

SN 96032.007

EMB Test Number 73-CBK-1

**Phillips Petroleum Company
Carbon Black Division
Toledo, Ohio**

**by
T.E. Eggleston**

July 1973

TRW / TRANSPORTATION &
ENVIRONMENTAL
OPERATIONS

Contract Number 68-02-0235

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TRW Transportation and Environmental Operations
Westgate Park
7600 Colshire Drive
McLean, Virginia 22101

Contract Number 68-02-0235

PREFACE

The work reported herein was conducted by TRW Environmental Services pursuant to the terms of Task Order No. 5, Contract No. 68-02-0235 with the Environmental Protection Agency. Mr. T. E. Eggleston served as Project Chief and Crew Leader. Mr. Winton E. Kelly, Office of Air Programs, Emission Testing Branch served as Project Officer. Mr. Leslie Evans, Office of Air Programs, Industrial Studies Branch served as Project Engineer.

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II. INTRODUCTION

Under the Clean Air Act of 1970, as amended, the Environmental Protection Agency is charged with the establishment of performance standards for new or modified stationary sources which may contribute significantly to air pollution. A performance standard is based on the best emission reduction systems which have been shown to be technically and economically feasible.

In order to set realistic performance standards, accurate data on pollutant emissions must be gathered from the stationary source category under consideration.

Phillips Petroleum Company's Toledo Philblack plant at Toledo, Ohio, was designated as a well-controlled stationary source in the Petrochemical Industry and was thereby selected by OAQPS for an emission testing program. Testing was conducted on the No. 1 off-gas boiler during the period April 9-12, 1973, by TRW Environmental personnel. The No. 1 Boiler is one of five units that handle the off-gas from two carbon black production lines at the Toledo Philblack plant. During testing, the boiler was operated such that a maximum amount of off-gas from Line 2 and a minimum amount of auxiliary natural gas were used.

Samples were collected simultaneously before and after the boiler to determine filterable and total particulates, sulfur dioxide, hydrogen sulfide, nitrogen oxides, carbon monoxide, and total and non-methane hydrocarbons.

III. SUMMARY AND DISCUSSION OF RESULTS

General

During the test series, the process operation was maintained at normal operating levels. No major upset or change was encountered. The baghouse and boiler were also operating normally.

Particulates

In reference to Table 1 for the inlet, its average emission concentration was calculated to be .1363 gr/DSCF. The emission concentration for Table 2, which is for the outlet, was calculated to be .0189 gr/DSCF. These values are for the total particulate catch. The average removal efficiency of the baghouse was computed to be 99.44 percent for the total particulate catch.

Test ABI-1 at the boiler inlet was discontinued after 70 minutes of the 180 minutes initially planned due to heavy snow and wind. This was an outdoor location without protection from the weather. Because of this, the data is not included in the summary of results.

Test ABE-3 was aborted due to a broken probe liner. The test was repeated at both locations the same day.

The testing for particulate matter was conducted under extremely difficult conditions, especially at the inlet location, but the conditions were not felt to have significantly affected the results.

The percent isokinetic values are above the allowable 110 percent as given in Method No. 5, Federal Register, December 23, 1971. However, due to the small size of the particles, this should have no significant effect on the results. The main reason for high isokinetic values was in the estimation of the moisture in the flue gas.

Test ABI-4 was interrupted for approximately 45 minutes due to another broken probe liner. The liner was broken while being moved from a horizontal sampling configuration to a vertical situation. The broken liner was removed, carefully cleaned and replaced with a new liner. During the vertical traverse, a plugging in the train was noticed approximately 15 minutes into the traverse. The problem was traced to a piece of scale that had lodged in the nozzle tip. The piece of scale was not included in the sample recovery, as this was felt to be an extremely unusual or "freak" occurrence.

A glass bottle from ABE-1 was broken and the amount of water collected was estimated from that picked up in other similar runs. The percent moisture in Test ABI-2 appears to be low when compared to other runs.

Hydrogen Sulfide

The emission rate values for the inlet and outlet are tabulated in Table 3. The average emission rate was determined to be .321 gr/DSCF or .332 lb/hr at the inlet testing location. No results were obtained for the testing at the boiler outlet because all samples that were collected exhibited irregular behavior during collection and analysis. During the sample collection, the reagent used in the impinger was expended, while none of the characteristic yellow precipitate was formed. During the analysis procedure, an excessive amount of iodine was required to react with the collected sample. Because of these difficulties the H_2S concentration at the boiler outlet could not be determined. One test was attempted with an isopropanol prescrubber, but the same results occurred. Some of these samples were retained by EPA for further investigation.

Sulfur Dioxide

The Table 4 of this summary contains the results from the sulfur dioxide testing. The average emission value for SO_2 was found to be 5.4×10^{-5} lb/DSCF or .793 lb/hr at the outlet location. No sulfur

dioxide was detected at the inlet since the rate was below the minimum detectable limit of 7×10^{-6} lb/DSCF or 45 ppm.

A sulfur balance around the boiler indicates that the sulfur as SO_2 at the outlet is more than 100 percent of the sulfur as H_2S at the boiler inlet.

Nitrogen Oxides

Table 5 is a summary of the results collected from the testing for nitrogen oxides. The average emission rates for the inlet were found to be 1.2×10^{-6} lb/DSCF or .0104 lb/hr while for the outlet the values were found to be 8.6×10^{-6} lb/DSCF and .126 lb/hr.

The values for nitrogen oxides are very consistent. The only problem encountered was on one sample from the inlet, this was 0 and was not included in the average emission rates for NO_x .

Hydrocarbons

The results from the testing for hydrocarbons are compiled in Table 6. The average emission rates for the inlet were found to be 10,000 ppm and 1826 lb/hr total hydrocarbons. The outlet hydrocarbon results show that the average emission rate is 72 ppm or 26.2 lb/hr and the non-methane hydrocarbons were found to be 34.2 ppm or 12.4 lb/hr. These analyses were completed utilizing a gas chromatograph. In these results CO was not reported as a hydrocarbon.

