PHASE II REPORT

APPLICABILITY OF ORGANIC SOLIDS TO THE DEVELOPMENT OF NEW TECHNIQUES FOR REMOVING OXIDES OF SULFUR FROM FLUE GASES

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PHASE II REPORT

APPLICABILITY OF ORGANIC SOLIDS TO THE DEVELOPMENT

OF

NEW TECHNIQUES FOR REMOVING OXIDES OF SULFUR FROM FLUE GASES

by

R. A. Meyers, A. Grunt and M. Gardner

Prepared for:

National Air Pollution Control Administration

Under Contract No. PH 22-68-46

1 September 1969

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1.0 INTRODUCTION AND SUMMARY

This Phase II addendum report describes the laboratory work performed by TRW Systems Group for the National Air Pollution Control Administration under Contract No. PH-22-68-46. The laboratory effort had the following objectives:

- Screen selected organic solids for ability to remove sulfur dioxide from synthetic flue gas, and
- Obtain more detailed data on the most promising organic solid (s)

As a result of a detailed literature search and conceptual analysis during Phase I of this program, a number of organic solids were identified as having capacity for the binding of sulfur dioxide. A principle accomplishment of this task was the identification of a number of waste materials which have high potential for removal of sulfur dioxide from flue gases. Organic materials were selected on the basis of:

- Known or postulated ability to take up sulfur dioxide,
- Price and availability,
- Thermal stability,
- Regenerability, and
- Mechanical properties.

Five organic materials were selected which offer promise for removing sulfur dioxide from flue gases:

1. Cellulosics,
2. Nigrosin,
3. Poly(N-vinylcarbazole),
4. Pyrolyzed poly(vinylchloride), and
5. Pyrolyzed poly(acrylonitrile).

The specific cellulosics selected were: waste newsprint, sawdust and cotton. Although a number of other inexpensive cellulosic based materials could have been evaluated, the limitations of Phase II required that the scope be narrowed.

As little or no data was available regarding the ability of the above-mentioned organic solids to sorb sulfur dioxide, a screening program was initiated for the assessment of the relative potential of the selected organic solids for the removal of sulfur dioxide from flue gases. The screening program involved determination of thermal stability at flue gas temperature (215°F and above) and ability to take up sulfur dioxide from moving flue gas streams at 215° to 300°F. All seven candidates (this includes the three cellulosics) were found to be thermally stable in flue gas, while all but nigrosin were found to take up substantial amounts of sulfur dioxide from synthetic flue gas streams. Of the solids which were evaluated, newsprint was found to be the most promising offering the following advantages over other sorbent systems for removal of sulfur dioxide:

- Selective sorption of sulfur dioxide from flue gas streams. (Little or no pickup of nitrogen, water carbon dioxide, oxygen, etc.).
- Low initial price (e.g., $3.00 per ton for office waste paper).
- The large scale use of waste cellulosics for sulfur dioxide removal could help solve a solid waste disposal problem.
- Ready availability of sorbent in areas proximate to power stations.
- Low attrition of sorbent (high mechanical strength, e.g., tensile strength of 80,000 psi compared with 65,000 psi for medium steel).
- Totally reversible sorption.
- Low temperature regeneration (350°F).

More detailed accumulation of data was performed on newsprint utilizing synthetic flue gases at temperatures encountered in flue gas streams (215° - 300°F). Both thermogravimetric analysis of weight increases of newsprint as a function of time in synthetic flue gas and chemical analysis of flue gas streams which had passed through packed beds of newsprint were performed to determine rate and capacity of newsprint for SO_2 sorption. In addition, regeneration studies were performed utilizing chemical analysis of desorption streams. The results may be summarized as follows:

- Newsprint picks up approximately 1-2% by weight sulfur dioxide in 20 minutes at a flow rate of ca., 200 vol/hr.
- Newsprint has a capacity of approximately 10% SO_2 by weight.
- Desorption streams of 16-26% by volume sulfur dioxide are obtained from newsprint at desorption temperatures of 350-450°F.

The results of the screening tests and more detailed accumulation of data, together with experimental procedures are presented in four sections:

2.0 Screening of Organic Solids,

3.0 More Detailed Accumulation of Data on Newsprint,

4.0 Appendix A - Thermogravimetric Analysis of SO_2 Sorption and

5.0 Appendix B - Sorption-Desorption Experiments - Analysis of Effluent Flue Gas

2.0 SCREENING OF ORGANIC SOLIDS

Five organic solids were identified in a detailed literature search and conceptual analysis which offered promise for removing sulfur dioxide from flue gas streams:

- Cellulosics
- Nigrosin
- Poly(N-vinylcarbazole) - PVK
- Pyrolyzed poly(vinylchloride) - PVC
- Pyrolyzed poly(acrylonitrile) - PAN

The specific cellulosics selected for this study were: waste newsprint, sawdust, and cotton. The chemical compositions of the above candidates as well as the postulated chemical and physical interactions with SO_2 are discussed in detail in the Phase I report. A sequential screening program was designed in order to determine which of the candidates should undergo more detailed characterization (Figure 1).

