

U.S. DEPARTMENT  
National Technical In

PB-268 232

DESTROYING CHEMICAL WASTES IN COMMERCIAL SCALE  
INCINERATORS.

U.S. ENVIRONMENTAL PROTECTION AGENCY

NOVEMBER 1976

DESTROYING CHEMICAL WASTES  
IN COMMERCIAL-SCALE INCINERATORS

*This final report (SW-122c.2) describes work performed  
for the Federal solid waste management program  
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U.S. ENVIRONMENTAL PROTECTION AGENCY

1977

<b>BIBLIOGRAPHIC DATA SHEET</b>	1. Report No. EPA/530/SW-122c.2	2.	3. Recipient's Accession No.
4. Title and Subtitle  Destroying Chemical Wastes in Commercial Scale Incinerators. Facility Report No. 2 - Surface Combustion Division, Midland-Ross Corporation		5. Report Date November 1976	
		6. Issue Date	
7. Author(s) J.W. Adams, J.C. Harris, P.L. Levins, J.L. Stauffer, K.E. Thrun, L. Woodland		8. Performing Organization Rept. No.	
9. Performing Organization Name and Address Arthur D. Little, Inc. Acorn Park Cambridge, Mass. 20460		10. Project/Task/Work Unit No. 78596-30	
		11. Contract/Grant No. 68-01-2966	
12. Sponsoring Organization Name and Address U.S. Environmental Protection Agency Office of Solid Waste Washington, D.C. 20460		13. Type of Report & Period Covered Facility Test Report	
		14.	
15. Supplementary Notes  Work done under TRW Systems, Inc., subcontract No. A82870 DNB-L			
16. Abstracts Tests were conducted at Surface Combustion Division, Midland-Ross Corporation, Toledo, Ohio, to determine the effectiveness of pyrolysis for treatment of three selected chemical wastes: centrifuged API separator bottoms, styrene production wastes and rubber manufacturing wastes. The average conversion of organic material in the waste to organic material in the gaseous pyrolyzer effluent was 70% for API waste, 60% for styrene waste, and 80% for rubber waste. In each case the vapor stream contained a wide variety of organic compounds from methane and acetylene to high boiling (500°C) aromatic liquids and tars, including appreciable concentration of polynuclear aromatic hydrocarbons. The residual ash in each case was 80% inorganic material. Test results suggest that pyrolysis is technically and economically feasible for rubber wastes, technically feasible but not economically attractive for the API waste, and not an appropriate treatment for the liquid styrene production waste.			
17. Key Words and Document Analysis. 17a. Descriptors Hazardous materials Waste disposal Industrial wastes Incinerators Chemical analysis and sampling Pyrolysis Styrene Rubber Petroleum Heat Recovery Environmental Impacts			
17b. Identifiers/Open-Ended Terms Surface Combustion API Separator bottoms			
17c. COSATI Field/Group Field 13/Group 13B (secondary assignment to Field 7/Group 7A)			
18. Availability Statement		19. Security Class (This Report) UNCLASSIFIED	21. No. of Pages
		20. Security Class (This Page) UNCLASSIFIED	22. Price PLA08 MFA01

This report as submitted by the grantee or contractor has been technically reviewed by the U.S. Environmental Protection Agency (EPA). Publication does not signify that the contents necessarily reflect the views and policies of EPA, nor does mention of commercial products constitute endorsement by the U.S. Government.

An environmental protection publication (SW-122c.2) in the solid waste management series.

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## FOREWORD

The tests described in this report are part of a program designed to evaluate the environmental, technical, and economic feasibility of disposing of industrial wastes via incineration. This objective is being pursued through a series of test burns conducted at commercial incinerators and with real-world industrial wastes. Approximately eight incineration facilities and seventeen different industrial wastes will be tested under this program. The incineration facilities were selected to represent the various design categories which appear most promising for industrial waste disposal. The wastes were selected on the basis of their suitability for disposal by incineration and their environmental priority.

This report describes the test conducted at Surface Combustion (Toledo, Ohio), which was the second facility of the series. A facility report similar to this one has been published for the first test which was conducted at the Marquardt liquid injection facility in Van Nuys, California. The facility reports are primarily of an objective nature presenting the equipment description, waste analysis, operational procedures, sampling techniques, analytical methods, emission data and cost information. Facility reports are published as soon as possible after the testing has been completed at a facility so that the raw data and basic results will be available to the public quickly.

In addition to the facility reports, a final report will also be prepared after all testing has been completed. In contrast to the facility reports which are primarily objective, the final report will provide a detailed subjective analysis on each test and the overall program.

## ACKNOWLEDGEMENTS

Arthur D. Little, Inc., is grateful to the Surface Combustion personnel for their cooperation in conducting these facility tests. Acknowledgement is also made of the extensive and fruitful interactions between ADL and TRW personnel during the initial phases of this program. The project is deeply indebted to Messrs. Alfred Lindsey and John Schaum, of the Office of Solid Waste Management Programs, U. S. Environmental Protection Agency, for their advice and technical direction.

## 1. SUMMARY

Pyrolysis is a technique with potential for effecting recovery of resources from chemical wastes of high organic content. Exploitation of that potential is becoming increasingly attractive as shortages of fossil fuels and chemical feedstocks increase. Furthermore, pyrolysis is one of a fairly small number of techniques which can be applied to tarry, semi-solid, or solid organic wastes. Consequently, pyrolysis was chosen as one of seven different thermal destruction methods to be investigated for their effectiveness in handling chemical wastes.

Tests were carried out at the pyrolysis unit located at the Toledo, Ohio facilities of the Surface Combustion Division of the Midland-Ross Corporation. The following chemical wastes were utilized.

- Petroleum refinery wastes (centrifuged API Separator Bottoms)
- Styrene production wastes
- Rubber manufacturing wastes

These wastes were selected for pyrolysis because, based on information obtained from the waste generators, it was anticipated that they would be tarry solids or highly viscous liquids with fairly high gross heating values (2800 - 5600 Kcal/Kg or 5000 - 10,000 Btu/lb), and containing only carbon, hydrogen and oxygen as substantial components. Of the wastes actually received for testing, only the rubber waste conformed to these expectations. The API separator bottoms had a high (70%) water content and high (13%) ash content; the heating value was only about 1400 Kcal/Kg (2500 Btu/lb). The styrene waste did have a high heating value (8900 Kcal/Kg or 16,000 Btu/lb) but was a mobile liquid suitable for combustion in a liquid injection incinerator or for use as fuel in a steam generator. The styrene waste received also contained almost 8% sulfur. The rubber waste was a solid with a water content of about 30% and an estimated heating value of 5500 Kcal/Kg (9800 Btu/lb). Only the rubber waste was truly representative of the type of waste for which pyrolysis might be expected to be a leading method of treatment.

Table 1-1 presents a brief overview of the test results.

The products of pyrolysis are a vapor stream and a residual ash or char. The effectiveness of a pyrolysis process is generally assessed in terms of the vapor stream, since this is expected to contain the recoverable resource(s) (energy content and/or organic chemicals of commercial value), while the ash or char is usually destined for disposal. For the three wastes tested at Surface Combustion, the average conversion of organic material in the waste feed to organic material in the vapor stream was 70% for API waste, 60% for styrene waste, and 80% for rubber waste. In each of these cases, the vapor stream was found to contain a wide variety of organic compounds, ranging from gases, at normal temperature and pressure, such as methane and acetylene, to high boiling (500°C) liquids and tars. The heavier, condensable components of these streams are aromatic compounds,

TABLE 1-1

## SUMMARY OF OPERATING CONDITIONS AND TEST RESULTS

	<u>API Waste</u>	<u>Styrene Waste</u>	<u>Rubber Waste</u>
<u>Operating Conditions</u>			
Temperature, °C	760	650-760	760
Waste Residence Time, min	12.5	12.5	15
Feed Rate, Kg/hr	14.7-25.3	5.3-10.0	7.3-12.1
<u>Distribution of Products</u>			
Organic Vapors (% of Total Feed)	9	57	27
Ash (% of Total Feed)	20	<2	20
Remainder	Water	Soot	Water & Soot
Percent of Organics in Feed which were Found in Vapor	70	60	80
Percent of Organics in Feed which were Found in Ash	30	<0.01	8
Ratio of Light ( $\leq C_6$ ) to Heavy Organics in Pyrolyzer Effluents	2.3	0.4	2.3

including appreciable concentrations of polynuclear aromatic hydrocarbons. In general, this chemical composition is similar to that of residual oils or the products obtained from coking of coal. These compositions do not appear to offer any possibilities for commercial recovery of specific organic chemicals for recycle as feedstocks, so that the resource recovery potential of pyrolysis of these wastes lies in the fuel value of the vapor stream. It can therefore be concluded that the extent of resource recovery, defined as conversion of organic material in the waste to a form suitable for conventional heat recovery systems, is 70% for API and 80% for rubber. For the styrene waste, no net benefit is achieved by pyrolysis since the waste itself could be used directly in a heat recovery system.

The residual ash in all tests was found to contain mostly (>80%) inorganic material. The average extents of conversion of organics in the waste feed to ash were: 3% for API, <0.01% for styrene, and 4% for rubber waste.

The results of these tests indicate that certain potential adverse environmental impacts must be evaluated in any large-scale recovery of the energy value of pyrolyzer effluents. Using the API and rubber wastes for example, the occurrence of  $>125 \text{ mg/m}^3$  of sulfur in the vapors could lead to problems in meeting emissions standards for sulfur oxides from combustion systems. Other potential problems are (1) the  $350\text{--}500 \text{ mg/m}^3$  of polynuclear aromatic hydrocarbons, a class which includes some species recognized as carcinogens and (2) the occurrence of small but detectable amounts of heavy metals such as the lead and zinc found in the API wastes. While these factors will have to be considered carefully in the design of an appropriate heat recovery system, they are by no means insurmountable problems. These problems will be similar to those encountered in coke making, gasification of coal, and the combustion of residual oils.

Capital and operating cost estimates prepared for three different sizes of pyrolysis systems to treat rubber waste indicated that the overall operating costs will be highly dependent upon the capacity of the system. Total estimated costs, including energy credits and capital related items, vary from \$117/metric ton for a unit capable of pyrolyzing 6000 metric tons/year of rubber waste to \$526/metric ton for the pyrolysis of 1,000 metric tons/year. Energy credits were \$48.10/metric ton based on energy costs of \$7.93/million Kcal (\$2.00/million Btu). Of the total costs, direct operating labor, utilities, maintenance and residual ash disposal account for approximately 60% while capital related items, depreciation, interest and taxes and insurance account for the remainder.

The overall conclusions, based on tests of these three specific wastes, are that pyrolysis is both technically and economically feasible as a method of treating rubber wastes. For the API waste, pyrolysis is technically feasible but probably not economically attractive compared with the alternative of combustion in a fluidized bed incinerator. For the styrene waste, pyrolysis has no advantages and some disadvantages compared to destruction in other types of incinerators or as a fuel in a steam generating boiler.

These conclusions are strictly applicable only to the particular wastes tested and might have been quite different, for example, if the water content of the API waste had been only 20%. Experience during this program has made it clear that reliable information as to the chemical and physical nature of the stream to be treated (and the range of variation expected) is absolutely essential in developing strategies for selection among thermal destruction processes.

## 2. INTRODUCTION

The U.S. Environmental Protection Agency has sponsored a program\* to evaluate the effectiveness of a variety of types of commercial thermal destruction facilities in destroying chemical wastes. Pyrolysis was selected as one method for testing because it offers the potential for recovery of resources from waste materials.

In a pyrolysis process, material is thermally decomposed in a non-oxidizing environment. If the starting material is a hydrocarbon, such as an alkane, the process is referred to as "cracking," since the products are alkanes and alkenes of lower molecular weights than the original hydrocarbon, plus some hydrogen. The usual objective is to convert a relatively high molecular weight hydrocarbon (or mixture) to a more convenient fuel form, especially one which burns more cleanly. In contrast to conventional incineration, which is intended to achieve complete oxidation, pyrolysis is intended to produce a product stream which contains a high energy content by virtue of its hydrocarbon concentration. This feature of the pyrolysis process is increasingly appealing with the advent of energy and raw materials shortages. In addition, pyrolysis can be applied to tarry, semi-solid, and solid organic chemical wastes that are not amenable to other treatment techniques.

The objective of this program was to evaluate the capabilities of commercial scale facilities. However, a full scale pyrolysis facility within the continental United States which would be available for this test program could not be located. Because of the high priority assigned to the resource recovery potential of pyrolysis, it was decided to conduct a series of tests using the pilot plant pyrolysis unit operated by the Surface Combustion Division of Midland-Ross Corporation in Toledo, Ohio. This facility is a rotary hearth pyrolyzer which is coupled to a rich fume incinerator for combustion of pyrolyzer effluent. This unit is used on a regular basis by Surface Combustion in determining the design conditions for the rotary hearth pyrolyzers which it manufactures. The pyrolysis unit and incinerator are described in detail in Section 3 of this report.

The chemical wastes selected for testing at this pyrolysis facility were three which, based on the information supplied by the waste generators, would be good candidates for resource recovery and/or would be difficult to treat in other types of thermal destruction facilities. The criteria for waste selection included:

- the waste should be a tarry, semi-solid or solid material that was difficult to handle in conventional thermal destruction facilities
- the waste should have a heating value high enough to make recovery of fuel value attractive

\*Contract No. 68-01-2966

- the waste should be composed primarily of carbon, hydrogen and/or oxygen, since the Surface Combustion facility was not equipped with systems for removal of chlorides, sulfur or nitrates from the incinerator effluent
- the waste should represent a high priority disposal problem in terms of potential hazardousness and/or annual volume generated.

The wastes selected for testing, and the descriptions of these wastes originally provided by the waste generators, were:

- Centrifuged API Separator Bottoms. A sludge, with a heating value of 2800-5600 Kcal/Kg (5,000-10,000 Btu/lb), containing water, benzene soluble organics (30-50%) and ppm levels of heavy metals.
- Tars from the Production of Styrene. A polymeric tarry material with a heating value of 2800-5600 Kcal/Kg (5,000-10,000 Btu/lb) containing styrene and ethyl benzene.
- Rubber Manufacturing Wastes. A solid material with a heating value of 2800-5600 Kcal/Kg (5,000-10,000 Btu/lb) containing SBR rubber, carbon black, plus salts, fatty acids, scrap, etc., from the coagulation of latex.

The materials actually received for testing differed substantially from expectations in the following ways:

- The API separator bottoms waste had a heating value of only about 1400 Kcal/Kg (2500 Btu/lb), because it contained 70% water and 13% ash.
- The styrene waste was a mobile liquid, not a tar, with a sulfur content approaching 8% by weight.
- The rubber waste contained about 30% water but was otherwise similar to expectations.

The difference between actual and predicted waste characteristics was not unexpected because it is well known that the composition of wastes from production processes varies according to raw materials composition, process operating conditions and product quality demands. Consequently, it was necessary to recognize that a high degree of flexibility had to be maintained in planning a program of this type and that the results, while generally typical of a generic class of chemical wastes, may vary widely depending upon the actual composition of the wastes being pyrolyzed.

