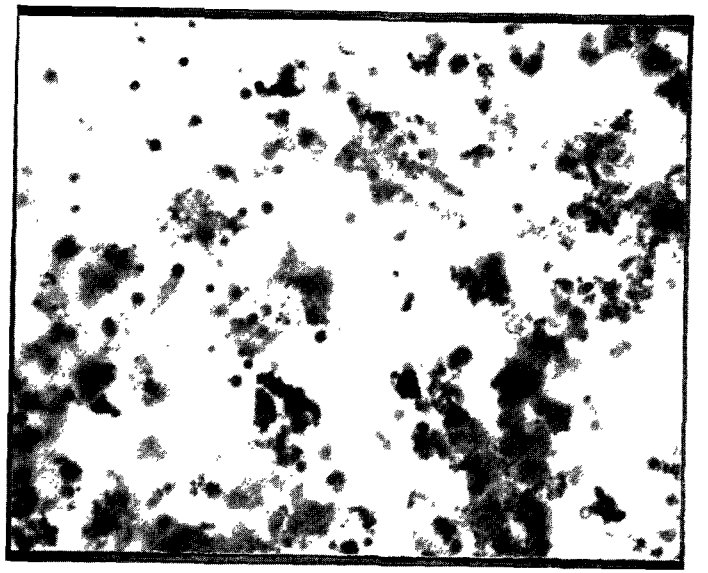


FIGURE 9

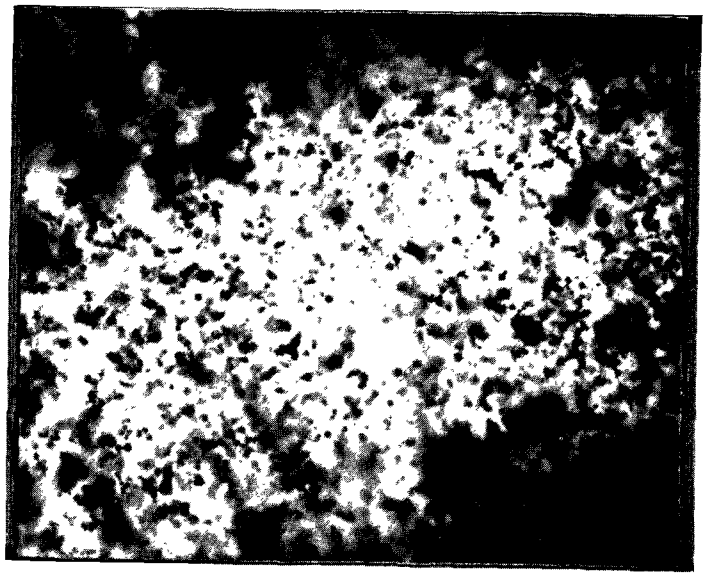


FIGURE 10

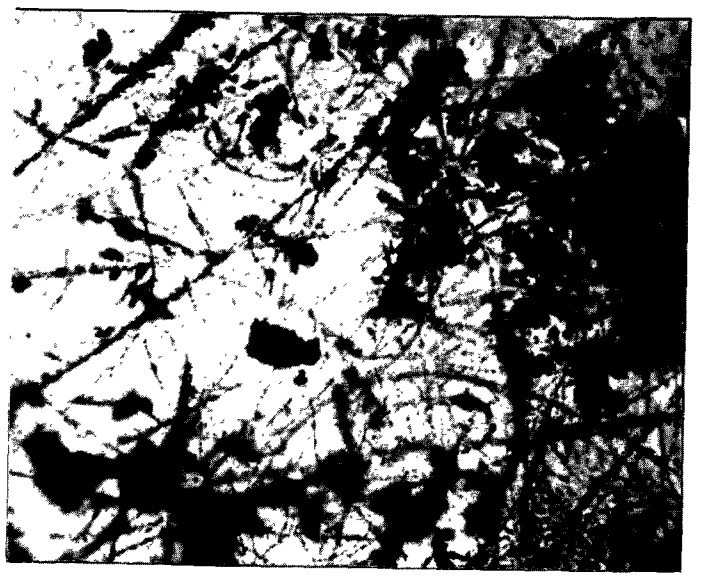


FIGURE 11

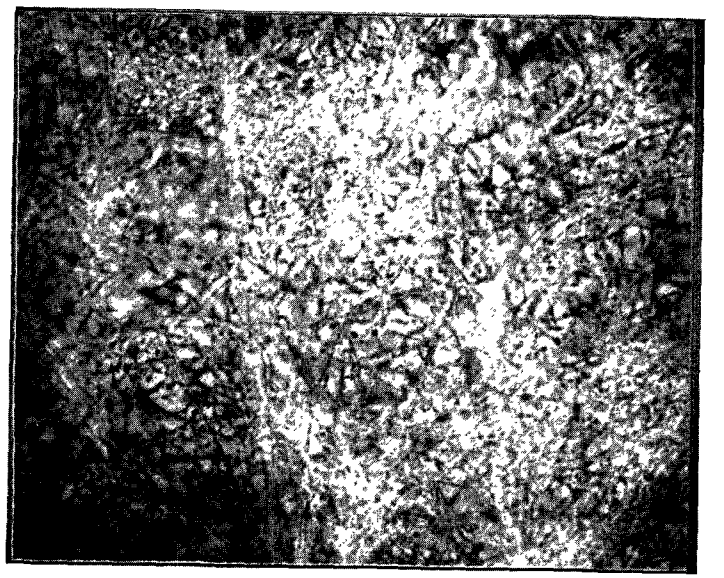


FIGURE 12

DEPOSITS ON STAGES 2,4,6
AND BACK-UP FILTER
STAG BREWERY

Figure 9 from Stag Brewery is from stage 2 taken at 406x. This shows lots of incompletely combusted coal, partially fused glassy material and Fe_2O_3 partially fused and coating other particles. Average particle size is approximately 5 microns. Figure 10 is from stage 4 taken at 406x. This shows fine fly ash spheres most of which are dark due to iron in solid solution and some minerals and fine carbonaceous particles. Average particle size is approximately 2 microns. Figure 11 is from stage 6 taken at 406x. This shows what appears to be black carbonaceous material which hit as a liquid or is particles suspended in a liquid. There is very little fly ash or else it is below 0.5 micron. Figure 12 is from the backup filter taken at 406x. This shows extremely fine liquid droplets with trapped fine carbonaceous particles and extremely fine sulfate particles.

Microscopic analysis of the filters has indicated that sulfate crystals form on the filters in increasing amounts on descending stages to the point where the backup filter sample is mostly sulfate. Personnel from IITRI have indicated that these crystals are ammonium sulfate and that they have grown on the filters. Figure 13 is from a backup filter from a test at General Motors, taken at 163x. Clearly, these crystals could not have passed through the Andersen impactor.

The mechanism for the formation of these crystals is still not understood. Apparently, there is a reaction between ammonia in the flue gases with sulfuric acid on the filters. To check that this reaction didn't take place from exposure sometime later, one backup filter was sealed in an air-tight enclosure at the test site and then examined immediately after opening the sample container. This sample also showed a large amount of crystals.

A few of these backup filters have been analyzed for acidity. Approximately 17% of the amount of sulfuric acid measured in the stack at General Motors was found to be entrained by the backup filter and by the total particulate filter on an EPA particulate run. Whether this is due to condensation and entrainment, or a gas-solid phase reaction, is not known. At these temperatures, 440°F in-stack and 350°F in the oven, sulfuric acid vapor should not condense.