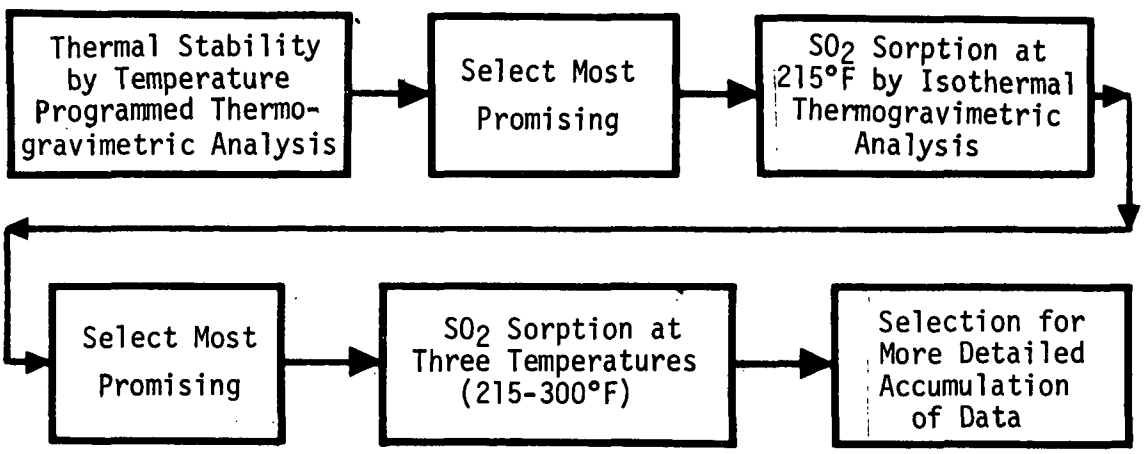

FIGURE 1. SCREENING OF ORGANIC SOLIDS

Thermogravimetric analyses in air and in synthetic flue gas were identical (Figure 2) and showed that all of the candidates were stable at flue gas temperatures. Two types each of pyrolyzed PVC and Nigrosin were evaluated.

The next sequential screening step involved measurement of weight uptake by temperature programmed thermogravimetric analysis in a synthetic flue gas. The flow rate was constant at 100 cc/min and it was assumed that weight uptake was due to sulfur dioxide (this was verified for newsprint and sawdust). All candidates were initially screened at 215°F. Poly(vinylcarbazole), sawdust, pyrolyzed poly(acrylonitrile), and newsprint picked up significant quantities of sulfur dioxide (Table 1). At this point, those organic solids which failed to pick up weight were eliminated (with the exception of cotton, as the cellulosics as a class had shown weight uptake to 215°F and it was thought possible that the cotton might show an improved weight uptake at elevated temperatures). In addition, poly(vinylcarbazole) was eliminated even though it took up sulfur dioxide because of its high price, e.g., in order for the PVK to be a candidate for further investigation, it would have had to show a marked improvement over the other far less expensive organic solids.

The next screening step involved determination of sulfur dioxide uptake at four temperatures (Table 2). Cotton, newsprint, sawdust, and pyrolyzed PAN picked up weight at temperatures of 215-300°F. The cellulosics did not pick up weight in some of the temperature regions, probably due to the operability of more than one sorption mechanism (Eqs. 1 and 2). Table 3 shows the regenerable uptake of SO_2 by newsprint and sawdust as compared with activated charcoal.

CHARGE TRANSFER

$$\text{CELLULOSE} + n\,SO_2 \rightleftharpoons \text{CELLULOSE}\cdot(SO_2)_n$$

$$K_{equil} = \frac{[\text{CELLULOSE}\cdot(SO_2)_n]^n}{[\text{CELLULOSE}]\,[SO_2]^n} \qquad (1)$$

CHEMICAL REACTION

$$\text{CELLULOSE}\text{-}(OH)_n + nSO_2 \rightleftharpoons \text{CELLULOSE}\text{-}(OSO_2H)_n$$

$$K_{equil} = \frac{[\text{CELLULOSE}\text{-}(OSO_2H)_n]^n}{[\text{CELLULOSE}\text{-}(OH)_n]\,[SO_2]^n} \qquad (2)$$

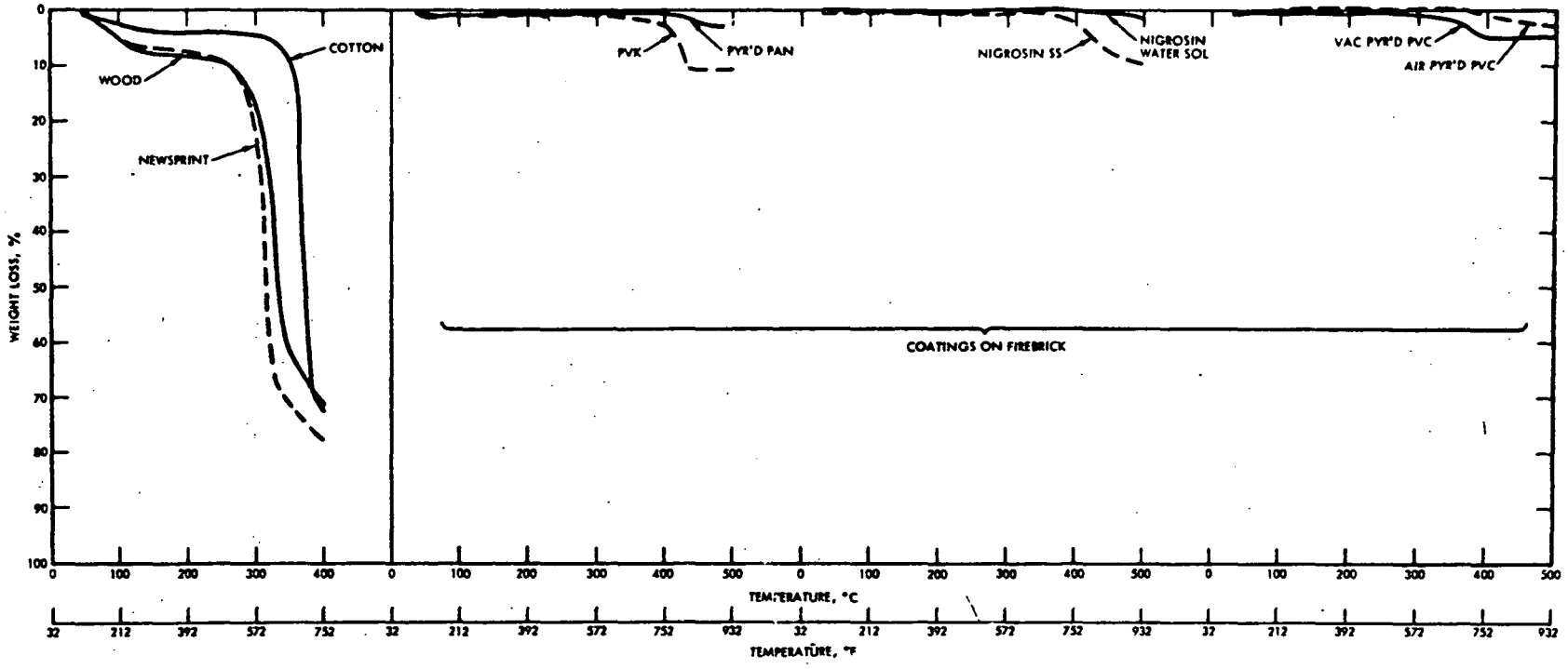

FIGURE 2. Thermogravimetric Analyses of Organic Solids in Air or in Synthetic Flue Gas