Some grab samples were taken in glass bottles, and the results from these samples confirmed those taken in the bags with one exception. No hydrogen was found in the bags, but small quantities were found in the bottles; however, quantitative analysis was not possible. Two samples and analyzed promptly on site by Phillips Petroleum indicated hydrogen at the inlet to be in the range of 15 percent. From the Orsat summary results on Table 10, the hydrogen, due to its reactivity, was absorbed in the oxygen absorbant in runs ABE-1 and ABI-1. The high oxygen values resulted due to the presence of hydrogen. The samples that were not

analyzed immediately showed a low oxygen content. This indicates that hydrogen was the reason for the erratic behavior of the orsat results, since the hydrogen permeated through the sample bags before the correct orsat analysis could be executed.

The comparative gas chromatography analysis by Phillips Petroleum Laboratory shown in Table 9, shows a breakdown of constituents in two samples analyzed immediately after collection.

Particle Sizing

The summary results for particle sizing is located on Table 7 and the graphic summary is on the following page. From the three tests, all conducted on the outlet, the results were plotted and found to range from a low mass median diameter of .09 microns to a high of .86.

The results are slightly scattered, but this is not unusual when measuring sub-micron particles.

Visible Emission Results

The summary for the visible emissions is tabulated on Table 8. The visible emissions data was observed on the outlet which exhausted two identical boilers, therefore, the readings are on the combined plume of the two boilers. The maximum opacity observed was 10 percent, and in all observations, the average opacity was less than five percent.

Carbon Monoxide

Measurement of carbon monoxide at the boiler outlet by non-dispersive infrared technique was attempted, but levels were in excess of the limit of the instrument (500 ppm). The readings were approximately 2000 ppm which is less than the minimum detectable on the orsat.

In order to relate the gas stream measurements at the boiler inlet and outlet to the process operation, the proportional product rate to Boiler No. 1 is used. The mass emissions in pounds per hour are divided by the proportional product rates to get emission factors. These are summarized for each sampling location in Tables 11 and 12.

The particulate emission factor at the boiler inlet can be used to calculate the product recovery efficiency of the baghouse. Based on the probe and filter portion of the catch, the collection efficiency ranges from 99.45 - 99.84 percent, with an average of 99.67 percent.

When the total catch is used, the collection efficiency ranges from 99.21 - 99.63 percent, with an average efficiency of 99.44 percent. In addition to the product recovery baghouse, the boiler itself exhibited a particulate removal efficiency. Based on the probe and filter catch, the particulate removal efficiency averaged 74.2 percent, while the removal efficiency averaged 76.2 percent based on the total particulate catch.

III. SUMMARY AND DISCUSSION OF RESULTS

TABLE 1
SUMMARY OF PARTICULATE RESULTS - INLET

| RUN NUMBER | ABI-2 | ABI-3 | ABI-4 | AVERAGE |
|--|---------|---------|---------|---------|
| Date | 4-11-73 | 4-12-73 | 4-12-73 | |
| Volume of Gas Sampled - DSCF ^a | 94.27 | 86.06 | 89.50 | 89.94 |
| Percent Moisture by Volume | 25.53 | 40.29 | 43.40 | 36.4 |
| Average Stack Temperature - °F | 423 | 430 | 430 | 427.7 |
| Stack Volumetric Flow Rate - DSCFM ^b | 8013 | 7218 | 7431 | 7554 |
| Stack Volumetric Flow Rate - ACFM ^c | 18026 | 20457 | 22221 | 20235 |
| Percent Isokinetic | 119 | 124 | 124 | 122 |
| Percent Excess Air | 5 | 4 | 4 | 4.3 |
| Total Product Rate - lb/hr. | 4262 | 4225 | 4225 | 4237 |
| Proportional Product Rate to Boiler 1 lb/hr. | 2620 | 2780 | 2750 | 2640 |
| <u>Particulates - probe, cyclone, and filter catch</u> | | | | |
| mg. | 628.5 | 383.3 | 1391.1 | 800.9 |
| gr/DSCF | .1040 | .0678 | .2373 | .1363 |
| gr/ACF | .0462 | .0239 | .0793 | .0498 |
| lb/hr | 7.1 | 4.2 | 15.1 | 8.8 |
| lb/lb product #1 Boiler | .00271 | .00160 | .00550 | .00327 |
| <u>Particulates Total Catch</u> | | | | |
| mg. | 1206.1 | 934.5 | 2004.5 | 1381.7 |
| gr/DSCF | .1996 | .1652 | .3420 | .2356 |
| gr/ACF | .0887 | .0583 | .1143 | .0871 |
| lb/hr | 13.7 | 10.2 | 21.8 | 15.23 |
| lb/lb product #1 Boiler | .00523 | .00376 | .00793 | .00564 |
| Percent Impinger Catch | 48.2 | 58.8 | 30.7 | 45.9 |

TABLE 2
SUMMARY OF PARTICULATE RESULTS - OUTLET

| RUN NUMBER | ABE-1 | ABE-2 | ABE-4 | AVERAGE |
|--|---------|---------|---------|---------|
| Date | 4-10-73 | 4-11-73 | 4-12-73 | |
| Volume of Gas Sampled - DSCF ^a | 114.37 | 87.44 | 104.69 | 102.17 |
| Percent Moisture by Volume | 31.18 | 32.02 | 31.52 | 31.57 |
| Average Stack Temperature - °F | 509 | 509 | 522 | 513 |
| Stack Volumetric Flow Rate - DSCM ^b | 14744 | 13718 | 15611 | 14691 |
| Stack Volumetric Flow Rate - ACFM ^c | 39799 | 37369 | 42822 | 39997 |
| Percent Isokinetic | 115 | 120 | 122 | 119 |
| Percent Excess Air | 170* | 20 | 16 | 18 |
| Percent Opacity | 3.75 | .58 | 1.64 | 1.99 |
| Total Product Rate - lb/hr. | 4264 | 4262 | 4225 | 4250 |
| Proportional Product Rate to Boiler 1 lb/hr. | 2420 | 2620 | 2780 | 2606 |
| Particulates - probe, cyclone, and filter catch | | | | |
| mg. | 113.0 | 103.0 | 151.4 | 122.5 |
| gr/DSCF | .0153 | .0186 | .0230 | .0189 |
| gr/ACF | .0057 | .0068 | .0084 | .0069 |
| lb/hr | 1.9 | 2.2 | 3.1 | 2.4 |
| lb/lb product #1 Boiler | .000785 | .000840 | .00113 | .000915 |
| Particulates Total Catch | | | | |
| mg. | 235.9 | 159.0 | 246.6 | 213.8 |
| gr/DSCF | .0319 | .0287 | .0375 | .0327 |
| gr/ACF | .0118 | .0105 | .0137 | .0120 |
| lb/hr | 4.0 | 3.4 | 5.0 | 4.13 |
| lb/lb product #1 Boiler | .00165 | .00130 | .00182 | .00159 |
| Percent Impinger Catch | 52.5 | 35.3 | 38.0 | 41.9 |

Footnotes for Tables 1 and 2 -

^a Dry Standard Cubic Feet at 70°F, 29.92 in Hg.