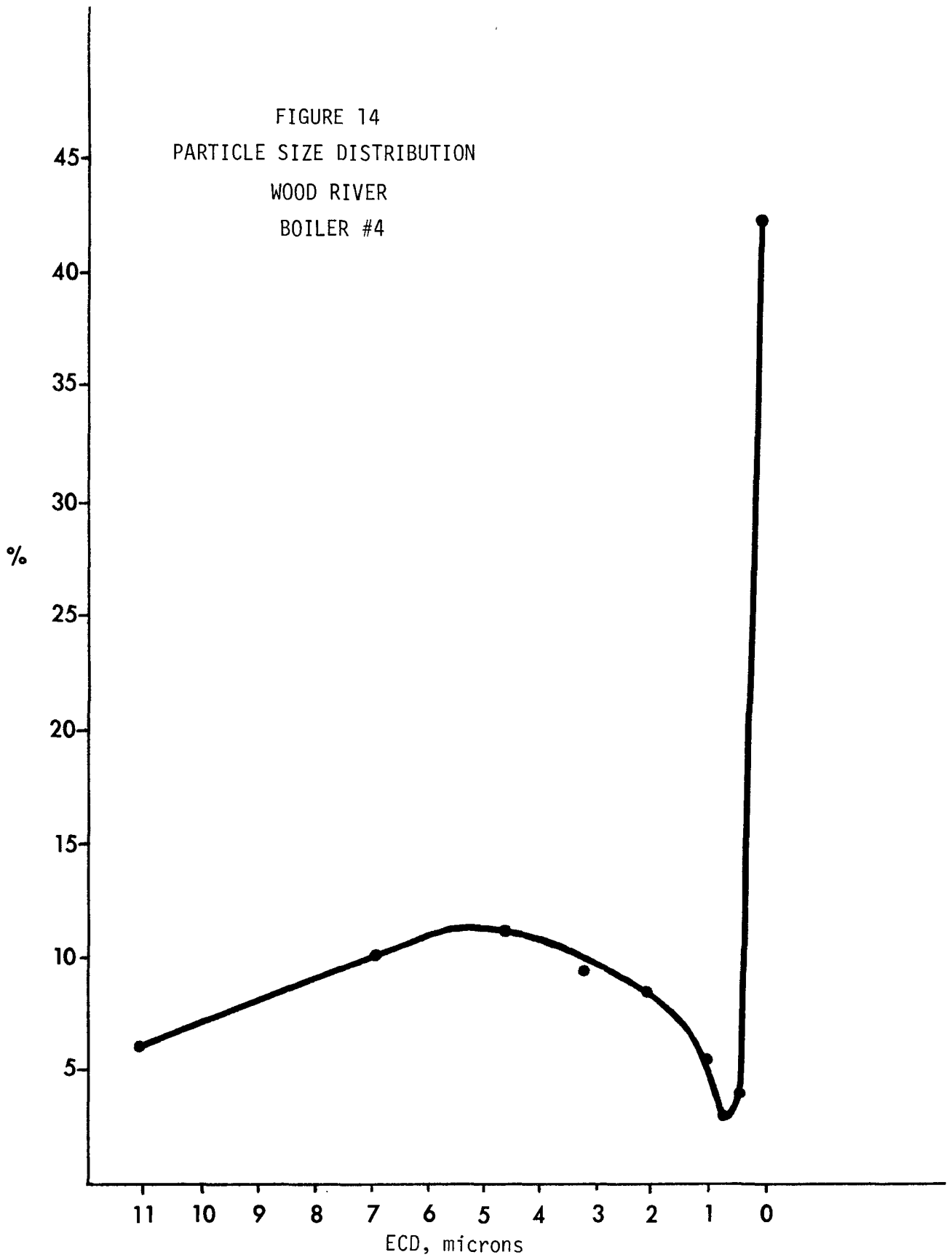
One test run indicated that temperature has some relationship to the amount of material in the backup filter. Two identical Andersen runs were made at General Motors with the sample head in the oven. The first test was with an oven

FIGURE 13
AMMONIUM SULFATE CRYSTALS
ON BACK-UP FILTER
GENERAL MOTORS

temperature of 300°F and the second with a temperature of 370°F. While the first 8 stages were very similar in weight, there was twice as much material collected on the backup filter in the first test than in the second.

The particle size distribution from all of the tests performed to date shows a bimodal distribution, generally with a peak around stages 4 or 5 and a large peak on the last backup stage. A typical curve is shown in Figure 14. The large amount of sulfate crystals on the backup indicates that perhaps 30% of that amount is sulfuric acid and should not be included. But even after this is subtracted there are two peaks, one around 5 microns and the other less than 0.7 micron.

FIGURE 14
PARTICLE SIZE DISTRIBUTION
WOOD RIVER
BOILER #4



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APPENDIX I

LABORATORY EVALUATION OF THE "SHELL" METHOD OF DETERMINATION OF SO_3

The "Shell" method for determination of sulfur trioxide (sulfuric acid) in flue gas is based on its selective condensation from the flue gas. This is achieved by utilizing the relatively high (60-90^{o}C) dew point of SO_3. At this temperature only the sulfuric acid condenses from the flue gas and therefore it can be determined rather easily.

Flue gas is drawn through the condenser at a rate of 2 liters per minute for 10-20 minutes depending upon the SO_3 level in the flue gas. Particulates are removed from the flue gas sample by using a plug of glass wool as a filter. At the end of the sampling period, the H_2SO_4 is washed out of the condenser with 5% solution of isopropyl alcohol in water. The combined washings were titrated with 0.02 N NaOH using bromophenol blue as indicator.