The test program involved pyrolysis of each of the three wastes under three different sets of conditions. On-line process instrumentation was used to determine the pyrolyzer operating condition and provide quantitative data on certain emissions. Samples were extracted from the pyrolyzer effluent for comprehensive analysis. Stack samples from the rich fume incinerator were collected to check on the environmental adequacy of the test. Details are in Section 4 of this report.

Detailed information on process and analytical data are recorded in Appendices A, B and C. The main body of the report presents data in a reduced form for assessment of the effectiveness of the pyrolysis process (Section 5). Also included in the report are estimates of capital and operating costs for pyrolysis of the API waste and rubber waste (Section 6).

### 3.0 PROCESS DESCRIPTION

#### 3.1 TEST FACILITY

The Surface Combustion pyrolysis/incineration system as used for this test program is shown schematically in Figure 3-1.

The test system included the following components:

- Pyrolyzer feed system
- Pyrolyzer
- Rich fume incinerator
- Induced draft fan and stack
- Inert gas generator

The pyrolyzer itself is the central piece of equipment in this system, but because of the physical nature of many of the chemical wastes treated by pyrolysis (e.g., semi-solid rubber waste), the feed system required also becomes a very important operating consideration.

Waste was fed to the pyrolyzer where it was decomposed into pyrolysis gas and a residual ash. This pyrolysis gas was sent to the rich fume incinerator where it was burned using 200-400% excess air. The effluent gas from the incinerator was diluted with room air to lower the temperature and discharged through the stack by an induced draft fan. An inert gas generator was used during the test program to supply relatively large quantities of inert gas to the pyrolyzer as a safety precaution during start-up and operation. (In a commercial operation it is anticipated that this inert gas would not be necessary.)

The schematic diagram of the pyrolysis system as shown in Figure 3-1 also indicates the location of the three sampling points.

##### 3.1.1 Rotary Hearth Pyrolyzer

The rotary hearth is 76 cm (2.5 ft) in diameter and 2.5 cm (1 inch) deep. A 15 cm (6 inches) diameter support pipe passes through the center of the hearth. The hearth speed can be varied from 1/2 to 3 revolutions per hour. The pyrolyzer is equipped with a 63,000 Kcal/hr (250,000 Btu/hr) burner. Two insulating boards, vertically mounted at 135° to each other at 2.5 cm (one inch) above the hearth, separate it into two zones. The burner is fired into the larger zone (hot zone) and the smaller zone (cold zone) is used for feeding waste and discharging residue. A plow mechanism is used to remove residue from the hearth. Temperature and pressure in the pyrolyzer are automatically controlled.

Pyrolysis of organic waste generates hydrocarbon vapors. Mixing of these vapors with oxygen can create hazardous conditions as it is possible to reach an explosive mixture of air and gases. The feed zone of the pyrolyzer was continuously purged with an inert gas during the operation to improve visibility and cool the feed zone as well as control the oxygen concentration. Pyrolyzer pressure was maintained slightly above atmospheric

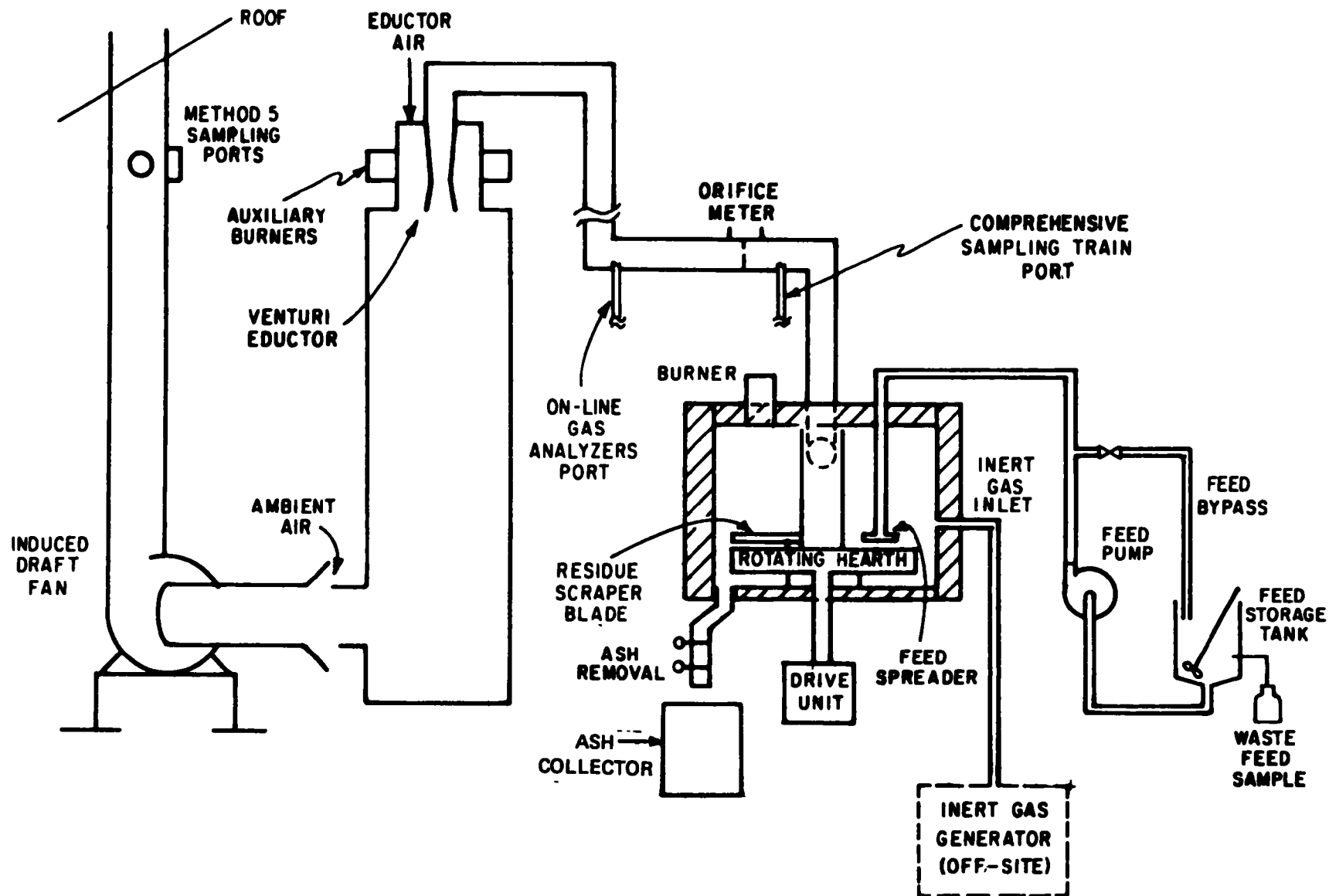


FIGURE 3-1 SCHEMATIC OF TEST PYROLYZER/INCINERATION SYSTEM

pressure by automatically controlling the position of the damper in the effluent gas duct. This positive pressure also reduced the chances of infiltration of air into the pyrolyzer. The burner system was modified so that burner flame-out would automatically cut off gas and air supply. A safety shield was installed in front of the glass observation port to protect personnel in case of rupture of the observation port. The pyrolyzer and incinerator burners were equipped with u.v. flame detectors and the temperature controllers had high limit contacts to shut the burner off in case the temperature exceeded the limit. When the burner is shut off, the air is turned off first (to exclude oxygen) and this, in sequence turns off the gas at the air/gas ratio regulator.

Oxygen concentration in the pyrolyzer was monitored continuously during the test program by an automatic-on line oxygen analyzer.

Figure 3-2 shows the pyrolyzer with shield over the glass window and the rubber waste feeder on top of the pyrolyzer. Figure 3-3 shows the pyrolyzer and the liquid feed tank.

A dimensional sketch for the rotary hearth pyrolyzer is shown in Figures 3-4 and 3-5. Figure 3-6 shows the process instrumentation used with the pyrolyzer.

### 3.1.2 Pyrolyzer Feed System

The liquid wastes, API separator bottoms and styrene tar, were fed by Moyno<sup>®</sup> pump. The feeding was done at room temperature. The piping arrangement for the liquid feed system is shown in Figure 3-7. The feed tank was equipped with a stirrer. The dimensions of the tank are 86 cm (34 inches) diameter and 61 cm (24 inches) height. The modified feed nozzle had a slot of size 0.32 cm x 20 cm (1/8" x 8") and a scraper attached to the nozzle for even distribution of waste on the hearth.

The rubber waste was fed by a specially designed mechanism. A pneumatic cylinder was used to operate a piston to push the waste through an orifice and then through a spreader nozzle. Manual feeding of the waste from the hopper into the feed cylinder was necessary in this test. (In the commercial unit, a kneader-extruder type feed system would be used.) The waste was distributed over the hearth by the 19 cm (7.5 inches) long feed nozzle. Width of the nozzle was varied for each test to give different layer thicknesses of waste on the hearth. A schematic of the rubber waste feeding mechanism used for this test is shown in Figure 3-8.

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\* Trademark of Robbins and Myers, Inc.

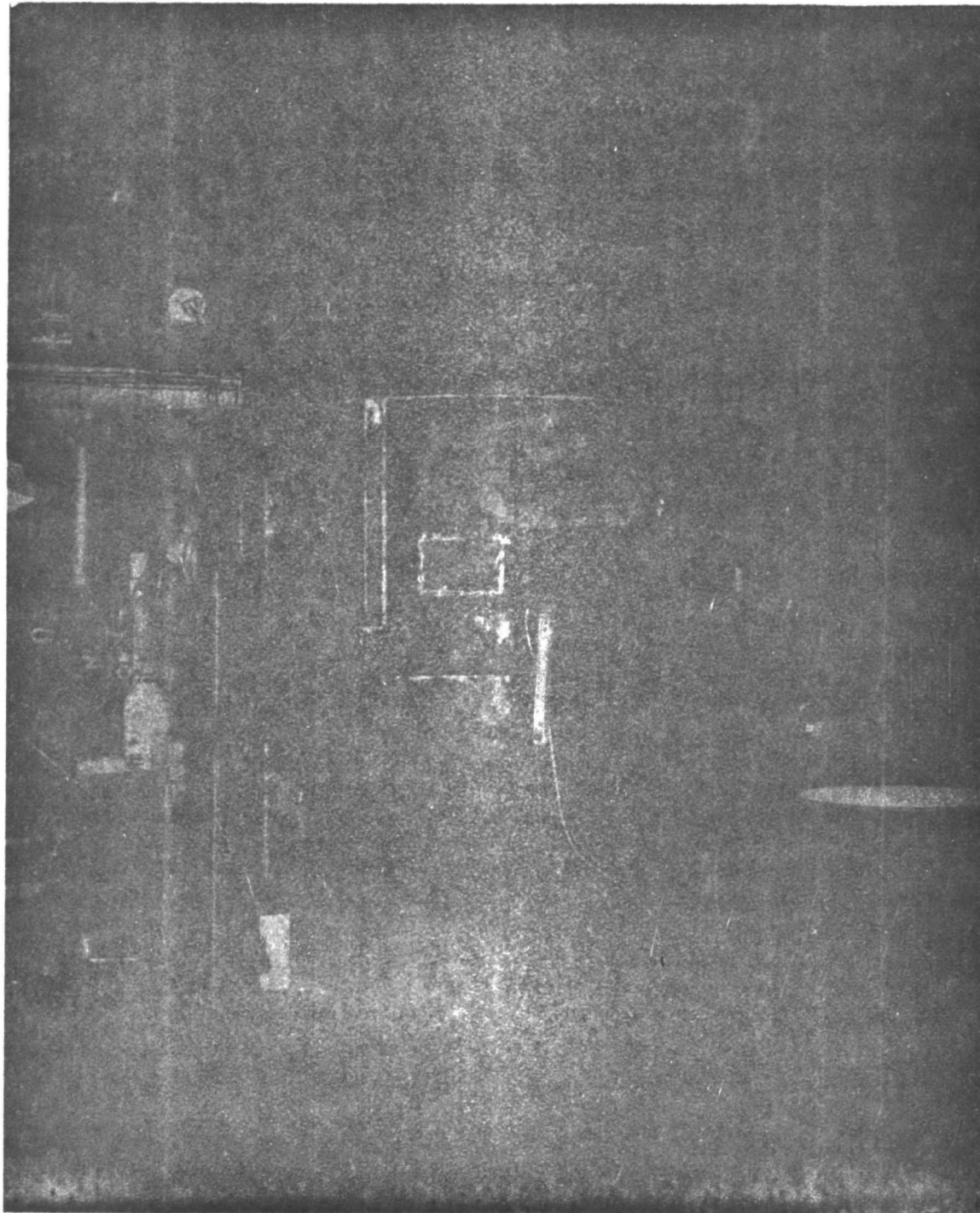


Figure 3 - 2. Pyrolyzer with Viewport Safety Shield and  
Rubber Waste Feed System