^b Dry Standard Cubic Feet per minute at 70°F, 29.92 in Hg.

^c Actual Cubic Feet per minute.

* Percent Excess Air Value in error due to hydrogen present in Orsat sample.

TABLE 3. SUMMARY OF RESULTS: H₂S

| DATE | INLET | | | | OUTLET | | | |
|---------|---------|----------|-----|-------|---------|----------|---------|-------|
| | RUN NO. | EMISSION | | | RUN NO. | EMISSION | | |
| | | gr/dscf | ppm | lb/hr | | gr/dscf | ppm | lb/hr |
| 4-10-73 | HBI-1a | 0.464 | 754 | ----- | | | | |
| 4-11-73 | HBI-2a | 0.336 | 546 | 0.385 | | | | |
| | HBI-2b | 0.223 | 362 | 0.255 | | | | |
| | HBI-2c | 0.269 | 437 | 0.308 | | | | |
| | HBI-2d | 0.297 | 483 | 0.340 | | | | |
| | | | | | | N O | D A T A | |
| 4-12-73 | HBI-3a | 0.205 | 334 | 0.217 | | | | |
| | HBI-3b | 0.299 | 486 | 0.313 | | | | |
| | HBI-3c | 0.492 | 800 | 0.515 | | | | |
| | HBI-3d | 0.306 | 497 | 0.320 | | | | |
| AVERAGE | | 0.321 | 522 | 0.332 | | | | |

TABLE 4. SUMMARY OF RESULTS: SO₂

| DATE | INLET | | | | OUTLET | | | |
|---------|---------|----------------------------|-----|-------|---------|----------------------------|-----|-------|
| | RUN NO. | EMISSION | | | RUN NO. | EMISSION | | |
| | | lb/dscf x 10 ⁻⁵ | ppm | lb/hr | | lb/dscf x 10 ⁻⁵ | ppm | lb/hr |
| 4-10-73 | ----- | ----- | --- | ----- | SBE-1a | 5.9 | 336 | 0.870 |
| 4-11-73 | SBI-2a | *0 | 0 | 0 | SBE-2a | 4.7 | 284 | 0.645 |
| | SBI-2b | 0 | 0 | 0 | SBE-2b | 5.7 | 344 | 0.782 |
| 4-12-73 | SBI-3a | 0 | 0 | 0 | SBE-3a | 4.8 | 290 | 0.749 |
| | SBI-3b | 0 | 0 | 0 | SBE-3b | 5.9 | 356 | 0.921 |
| AVERAGE | | 0 | 0 | 0 | | 5.4 | 322 | 0.793 |

* 0 indicates below detection limit. For this testing, minimum detection is approximately 7×10^{-6} lb/dscf, or 45 ppm.

TABLE 5. SUMMARY OF RESULTS: NO_x

| DATE | INLET | | | | OUTLET | | | |
|---------|---------|----------------------------|------|--------|---------|----------------------------|------|-------|
| | RUN NO. | EMISSION | | | RUN NO. | EMISSION | | |
| | | lb/dscf x 10 ⁻⁶ | ppm | lb/hr | | lb/dscf x 10 ⁻⁶ | ppm | lb/hr |
| 4-10-73 | NBI-1a | 0.80 | 6.7 | ----- | NBE-1a | 8.9 | 74.8 | 0.131 |
| 4-11-73 | NBI-2a | 1.1 | 9.3 | 0.0088 | NBE-2a | 8.7 | 73.2 | 0.119 |
| | NBI-2b | 1.6 | 13.5 | 0.013 | NBE-2b | 8.9 | 74.8 | 0.122 |
| 4-12-73 | NBI-3a | 1.2 | 10.1 | 0.0088 | NBE-3a | 8.9 | 74.8 | 0.139 |
| | NBI-3b | 1.5 | 12.6 | 0.011 | NBE-3b | 7.5 | 63.1 | 0.117 |
| AVERAGE | | 1.2 | 10.4 | 0.0104 | | 8.6 | 72.1 | 0.126 |

TABLE 6. SUMMARY OF RESULTS: HYDROCARBONS

| DATE | INLET | | | | | OUTLET | | | | |
|---------|---------|--------------------|--------|--------------------------|--------|---------|--------------------|--------|--------------------------|--------|
| | RUN NO. | EMISSION | | | | RUN NO. | EMISSION | | | |
| | | TOTAL HYDROCARBONS | | NON-METHANE HYDROCARBONS | | | TOTAL HYDROCARBONS | | NON-METHANE HYDROCARBONS | |
| | | ppm* | lb/hr* | ppm* | lb/hr* | | ppm* | lb/hr* | ppm* | lb/hr* |
| 4-10-73 | ----- | ----- | ----- | --- | ---- | BBE-1a | 125 | 44.5 | 50 | 17.8 |
| 4-11-73 | BBI-2a | 10000 | 1937 | 0 | 0 | BBE-2a | 45 | 14.9 | 29 | 9.6 |
| 4-12-73 | BBI-3b | 10000 | 1770 | 0 | 0 | BBE-3a | 70 | 26.4 | 30 | 11.3 |
| | BBI-3c | 10000 | 1770 | 0 | 0 | BBE-3c | 50 | 18.9 | 27 | 10.2 |
| | ----- | ----- | ----- | --- | ---- | BBE-3d | 70 | 26.4 | 35 | 13.2 |
| AVERAGE | | 10000 | 1826 | 0 | 0 | | 72 | 26.2 | 34.2 | 12.4 |

* Hydrocarbons measured and calculated as Methane (CH₄).