The laboratory evaluation of this method had a dual purpose. The first was to check the accuracy of the method under the experimental conditions and secondly, to determine which of the experimental parameters may affect the performance of this method. For the latter, stack conditions had to be simulated in a way which would allow adjustment of each parameter to predetermined levels. The accuracy of the method was tested by duplicating the experimental work of E.S. Lisle and J.D. Sensenbaugh[1]. The effect of the experimental conditions on the accuracy of the method was evaluated by using the Plackett-Burman [2] statistical design of screening process variables. This method is based on balanced incomplete blocks. A good example of applying this method to a chemical process has been published by R.A. Stowe and R.P. Mayer[3]. With this method it is possible to effectively screen all the experimental parameters and to find out which of them most likely will affect the overall process, by performing only a small fraction of experimental work usually required for other methods of screening variables. For example, a complete factorial design for fifteen variables at two levels requires 32,768 experiments; with the Plackett-

Burman method the same number of variables can be screened effectively with only 16 experiments[3]. It should be emphasized, however, that this method does not optimize the process; it only indicates which of the parameters do not affect the process.

EXPERIMENTAL

The experimental set-up used in this study is given in Figure 1. A special condenser thermostated at 60-90°C was used for the collection of the condensed H_2SO_4. The simulated flue gas is introduced at the end of the condenser which consists of a spiral glass tube followed by a coarse glass fritted disc. Both the spiral and the glass fritted disc are kept at constant temperature (60-90°C) by circulating water from a heating bath. The H_2SO_4 generator consists of a quartz tubing heated electrically by a spiral of nichrome wire insulated by several layers of asbestos tape. With this arrangement the temperature of the H_2SO_4 generated can be adjusted at the desired level and kept constant within 10°F. Dilute sulfuric acid solution is added at a constant rate by a peristaltic pump through a hypodermic needle and serum cap in the top opening of the H_2SO_4 generator. The rate of H_2SO_4 addition can be altered by using pump tubes of different diameter. The flow rate of the gases (O_2, N_2, SO_2) was adjusted and maintained at the proper levels with a combination of valves and rotometers. The total flow was checked by a rotometer at the outlet of the condenser.

PROCEDURE

The H_2SO_4 generator was calibrated by titrating the amount of acid delivered by the peristaltic pump at the upper end of the generator for a certain period of time (about 10 minutes) for the two pump tubes and the two H_2SO_4 solutions used throughout the experimental work. The nominal flow rates of the pump tubes used were 0.42 and 0.70 cc/min and the normality of the H_2SO_4 solutions was 0.01 and 0.03 N. Tables 1, 2, 3 and 4 give the calibration of the H_2SO_4 generator for the above flow rates and the sulfuric acid solutions. The results are expressed in μ. equiv/min. The actual experiments were conducted in a similar manner. Sulfuric acid solution was delivered to the H_2SO_4 generator by the pump for about ten minutes and collected in the condenser. The condensed H_2SO_4 was washed out of the condenser with 5% isopropyl alcohol in water and the combined washings