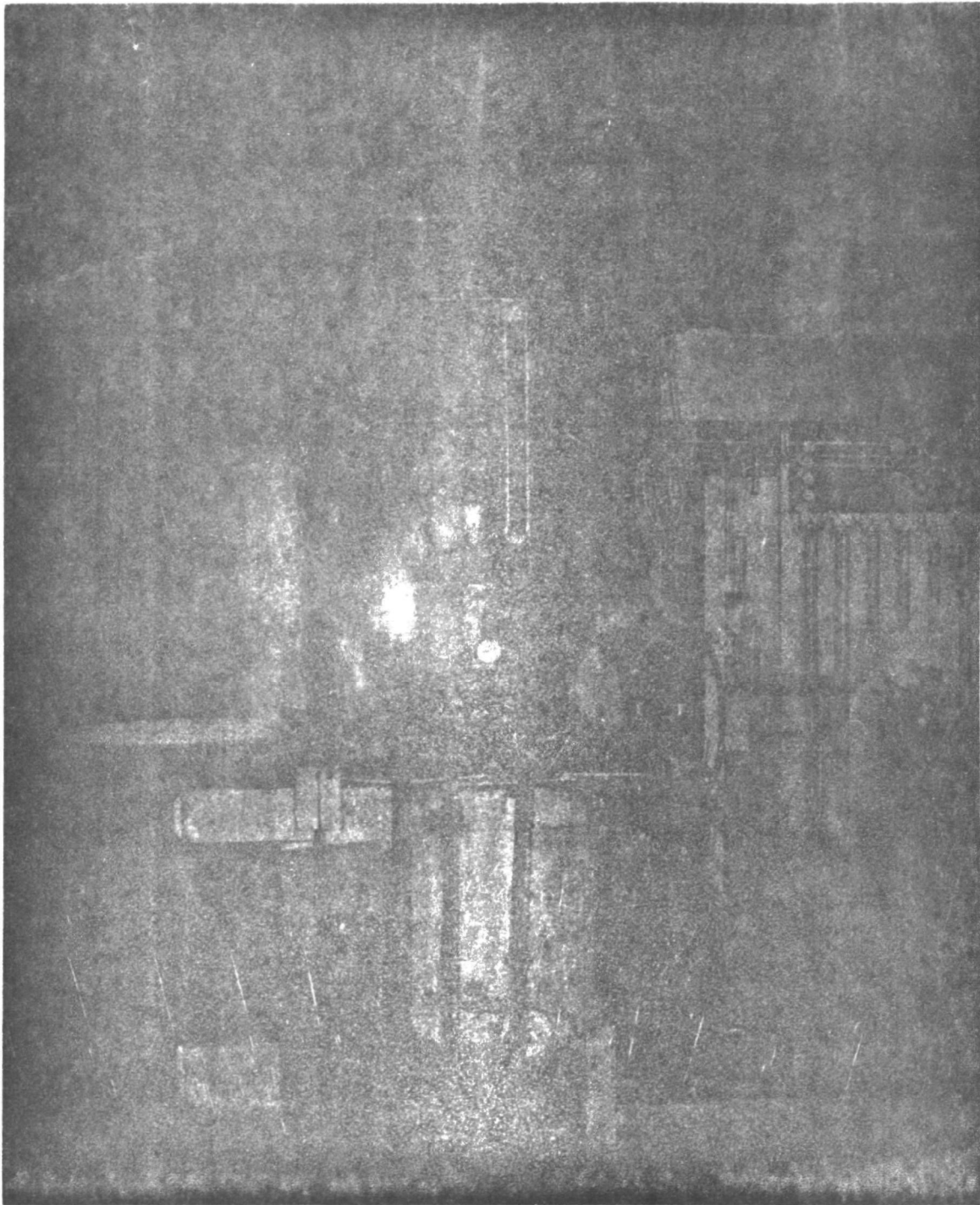
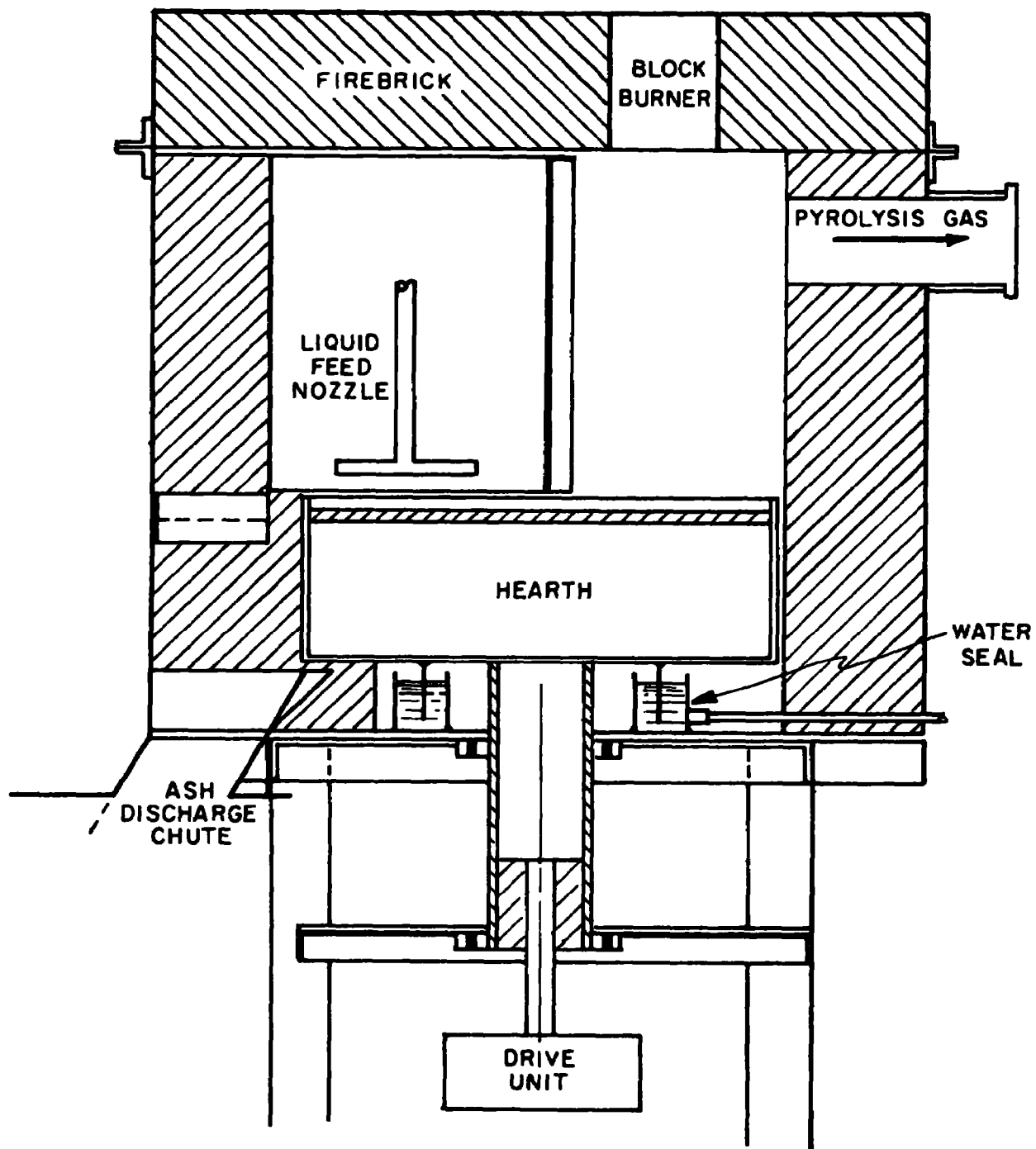
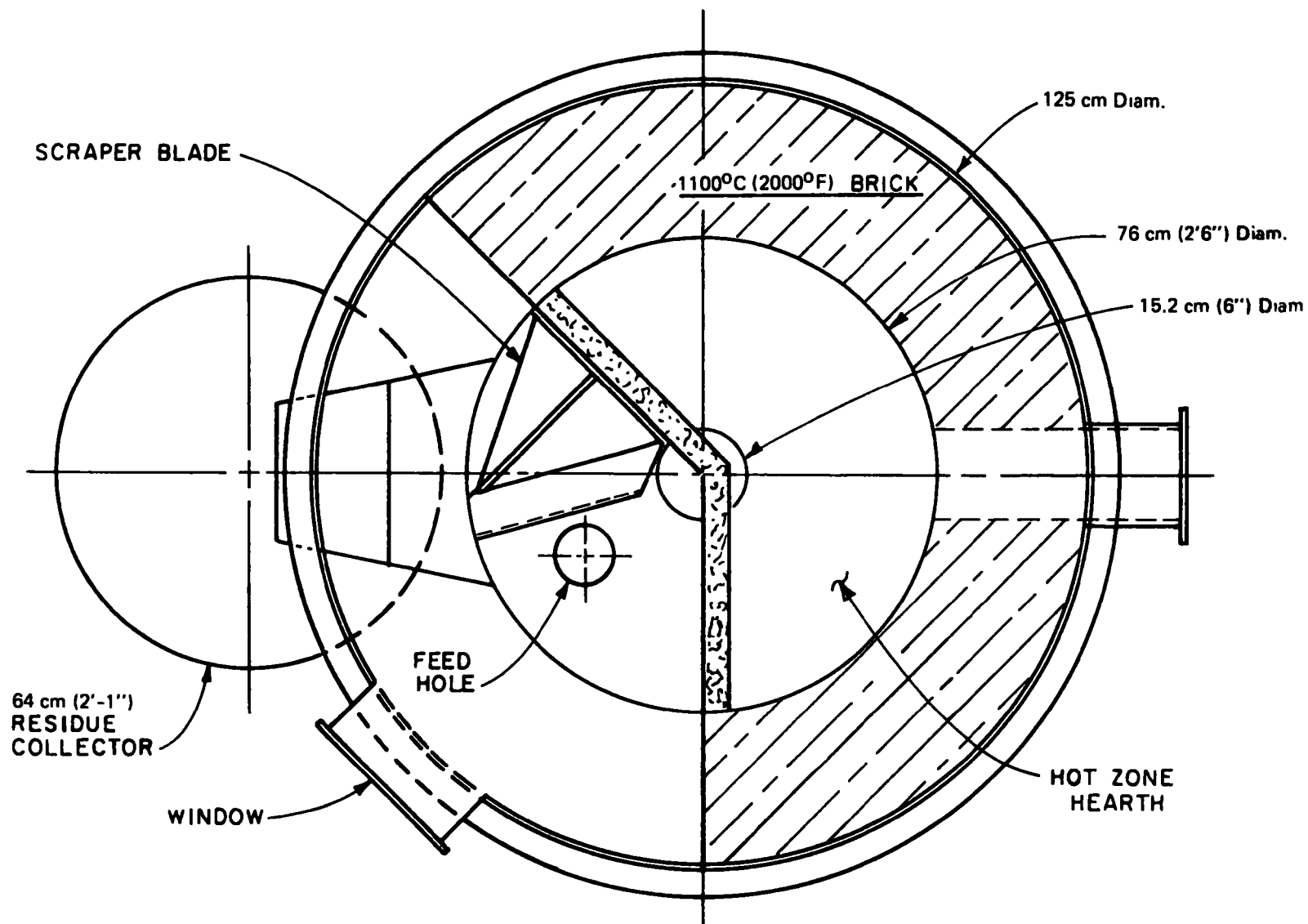