TABLE 7
PARTICLE SIZING RESULTS*

| DATE | RUN NO. | MASS MEDIAN DIAMETER- μ |
|---------|---------|-----------------------------------|
| | | |
| 4-11-73 | PBE-1 | 0.86 |
| 4-12-73 | PBE-2 | 0.24 |
| 4-12-73 | PBE-3 | 0.09 |

*Summary plot of particle sizing results on following page.

TABLE 8
VISIBLE EMISSION RESULTS

| DATE | TIME | % Opacity | | |
|---------|-----------|-----------|---------|---------|
| | | Minimum | Maximum | Average |
| 4-10-73 | 1525-1650 | 0 | 5 | <5 |
| 4-11-73 | 1430-1500 | 0 | 5 | <5 |
| 4-12-73 | 1600-1630 | 0 | 10 | <5 |

TABLE 9
COMPARATIVE GAS CHROMATOGRAPHIC ANALYSIS*

| DATE | RUN NO. | % CO ₂ | % C ₂ H ₂ | % H ₂ | % CH ₄ | % CO | % N ₂ +Ar | % ATOM C |
|---------|---------|-------------------|---------------------------------|------------------|-------------------|------|----------------------|----------|
| 4-10-73 | BBI-2a | 4.60 | 1.24 | 15.5 | 0.94 | 13.1 | 64.7 | 21.1 |
| 4-10-73 | BBI-2b | 4.57 | 1.24 | 14.9 | 0.93 | 13.0 | 65.3 | 20.1 |

* Performed by Phillips Petroleum on site immediately after sample collection in glass.

TABLE 10. SUMMARY OF ORSAT DATA

| DATE | RUN NO. | %CO ₂ | %O ₂ | %CO | %N ₂ |
|---------|---------|------------------|-----------------|------|-----------------|
| 4-10-73 | ABI-1 | 4.4 | 15.5 | 11.2 | 68.9 |
| 4-10-73 | ABE-1 | 10.7 | 12.8 | 0 | 76.5 |
| 4-11-73 | ABI-2 | 4.2 | .6 | 7.7 | 87.5 |
| 4-11-73 | ABI-2a | 4.1 | 2.0 | 5.3 | 88.6 |
| 4-11-73 | ABE-2 | 10.2 | 4.0 | 0 | 85.8 |
| 4-11-73 | ABE-2a | 10.5 | 3.6 | 0 | 85.9 |
| 4-12-73 | ABI-3 | 4.4 | .5 | 13.4 | 81.7 |
| 4-12-73 | ABE-3 | 10.7 | 3.1 | 0 | 86.2 |
| 4-12-73 | ABE-3a | 10.7 | 2.3 | 0 | 87.0 |
| 4-12-73 | ABE-3b | 10.7 | 3.2 | 0 | 86.1 |

TABLE 11. PROCESS EMISSION RATES, LB/LB PRODUCT

| BOILER INLET | | | | | |
|---|---------|------------------------|------------------------|---------|------------------------|
| RUN NO. | 1 | 2 | 3 | 4 | AVERAGE |
| DATE | 4-10-73 | 4-11-73 | 4-12-73 | 4-12-73 | |
| <u>PARTICULATE</u> , Probe and Filter | - | 0.00271 | 0.00160 | 0.00550 | 0.00327 |
| Total | - | 0.00523 | 0.00376 | 0.00793 | 0.00564 |
| SULFUR DIOXIDE | - | 0 | 0 | 0 | 0 |
| HYDROGEN SULFIDE | - | 0.000123 | 0.000138 | - | 0.000130 |
| <u>NITROGEN OXIDES</u> (as NO ₂) | - | 4.2 x 10 ⁻⁶ | 3.6 x 10 ⁻⁶ | - | 3.9 x 10 ⁻⁶ |
| <u>HYDROCARBONS</u> (as CH ₄) Total* | - | 0.740 | 0.637 | - | 0.688 |
| Non-Methane | | 0 | 0 | - | 0 |
| CARBON MONOXIDE | - | 0.867 | 1.518 | - | 1.193 |

*These numbers do not include CO as a Hydrocarbon.

TABLE 12. PROCESS EMISSION RATES, LB/LB PRODUCT

| BOILER OUTLET | | | | | |
|---|---------------------------------------|-----------------------|-----------------------|---------|-----------------------|
| RUN NO. | 1 | 2 | 3 | 4 | AVERAGE |
| DATE | 4-10-73 | 4-11-73 | 4-12-73 | 4-12-73 | |
| <u>PARTICULATE,</u> Probe and Filter | 0.000785 | 0.000840 | ----- | 0.00113 | 0.000915 |
| Total | 0.00165 | 0.00130 | ----- | 0.00182 | 0.00159 |
| SULFUR DIOXIDE | 0.000360 | 0.000268 | 0.000300 | ----- | 0.000309 |
| HYDROGEN SULFIDE | ----- | ----- | ----- | ----- | ----- |
| <u>NITROGEN OXIDES</u> (as NO ₂) | 54.2×10^{-6} | 45.8×10^{-6} | 46.0×10^{-6} | ----- | 48.7×10^{-6} |
| <u>HYDROCARBONS</u> (as CH ₄) | | | | | |
| Total* | 0.0184 | 0.00570 | 0.00860 | ----- | 0.0109 |
| Non-Methane | 0.00735 | 0.00404 | 0.00417 | ----- | 0.00515 |
| <u>CARBON MONOXIDE</u> | In all cases CO is > 0.075 but < 0.30 | | | | |

*These numbers do not include CO as a Hydrocarbon.