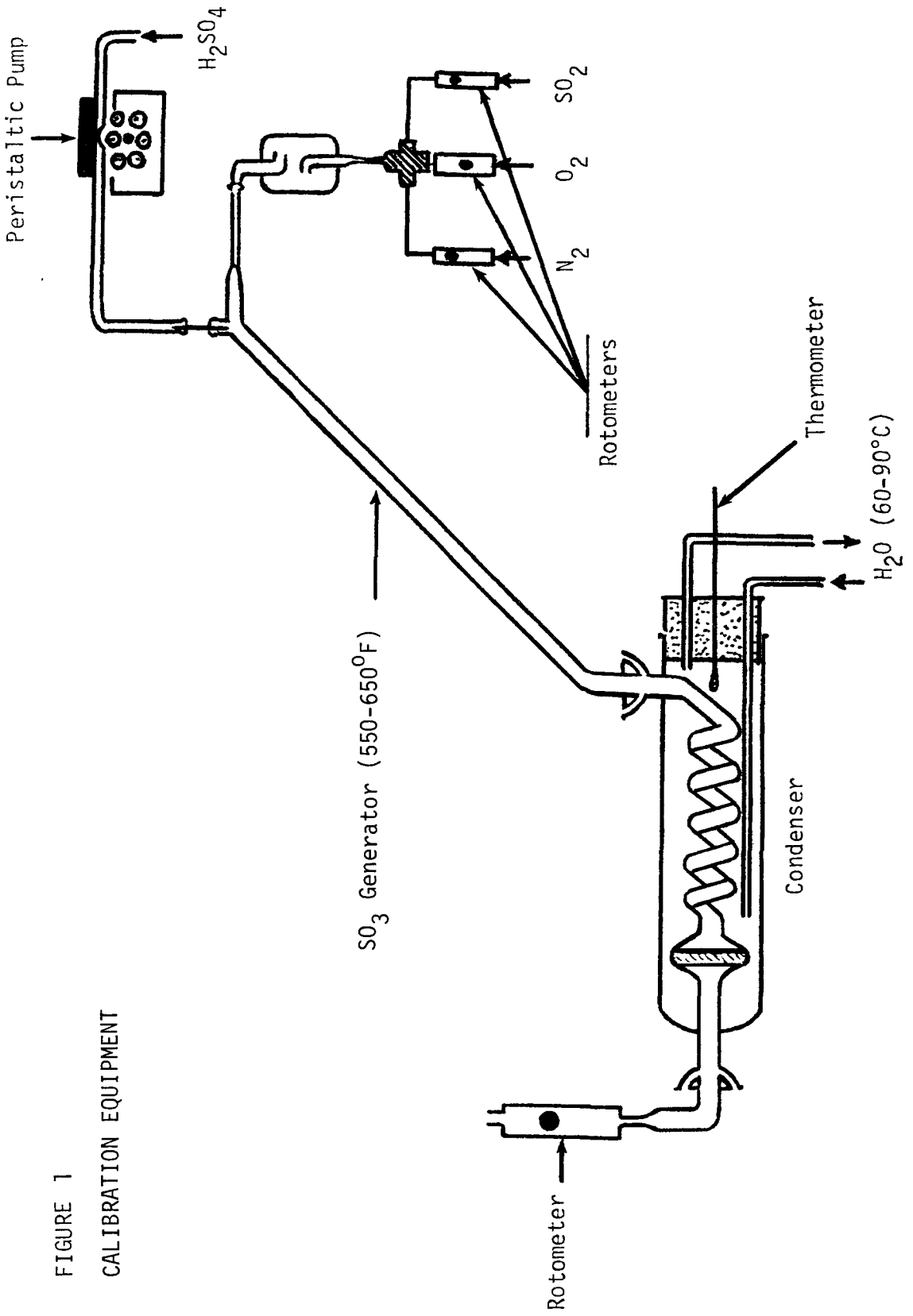

FIGURE 1

CALIBRATION EQUIPMENT

Table 1

CALIBRATION OF THE SO_3 GENERATOR
Nominal Pump Rate = 0.42 cc/min
Normality of H_2SO_4 Solution = 0.01 N

Run #	Time Sec.	cc of 0.02 N NaOH	μ equil/min
1	601.7	2.58	5.14
2	599.7	2.42	4.84
3	599.5	2.45	4.90
4	600.3	2.30	4.60
5	600.4	2.32	4.63
		Average	4.82 ± 0.22 μ. equiv/min

Table 2

CALIBRATION OF THE SO_3 GENERATOR
Nominal Pump Rate = 0.42 cc/min
Normality of H_2SO_4 Solution = 0.03 N

Run #	Time Sec.	ml of 0.2 N NaOH	μ equil/min
1	601.0	7.46	14.89
2	601.5	7.82	15.60
3	600.4	7.52	15.03
4	600.5	7.66	15.30
		Average	15.20 ± 0.31 μ. equiv/min

TABLE 3

CALIBRATION OF THE SO_3 GENERATOR
Nominal Pump Rate = 0.70 cc/min
Normality of H_2SO_4 Solution = 0.01 N

Run #	Time Sec.	Titrant ml of 0.02 N NaOH	μ equil/min
1	630.6	4.60	8.15
2	600.0	4.46	8.92
3	689.8	4.98	8.66
4	600.7	4.44	8.87
5	600.4	4.42	8.83
		Average	8.81 ± 0.10 μ.equiv/min

TABLE 4

CALIBRATION OF THE SO_3 GENERATOR
Nominal Pump Rate = 0.70 cc/min
Normality of H_2SO_4 Solution = 0.03 N

Run #	Time Sec.	Titrant cc of 0.02 N NaOH	μ equil/min
1	599.9	12.80	25.60
2	600.2	12.23	24.45
3	599.2	12.26	24.55
4	600.7	12.42	24.81
5	602.2	13.24	26.38
		Average	25.16 ± 0.82 μ.equiv/min

were titrated with 0.02 N NaOH. Throughout this work all the experimental parameters were varied at two levels: one high level and one low level designated here as (+) or (-) respectively. Table 5 gives all the experimental parameters examined in this study and their respective high and low values.

The resulting efficiency of collection of the generated H_2SO_4 vapors was determined by dividing the recovered amount of H_2SO_4 by the amount of SO_3 delivered into the system (Tables 1 and 4).

RESULTS AND DISCUSSION

As it was mentioned previously, the purpose of this study was to first determine the efficiency of the system under the recommended conditions and secondly to screen all the experimental parameters and determine which of them affect the efficiency of the system.