TABLE 1

SO_2 SORPTION AT 215°F OF ORGANIC SOLIDS BY ISOTHERMAL THERMOGRAVIMETRIC ANALYSIS IN SYNTHETIC FLUE GAS[a]

ORGANIC SOLID	% UNCORRECTED WEIGHT UPTAKE OF SO_2[b,c] in 3 hr. periods
Newsprint	1.9
Sawdust	3.0
Cotton	-0-
Pyrolyzed PVC	2.2
Pyrolyzed PAN	-0-
Poly(N-vinylcarbazole)	3.0
Nigrosin	-2.0

[a]The synthetic flue gas with composition: 0.57% SO_2, 0.07% NO_2, 18.07% CO_2 and 2.80% O_2 was humidified by passage through a humidifier maintained at 40°C. Flow rate was 100 cc/min.

[b]No correction was made for the slow thermal loss of the organic solids maintained at 215°F.

[c]Weight uptake taken to be due entirely to SO_2.

TABLE 2

SO_2 SORPTION AT FOUR TEMPERATURES OF ORGANIC SOLIDS BY ISOTHERMAL THERMOGRAVIMETRIC ANALYSIS[a]

ORGANIC SOLID	TEMP ^oF	UNCORRECTED % WEIGHT UPTAKE OF SO_2[b] in 3 hr. time periods	
Newsprint	215	1.9	2.6[c]
	250	-0-	0.9[c]
	275	1.8	2.8[c]
	300	-0-	1.0[c]
Cotton	215	-0-	
	250	0.5	
	275	-0-	
	300	0.4	
Sawdust	215	3.0	
	250	-0-	
	275	-0-	
	300	-0-	
Pyrolized PAN	215	2.2	
	250	1.9	
	275	3.2	
	300	1.4	

[a]The synthetic flue gas with composition: 0.57% SO_2, 0.07% NO_2, 18.07% CO_2 and 2.80% O_2 was humidified by passage through a humidifier maintained at 40°C. Flow rate was 100 cc/min.

[b]Weight uptake taken to be due entirely to SO_2.

[c]Corrected weight %, correction factors determined from isothermal runs in nitrogen: + 0.23%/hr. at 215°F, + 0.30%/hr. at 250°F, + 0.32%/hr at 275°F + 0.33%/hr. at 300°F.

TABLE 3. SO_2 Uptake of Cellulosics and Charcoals

Sorbent	Surface Area m^2/g	Initial SO_2 Uptake [a, d]		Regeneration I [a, b, d]		Regeneration II [a, b, d]	
		g/g sorb. x 10^{-2}	g/m^2 sorb. x 10^{-2}	g/g sorb. x 10^{-2}	g/m^2 sorb. x 10^{-2}	g/g sorb. x 10^{-2}	g/m^2 sorb. x 10^{-4}
Sawdust	0.5[c]	5.9	11.8	4.0	8.0	4.3	8.6
Newsprint	1.3[c]	4.0	3.1	4.6	3.5	5.0	3.9
Activated Charcoal	1000 (Approx)	7.9	.0079	--	--	--	--
Carbon Black	--	0	--	--	--	--	--

[a] 30% SO_2 in N_2 stream through sorbent at 215°F space velocity approximately 25 hr^{-1} (neglecting volume occupied by sorbent)

[b] N_2 stream at 350°F for 5 minutes, space velocity approximately 250 hr^{-1} (neglecting volume occupied by sorbent)

[c] BET method with Krypton

[d] SO_2 uptake was determined by first passing a very small amount of N_2 gas through material at room temperature to remove unsorbed and loosely bonded SO_2, then flushing with N_2 at 350°F passing effluent through std. base and back titrating with H_2SO_4.

3.0 MORE DETAILED ACCUMULATION OF DATA ON NEWSPRINT

At this point, newsprint was selected as the prime candidate due to the fact that it had a leading weight uptake and was, by far, the least expensive and most easily obtainable in quantity of all the organic solids in contention. More detailed determination of rate of sorption of sulfur dioxide as a function of temperature and sulfur dioxide concentration was determined for newsprint by thermogravimetric analysis (Table 4).

It should be indicated that while the TGA technique, as applied to cellulosics for SO_2 uptake, is exploratory in nature, a definite increase in weight of cellulosics in synthetic flue gas is noted, which clearly shows affinity of the specific cellulosic materials for SO_2 at flue gas temperature.

The results indicate that paper picks up sulfur dioxide from flue gas streams containing 0.57-2.0% v/v sulfur dioxide at temperatures of 215-302°F with an ultimate capacity for sulfur dioxide of 10.2% by weight. The rate data shown in the tables were obtained under diffusion limited conditions at approximately 10 $vol/\underline{hr}^{-1}$ wherein the laminar layer about the paper sample greatly influences the rate of diffusion controlled sorption of SO_2.