CUMULATIVE WEIGHT PERCENT LESS THAN STATED MICRON SIZE

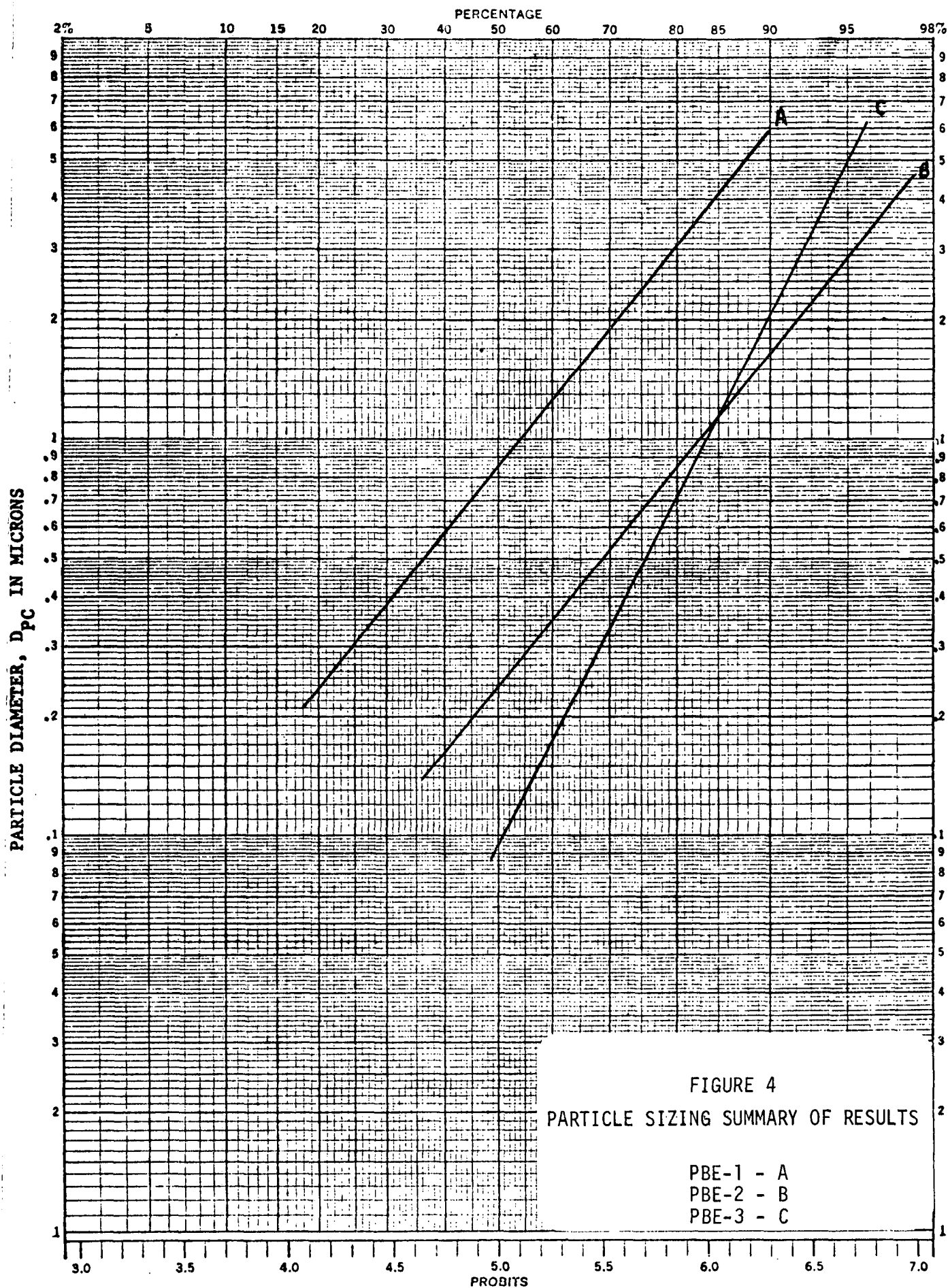


FIGURE 4
PARTICLE SIZING SUMMARY OF RESULTS

PBE-1 - A
PBE-2 - B
PBE-3 - C

IV. PROCESS DESCRIPTION AND OPERATION

The fundamental steps in carbon black manufacturing by the furnace process are as follows:

- A. Production of the black from feed stock.
- B. Separation of the black from the gas stream.
- C. Final conversion of the black to a marketable product.

In the furnace process, carbon black is produced by burning a mixture of gas and heavy aromatic oil. This feed is preheated and injected into a reactor with a limited supply of combustion air. The flue gases and entrained carbon from the reactor (furnace) are cooled by heat exchange against the reactor feed and water quenching. This stream (450-500°F) is then sent to bag filters for carbon black recovery. The recovered carbon black is transported to a finishing area by screw or pneumatic conveyors. In the finishing area the black is passed through a pulverizer to break up lumps. This produces a 5 to 12 lb/cu. ft. bulk density. This petuitizing is done with water in a paddle or pin type mixer. The resulting wet product (30-40 wt. percent water) is sent to driers. The dried product is then screened and sent to storage. Figure 1 presents a block flow diagram for carbon black manufacture by the furnace process. The major source of air pollution from the process is the process vent or "off-gas" shown as stream 4 on the diagram.

The Toledo Philblack plant has two independent process lines. The off-gases are combusted in two parallel CO-boilers and two indirect combustor driers. If the total heat available in the off-gas stream were used for these purposes, more steam would be generated than could be used in the plant. The excess off-gas is therefore incinerated and no unburned gases are vented except in an emergency.

The equipment tested was the No. 1 CO-boiler. The boiler was a standard Babcock and Wilcox small-tube, twin drum, water-tube boiler,