Figure 3 - 3. Pyrolyzer with Liquid Waste Feed Tank



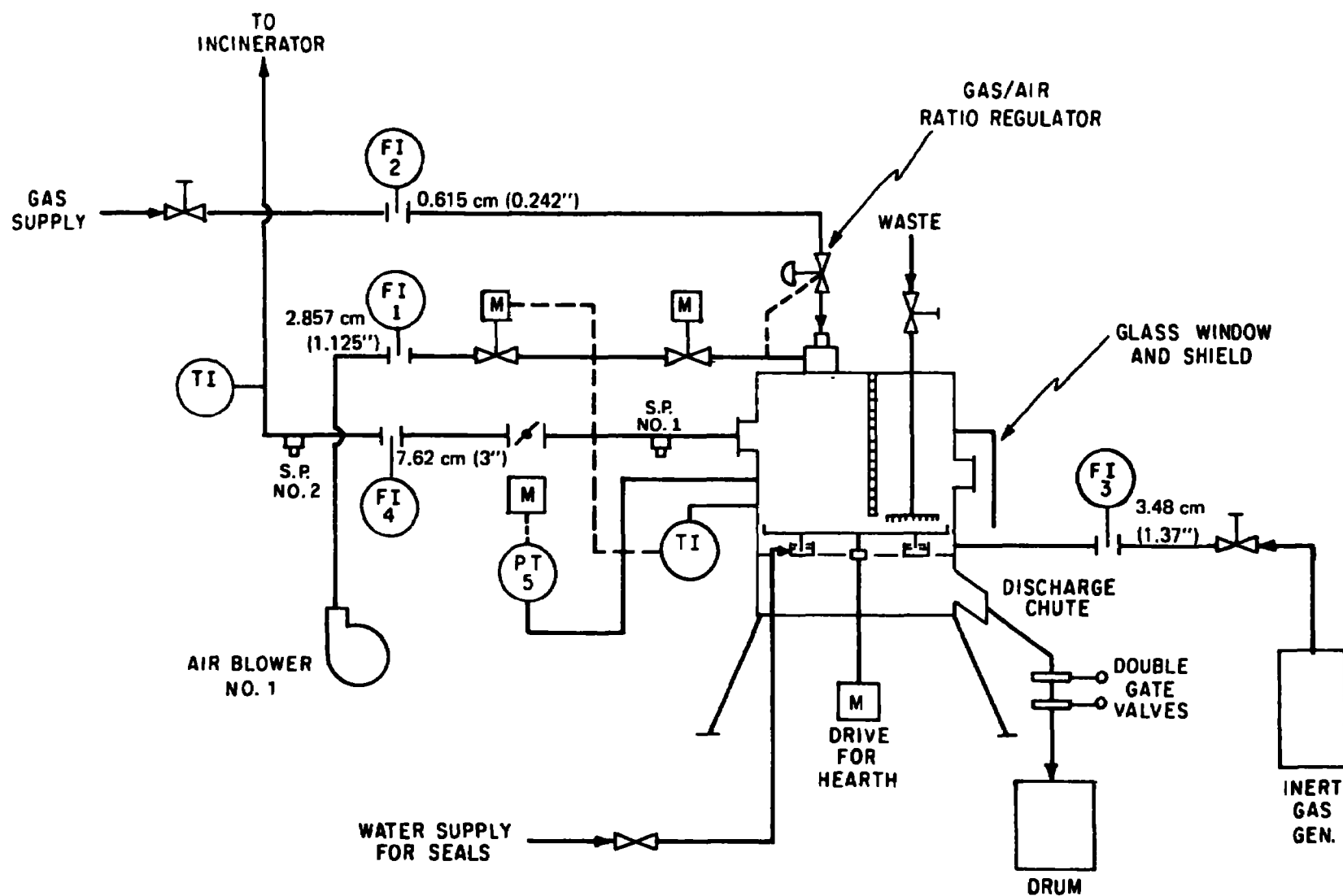
SOURCE: SURFACE COMBUSTION

FIGURE 3-4 SIDE VIEW ROTARY HEARTH PYROLYZER



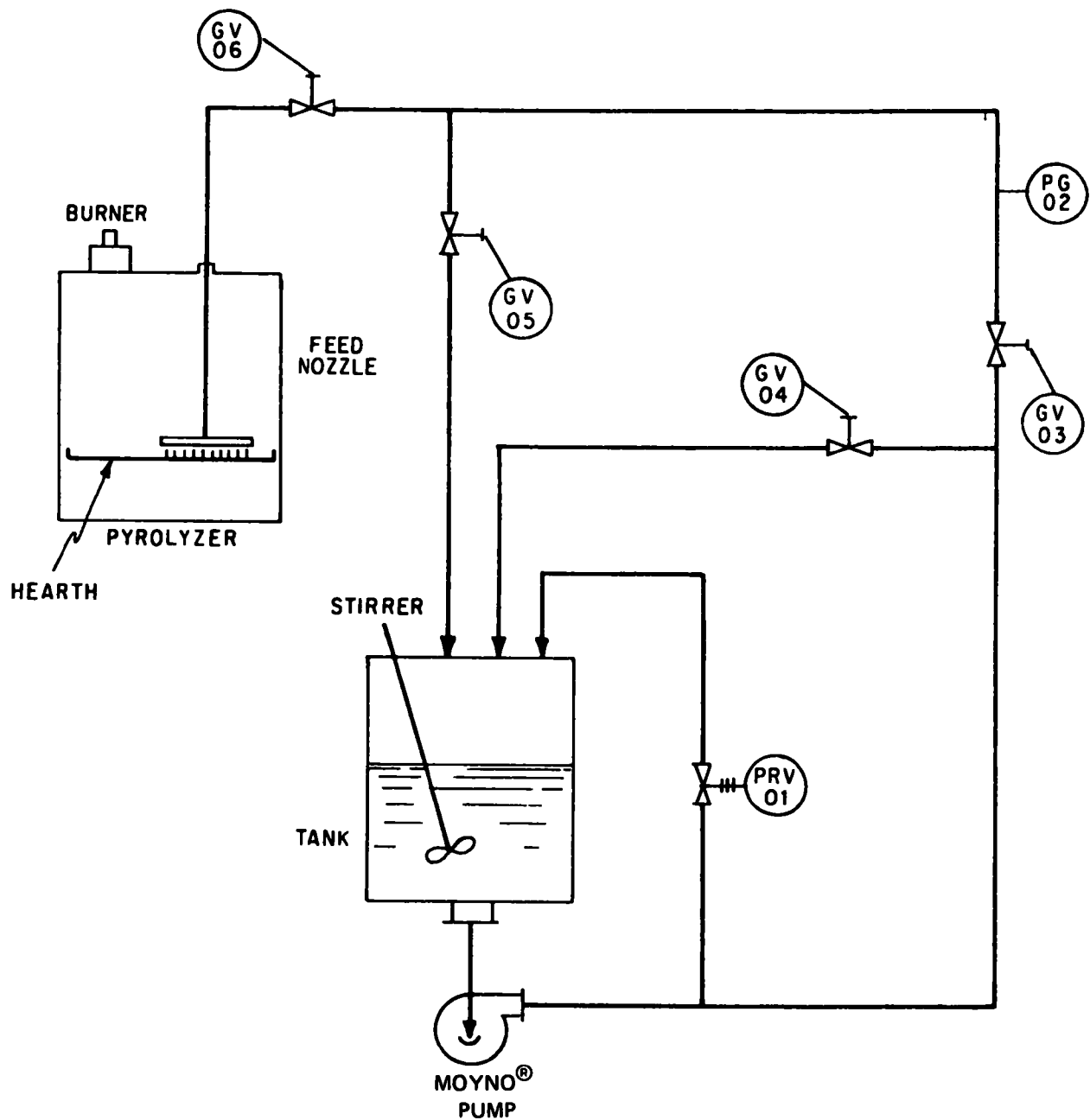
SOURCE SURFACE COMBUSTION

FIGURE 3-5 TOP VIEW ROTARY HEARTH PYROLYZER



SOURCE: SURFACE COMBUSTION

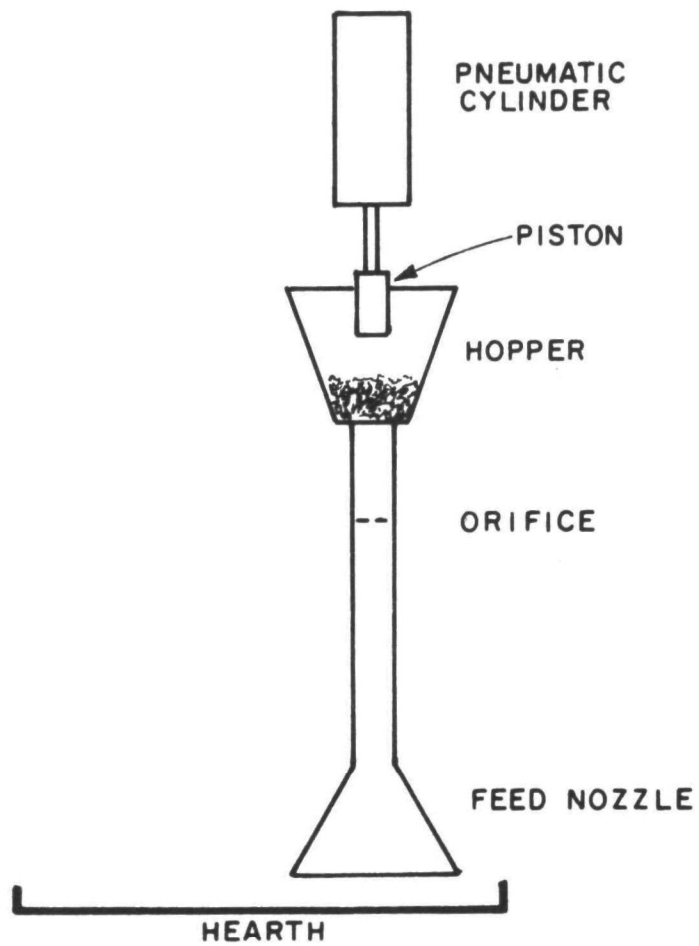
FIGURE 3-6 PROCESS INSTRUMENTATION FOR PYROLYZER



GV - GATE VALVE OR GLOBE VALVE  
 PRV - PRESSURE RELIEF VALVE

SOURCE . SURFACE COMBUSTION

FIGURE 3-7 PYROLYZER LIQUID FEED SYSTEM



SOURCE: SURFACE COMBUSTION

**FIGURE 3-8 RUBBER WASTE FEEDING SYSTEM**

### 3.1.3 Rich Fume Incinerator

The rich fume incinerator is equipped with two throat mix burners of 126,000 Kcal/hr (500,000 Btu/hr) capacity each. Auxiliary fuel was also used in the incinerator in this test series. The incinerator is equipped with temperature controller and high limit safety shut-off instrumentation. The burners are mounted at the top and gases flow downward and are exhausted by an induced draft fan after dilution with ambient air. The fan capacity is about 113 std. m<sup>3</sup>/min (4,000 scfm). (In a commercial unit the rich fume incinerator would be followed by a heat recovery boiler rather than the air dilution system used in the test program.)

### 3.2 PROCESS PARAMETERS

Table 3-1 summarizes the test conditions for three runs of each type of waste (API separator bottoms, styrene tar and rubber waste) and one background burn (no waste feed).

Gas and air flows into the system were monitored using orifice plates (as shown in Figure 3-6) and water manometers. Due to the limited accuracy with which it was possible to read the manometers ( $\pm 10\%$ ), the accuracy of the flow rates of gas and air were also about  $\pm 10\%$ . Pyrolyzer gas flow was, likewise, measured by orifice plate and manometer. Temperatures were measured by thermocouples and were recorded by strip chart recorders. Waste feed rate was measured by monitoring feed tank level for liquids and timing the piston strokes for rubber waste. The overall average feed rate was checked by weighing the waste between runs.

In the original test program, it was anticipated that several residence times and pyrolyzer temperatures would be tested for each feed. When the testing was actually conducted, however, it was necessary to use the maximum pyrolyzer temperature 760°C (1400°F), and the maximum hearth speed (3 revolutions per hour) in most cases in order to adequately destroy the wastes. The maximum hearth speed was necessary in order to spread the wastes thinly enough on the hearth to allow their complete pyrolysis. With a pyrolyzer temperature of 760°C (1400°F) the temperature of the pyrolysis gas ranged from 590-650°C (1000-1200°F). The variable changed with each run was, therefore, the waste feed rate. The waste feed rate was varied to find the maximum feed rate consistent with an acceptable ash while operating at maximum temperature and minimum residence time.

In the case of the rubber waste, it was also necessary to determine what nozzle opening was needed to produce a thin enough layer of rubber waste on the hearth to allow it to be adequately pyrolyzed.

TABLE 3-1 SUMMARY OF PYROLYSIS TEST CONDITIONS

Date	Run No.	Waste	Feed Rate		Feeder	Inert Gas Flow		Pyro. Temp		Hearth Speed (rph)	Residence in Hot Zone (mins)	Incinerator Temperature		% Residue
			Kg/hr	(lbs/hr)		m <sup>3</sup> /hr	(SCFH)	(°C)	(°F)			(°C)	(°F)	
1-28/76	1	API Separator Bottoms	16.7	(36.7)	0.32cmx15cm Nozzle, Moyno Pump	42.5	(1500)	760	1400	3	12.5	830	1520	19.4
1-29/76	2	"	14.7	(32.4)	0.32cmx20cm Nozzle, Moyno Pump	42.5	(1500)	760	1400	3	12.5	830	1520	19.7
1-30-76	3	"	25.3	(55.6)	"	42.5	(1500)	760	1400	3	12.5	830	1520	37.5
2-2-76	4	Styrene Tar	5.3	(11.7)	"	44.7	(1580)	760	1400	3	12.5	830	1520	1.4
2-3-76	5	"	7.4	(16.3)	"	42.5	(1500)	650	1200	3	12.5	830	1520	2.9
2-4-76	6	"	10.0	(22.0)	"	42.5	(1500)	760	1400	3	12.5	880	1610	0.5
2-5-76	7	No Feed	-	-	-	42.5	(1500)	760	1400	3	12.5	825	1515	-
2-17-76	8	Rubber Waste	12.2	(26.8)	1.28cmx19cm Nozzle	42.5	(1500)	760	1400	2.5	15	825	1515	29.9 (90-95% Lumps)
2-18-76	9	"	9.4	(20.7)	0.96cmx19cm Nozzle	35.4	(1250)	760	1400	2.5	15	820	1510	17.5 (60-70% Lumps)
2-18-76	10	"	7.3	(16.0)	0.64cmx19cm Nozzle	34.7	(1225)	760	1400	2.5	15	820	1510	12.5 (5 to 10% Lumps)

## 4. TEST DESCRIPTION

### 4.1 WASTES TESTED

The three wastes selected for testing at Surface Combustion were API Separator Bottoms, tars from the production of styrene, and rubber manufacturing wastes. Survey samples were received well in advance of the tests and analyzed in order to determine appropriate sampling procedures. The results of those survey analyses are summarized below.\*

#### 4.1.1 API Waste

The API waste was a grey-black, shiny goo which had a strong and somewhat irritating odor. The waste was about 69% by weight water and had an ash content of 11%. Elemental analyses showed the following composition for the wet waste: C, 12.07%; H, 8.80%; N, 0.30%; and S, 1.44%. Examination of the waste by X-ray fluorescence revealed Ca and Fe; smaller amounts of Cu and Zn, plus traces of K, Cl, S, Ti, Sr, Pb, Ni and Si.

The organic portion of the waste was found by mass spectrometry to consist of a complex mixture of hydrocarbons, with a substantial aliphatic component.

The higher heating value of the waste was estimated at 1390 Kcal/kg (2500 Btu/lb).

#### 4.1.2 Styrene Waste

The styrene waste was a brown-black viscous liquid with some suspended particulate. It had a pungent odor. The ash content was 0.9%. Elemental analysis showed the following composition: C, 85.04%; H, 7.41%; N, 0.03%; and S, 7.07%. Examination of the waste by X-ray fluorescence revealed sulfur, but no trace metals.

The organic portion of the waste was found by mass spectrometry to consist of a complex mixture of hydrocarbons, largely aromatic and of fairly high molecular weight.

The higher heating value of the waste was found to be  $8.9 \times 10^3$  Kcal/kg ( $16 \times 10^3$  Btu/lb).

#### 4.1.3 Rubber Waste

The rubber waste was composed of slightly sticky black lumps of various sizes. The waste had an ash content of 3.1%. Elemental analysis showed the following composition: C, 73.9%; H, 9.40%; N, 0.09%; and S, 0.54%. Examination of the waste by X-ray fluorescence revealed small amounts of Ca, Cl and Fe, plus traces of Zn, K, S, Pb, Sr, and Ni.

The organic portion of the waste was found to consist largely of polymeric, aromatic materials.

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\* Results of analyses of representative samples of the wastes actually tested are presented in Chapter 5 and in Appendix B.

The higher heating value of the waste was found to be  $7.8 \times 10^3$  Kcal/kg ( $14 \times 10^3$  Btu/lb).

#### 4.2 OPERATIONAL PROCEDURES

Detailed operating procedures, including a test plan and safety plan, were reviewed and approved prior to arrival of the sampling team on-site. A brief summary of the operating procedure follows:

##### Test Procedure

- Fill waste feed tank
- Ignite auxiliary fuel and allow system to reach thermal equilibrium.
- Activate on-line instruments.
- Begin waste feed and allow system to reach equilibrium, as shown by on-line instruments.
- Collect pyrolysis zone and stack samples.
- Discontinue waste feed.
- Maintain temperature with auxiliary fuel for about 30 min.
- Shut down system.
- Collect residue from pyrolyzer hearth.

#### 4.3 SAMPLING METHODS

Sampling methods used in the tests at Surface Combustion are described briefly below.

Five distinct samples were taken during each waste test:

- Composite sample of waste feed material.
- Sample of pyrolysis zone effluent fed to on-line instruments for continuous monitoring of test.
- Grab sample of pyrolysis zone effluent to evaluate process effectiveness.

- Grab sample of stack gases to verify that gaseous effluents were within local emission regulations.
- Sample of solid residue from the pyrolysis zone.

The locations of sampling points are shown in Figure 3-1.

#### 4.3.1 Waste Feed Sample

A composite sample of the waste feed was obtained by collecting a portion of the material in the waste feed drum during each test. The three feed samples were blended to yield one representative sample (REP) for each waste.

#### 4.3.2 On-line Gas Monitoring

A portion of the pyrolyzer effluent was sampled through a 1.27 cm (0.5") stainless steel probe and passed through an ice-cooled knock-out trap, then through a heated Teflon<sup>®</sup>\* line to a gas conditioning system. The gas conditioner was designed to deliver a cool, dry, particulate-free sample to the CO, CO<sub>2</sub>, O<sub>2</sub>, and NO<sub>x</sub> analyzers. A fraction of the sample was also supplied, untreated, to the hydrocarbon analyzer.

The instruments used and their ranges were:

Hydrocarbons	Beckman Model 402	0.05 ppm- 10%
Carbon Monoxide	Beckman Model 865	2-220 ppm
Carbon Dioxide	Beckman Model 864	0.05 - 20%
Oxygen	Taylor OA 273	0.05 - 100%
Nitrogen Oxides	Thermo Electron Model 10A	0.05 ppm- 1%

#### 4.3.3 Pyrolysis Zone Grab Sample

The train used for collecting this sample is shown schematically in Figure 4-1 and in the photograph in Figure 4-2. The principal components in this comprehensive sampling train were:

- a 1.27 cm (0.5") quartz sampling probe,
- a knock-out trap consisting of ice-cooled impingers to collect readily condensable organics,

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\* Trademark of E. I. du Pont de Nemours and Company.