Sorption and desorption profiles were determined for packed beds of shredded newsprint. Synthetic flue gas was passed through a packed bed of paper maintained at 215°F at a velocity of 180 vols/hr for a period of 20 minutes. The sorption of SO_2 from the stream was followed by chemical analysis of the effluent flue gas. The results are shown in Figures 3 and 4. The figures show initial rapid uptake of sulfur dioxide during the first five to six minutes of sorption followed by a slower but indeed, significant uptake out to the termination of the experiment.

Desorption of sulfur dioxide from newsprint was found to give 22-26% v/v SO_2 in the desorption gas stream at 450°F and 16.5% maximum percent SO_2 v/v in the desorption gas stream at 350°F (See Table 5).

TABLE 4

DETERMINATION OF RATE OF SORPTION OF SULFUR DIOXIDE BY NEWSPRINT AS A FUNCTION OF SULFUR DIOXIDE CONCENTRATION AND TEMPERATURE

RUN NO.	SO_2 CONTENT	TEMP., °F	CORRECTED [a, b] $\Delta W/\Delta t$, mg/hr	TOTAL % WEIGHT UPTAKE OVER 7 HOUR PERIOD
1	0.57%	216	1.74	---
2	(0.0149 mg/ml)	248	2.00	---
3		302	0.62	---
4		216	2.34	---
5	1.0%	248	0.81	---
6	(0.0262 mg/ml)	302	0.38	---
7	2.0%	216	4.20	---
8	(0.0524 mg/ml)	248	1.77	---
9		302	1.59	---
10	0.57% (0.0149 mg/ml)	216	2.48	---
11		216	----	10.2[d]

[a] Correction factors for paper weight loss at temperature, determined in dry nitrogen (+ 0.67 mg/hr at 215°F, + 0.87 mg/hr at 248°F and + 0.95 mg/hr at 302°F).

[b] 100 cc/min flue gas flow for three hour time period the rate remains constant. Synthetic flue gas passed through humidifier at 40°C.

[c] Weight newsprint, approx. 290 mg.

[d] This sample was exposed to flue gas until rate leveled off to zero for ultimate capacity determination.

TABLE 5

DESORPTION OF SO_2 FROM NEWSPRINT

DESORPTION - RUN 1[a] (450°F)				*DESORPTION - RUN 2[a] (450°F)*				*DESORPTION - RUN 3[a] (350°F)*			
Sample	Time of Flow, Min	% Volume SO2 in Effluent Gas	SO_2 (mg)	Sample	Time of Flow, Min	% Volume SO2 in Effluent Gas	SO_2 (mg)	Sample	Time of Flow, Min	% Volume SO2 in Effluent Gas	SO_2 (mg)
Preflow	(Expansion)	26.49	115.17	Preflow	(Expansion)	21.90	57.50	Preflow	(Expansion)	16.50	40.00
1	1	4.33	16.48	1	1	13.00	20.58	1	1	12.30	15.58
2	2	1.08	13.23	2	1	9.20	17.82	2	1	9.60	16.10
3	2	0.36	2.24	3	1	4.80	10.43	3	1	3.60	7.74
4	1	0.47	1.50	4	1	1.70	4.10	4	1	4.10	8.48
5	1	1.27	0.54	5	1	1.00	2.30	5	1	0.90	2.08
6	1	0.98	0.86	6	1	1.50	3.62	6	1	1.70	3.94
7	1	0.45	0.45	7	1	0.70	1.66	7	1	0.70	1.63
8	1	0.36	1.28	8	1	1.10	2.43	8	1	0.80	1.76
9	1	0.32	0.38	9	1	0.70	1.44	9	1	0.60	1.31
10	1	0.28	0.29	10	1	0.60	1.47	10	1	0.70	1.38
11	1	0.17	0.19	11	1	0.50	1.12	11	1	0.50	1.06
12	1	0.11	0.48	12	1	0.80	1.66	12	1	0.50	1.06
13	1	0.13	0.29	13	1	0.50	1.12	13	1	0.30	0.80
				14	1	0.20	0.70	14	1	0.40	0.90
				15	1	0.20	0.70	15	1	0.30	0.80

[a]Conditions

	Run 1	Run 2	Run 3
Col. length	8 ft	8 ft	8 ft
Ullage	102 cc	102 cc	102 cc
Total Volume	109 cc	110 cc	111 cc
Weight Newsprint	9.73 g	8.32 g	8.32 g
Pulp Density	1.28 g/cc	1.04 g/cc	1.04 g/cc
Regen. Temperature	450°F	450°F	350°F

SO_2 sorption stream for these experiments was a 30% SO_2 in nitrogen mixture.