Table 6 summarizes the results obtained by using the system under the recommended conditions. No SO_2 was used in these experiments because the main purpose was to determine the efficiency of collection of H_2SO_4 from flue gas. These experiments were performed in the manner recommended by Lisle and Sensenbaugh[1]. The samples were introduced in the evaporator by a syringe through the serum cap without the use of the proportioning pump. The average recovery was found to be equal to $100.1 \pm 6.5\%$. It should be noted, however, that no extra effort was made to optimize any of the experimental conditions and therefore these results represent data obtained by a casual application of this method. A close inspection of the results tabulated in Table 6 shows that sources of positive (recoveries > 100%) and negative (recoveries < 100%) errors do exist and therefore an examination of the parameters affecting the accuracy of the method appeared to be necessary. The parameters listed in Table 5 were tested by the method of Plackett and Burman by using a matrix for sixteen runs and fifteen variables. Figure 2 gives the Plackett-Burman matrix used in this study. Five out of the fifteen variables were blank "dummy" tests from which the standard error of the method was calculated.

The statistical analysis of the results is given in Table 7. In this table, confidence levels are shown only to the 70% level; the remaining variables are considered to have an insignificant effect on the method within the studied ranges. Therefore from the ten variables studied only four may have an effect on the ac-

TABLE 5

VARIABLES CHOSEN FOR STUDY

Code letter of the variable	Variable	Levels Low(-)	Levels High(+)
A	Temperature of Condenser (°C)	60 ± 3	90 ± 3
B	Temperature of Evaporator (°F)	550	650
C	O_2/SO_2 Ratio	149	223
(D)	Dummy	---	---
E	Total Flow (liter/min)	2	4
F	Flow Rate of H_2SO_4 Solution (cc/min)	0.42	0.70
(G)	Dummy	---	---
H	Elapsed Time Prior to Rinsing the Condenser (min)	1	10
I	Volume of Solvent for Each Washing (ml)	10	25
J	Total Volume of Solvent Used for Each Experiment (ml)	135	185
K	Size of Hypodermic Needle (gauge)	26	20
(L)	Dummy	---	---
M	Normality of H_2SO_4 Solution	0.01	0.03
(N)	Dummy	---	---
(O)	Dummy	---	---

TABLE 6

COLLECTION EFFICIENCY OF THE METHOD
OF SO_3 DETERMINATION IN FLUE GASES

Volume of acid = 4.0 mil; total flow = 3.96 liter/min; nitrogen to oxygen ratio 20:1; condenser's temperature 85°C; temperature of H_2SO_4 generator 600°F.

	m. equiv. H_2SO_4		
Run #	Taken	Found	% Recovery
1	0.040	0.039	97.50
2	0.040	0.042	105.00
3	0.040	0.041	102.50
4	0.040	0.047	117.50
5	0.080	0.077	96.25
6	0.080	0.077	96.25
7	0.080	0.074	92.50
8	0.080	0.077	96.25
9	0.120	0.123	102.50
10	0.120	0.119	99.17
11	0.120	0.120	100.00
12	0.120	0.115	95.83

Average % Recovery 100.0 $\pm$ 6.5

PLACKETT-BURMAN MATRIX FOR DETERMINING
THE EFFECTS OF FIFTEEN VARIABLES
AT TWO LEVELS USING SIXTEEN RUNS

Random Number	Run Order	Variable																
		A	B	C	(D)	E	F	(G)	H	I	J	K	(L)	M	(N)	(O)		% Recovery
1	1	+	+	+	+	−	+	−	+	+	−	−	+	−	−	−		92.4
2	2	+	+	+	−	+	−	+	+	−	−	+	−	−	−	+		93.9
3	3	+	+	−	+	−	+	+	−	−	+	−	−	−	+	+		92.5
4	8	+	−	+	−	+	+	−	−	+	−	−	−	+	+	+		94.1
5	4	−	+	−	+	+	−	−	+	−	−	−	+	+	+	+		87.4
6	6	+	−	+	+	−	−	+	−	−	−	+	+	+	+	−		86.6
7	9	−	+	+	−	−	+	−	−	−	+	+	+	+	−	+		83.1
8	12	+	+	−	−	+	−	−	−	+	+	+	+	−	+	−		109.3
9	10	+	−	−	+	−	−	−	+	+	+	+	−	+	−	+		87.3
10	13	−	−	+	−	−	−	+	+	+	+	−	+	−	+	+		102.1
11	14	−	+	−	−	−	+	+	+	+	−	+	−	+	+	−		83.7
12	16	+	−	−	−	+	+	+	+	−	+	−	+	+	−	−		96.4
13	5	−	−	−	+	+	+	+	−	+	−	+	+	−	−	+		98.5
14	7	−	−	+	+	+	+	−	+	−	+	+	−	−	+	−		107.5
15	11	−	+	+	+	+	−	+	−	+	+	−	−	+	−	−		81.8
16	15	−	−	−	−	−	−	−	−	−	−	−	−	−	−	−		84.3