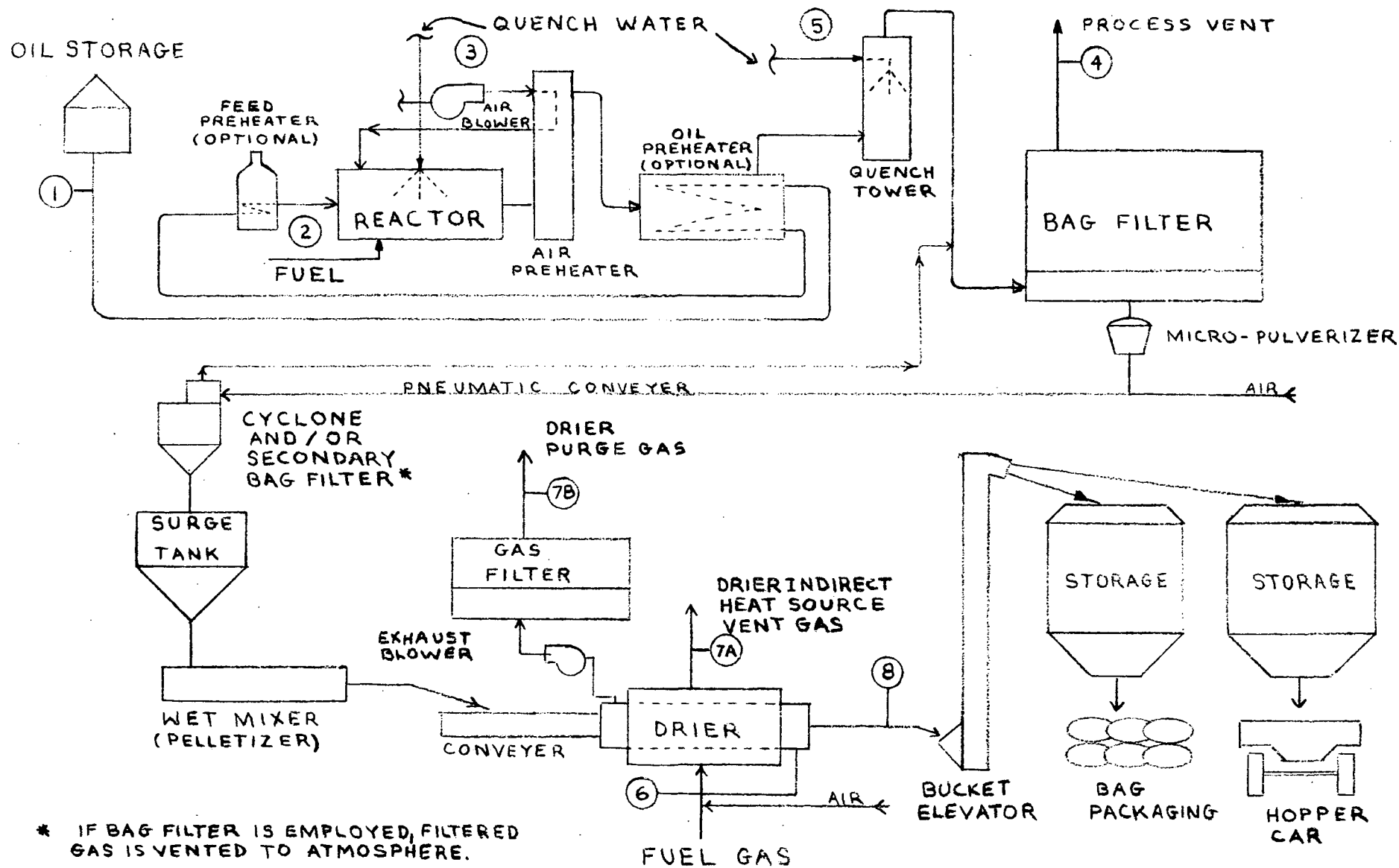


FIGURE 1

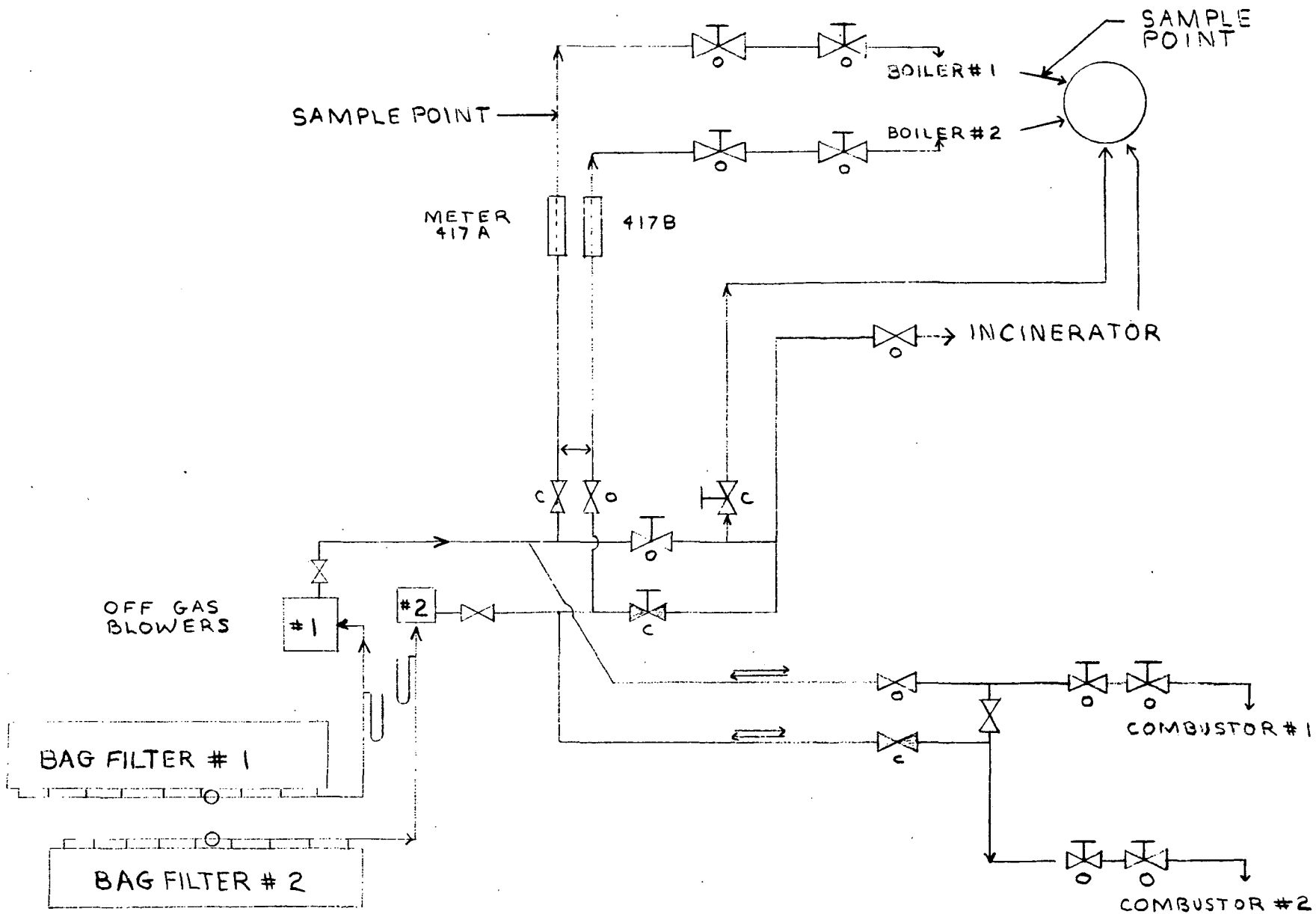


FIGURE 2

with water cooled combustion chamber. The boiler is designed to produce 45,000 lb/hour of 400 psig, 650°F steam. An auxiliary support fuel (natural gas) must be burned with the off-gas to prevent flame-outs. The boiler is operated with about 20 percent of the total heating value of the mixed fuel from natural gas.