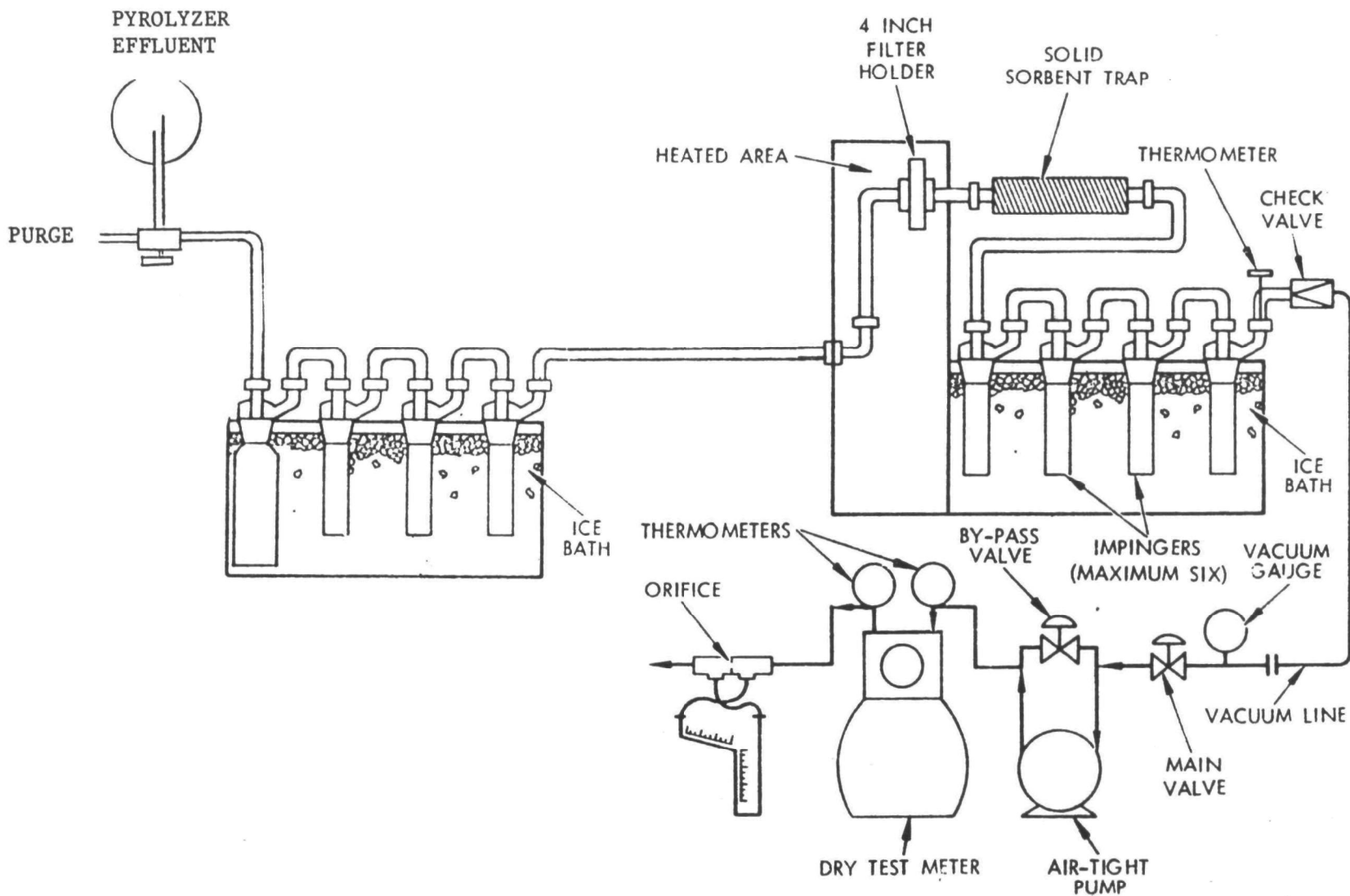


Figure 4-1. Modified Hot Zone Sampling Train

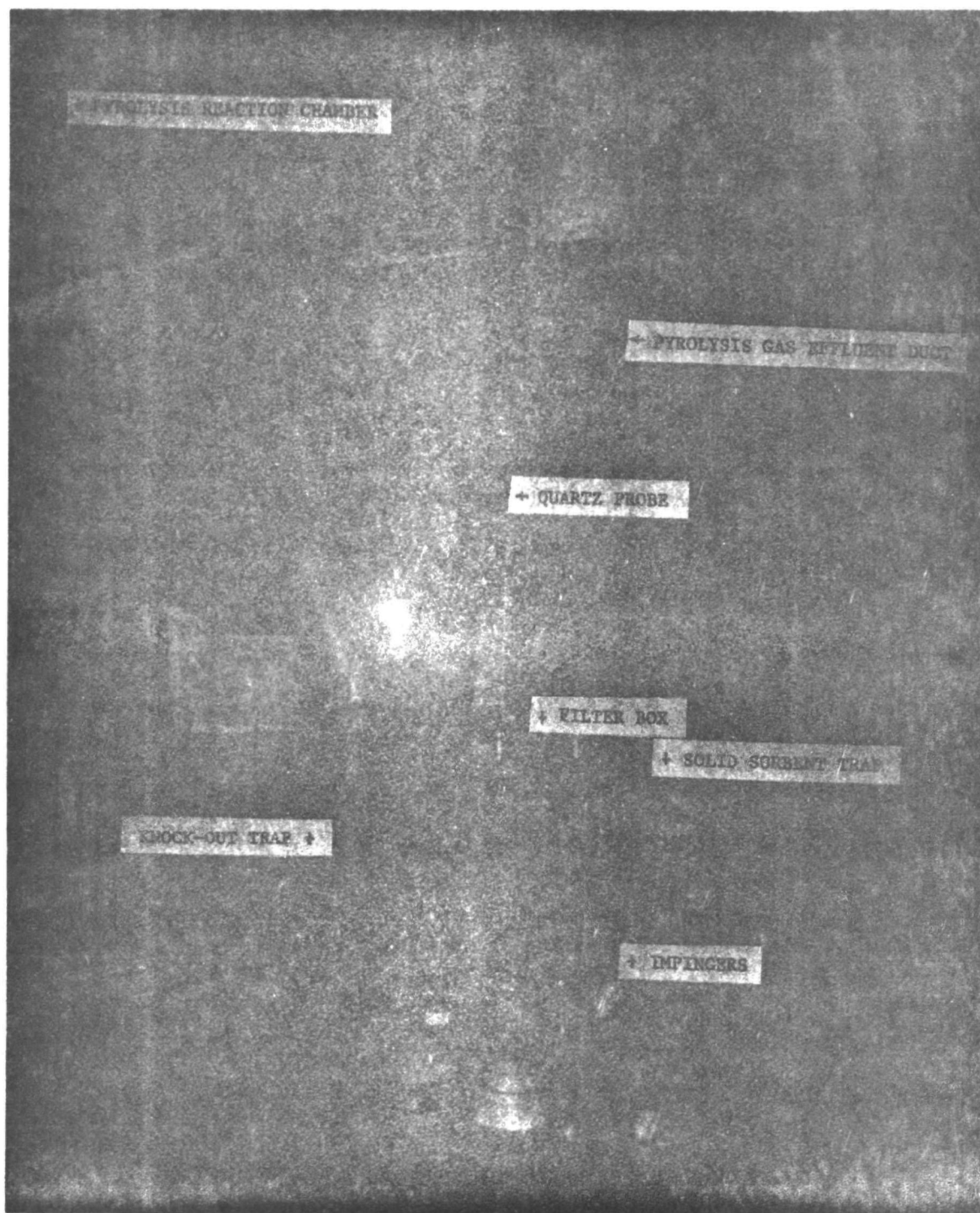


Figure 4 - 2. Sampling Train for Grab Sample of  
Pyrolysis Zone Effluent

- a quartz fiber filter,
- a sorbent trap filled with XAD-2<sup>®\*</sup> resin to collect organics of moderate volatility,
- impingers containing aqueous sodium hydroxide to collect acidic gases.

In addition, a portion of the pyrolyzer effluent was collected in gas sampling bulbs from the bypass line of the hydrocarbon analyzer. This allowed identification of effluent components too volatile for collection in the comprehensive sampling train.

#### 4.3.4 Stack Gas Grab Sample

The stack gas effluent was sampled isokinetically, according to the EPA Method 5 procedure, along two perpendicular traverses at 8 points per traverse. The train was a typical EPA Method 5 type, the RAC Staksamplr.<sup>®†</sup> The impingers contained aqueous NaOH to trap acidic sulfur gases. In addition, length of stain tubes were used to provide real-time estimates of sulfur dioxide concentration in the stack effluent.

#### 4.3.5 Ash Sample

The solid residue from the hearth was composited after each run and an aliquot taken for analysis.

### 4.4 ANALYSIS TECHNIQUES

#### 4.4.1 Extractions and Sample Preparation

A detailed description of the specific solvents and techniques used for the Surface Combustion Samples is given in Appendix A.

#### 4.4.2 Analytical Methods

The techniques which were chosen for evaluation of the effectiveness of thermal destruction of industrial wastes were:

Low Resolution Mass Spectrometry (LRMS)  
Infrared Spectrometry (IR)  
Gas Chromatography/Mass Spectrometry (GC/MS)  
Elemental Analysis

Inorganic Analyses were done by:

X-ray Fluorescence (XRF)  
Spark Source Mass Spectrometry (SSMS)  
Atomic Absorption Spectroscopy (AAS)  
Specific Ion Electrode Methods (SIE)

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\* Trademark of Rohm and Haas Company, † Trademark of Research Appliance Corp.

These techniques were applied to the Surface Combustion samples where appropriate.

In addition, a number of analytical techniques were added because of the special features of the pyrolysis process. Because the pyrolysis process is intended to allow resource recovery through conversion of waste to readily utilized fuels, several techniques were utilized to reveal the distribution of boiling points and/or molecular weights of feed and effluent samples. These techniques, which are described in Appendix A, were:

- Thermogravimetric Analysis (TGA)
- Boiling point distribution curves
- Gel Permeation Chromatography (GPC)

#### 4.5 PROBLEMS ENCOUNTERED

##### 4.5.1 Facility-related

Surface Combustion had originally intended to run the waste tests with the pyrolyzer at slightly negative pressure using cylinder nitrogen to provide an inert atmosphere in the pyrolysis zone. During one of the check-out burns on styrene waste, however, it appeared that these operating conditions were inadequate. Air leaked into the pyrolyzer, causing a minor explosion and rupture of the pyrolyzer viewing port.

As a result of this, conditions for the set of 10 tests were altered. The pyrolyzer was operated at a slightly positive pressure, using flue gas (DX-gas) as an inert medium. The DX-gas was created by combustion of natural gas. In addition, Surface Combustion installed an on-line oxygen monitor. If the oxygen level in the pyrolysis zone exceeded 0.5%, the pyrolysis unit was to be shut down.

##### 4.5.2 Waste-related

The waste-related problems encountered were primarily associated with the waste feed system. The API waste was found to contain occasional lumps, which clogged the waste feed system. Also, appreciable difficulties were encountered in devising a system which would feed the solid, but compressible, rubber waste.

During the styrene waste tests, there was occasional plugging of the pyrolysis zone sample lines due to condensation of effluent.

## 5. TEST RESULTS

### 5.1 INTRODUCTION

Process and analytical data are presented in detail in the Appendices. In this section, the data are presented in a reduced form, which facilitates assessment of the effectiveness of the pyrolysis process for treatment of each waste tested. The techniques used for reduction of the data are described briefly below. Throughout, gas volumes refer to standard conditions of 21.1°C (70°F) and 760 mm of mercury (29.92" of mercury).

#### 5.1.1 On-Line Hydrocarbon Analyzer Data

The hydrocarbon analyzer provided an on-line estimate of the concentration of gaseous ( $MW \leq \sim 100$ ) hydrocarbons as % by volume of  $CH_4$ . The results of analyses of gas bulb samples provided estimates of the average molecular weight and carbon number of the hydrocarbon material in the volatile pyrolyzer effluent. These estimates were used to convert "ppm by volume as  $CH_4$ " to " $mg/m^3$  of gaseous hydrocarbon." The " $mg/m^3$ " values were combined with the pyrolyzer effluent flow rate ( $m^3/hr$ ) to calculate the production of gaseous hydrocarbons in  $Kg/hr$ .

#### 5.1.2 Grab Samples of Pyrolyzer Effluent

For these samples, gravimetric determinations were made in ADL laboratories. These were combined with ADL data on the volume of effluent sampled plus Surface Combustion data on the total pyrolyzer effluent flow to give reduced values in units of  $mg/m^3$  and  $Kg/hr$ .

In the discussion which follows, the syllable, "GOO," refers to material collected in the Knockout trap and on the filter of the sampling train (Figure 4-1). The syllable, "ST," refers to the sorbent trap in that train. Together, GOO and ST include the readily condensable ( $MW > 100$ ) fractions of pyrolyzer effluent.

The syllable, -P-, in a sample code always indicates a portion of the pyrolysis zone effluent.

#### 5.1.3 Grab Samples of Stack Effluent

In this section of the report, all stack effluent data are presented in units of  $mg/m^3$ , based on ADL measurements of volume sampled and quantities of material collected. The syllable, -S-, in a sample code always indicates a portion of the stack effluent.

#### 5.1.4 Selection of "Typical" Waste Tests

Preliminary analyses of all the effluent samples collected during the tests showed that the samples obtained from the three tests on each waste had similar composition. Consequently, a set of samples corresponding to one test condition for each waste was selected for detailed chemical analysis. Selection criteria are specified in Appendix B.

## 5.2 TESTS ON API WASTE

### 5.2.1 Operating Conditions

Table 5-1 presents the operating parameters for the three tests on API wastes.

It is clear that the major difference among tests is the waste feed rate and the waste layer thickness. The temperature was maintained at the accessible maximum of 760°C (1400°F). The residence time was maintained at 12.5 min. throughout the tests.

### 5.2.2 Distribution of Pyrolyzer Effluent

In Table 5-2 are presented the data showing how the total mass of API waste feed was distributed among pyrolyzer effluent samples in the three tests.

The data indicate, first, that the total quantity of feed accounted for by the effluent samples (27 to 42%) was low. The loss is primarily due to the water (70 + 5% by weight) in the waste feed. The percent accounted for in the 3-API test is higher than in the other two tests. This is because the large quantity of ASH collected in the 3-API test contained a considerable amount of water. Other factors which contribute to the apparent loss of waste feed material are losses on the walls of the pyrolyzer effluent duct and losses in handling of the collected samples.

The data also indicate that the particular pyrolysis system used in these tests has an effective capacity of about 17 Kg/hr (37 lbs/hr) for the API waste. When the waste feed was increased to 25 Kg/hr (55 lbs/hr), the system appeared to be overloaded. This is evidenced by the fact that the absolute yield of volatile pyrolysis products (GOO plus ST plus gaseous hydrocarbons) decreased in the 3-API test while the yield of ASH increased dramatically.

### 5.2.3 Fate of Organic Components of the Waste

#### 5.2.3.1 Quantitative

The analyses showed that the API waste contained 13.0% by weight of organic material (material extractable with methylene chloride). It is the fate of this organic portion of the waste which is of primary importance in assessing the effectiveness of the pyrolysis process.

Table 5-1

OPERATING CONDITIONS FOR TESTS ON API WASTES

	<u>1-API</u>	<u>2-API</u>	<u>3-API</u>
Pyrolyzer Temperature	760°C (1400°F)	760°C (1400°F)	760°C (1400°F)
Residence Time in Pyrolysis Zone	12.5 min	12.5 min	12.5 min
Layer Thickness	2.54 cm (1 in)	1.27 cm (0.5 in)	1.91 cm (0.75 in)
Inert Gas Flow	0.0118 m <sup>3</sup> /sec (1500 SCFH)	0.0118 m <sup>3</sup> /sec (1500 SCFH)	0.0118 m <sup>3</sup> /sec (1500 SCFH)
Feed Rate	16.7 Kg/hr (36.7 lbs/hr)	14.7 Kg/hr (32.4 lbs/hr)	25.3 Kg/hr (55.6 lbs/hr)
Pyrolyzer Effluent Flow	.0303 m <sup>3</sup> /sec (3850 SCFH)	0.0317 m <sup>3</sup> /sec (4030 SCFH)	0.0342 m <sup>3</sup> /sec (4360 SCFH)
Pyrolyzer Effluent Temperature	582°C (1080°F)	577°C (1070°F)	582°C (1080°F)
Stack Gas Flow*	1.00 m <sup>3</sup> /sec (1.27 x 10 <sup>5</sup> SCFH)	0.98 m <sup>3</sup> /sec (1.25 x 10 <sup>5</sup> SCFH)	0.89 m <sup>3</sup> /sec (1.13 x 10 <sup>5</sup> SCFH)
Stack Gas Temperature	355°C (671°F)	362°C (684°F)	355°C (671°F)

\*ADL values--all other data by Surface Combustion.