− = LOW
+ = HIGH

FIGURE 2

TABLE 7 - LIST OF PARAMETERS

Variable Code	Variable Name	Effect* (+) to (-)	Relative Significance t-Test**	Relative Significance % Confidence Level
A	Temp. of Condenser	0.0289	1.018	--
B	Temp. of Evaporator	-0.0421	1.482	80
C	O_2/SO_2 Ratio	0.0014	0.049	--
(D)	Dummy	-0.0149	-----	--
E	Total Flow	0.0699	2.464	95
F	Flow Rate of H_2SO_4 Soln.	-0.0110	0.387	--
(G)	Dummy	-0.0136	-----	--
H	Elapsed Time Prior to Rinsing	0.0244	0.860	--
I	Volume of Solvent for Each Washing	0.0231	0.814	--
J	Total Volume of Solvent for Each Exp't.	0.0501	1.766	85
K	Size of Hypodermic Needle	0.0224	0.789	--
(L)	Dummy	0.0147	-----	--
M	Normality of H_2SO_4 Solution	-0.0989	3.486	99
(N)	Dummy	0.0581	-----	--
(O)	Dummy	-0.0051	-----	--

*The effect of each parameter is the difference of the average high (+) minus the average low (-) response. For example, the effect of A is $E_A = \frac{\Sigma R(+)}{8} - \frac{\Sigma R(-)}{8}$

**t-Test value of a variable is given by $t = \frac{Ev}{S.E._{eff}}$, where Ev is the effect of variable v and $S.E._{eff}$ is the standard error effect given by $S.E._{eff} = \sqrt{\Sigma (E_d)^2}$ where Ed is the dummy effect and n the number of the dummy variables

curacy of the method and should be studied further for the optimization of the total system. These four variables are the temperature of the evaporator (B), the total flow (E), the total volume of washing solution (J) and the normality of the H_2SO_4 solution used (M). It should be noted at this point that from these four variables, the two (B and M) are very closely related with the experimental conditions used for generating simulated flue gas in the laboratory and therefore may not be associated with the application of the method in the determination of SO_3 in real flue gas. The other two (E and J) are associated with the method and appear to be the most significant parameters which may affect the accuracy of the SO_3 determination in flue gas. The total flow (parameter E) most likely affects the condensation of SO_3 from the flue gas and the total volume of washing solution (parameter J) is related with the efficient washing of the condensed H_2SO_4. These two parameters are most likely the ones on which proper attention should be given in the application of this method for determination of SO_3 in flue gas.

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3. R.A. Stowe and R.P. Mayer, Ind. and Eng. Chem. 58, 36 (1966).

APPENDIX II

RAPS - TASK 56 PARTICULATE INVENTORY: SIZE - FILE

SOURCE OPERATION	SCC - CODE	PARTICULATE SIZE RANGE					
		3-7 μ	1-3μ	.5-1μ	.1-.5μ	.05-.1μ	.01-.05μ
Asphalt Dryers	3-05-002-01	.190	.131	.022	.0009	.0001	0
Asphalt Vent Lines	3-05-002-02	.232	.0735	.0041	.0004	0	0
Cement Kilns	3-05-006-03	.165	.103	.0165	.0054	.0001	0
	3-05-007-01	.165	.103	.0165	.0054	.0001	0
Fertilizer Granulators and Dryers	3-01-027-06	.040	.0182	.0043	.0023	.0002	0
Iron and Steel: BOF	3-03-009-03	0	.060	.360	.578	.002	0
Elec. arc	3-03-009-05	.13	.200	.120	.210	.060	.070
sintering	3-03-008-03	.050	.027	.0056	.0024	0	0
Iron Foundry Cupolas	3-04-003-01	.065	.060	.020	.0230	.0035	.0035
Kraft Pulp Mill: B.F. Boiler	1-02-009-02	.120	.083	.018	.0080	.0003	.0001
Lime Plant: Sec. sources	3-05-016-01	.320	.350	.045	.005	0	0
	3-05-016-02	.320	.350	.045	.005	0	0
Municipal Incinerators	5-01-001-01	.050	.075	.035	.045	.010	.015
	5-01-001-02	.050	.075	.035	.045	.010	.015