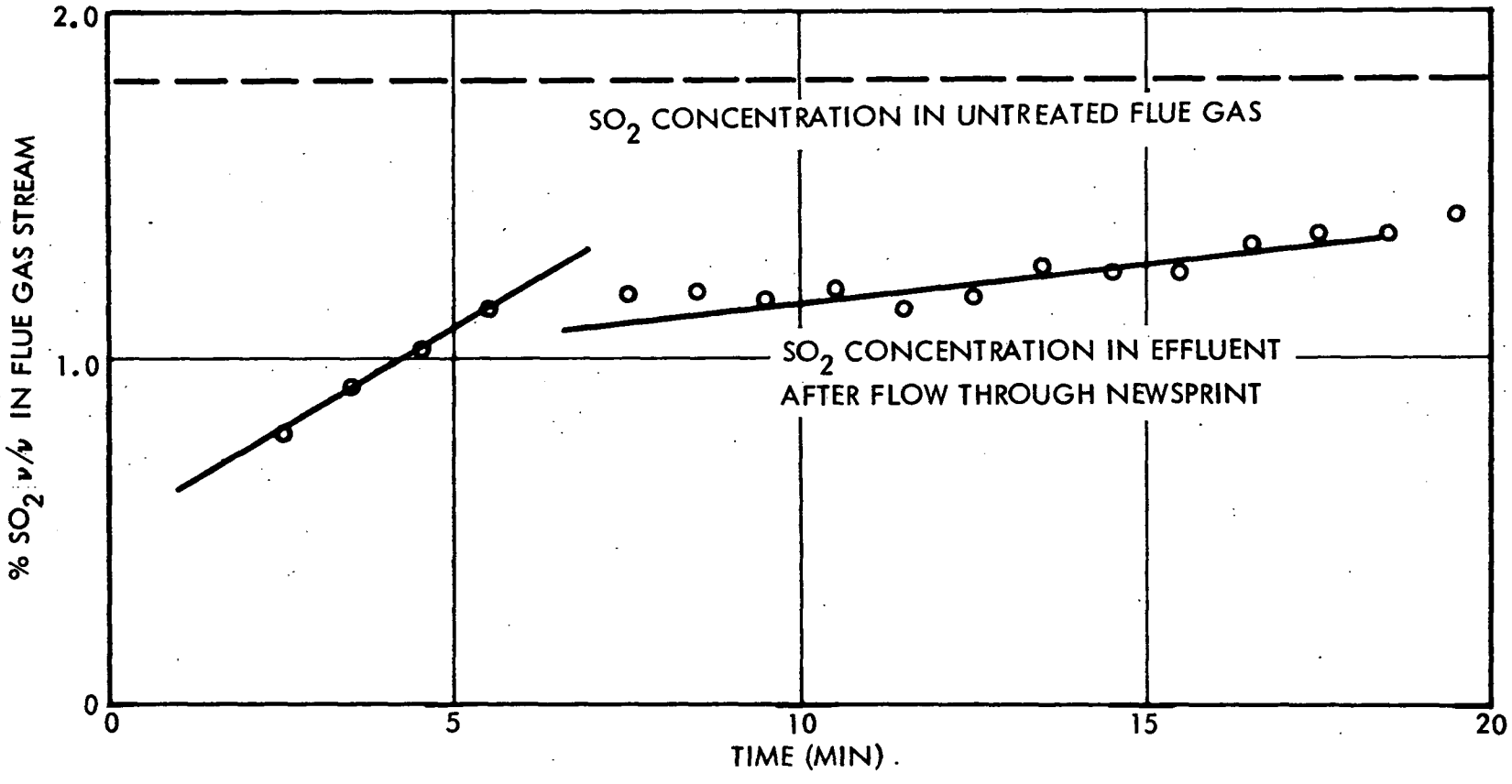

FIGURE 3. SO_2 CONCENTRATION IN FLUE GAS EFFLUENT

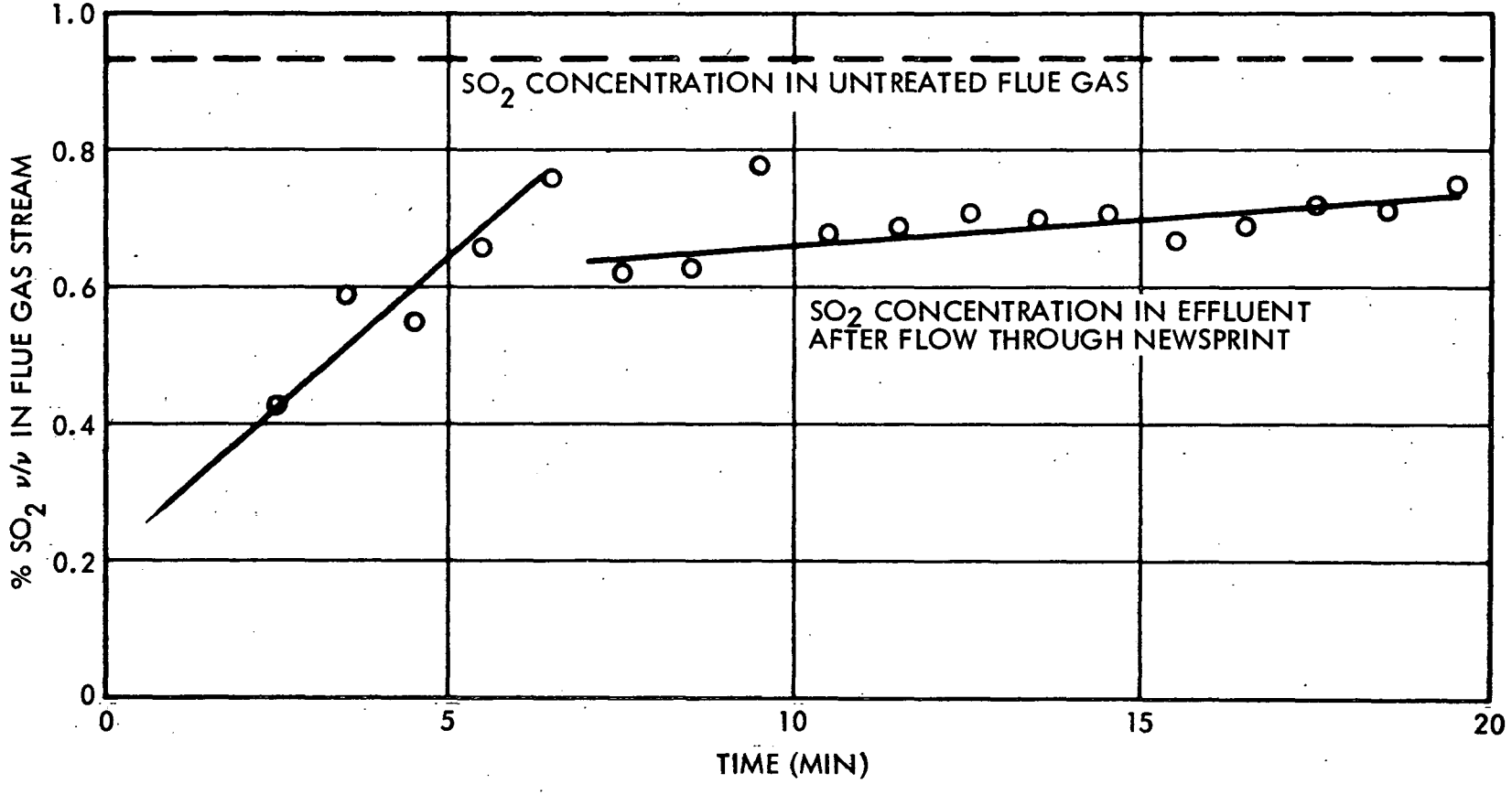

FIGURE 4. SO_2 CONCENTRATION IN DRY GAS EFFLUENT

4.0 APPENDIX A - THERMOGRAVIMETRIC ANALYSIS OF SO_2 SORPTION

A thermogravimetric analysis apparatus (TGA) was employed to monitor the changes in weight of samples of newsprint as a simulated "flue gas" was passed over the paper under isothermal conditions. The rate of weight gain was recorded as a function of time on an X-Y recorder. The weight gain data was employed to determine sorption rates at various temperatures.