Table 5-2

TOTAL QUANTITIES OF PYROLYZER EFFLUENTS FROM API WASTE TREATMENT\*

Feed Rate	Kg/hr	1-API	2-API	3-API
		16.7	14.7	25.3
-P-ASH	Kg/hr	3.24	2.90	9.48
	% of Feed	19.4	19.7	37.5
-P-GOO	mg/m <sup>3</sup>	2410	2726	2285
	Kg/hr	0.263	0.311	0.281
	% of feed	1.6	1.9	1.1
-P-ST	mg/m <sup>3</sup>	1320	1294	910
	Kg/hr	0.144	0.148	0.112
	% of feed	0.9	1.0	0.4
-P-Gaseous Hydrocarbons	mg/m <sup>3</sup>	7270	6885	6532
	Kg/hr	0.793	0.786	0.804
	% of feed	4.7	5.3	3.2
TOTAL	% of feed	26.6	27.9	42.2

\* "P-ASH" is the solid residue remaining on the hearth after pyrolysis. Together, "P-GOO" (the condensable organics in the pyrolyzer vapor stream effluent), "P-ST" (the organics trapped by the solid sorbent) and "P-Gaseous Hydrocarbons" (the true volatiles) constitute the portion of pyrolyzer effluent delivered to the heat recovery system.

Table 5-3 shows how the organic material is distributed among the various effluent fractions. For the 2-API test, which was selected as typical, the total recovery of organics was 85%. This probably represents complete recovery within experimental error. Of the total organic effluent, 27% was in the ASH, 14.9% in the GOO, 9.1% in the sorbent trap, and 49% in the gaseous hydrocarbon fraction. The total amount of waste organic material which was converted to a form suitable for introduction to the rich fume incineration was thus 73%.

#### 5.2.3.2 Qualitative

Table 5-4 summarizes the results of the LRMS analyses of the various samples from the 2-API test. These data have been normalized to reflect the total amount of organic effluent found in each fraction. (Normalized values do not add to 100% because some components in each sample were present at concentrations too low for compound identification.)

The organic material in the waste feed (REP-SOL fraction) consisted largely of unsaturated aliphatic hydrocarbons (42.7%) and aromatic hydrocarbons (39%) of up to three fused rings (anthracene and phenanthrene). The higher molecular weight polynuclear aromatic hydrocarbons, such as pyrene, were not found in the waste.

The total volatile effluent (GOO-SOL plus ST plus gaseous hydrocarbons) was found to have an aliphatic component very close to that of the feed (43.1%). This consisted of roughly equal parts of methane ( $\text{CH}_4$ ) and acetylene ( $\text{C}_2\text{H}_2$ ) in the gaseous hydrocarbon fraction.

The volatile effluent is seen to contain relatively more unsubstituted aromatics than the waste. Furthermore, the volatile effluent contains detectable levels of polynuclear aromatic hydrocarbons. Table 5-4 lists individual concentrations for five species which were chosen as indicators of polynuclear aromatics; these account for 2.9% of the organics in the volatile effluent from the pyrolyzer.

A small quantity (0.2%) of high molecular weight oxygenated aromatic material was found in the volatile effluent samples. These materials may have been formed by partial oxidation of waste material in the direct-fired pyrolyzer.

In contrast to the volatile effluent, the ASH was found to contain very little purely aliphatic organic material. The ASH was highly enriched in alkyl substituted aromatics (e.g., methyl naphthalenes, phenyl alkanes), which account for the high degree of aliphatic character in the IR spectrum of this material. The ASH also contained small amounts of polynuclear aromatics.

Table 5-3

ORGANIC MATERIAL IN PYROLYZER EFFLUENTS FROM API WASTE TESTS \*

	<u>1-API</u>	<u>2-API</u>	<u>3-API</u>
<u>ASH-SOL</u>			
Kg/hr	0.56	0.44	1.82
% of Organic Effluent	33	27	61
<u>G00-SOL</u>			
Kg/hr	0.204	0.243	0.25
% of Organic Effluent	12	14.9	8.4
<u>ST</u>			
Kg/hr	0.144	0.148	0.122
% of Organic Effluent	8.5	9.1	3.7
<u>GASEOUS HYDROCARBONS</u>			
Kg/hr	0.79	0.80	0.81
% of Organic Effluent	47	49	27
<u>TOTAL ORGANIC EFFLUENT</u>			
Kg/hr	1.70	1.63	2.99
<u>ORGANIC FEED RATE **</u>			
Kg/hr	2.17	1.91	3.29
<u>TOTAL RECOVERY</u>	78%	85%	91%
<u>OF ORGANICS</u>			

\* "P-ASH" is the solid residue remaining on the hearth after pyrolysis. Together, "P-G00" (the condensable organics in the pyrolyzer vapor stream effluent), "P-ST" (the organics trapped by the solid sorbent) and "P-Gaseous Hydrocarbons" (the true volatiles) constitute the portion of pyrolyzer effluent delivered to the heat recovery system.

\*\* 13% by weight of total feed, based on amount extracted from REP sample with methylene chloride.

Table 5-4

NORMALIZED DISTRIBUTION OF TOTAL PYROLYZER EFFLUENT\*  
BY CHEMICAL CLASS OF MAJOR COMPONENTS FOR 2-API TEST

<u>Class</u>	<u>% REP</u>	<u>PERCENT OF EFFLUENT</u>			<u>Total Volatile Effluent</u>	<u>Total Effluent</u>
		<u>P-ASH-SOL</u>	<u>P-GOO-SOL + P-ST</u>	<u>Gaseous HC's</u>		
1. Aliphatics	42.7	0	0	43.1	43.1	43.1
2. Unsubstituted Aromatics of $\leq 3$ Fused Rings	1.4	1.2	4.3	4.4	8.7	9.9
3. Substituted Aromatics of $\leq 3$ Fused Rings	37.6	21.5	6.8	1.5	8.3	29.8
4. Polynuclear Aromatics:						
Pyrene	0	0	1.3		1.3	1.3
Benzpyrene	0	0	0.5		0.5	0.5
Chrysene/ Benzanthracene	0	0	0.5		0.5	0.5
Benzfluoranthene	0	0.3	0.6		0.6	0.9
5. Micellaneous Aromatics	4.4	1.4	4.4		4.4	5.8
6. Diphenyl Thiophene	1.0	0.3	0.1		0.1	0.4
7. Oxygenated Aromatics	0	0	0.2		0.2	0.2
<u>TOTAL</u>	87.1	24.7	18.7		67.7	92.4

\*"P-ASH" is the solid residue remaining on the hearth after pyrolysis. Together, "P-GOO" (the condensable organics in the pyrolyzer vapor stream effluent), "P-ST" (the organics trapped by the solid sorbent) and "P-Gaseous Hydrocarbons" (the true volatiles) constitute the portion of pyrolyzer effluent delivered to the heat recovery system.

#### 5.2.3.3 Physical Properties of Pyrolyzer Effluent

The TGA and the boiling point distribution curves indicate that the condensable portion of pyrolyzer effluent includes components which boil in the range of 150 to 500°C (300 to 930°F). These, in comparison to typical petroleum products, correspond to the boiling point ranges of kerosene and diesel oil (150 to 300°C) and heavier oils.

The total volatile effluent from the pyrolyzer is about one-third by weight of these high-boiling species and about two-thirds very low boiling species (methane and acetylene).

#### 5.2.4 Fate of Inorganic Components of the Waste

##### 5.2.4.1 Sulfur

Elemental analysis of the REP sample indicated that the waste contained 1.5% by weight of sulfur.

Most of the sulfur in the waste feed was found in the ASH portion of the pyrolyzer zone. Analysis of the 2-API-P-I impinger solution indicated a total sulfur concentration of 136 mg/m<sup>3</sup> of volatile pyrolyzer effluent. This is consistent with the results of the gas bulb analysis which showed < 70 ppm by volume of volatile sulfur species.

The diphenyl thiophene found in the waste and effluent samples accounts for less than 10% of the total sulfur.

##### 5.2.4.2 Trace Elements

The SSMS analysis of the O-API-REP sample showed that the waste contained some 63 elements, including a number of rare earth elements at very low concentrations. A number of elements that were found at substantial concentrations (> 100 ppm) are recognized as potentially hazardous. These include zinc (1000 ppm), chromium (420 ppm), fluorine (240 ppm), and lead (210 ppm).

Analysis of the ASH fraction of the pyrolyzer effluent by SSMS revealed that all of the trace elements were enriched in this sample. In fact, the concentrations found in the ASH could account, within experimental error, for all of the trace materials in the waste feed. However, analysis of stack gas samples indicated that small quantities of some elements were found in the pyrolyzer effluent gas.

#### 5.2.5 Analysis of Stack Gases

The objective of this test program was the evaluation of the pyrolysis process, per se, not the rich fume incinerator in which the pyrolyzer effluent was burned. A small number of analyses were, however, performed on the incinerator stack gases.

#### 5.2.5.1 Particulate Loading

The stack particulate loading, determined according to the EPA Method 5, was 87.6 mg/m<sup>3</sup> for the 2-API test and 23.0 mg/m<sup>3</sup> for the 3-API test. (The filter from the 1-API test disintegrated and could not be weighed.) These particulate loadings are well within stationary source standards of 180 mg/m<sup>3</sup> for incinerators larger than 50 tons/day capacity.

#### 5.2.5.2 Sulfur Dioxide

The sulfur dioxide level of the stack gases was found to be 30 to 50 ppm by analysis with Gastec® tubes during the test. Analysis of the 2-API-S-I impinger samples for total sulfur indicated a stack gas loading of 47 mg/m<sup>3</sup>, as S, or 33 ppm as SO<sub>2</sub>.

#### 5.2.5.3 Trace Elements

One-half of the 2-API-S-F filter sample was analyzed directly by SSMS. After the background due to the filter material had been subtracted, the elements identified were: lead at about 0.05 mg/m<sup>3</sup> and zinc at 0.05 mg/m<sup>3</sup> of stack gas. These concentrations represent less than 5% of the amount present in the waste feed.

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\* Trademark of Bendix Environmental Science Division.

### 5.3 TESTS ON STYRENE WASTE

#### 5.3.1 Operating Conditions

Table 5-5 presents the operating parameters for the three tests on styrene wastes. The major differences among tests are the waste feed rate and the pyrolyzer temperature. The rate at which waste could be fed was limited by the capacity of the rich fume incineration used as an afterburner.

#### 5.3.2 Distribution of Pyrolyzer Effluent

In Table 5-6 are presented the data which show how the total mass of styrene waste feed was distributed among pyrolyzer effluent samples in the three tests.

The total percentages of feed accounted for in the pyrolyzer effluent from the styrene tests are considerably higher than those for the API tests. This is because the styrene waste contained very little water. A major contribution to the 20-35% net loss of material is deposition of the pyrolyzer effluent (soot) on the walls of the system. Some losses are also due to sample handling.

A significant feature of the data in Table 5-6 is that very little residue (ASH) is formed during pyrolysis of the styrene wastes.

#### 5.3.3 Fate of Organic Components of the Waste

##### 5.3.3.1 Quantitative

The analyses showed that the styrene waste contained 98% by weight of organic material (material extractable with methylene chloride). It is the fate of this organic portion of the waste which is of primary importance in assessing the effectiveness of the pyrolysis process.

Table 5-7 shows how the organic material is distributed among the various effluent fractions. The total recovery of organics was lower than in the API waste tests. Evidence obtained in the "background" test indicates that substantial quantities of material were deposited in the ductwork of the pyrolysis system.

For the 6-STY test which was selected as typical, the total recovery of organics was 59%. Of the total organic effluent, 1.7% was in the ASH, 52.4% in the GOO, 19.2% in the sorbent trap, and 26.7% in the gaseous hydrocarbon fraction.

Table 5-5

OPERATING CONDITIONS FOR TESTS ON STYRENE WASTES

	<u>4-STY</u>	<u>5-STY</u>	<u>6-STY</u>
Pyrolyzer Temperature	760°C (1400°F)	650°C (1200°F)	760°C (1400°F)
Residence Time in Pyrolysis Zone	12.5 min	12.5 min	12.5 min
Inert Gas Flow	0.0124 m <sup>3</sup> /sec (1580 SCFH)	0.0118 m <sup>3</sup> /sec (1500 SCFH)	0.0118 m <sup>3</sup> /sec (1500 SCFH)
Feed Rate	5.32 Kg/hr (11.7 lb/hr)	7.41 Kg/hr (16.3 lb/hr)	10.0 Kg/hr (220 lb/hr)
Pyrolyzer Effluent Flow	0.0303 m <sup>3</sup> /sec (3850 SCFH)	0.0275 m <sup>3</sup> /sec (3500 SCFH)	0.0313 m <sup>3</sup> /sec (3980 SCFH)
Pyrolyzer Effluent Temperature	560°C (1050°F)	550°C (1020°F)	600°C (1115°F)
Stack Gas Flow*	0.96 m <sup>3</sup> /sec (1.22 x 10 <sup>5</sup> SCFH)	0.96 m <sup>3</sup> /sec (1.22 x 10 <sup>5</sup> SCFH)	0.89 m <sup>3</sup> /sec (1.13 x 10 <sup>5</sup> SCFH)
Stack Gas Temperature	360°C (690°F)	365°C (685°F)	410°C (775°F)

\*ADL values - all other data by Surface Combustion

Table 5-6

TOTAL QUANTITIES OF PYROLYZER EFFLUENTSFROM STYRENE WASTE TESTS\*

		4-STY	5-STY	6-STY
FEED RATE	Kg/hr	5.32	7.41	10.0
ASH	Kg/hr	0.075	0.215	0.050
	% of Feed	1.4	2.9	0.5
GOO	mg/m <sup>3</sup>	15,980	33,014	33,093
	Kg/hr	1.74	3.27	3.73
	% of Feed	32.7	44.1	37.3
ST	mg/m <sup>3</sup>	7,048	(sample lost)	9,721
	Kg/hr	0.769	-	1.09
	% of Feed	14.5	-	10.9
GASEOUS HYDROCARBONS	mg/m <sup>3</sup>	14,330	13,490	13,595
	Kg/hr	1.56	1.33	1.53
	% of Feed	29.4	17.9	15.3
TOTAL	% of Feed	78	64.9	64

\* "P-ASH" is the solid residue remaining on the hearth after pyrolysis. Together, "P-GOO" (the condensable organics in the pyrolyzer vapor stream effluent), "P-ST" (the organics trapped by the solid sorbent and "P-Gaseous Hydrocarbons" (the true volatiles) constitute the portion of pyrolyzer effluent delivered to the heat recovery system.