RAPS - TASK 56 PARTICULATE INVENTORY: SIZE - FILE

SOURCE OPERATION	SCC CODE	PARTICULATE SIZE RANGE					
		3-7μ	1-3μ	.5-1μ	.1-.5μ	.05-.1μ	.01-.05μ
Elec. Util: Pulv. coal	1-01-002-01	.160	.100	.021	.0087	.0002	
	1-01-002-02	.160	.100	.021	.0087	.0002	0
Stoker coal	1-01-001-02	.180	.048	.009	.0029	0	0
	1-01-002-08	.180	.048	.009	.0029	0	0
Cyclone coal	1-01-002-03	.250	.220	.055	.0244	.0005	0
Gas	1-01-006-01	0	.90	0	0	0	0
	1-01-006-02	0	.90	0	0	0	0
Oil	1-01-004-01	.90	0	0	0	0	0
	1-01-005-01	.90	0	0	0	0	0
	1-01-005-02	.90	0	0	0	0	0
	1-01-005-03	.90	0	0	0	0	0
Industrial: Pulv. coal	1-02-002-01	.100	.0195	.0005	0	0	0
	1-02-002-02	.100	.0195	.0005	0	0	0
	1-02-002-08	.100	.0195	.0005	0	0	0
	1-02-002-12	.100	.0195	.0005	0	0	0
Stoker coal	1-02-002-04	.050	.0176	.0019	.0005	0	0
	1-02-002-09	.050	.0176	.0019	.0005	0	0
	1-02-002-11	.050	.0176	.0019	.0005	0	0
	1-02-002-13	.050	.0176	.0019	.0005	0	0

RAPS - TASK 56 PARTICULATE INVENTORY: SIZE - FILE

SOURCE OPERATION	SCC CODE	PARTICULATE SIZE RANGE					
		3-7 μ	1-3μ	.5-1μ	.1-.5μ	.05-.1μ	.01-.05μ
Gas	1-02-006-01	0	.90	0	0	0	0
	1-02-006-02	0	.90	0	0	0	0
	1-02-006-03	0	.90	0	0	0	0
	1-02-067-01	0	.90	0	0	0	0
	1-02-010-03	0	.90	0	0	0	0
Oil	1-02-004-01	.90	0	0	0	0	0
	1-02-004-02	.90	0	0	0	0	0
	1-02-004-03	.90	0	0	0	0	0
	1-02-005-01	.90	0	0	0	0	0
	1-02-005-02	.90	0	0	0	0	0
	1-02-005-03	.90	0	0	0	0	0

RAPS - TASK 56 PARTICULATE INVENTORY: EFCNCY-FILE

CONTROL DEVICE/METHOD	DEVICE ID	PARTICLE SIZE RANGE					
		3-7μ	1-3μ	.5-1μ	.1-.5μ	.05-.1μ	.01-05μ
No Equipment	000	0	0	0	0	0	0
Net Scrubber: high efficiency	001	.9997	.91	.74	.38	.08	.02
med. efficiency	002	.98	.79	.57	.26	.05	0
low efficiency	003	.90	.69	.39	.14	.01	0
Centrifugal Collector: high eff.	007	.68	.43	.18	.06	0	0
med. eff.	008	.44	.17	.03	.01	0	0
Elec. Precipitator: high eff.	010	.991	.984	.977	.962	.945	.918
med. eff.	011	.93	.89	.83	.71	.59	.45
low eff.	012	.90	.75	.60	.45	.22	.12
Fabric Filter: high temp.	016	.9999	.995	.982	.967	.958	.956

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16. ABSTRACT

In conjunction with the Regional Air Pollution Study being conducted in the St. Louis Air Quality Control Region (AQCR), a methodology for estimating the amount of sulfur trioxide (SO_3) emitted by combustion sources was developed. It is based on SO_2/SO_3 ratios determined both experimentally and from literature surveys. The most likely value appears to be 1.85% of the SO_2 emissions. On this basis, about 22,000 tons of SO_3 are emitted yearly from combustion sources

A fine particle size inventory for the area was also developed. The inventory gives a breakdown of particulate emissions in the range of 7 to .01 microns, based on production rates and collection efficiencies for point sources in the St. Louis AQCR. The information on the SO_2/SO_3 ratios and the particle size breakdown is stored in the RAPS Data Handling System.

17. KEY WORDS AND DOCUMENT ANALYSIS		
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