An Aminco TGA was employed for the measurements of weight change. The simulated flue gas was prepared by passing a dry gas of the proper composition through a humidification chamber, where the gas stream was saturated with water vapor at the temperature maintained by the surrounding oven (40°C in this study). The lines from the oven to the TGA apparatus were insulated and heated to a temperature above the temperature maintained by the oven in order to prevent condensation of water in the lines. The gas flow into the humidification chamber was maintained at 100 cm^3 per minute by a metering valve. This flow rate provided a quantity of SO_2 which was greatly in excess of the quantity sorbed in any given time period.

The standard sample holder (Figure 5) was used for screening, while a special sample holder (Figure 6) was fabricated for the TGA balance for more detailed accumulation of data experiments. This holder is essentially a glass cage and suspension rod designed to maintain the sample in the desired cylindrical geometry throughout the experiment. The gas flow enters the furnace tube from the inlet tube at the bottom, passes around the sample, and exists from the apparatus.

The synthetic flue gas mixtures containing 1% and 2% SO_2 were prepared from the stock mixture purchased from Matheson, which has been described above, by adding down pressures of pure SO_2 to a storage tank, then filling to the required final pressure with the purchased flue gas mixture containing 0.57% of SO_2. A sufficient quantity of gas mixture was prepared at each SO_2 concentration to complete the scheduled runs at that composition. This method of preparing the synthetic flue gas supply was preferred to the method of mixing metered flows of component gases because of the greater certainty of maintaining a known gas composition throughout each experimental run.

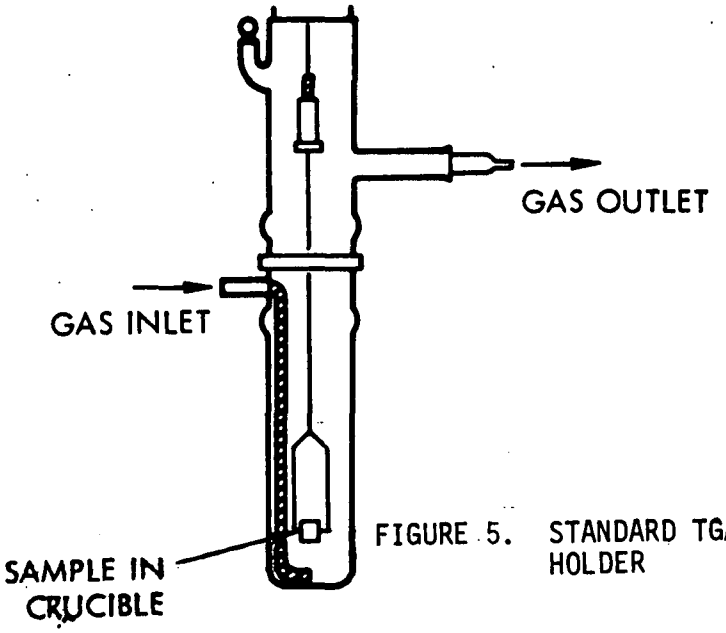

FIGURE 5. STANDARD TGA SAMPLE HOLDER

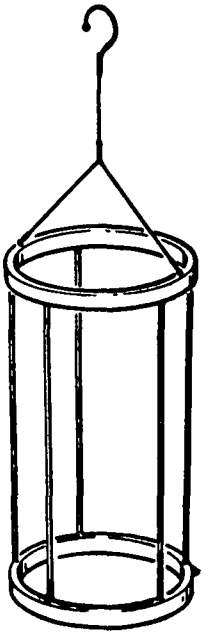

FIGURE 6. QUARTZ CAGE FOR ISOTHERMAL TGA EXPERIMENTS

The samples were rectangular pieces of newsprint 70. mm (2.8 inches) by 80. mm (3.1 inches). No preconditioning of any kind was carried out. Each sample weighed approximately 290 mg (6×10^{-4} lbs) with the exception of the capacity run for which a 550/mg sample was utilized.

Results

A series of 10 TGA runs of 3 hours duration were carried out at 102°C (115°F), 120°C (248°F), and 150°C (302°F) at the SO_2 concentrations of 0.57, 1.0 and 2.0 volume percent. In addition to these runs, a single run of 10 hours duration was carried out at 102°C and an SO_2 concentration of 0.57 volume percent. Data from all eleven TGA runs are tabulated in Table 4.

Correlation of rate of weight gain with increasing SO_2 concentration was generally masked by variations in paper surface characteristics such as, variations in amount of ink. Consequently, it was evident that further treatment of the data, such as calculation desorption kinetics or activation energies was not warranted.

The approximate capacity of the newspaper form of cellulosic was determined by TGA in run #11. For this determination, sample size was maximized (551 mg) maintaining 100 mg full scale on the TGA apparatus balance.

5.0 APPENDIX B - SORPTION-DESORPTION EXPERIMENTS - ANALYSIS OF EFFLUENT FLUE GAS

Experimental work was performed on packed columns of paper pulp. The sorption profiles reveal large initial sorption with lower level sorption continuing for the duration of the 20-minute runs. The desorption profiles indicate that a high percentage of the sorbed SO_2 is desorbed by a thermal mechanism and that repeated use of the columns are possible.