Table 5-7

ORGANIC MATERIAL IN PYROLYZER EFFLUENT  
FRACTIONS FROM STYRENE TESTS \*

	<u>4-STY</u>	<u>5-STY</u>	<u>6-STY</u>
<u>ASH-SOL</u>			
Kg/hr	0.035	0.040	0.096
% of Organic Effluent	0.9	0.9	1.7
<u>GOO-SOL</u>			
Kg/hr	1.59	2.86	3.01
% of Organic Effluent	40.2	67.6	52.4
<u>ST</u>			
Kg/hr	0.769	(lost)	1.10
% of Organic Effluent	19.4		19.2
<u>GASEOUS HYDROCARBONS</u>			
Kg/hr	1.56	1.33	1.53
% of Organic Effluent	39.4	31.4	26.7
<u>TOTAL ORGANIC EFFLUENT</u>			
Kg/hr	3.95	4.23	5.74
<u>ORGANIC FEED RATE**</u>			
Kg/hr	5.22	7.26	9.80
<u>TOTAL RECOVERY</u> <u>OF ORGANICS</u>	76%	58%	59%

\* "P-ASH" is the solid residue remaining on the hearth after pyrolysis. Together, "P-GOO" (the condensable organics in the pyrolyzer vapor stream effluent), "P-ST" (the organics trapped by the solid sorbent and "P-Gaseous Hydrocarbons" (the true volatiles) constitute the portion of pyrolyzer effluent delivered to the heat recovery system.

\*\* 98% by weight of total feed, based on amount extracted from REP sample with methylene chloride.

#### 5.3.3.2 Qualitative

Table 5-8 summarizes the results of the LRMS analyses of the various samples from the 6-STY test. These data have been normalized to reflect the total amount of organic effluent found in each fraction. (Values do not add to 100% because not all of the waste components fall into the seven selected classes.)

The waste feed consisted largely of unsubstituted (27.8%) and substituted (59.9%) aromatic species of up to three fused rings. No purely aliphatic species were identified, nor were any higher molecular weight polynuclear aromatics found.

The total volatile effluent (GOO-SOL plus ST plus gaseous hydrocarbons) was found to contain 18.4% aliphatic material. This was mainly methane and acetylene in the gaseous hydrocarbon fraction. The fact that the ratio of unsubstituted to substituted aromatics is dramatically increased in the effluent suggests that the aliphatic material arose from alkyl sidechains of components in the waste feed.

In addition to the low molecular weight aromatics, pyrene (four fused rings) is found in the effluent at a concentration of 1.6%. It is probable that other polynuclear aromatics are also present at low levels.

The data suggest that diphenyl thiophene is formed during pyrolysis, since the quantity found in the effluent exceeds that in the waste feed. In contrast to the API tests, the styrene tests yielded ASH with very little organic material.

#### 5.3.3.3 Physical Properties of Pyrolyzer Effluent

The TGA and the boiling point distribution curves for the 6-STY samples indicate that the condensable portion of pyrolyzer effluent has a boiling point range of 150 to 500°C (300 to 900°F). This spans the range covered by diesel oil and kerosene (150 to 300°C) and heavier oils.

The total volatile effluent from the pyrolyzer is about 60% by weight of these high boiling species and about 18% very low boiling species (methane and xylene in the gaseous hydrocarbon fraction).

#### 5.3.4 Fate of Inorganic Components of the Waste

##### 5.3.4.1 Sulfur

Elemental analysis of the REP sample indicated that the waste contained 7.68% by weight of sulfur. The diphenyl thiophene in the waste accounts for less than 2% of the total sulfur content. Most of the sulfur is present as the free element (S<sub>8</sub>).

Table 5-8

NORMALIZED DISTRIBUTION OF TOTAL PYROLYZER EFFLUENT BY  
CHEMICAL CLASS OF MAJOR COMPONENTS FOR 6-STY TEST

<u>Class</u>	<u>% REP</u>	<u>PERCENT OF EFFLUENT</u>			<u>Total Volatile Effluent</u>	<u>Total Effluent</u>
		<u>ASH-SOL</u>	<u>GOO-SOL + ST</u>	<u>Gaseous HC's</u>		
1. Aliphatics	0	0	0	18.4	18.4	18.4
2. Unsubstituted Aromatics of ≤ 3 Fused Rings	27.8	0.5	24.0	17.4	41.4	41.9
3. Substituted Aromatics of ≤ 3 Fused Rings	59.9	1.0	28.2	3.5	31.7	32.7
4. Pyrene	0	0	1.6	0	1.6	1.6
Benzpyrene	0	0	0	0	0	0
Chrysene/ Benzanthracene	0	0	0	0	0	0
Benzfluoranthene	0	0	0	0	0	0
5. Miscellaneous Aromatics	3.4	0.005	2.5	0	2.5	2.5
6. Diphenyl Thiophene	1.1	0.08	5.1	0	5.1	5.2
7. Oxygenated Aromatics	0	0	0	0	0	0
TOTAL	92.2	1.6	61.4	39.3	100.7	102.3

In the pyrolyzer gaseous effluent fractions, most of the sulfur appears as carbon disulfide (1330 ppm), carbonyl sulfide (400 ppm) and sulfur dioxide (200 ppm). These components account for 68% of the sulfur in the waste feed. In addition, some sulfur is found in the ASH sample.

Analysis of the 6-STY-P-I impinger solution indicated that 753 mg/m<sup>3</sup> of sulfur, as S, was present as acidic volatile species in the pyrolyzer effluent. This value agrees (within 10%) with the total concentrations of SO<sub>2</sub> and COS (821 mg/m<sup>3</sup> as S) estimated from the gas bulb analyses.

#### 5.3.4.2 Trace Elements

The SSMS analysis of the 0-STY-REP sample showed that the waste contained only low levels of the metals generally recognized as hazardous. These included zinc (1.7 ppm), chromium (0.19 ppm), and lead (0.11 ppm). All of these were found to be concentrated in the 6-STY-P-ASH sample. The levels of trace metals found in the ASH could account, within experimental error, for the total quantities in the waste feed.

#### 5.3.5 Analysis of Stack Gases

The objective of this test program was the evaluation of the pyrolysis process, per se, not the rich fume incinerator in which the pyrolyzer effluent was burned. A small number of analyses were, however, performed on the incinerator stack gases.

##### 5.3.5.1 Particulate Loading

The stack particulate loading, determined according to EPA Method 5, was 27.5 mg/m<sup>3</sup> for the 5-STY test and 43.2 mg/m<sup>3</sup> for the 6-STY test. (The filter from the 4-STY test disintegrated and could not be weighed.)

##### 5.3.5.2 Sulfur Dioxide

The SO<sub>2</sub> level of the stack gases was found to be 100-200 ppm by analysis with Gastec<sup>®</sup> tubes during the test. Analysis of the 6-STY-S-I impinger samples for total sulfur indicated a stack gas loading of 126 mg/m<sup>3</sup> as S or 88 ppm as SO<sub>2</sub>.

##### 5.3.5.3 Trace Elements

One-half of the 6-STY-S-F filter sample was analyzed by SSMS. After the background due to the filter material had been subtracted, the only element found at significant concentration was sulfur at about 2 mg/m<sup>3</sup> of stack gas.

## 5.4 TESTS ON RUBBER WASTES

### 5.4.1 Operating Conditions

Table 5-9 presents the operating parameters for the three tests on rubber wastes. The major differences among tests are the waste feed rate and the waste layer thickness.

### 5.4.2 Distribution of Pyrolyzer Effluent

In Table 5-10 are presented the data which show how the total mass of rubber waste feed was distributed among pyrolyzer effluent samples in the three tests.

The data show that the total quantity of feed accounted for by the effluent samples averaged 44%. The lower recoveries are in large part due to the fact that the waste contained  $30 \pm 5\%$  water. Other sources of loss are deposition of material in the pyrolysis system and sample manipulations.

### 5.4.3 Fate of Organic Components of the Waste

#### 5.4.3.1 Quantitative

The rubber waste material was found to contain 33% by weight of organic material extractable with methylene chloride, 36% residue on extraction and 30% water. It is the organic portion of the waste which is of primary importance in assessing the effectiveness of the pyrolysis process.

Table 5-11 shows the distribution of organic material among the various effluent fractions. The total recovery of organics was 79% for 9-RUB, which probably represents complete recovery within experimental error. Of the total organic effluent, 12.1% was in the ASH, 12.7% in the G00, 6.8% in the sorbent trap, and 68.4% in the gaseous hydrocarbon fraction.

Table 5-9

OPERATING CONDITIONS FOR TESTS ON RUBBER WASTES

	<u>8-RUB</u>	<u>9-RUB</u>	<u>10-RUB</u>
Pyrolyzer Temperature	760°C (1400°F)	760°C (1400°F)	760°C (1400°F)
Residence Time in Pyrolysis Zone	15 min	15 min	15 min
Layer Thickness	1.73 cm (~0.68 in)	1.42 cm (~0.56 in)	1.09 cm (~0.43 in)
Inert Gas Flow	(0.0118 m <sup>3</sup> /sec) (1500 SCFH)	(0.0098 m <sup>3</sup> /sec) (1250 SCFH)	(0.0096 m <sup>3</sup> /sec) (1225 SCFH)
Feed Rate	12.1 Kg/hr (26.7 lbs/hr)	9.41 Kg/hr (20.7 lbs/hr)	7.27 Kg/hr (16.0 lbs/hr)
Pyrolyzer Effluent Flow	0.0286 m <sup>3</sup> /sec (3640 SCFH)	0.0260 m <sup>3</sup> /sec (3300 SCFH)	0.0261 m <sup>3</sup> /sec (3320 SCFH)
Pyrolyzer Effluent Temperature	640°C (1180°F)	620°C (1150°C)	640°C (1180°F)
Stack Gas Flow*	0.94 m <sup>3</sup> /sec (1.19 x 10 <sup>5</sup> SCFH)	0.97 m <sup>3</sup> /sec (1.23 x 10 <sup>5</sup> SCFH)	0.96 m <sup>3</sup> /sec (1.22 x 10 <sup>5</sup> SCFH)
Stack Gas Temperature	342°C (660°F)	337°C (640°F)	323°C (630°F)

\* ADL values - all other data by Surface Combustion

Table 5-10

TOTAL QUANTITIES OF EFFLUENTS FROM RUBBER WASTE TESTS\*

	8-RUB	9-RUB	10-RUB
Feed Rate Kg/hr	12.1	9.41	7.27
ASH Kg/hr	3.62	1.64	0.908
% of feed	29.9	17.4	12.5
GOO mg/m <sup>3</sup>	4,840	4,825	6,020
Kg/hr	0.498	0.452	0.566
% of feed	4.1	4.8	7.8
ST mg/m <sup>3</sup>	1,070	1,820	1,220
Kg/hr	0.110	0.170	0.115
% of feed	0.9	1.8	1.6
Gaseous Hydrocarbons mg/m <sup>3</sup>	18,040	18,190	14,300
Kg/hr	1.86	1.70	1.34
% of feed	15.4	18.1	18.4
TOTAL % of Feed	50.3	42.1	40.3

\* "P-ASH" is the solid residue remaining on the hearth after pyrolysis. Together, "P-GOO" (the condensable organics in the pyrolyzer vapor stream effluent), "P-ST" (the organics trapped by the solid sorbent and "P-Gaseous Hydrocarbons" (the true volatiles) constitute the portion of pyrolyzer effluent delivered to the heat recovery system.

Table 5-11

ORGANIC MATERIAL IN PYROLYZER EFFLUENT  
FRACTIONS FROM RUBBER TESTS\*

	<u>8-Rub</u>	<u>9-Rub</u>	<u>10-rub</u>
<u>Ash-Sol</u>			
Kg/hr	1.66	0.302	0.087
% of Organic Effluent	41.5	12.1	4.4
<u>Goo-Sol</u>			
Kg/hr	0.373	0.315	0.416
% of Organic Effluent	9.3	12.7	21.2
<u>ST</u>			
Kg/hr	0.110	0.170	0.115
% of Organic Effluent	2.7	6.8	5.9
<u>Gaseous Hydrocarbons</u>			
Kg/hr	1.86	1.70	1.34
% of Organic Effluent	46.5	68.4	68.4
<u>Total Organic Effluent</u>			
Kg/hr	4.003	2.487	1.958
<u>Organic Feed Rate**</u>			
Kg/hr	4.029	3.136	2.421
Total Recovery of Organics	99%	79%	81%

\* "P-ASH" is the solid residue remaining on the hearth after pyrolysis. Together, "P-GOO" (the condensable organics in the pyrolyzer vapor stream effluent), "P-ST" (the organics trapped by the solid sorbent) and "P-Gaseous Hydrocarbons" (the true volatiles) constitute the portion of pyrolyzer effluent delivered to the heat recovery system.

\*\* 33.3% of total feed, based on amount extracted from REP sample with methylene chloride.

#### 5.4.3.2 Qualitative

The organic extracts of the REP, ASH, GOO, and ST samples were analyzed by gel permeation chromatography (GPC) to determine the molecular weight distribution. The results, normalized to reflect the percent of total organic effluent in each fraction, are:

Z of material in molecular weight class			
	MW: $10^6 - 10^4$	$\sim 10^3$	$\sim 10^2$
<u>FEED</u>			
O-RUB-REP	35	27	37
<u>EFFLUENT</u>			
9-REP-P-ASH-SOL	4.7	6.0	0.2
9-RUB-P-GOO-SOL		4.1	8.6
9-RUB-P-ST			0.9

When the GPC data are combined with the result that 68.4% of the total organic effluent was in the gaseous hydrocarbon fraction (MW <100), the total molecular weight distribution of the pyrolyzer organic effluent becomes:

	<u>Including ASH</u>	<u>Excluding ASH</u>
MW $10^6 - 10^4$	4.7%	0%
MW $\sim 10^3$	10.1%	5%
MW $< \sim 10^2$	78.1%	95%

Table 5-12 summarizes the results of the LRMS analyses of the feed and effluent samples for the 9-RUB test. The LRMS analysis is limited to compounds which are reasonably volatile and therefore "sees" only compounds with molecular weights less than about 500. The data in Table 5-12 have been normalized to reflect the amount of total organic effluent which is contained in the low molecular weight range of each fraction. (The normalized values do not add to 100% because not all components of each sample are in that molecular weight range.)

As was the case for the other two wastes, the aliphatic content of the rubber pyrolysis effluent does not exceed that in the waste feed. In addition to the low molecular weight aliphatics indicated in Table 5-12, the high ( $>10^4$ ) molecular weight fraction of the rubber waste feed was also predominantly aliphatic, as shown by the lack of response to the UV detector in the GPC analysis. Overall, therefore, the aliphatic material in the pyrolyzer effluent is only about one-third of that in the waste feed. It seems that a substantial amount of the high ( $>10^4$ ) molecular weight material in the waste has been converted, during pyrolysis, to low

Table 5-12  
NORMALIZED DISTRIBUTION OF PYROLYZER EFFLUENT  
BY CHEMICAL CLASS OF MAJOR COMPONENTS FOR 9-RUB TEST \*

<u>Class</u>	<u>% REP-SOL</u>	<u>P-ASH-SOL</u>	<u>PERCENT OF EFFLUENT</u>			
			<u>P-GOO-SOL P<sup>+</sup>-ST</u>	<u>Gaseous HC's</u>	<u>Total Volatile Effluent</u>	<u>Total Effluent</u>
1. Aliphatics	18.0	0	0	20.2	20.2	20.2
2. Unsubstituted Aromatics of <u>≤</u> 3 Fused Rings	0	0.4	5.5	48.2	53.7	54.1
3. Substituted Aromatics of <u>≤</u> 3 Fused Rings	1.5	0.8	3.6	0	3.6	4.4
4. Pyrene	0	0	1.1	0	1.0	1.0
Benzpyrene	0	0	0.3	0	0.3	0.3
Chrysene/Benzanthracene	0	0	0.5	0	0.5	0.5
Benzfluoranthene	0	0	0.3	0	0.3	0.3
5. Miscellaneous Aromatics	2.1	0	2.4	0	2.4	2.4
6. Diphenyl Thiophene	0	0	0	0	0	0
7. Oxygenated Aromatics	9.6	0	0.1	0	0.1	0.1
<u>TOTAL</u>	31.2	1.2	13.7	68.4	82.1	83.3

\* "P-ASH" is the solid residue remaining on the hearth after pyrolysis. Together, "P-GOO" (the condensable organics in the pyrolyzer vapor stream effluent), "P-ST" (the organics trapped by the solid sorbent) and "P-Gaseous Hydrocarbons" (the true volatiles) constitute the portion of pyrolyzer effluent delivered to the heat recovery system.

molecular weight unsubstituted aromatics (up to 3 fused rings).