The manifold system used to direct the off-gas to the various combustion devices is shown in Figure 2. During testing, the off-gas from Line 2 was burned in the two boilers and the off-gas from Line 1 was burned in the process driers and the incinerator. Boiler No. 1 was operated with minimum natural gas and maximum off-gas. The remainder of the Line 2 off-gas was burned in Boiler No. 2. The total off-gas from Line 2 was measured by two venturi meters shown in Figure 2.

Line 2 was producing carbon black Grade N330 (the largest volume grade made at this location) during the test. The production rate during the test was measured by collecting the material produced each day in a separate storage bin. At the end of a timed collection period, the collected black was bagged and weighed.

The Line 2 and Boiler No. 1 operating conditions recorded during testing are summarized in Table 14.

The proportional product rate to Boiler No. 1 was calculated by multiplying the total product rate from Line 2 by the ratio: off-gas to Boiler No. 1 / total off-gas from Line 2. As can be seen from Table 14, the average product rate that can be associated with the off-gas to Boiler No. 1 is 2640 lb/hour.

The age of the bags in each of the eight compartments at the time of testing is given in Table 13.

TABLE 13. LINE 2 BAGHOUSE BAG AGE

| | |
|----------------|-----------|
| 2 Compartments | 2 Months |
| 1 Compartment | 3 Months |
| 3 Compartments | 4 Months |
| 1 Compartment | 5 Months |
| 1 Compartment | 15 Months |

TABLE 14. LINE 2 AND BOILER NO. 1

| RUN NO. | 1 | 2 | 3 | 4 | AVERAGE |
|---|---------|----------|----------|----------|----------|
| DATE | 4-10-73 | 4-11-73 | 4-12-73 | 4-12-73 | |
| <u>PROCESS DATA: (Line 2)</u> | | | | | |
| Pressure Drop in Baghouse (in H ₂ O) | 3.0 | 3.3 | 3.3 | 3.6 | 3.3 |
| Outlet Temp. (°F) | 455 | 455 | 456 | 460 | 458 |
| Off-Gas To: (Wet SCFH) | | | | | |
| Boiler No. 1 | 517,000 | 645,000 | 720,000 | 675,000 | 639,000 |
| Boiler No. 2 | 392,000 | 405,000 | 375,000 | 363,000 | 384,000 |
| Total | 909,000 | 1050,000 | 1095,000 | 1038,000 | 1023,000 |
| Total Product Rate (lb/hr) | 4,264 | 4,262 | 4,225 | 4,225 | 4,244 |
| Proportional Product Rate to Boiler No. 1* (lb/hr) | 2,420 | 2,620 | 2,780 | 2,750 | 2,640 |
| <u>BOILER NO. 1 DATA</u> | | | | | |
| Steam Flow (lb/hr) | 32,250 | 21,500 | 33,500 | 31,900 | 32,300 |
| Steam Temp. (°F) | 663 | 660 | 668 | 662 | 663 |
| Boiler Press. (psig) | 393 | 393 | 392 | 389 | 392 |
| Header Press. (psig) | 391 | 392 | 392 | 392 | 392 |
| Fuel Flow (SCFH) | 7,240 | 7,250 | 7,280 | 7,110 | 7,230 |
| Air Flow (SCFH) | 870,000 | 874,000 | 935,000 | 935,000 | 903,000 |
| Outlet Gas Temp. (°F) | 525 | 531 | 535 | 537 | 532 |
| Percent O ₂ by volume, dry | 4.5 | 5.4 | 5.4 | 5.5 | 5.2 |
| Percent Combustibles (as H ₂) | 0.100 | 0.000 | 0.004 | 0.000 | 0.026 |

*Based on ratio of off-gas to Boiler No. 1.

The average operating pressure drop across the baghouse was 3.3 inches of H₂O.