Sorption Experiments

The test apparatus utilized in the sorption experiments is presented schematically in Figure 7. The system consisted of two cylinders containing N_2 gas and synthetically-blended flue gas, respectively, a Fischer-Porter flowmeter, a metering valve, a humidifier (omitted for Run Number 2), a two-way valve, the sorption column system, the column by-pass system, an oven with an upper temperature limit of 400°C, a two-way stopcock, two pyrex glass scrubber traps, a bubble meter, and the necessary stainless steel lines and valves to construct the diagrammed system. The sorption experiments were conducted by the following procedure:

a. The system was constructed as diagrammed.

b. The oven temperature was allowed to reach equilibrium at the required sorption temperature.

c. The flow rate was established using flue gas through the by-pass system to determine approximate pressure and valve settings.

d. The scrubber traps were filled with 150 cc of 3% H_2O_2 solution.

e. The column valves are opened and the flue gas flow is diverted through the column system. Timing begins when flow is seen in the scrubber system.

f. Utilizing the two-way stopcock, the flue gas flow from the column is diverted to a fresh scrubber trap at one-minute intervals. During the one-minute periods, the scrubber trap is removed from the system, the sample recovered and fresh peroxide replaced in the trap. Once during each interval, a flow rate reading is taken.

g. Sampling continues at one-minute intervals for 20 minutes. The samples are analyzed by the procedure given in the following paragraph.

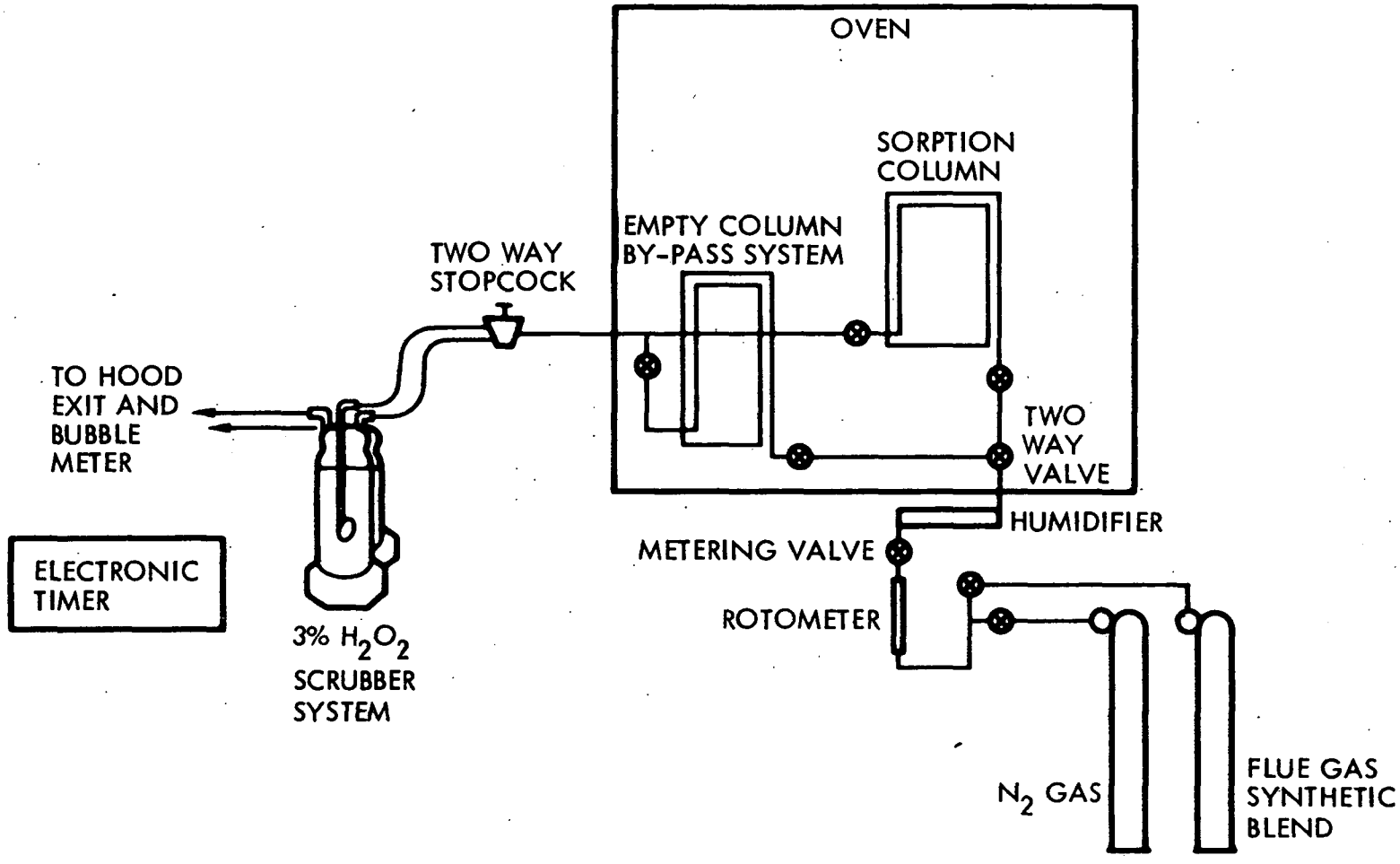

FIGURE 7. Sorption Test Apparatus

The samples obtained in Step f. above are analyzed by the following procedure:

a. The solutions are rinsed from the scrubbers into 250 cc beakers.

b. The solutions are reduced in volume from 150 cc to approximately 5 cc by boiling.

c. 4 cc of acetone and 3 drops of Sulfonazo III indicator solution is added to the solution.

d. The resulting solution is titrated with 0.01 N $Ba(ClO_4)_2$ solution to a blue end point.

e. Calculations are as follows:

A. $(\text{mg } SO_2 \text{ recovered}) = T \times N \times 64.0$

where:

T = $Ba(ClO_4)_2$ titer (ml)

N = Normality of $Ba(ClO_4)_2$ solution.

B. $$\% \text{ Vol } SO_2 = \frac{\frac{A}{2.927}}{\frac{A}{2.927} + C}$$

where:

A = mg SO_2 recovered

C = Volume of gas passed through the system in the one-minute period

2.927 = SO_2 gas density mg/cc.

Desorption Experiments

The desorption apparatus is presented schematically in Figure 8. The system consists of two cylinders containing N_2 gas and 30% SO_2 - N_2 gas blend respectively, an oven capable of 400°C temperature, desorption column systems, a two stopcock, dual pyrex glass scrubbers, a marionette bottle equipped with a two-way stopcock and a graduated cylinder. The desorption experiments were conducted by the following procedure:

a. The oven was allowed to reach thermal equilibrium at approximately 102°C.

b. A flow of 30% SO_2 gas blend was initiated through the column and maintained at 25 column volumes per hour for 90 minutes. Saturation is assumed.

c. The column is reduced to ambient temperature and 200 cc of gas displaced with N_2 utilizing the scrubber system. A sample is taken to determine the percentage of ellage swept by this procedure.

d. The column is isolated and the oven allowed to thermally equilibrate at desorption temperature.

e. When the column is at temperature, the valve connecting column and scrubber system is opened and the gas allowed to expand through the scrubber.

f. A slow N_2 gas flow is initiated and 15 samples are obtained at one-minute intervals as described in this sorption procedure. The volume of N_2 passed per minute is recorded by reading the graduated cylinders at the completion of each one-minute sample period.

g. The samples are analyzed and SO_2 recovered calculated by the methods given for the sorption procedure.

The results for the sorption runs are given in Figures 3 and 4. The results are reported in % vol/vol SO_2 as a function of time.

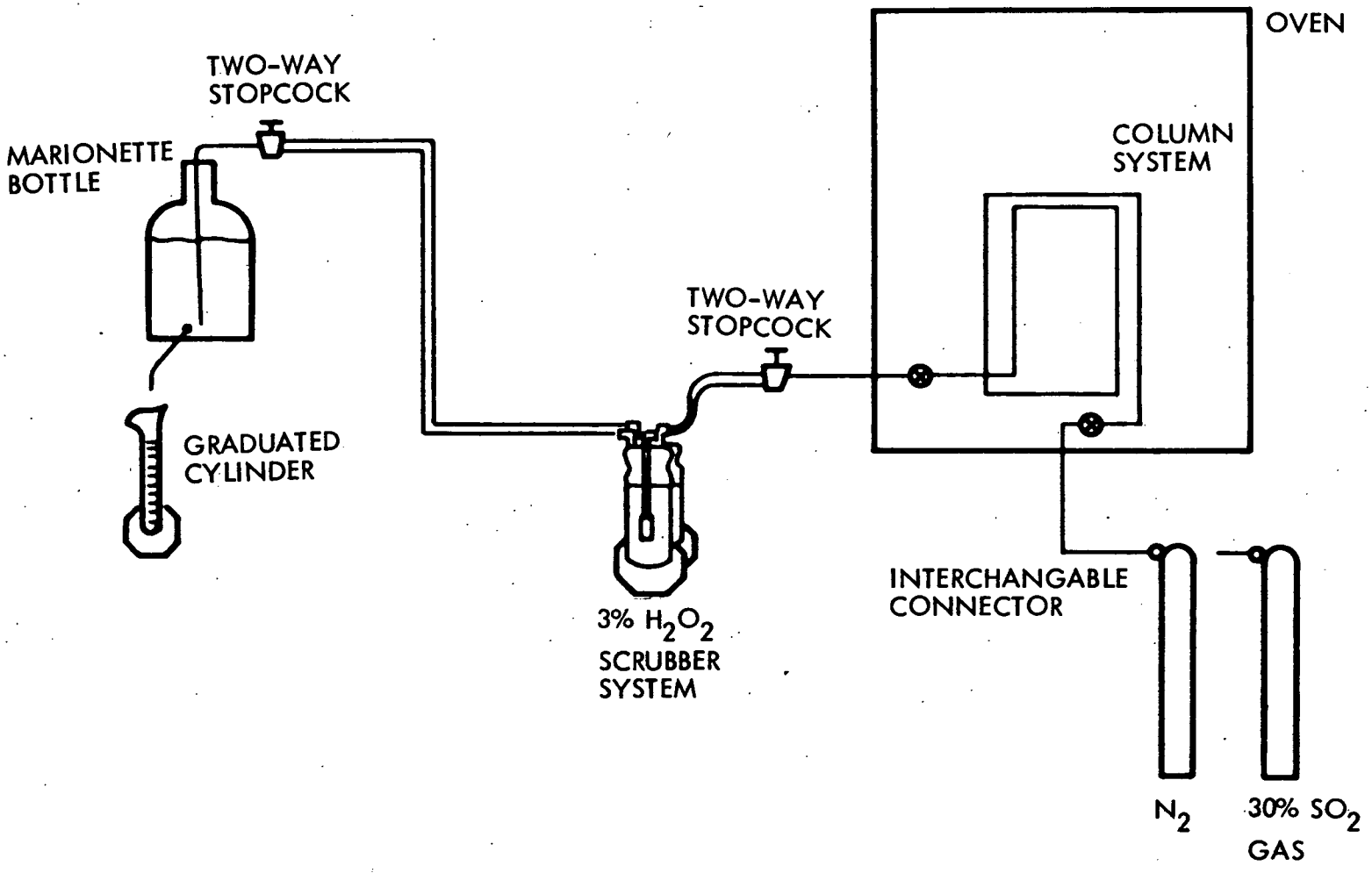

FIGURE 8. DESORPTION TEST APPARATUS