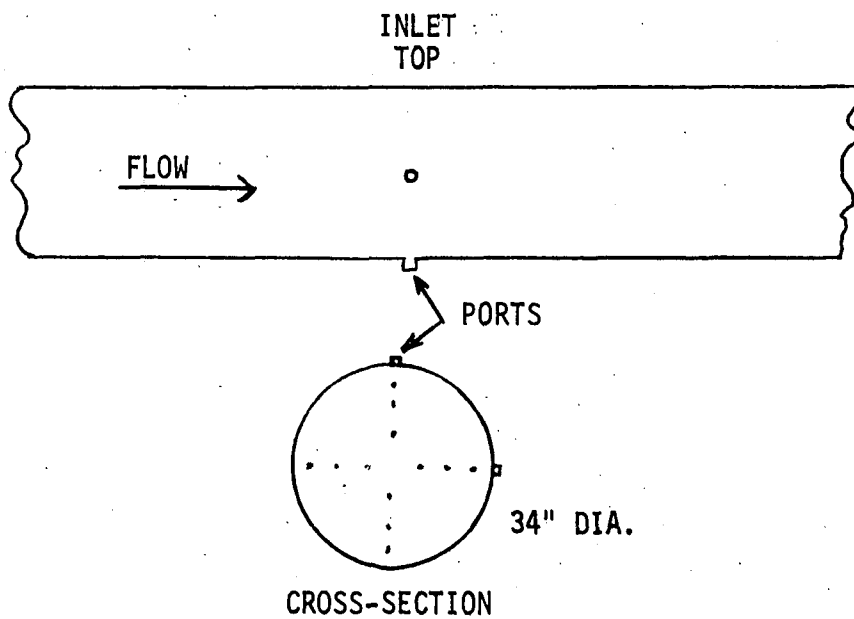
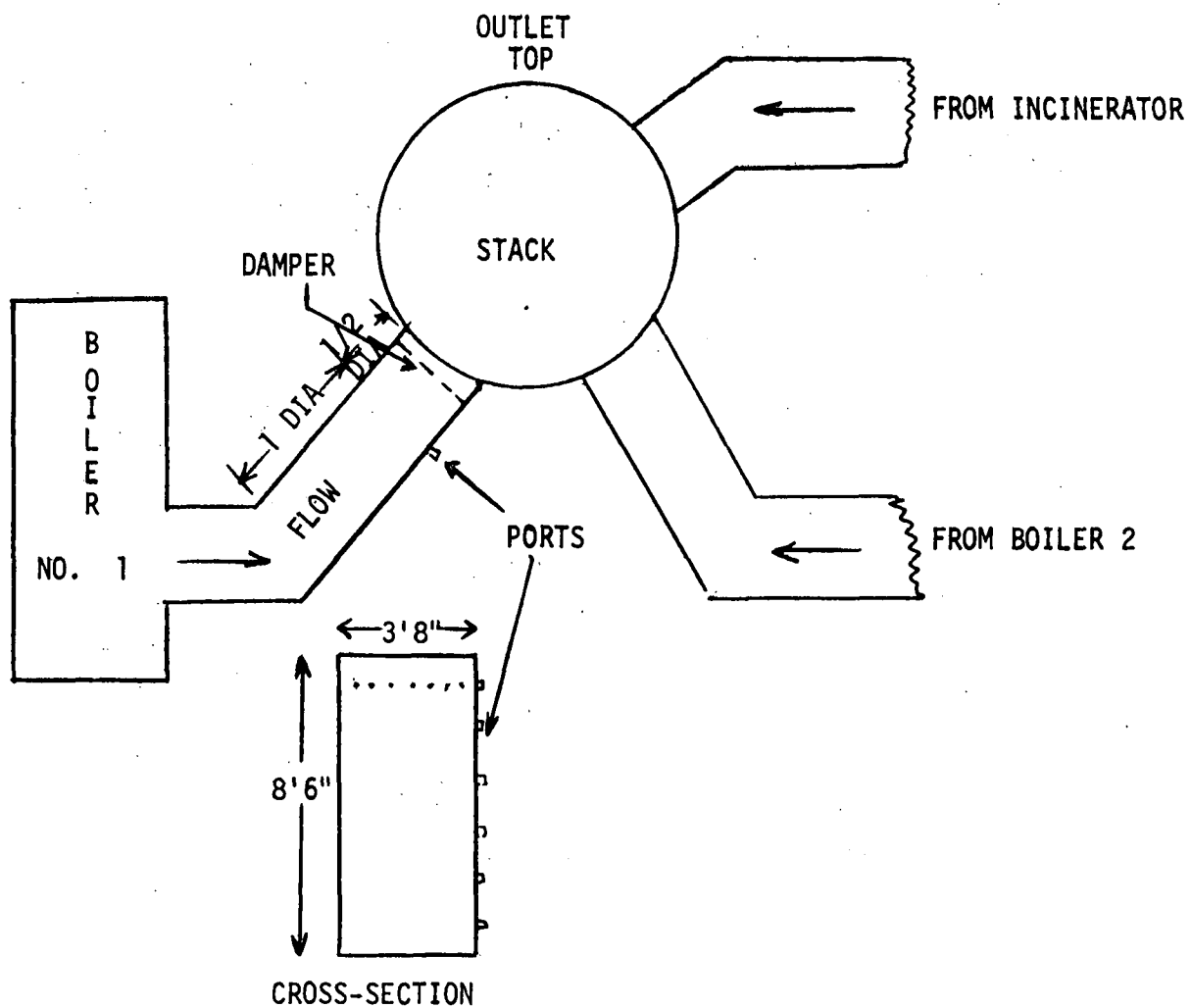
Analysis of the data from Table 14 shows that both the product collector and the No. 1 Boiler were operating at normal and uniform levels during testing.

V. LOCATION OF SAMPLING POINTS

Sampling was conducted in a circular duct at the inlet location and in a rectangular duct at the outlet location (see Figure 3).

The inlet location was ideal in relation to flue gas obstructions. Sampling was carried out on one vertical and one horizontal traverse. The closest disturbance in either direction was in excess of eight diameters. As per Method 1, Federal Register, December 23, 1971, twelve points were used in the traverse.

The outlet sampling location was less than ideal. Since the exhausts from two boilers and one incinerator are combined into the stack, the breaching from Boiler 1 to the stack had to be the sampling location. The equivalent diameter of this duct is 5.12 feet. There was approximately one diameter upstream and one-half diameter downstream from the nearest disturbances at this location. The location does not meet the minimum requirements of Method 1, but no alternative was available. There were 42 points used in the traverse at this location.



LOCATION OF SAMPLING PORTS

FIGURE 3

VI. SAMPLING AND ANALYTICAL PROCEDURES

Test methods were in accordance with standard methods as published in the Federal Register, Volume 36, Number 159, Part II, December 23, 1971, and other EPA methods. The following is a breakdown of the methods used in evaluating the various test parameters:

Particulates

Methods 1, 2, 5, Federal Register, December 23, 1971. In addition to this, the impinger solutions were analyzed for mass.

Sulfur Dioxide

Method 6, Federal Register, December 23, 1971.

Molecular Weight (Orsat Analysis)

Method 3, Federal Register, December 23, 1971.

Nitrogen Oxides

Method 7, Federal Register, December 23, 1971.

Hydrogen Sulfide

Method 11, Federal Register, June, 11, 1973.

Carbon Monoxide

Method 10, Federal Register, June 11, 1973.

Hydrocarbons

The parameters for Hydrocarbons were evaluated by utilizing a flame ionization gas chromatograph. Total hydrocarbons and non-methane hydrocarbons were measured extracting a gas sample from an integrated bag sample and injecting into the gas chromatograph. Calibration of the

detector is accomplished by introducing a standard sample through the gas sampling valve and the response measured using the same recorder as used for the sample determinations.

Two modes are used in determining total hydrocarbons and non-methane hydrocarbons. In one mode the injected sample by-passes the column and goes directly to the flame detector giving a peak for total organics. In the second mode the sample goes through the column where methane and other materials are individually determined.

The actual values were determined by reference to the known concentration of the standard calibration sample.

Particle Size Determination

A Brink Cascade, followed by a 47 millimeter glass fiber filter, was mounted on a probe and connected to a vacuum pump by a length of rubber tubing. The sampler was then placed in the stack to obtain thermal equilibrium before the actual sampling was completed. The amount of particulate material collected on each plate and on the glass fiber filter was determined in the laboratory analysis.