In addition to the  $\leq 3$ -ring aromatics, there is evidence for the formation of higher polynuclear aromatics. The total concentration of the four polynuclears selected as indicators is 2.1% of the organic effluent.

#### 5.4.3.3 Physical Properties of Pyrolyzer Effluent

The TGA results for the 9-RUB samples indicate that both the ASH and G00 fractions of pyrolyzer effluent contain appreciable amounts of non-volatile material. This is consistent with the GPC data showing substantial concentrations in these fractions of material with molecular weights  $\geq 1000$ .

On the other hand, the rubber waste test resulted in a very substantial portion of the feed being converted to very volatile species (methane and benzene).

#### 5.4.4 Fate of Inorganic Components of the Waste

A total of 61 elements were detected in the 0-RUB-REP sample by SSMS. Among the elements found at significant concentrations which are generally recognized as potentially hazardous were: chromium (130 ppm), lead (62 ppm), zinc (53 ppm), and fluorine (20 ppm). In a separate analysis, mercury was found at a level of 0.3 ppm.

Analysis of the ASH fraction of the pyrolyzer effluent by SSMS revealed that all of the trace metals were enriched in this sample. In fact, the concentrations found in the ASH can account, within experimental error for all of the trace elements in the waste feed.

#### 5.4.5 Analysis of Stack Gases

The objective of this test program was the evaluation of the pyrolysis process, *per se*, not the rich fume incineration in which the pyrolyzer effluent was burned. A small number of analyses were, however, performed on the incineration stack gases.

##### 5.4.5.1 Particulate Loading

The stack particulate loading, determined according to EPA Method 5, was  $10.3 \text{ mg/m}^3$  for the 8-RUB,  $14.0 \text{ mg/m}^3$  for the 9-RUB, and  $9.1 \text{ mg/m}^3$  for the 10-RUB test.

##### 5.4.5.2 Sulfur Dioxide

Analysis of the 9-RUB-S-I impinger sample for total sulfur indicated a stack gas loading of  $39 \text{ mg/m}^3$  as S, or 25 ppm as  $\text{SO}_2$ .

##### 5.4.5.3 Trace Elements

One-half of the 9-RUB-S-F filter sample was analyzed directly by SSMS. After the background due to the filter material had been subtracted, no trace elements were identified in the stack gas particulate sample.

## 5.5 SURFACE COMBUSTION BACKGROUND (SCB) TEST

The "background" test at Surface Combustion was made after the styrene test. In retrospect, this decision may have been unwise. All of the analytical data indicate that the samples collected during the "background" burn were, though lower in quantity, qualitatively similar to those of the styrene waste tests immediately preceding. (Appendix B) For this reason, a detailed analysis of the effluent from the SCB burn is not presented here.

The difficulty encountered in attempting to obtain a background sample reemphasizes the fact that the "pyrolysis gas" produced from all three wastes in fact contains substantial amounts of rather non-volatile materials. These components of the pyrolyzer effluent begin to condense if the temperature of the "pyrolysis gas" drops much below 500°C. Besides causing potential plugging problems, this accumulation of material in the ductwork produces "memory" effects in the pyrolysis system.

## 6. WASTE INCINERATION COST

Individual economic analyses were prepared for pyrolysis and incineration (with heat recovery) facilities of several different capacities for the destruction of rubber waste. An economic analysis was also prepared for the pyrolysis of an API separator bottoms waste. An economic analysis was not prepared for the pyrolysis of styrene waste because the physical form of this waste (liquid) would make it amenable to direct combustion in heat recovery equipment.

Each of these economic analyses was based on the "close coupling" of the pyrolyzer to a pyrolysis gas incinerator to preclude loss of sensible heat and condensation of high molecular weight organics in the duct between the pyrolyzer and incinerator.

The quantity of each type of waste to be destroyed is based on the following estimates of waste generation from single sources:

	<u>Size Productions Units</u>		<u>Waste Generated (Metric tons/yr)</u>
Refinery API separator bottoms	Crude oil capacity	50,000 bbl/day	300
Rubber Waste			
(Small Plant)	SBR Rubber	125,000 metric tons/yr	1000
(Large Plant)	SBR Rubber	250,000 "	2000
(From several Plants)	SBR Rubber	750,000 "	6000

The size of pyrolyzer required for these wastes (with the exception of the 6000 metric ton/yr pyrolyzer) is smaller than that normally built by Surface Combustion so the equipment cost estimates supplied for the pyrolyzer and rich fume incinerator were scaled down from the larger units.

As can be seen in the operating cost estimates, the smaller units are much more expensive to operate than the larger units. Net operating costs range from about \$117 to \$526 per ton of rubber waste (corresponding to 6000 and 1000 metric tons/yr of rubber waste treatment capacity) up to \$895 per metric ton of API separator bottoms waste at 300 metric tons/yr.

### 6.1 CAPITAL INVESTMENT

The equipment costs for the pyrolyzer and fume incinerator plus the necessary (uninstalled) instrumentation were supplied by Surface Combustion.

The cost of the other major pieces of equipment were estimated by ADL using cost data reported in the literature \* \*\* and updated using the Marshall and Swift Equipment (M&S) Index to a base of 460 (March 1976).

Each estimate is based on a system that includes waste storage, a feed system, the pyrolyzer, fume incinerator and heat recovery. However, no costs were included for air pollution control should it be required for particulates or sulfur oxides.

In the case of the API separator bottoms, the storage and feed system is relatively simple i.e., storage tank for about seven days waste and a progressing cavity or gear type feed pump.

The rubber waste, on the other hand, would require a much more sophisticated feed system. For the estimates, an extrusion type feeder has been assumed to discharge directly into the pyrolyzer. A belt conveyor would carry the rubber waste from the storage hopper to the feeder and pyrolyzer.

A certain portion of the piping and wiring of the system would be done during the construction of the equipment, but additional piping and wiring would be necessary at the construction site.

The estimate of capital investment requirements for three different capacities of rubber waste and one capacity of API separator bottoms waste are given in Tables 6-1, 6-3, 6-5 and 6-7.

## 6.2 OPERATING COSTS

The operating costs for three different capacity pyrolysis/incineration/heat recovery systems for handling rubber waste and a smaller system for handling API bottoms are presented in Tables 6-2, 6-4, 6-6 and 6-8. The operating costs for these four systems are summarized below:

<u>Waste</u>	<u>Waste Treated (metric tons/yr)</u>	<u>Net Operating Cost (\$/metric ton)</u>
Rubber Waste		
From Several Plants Combined	6000	\$117.17
From a Large Plant	2000	295.69
From a Small Plant	1000	525.89
API Bottoms Waste	300	894.51

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\* K. M. Guthrie, Process Plant Estimating Evaluation and Control, Craftsman Book Co. of America, Solano Beach, California (1974)

\*\* C. Dryden and R. Furlow, Chemical Engineering Costs, Ohio State University, Columbus, Ohio 1966

**TABLE 6-1**  
**CAPITAL INVESTMENT FOR PYROLYSIS, INCINERATION AND HEAT RECOVERY**  
**FOR 6000 METRIC TONS/YR OF RUBBER WASTE**

**Basis: 750 Kg/hr, 24 hrs/day, 330 days/yr**

<u>Purchased Equipment</u>	<u>Size</u>	<u>Cost (March 1976\$)</u>
Forced Draft Blower	7,500 scfm at 2 psi	10,000
Rotary Hearth Pyrolyzer	10 ft diameter	230,000
Incinerator Burner	30 million Btu/hr	36,000
Instrumentation Package		11,000
Extruder/Feeder	1,500 lbs/hr	75,000
Feed Storage	3,000 cuft (5 days)	7,000
Feed Conveyor (Belt)	100 ft	5,000
Heat Recovery Boiler	30 million Btu/hr	<u>105,000</u>
Purchased Equipment Cost		\$479,000
Installed Equipment Cost (IEC)		550,000
Piping (40% IEC)		220,000
Foundations (5% IEC)		28,000
Buildings and Structures (25% IEC)		138,000
Electrical (Including Instruments)		<u>50,000</u>
Total Physical Plant Cost (TPPC)		\$986,000
Engineering and Construction 30% TPPC		297,000
Contingency 20% TPPC		<u>197,000</u>
Total Capital Investment		\$1,480,000
Round to		\$1,500,000

TABLE 6-2

## OPERATING COST FOR PYROLYSIS, INCINERATION AND HEAT RECOVERY

## FOR 6000 METRIC TONS/YR OF RUBBER WASTE

Basis: Fixed Capital Investment (FCI) \$1,500,000  
 750 kg/hr Waste  
 Operation 24 hrs/day 330 days/yr  
 Rubber Waste Heat Value 5500 K Cal/kg (9800 Btu/lb) at 30% water  
 90% Conversion of Organics in Waste to Pyrolysis Gas

<u>Variable Costs</u>	<u>\$/Unit</u>	<u>Units per 2000 Metric Ton Waste</u>	<u>\$ per Metric Ton Waste</u>	<u>Annual Cost (\$)</u>
Operating Labor*			52.13	312,800
Utilities				
Oil or Gas	7.93/MillionKCal (2.00/MillionBtu)	2.66	21.11	126,700
Electricity*	0.015/kwh	250	3.75	22,500
Maintenance(8% FCI)			20.00	120,000
Solid Waste Disposal (12% Input)	6.50/Metric Ton	0.12	<u>0.78</u>	<u>4,700</u>
TOTAL VARIABLE COSTS			\$97.77	\$586,700
<u>Fixed Costs</u>				
Depreciation (15% FCI)			37.50	225,000
Cost of Capital (10% FCI)			25.00	150,000
Taxes and Ins. ( 2% FCI)			<u>5.00</u>	<u>30,000</u>
		Total Fixed Cost	\$67.50	\$405,000
		Total Operating Cost	\$165.27	\$991,700
<u>Credit for Recovered Heat (at 80% boiler efficiency)</u>				
From Rubber Waste	7.93/Million Kcal	3.96	31.40	188,400
From Auxiliary Fuel	7.93/Million Kcal	2.11	<u>16.70</u>	<u>100,100</u>
		Total Credit	\$48.10	\$288,500
<u>Net Operating Cost</u>			\$117.17	\$703,000

\* See footnotes to Table 6-2

FOOTNOTES TO TABLE 6-2

<u>Operating Labor</u>		<u>Annual Cost</u>
Pyrolyzer Feed System Operator	1 x 24 x 365 x \$7.00 =	\$ 61,300
Pyrolyzer/Incinerator Operator	1 x 24 x 365 x 7.50 =	65,800
Helper	1 x 24 x 365 x 6.50 =	<u>56,900</u>
	Direct Labor	\$184,000
Supervision (15% Direct Labor)		27,600
Supplies (20% Direct Labor)		36,800
Payroll Related Expense (35% Direct Labor)		64,400
Total Operating Labor		<u>\$312,800</u>

Electric Power

Forced Draft Blower	75KW	$185 \text{ Kwh/hr} \times \frac{1000}{750} = 250 \text{ kwh/metric ton}$ <p>Rubber Waste</p>
Extruder/Feeder	65KW	
Rotary Hearth	40KW	
Waste Conveyor	5KW	

TABLE 6-3  
CAPITAL INVESTMENT FOR PYROLYSIS, INCINERATION AND HEAT RECOVERY  
FOR 2000 METRIC TONS/YR OF RUBBER WASTE

Basis: 250 Kg/hr, 24 hrs/day, 330 days/yr

<u>Purchased Equipment</u>	<u>Size</u>	<u>Cost (March 1976\$)</u>
Forced Draft Blower	2,500 scfm at 2 psi	5,500
Rotary Hearth Pyrolyzer	6 ft diameter	150,000
Incinerator Burner	12 million Btu/hr	12,500
Instrumentation Package		11,000
Extruder/Feeder	600 lbs/hr	40,000
Feed Storage	1,000 cuft (5 days)	4,000
Feed Conveyor (Belt)	100 ft	5,000
Heat Recovery (Boiler)	10 million Btu/hr	<u>50,000</u>
Purchased Equipment Cost (PEC)		\$278,000
Installed Equipment Cost (IEC)		320,000
Piping (40% IEC)		128,000
Foundations (5% IEC)		16,000
Buildings and Structures (30% IEC)		96,000
Electrical (Including Instruments)		<u>50,000</u>
Total Physical Plant Cost (TPPC)		\$610,000
Engineering and Construction 30% TPPC		183,000
Contingency 20% TPPC		<u>122,000</u>
Total Capital Investment		\$915,000
Round to		\$920,000

TABLE 6-4

OPERATING COST FOR PYROLYSIS, INCINERATION AND HEAT RECOVERY  
FOR 2000 METRIC TONS/YR OF RUBBER WASTE

Basis: Fixed Capital Investment (FCI) \$920,000

250 Kg/hr Rubber Waste

Operation 24 hrs/day 330 days/yr

Rubber Waste Heat Value 5500 KCal/kg (9,800 Btu/lb) at 30% Water

90% Conversion of Organics in Waste to Pyrolysis Gas

<u>Variable Costs</u>	<u>\$/Unit</u>	<u>Units per Metric Ton Waste</u>	<u>\$ per Metric Ton Waste</u>	<u>Annual Cost (\$)</u>
Operating Labor*			156.40	312,800
Utilities				
Oil or Gas	7.93/Million KCal (2.00/Million Btu)	2.66	21.11	42,100
Electricity*	0.015/kwh	300	4.50	9,000
Maintenance (8% FCI)			36.80	73,600
Solid Waste Disposal (12% input)	6.50/metric ton	0.12	<u>0.78</u>	<u>1,600</u>
Total Variable Costs			\$219.59	\$439,100
<u>Fixed Costs</u>				
Depreciation	(15% FCI)		69.00	138,000
Cost of Capital	(10% FCI)		46.00	92,000
Taxes and Insurance (2% FCI)			<u>9.20</u>	<u>18,400</u>
Total Fixed Costs			\$124.20	\$248,000
Total Operating Costs			\$343.79	\$687,500
<u>Credit for Recovered Heat (80% Boiler Efficiency)</u>				
From Rubber Waste	7.93/Million KCal	3.96	31.40	63,000
From Auxiliary Fuel	7.93/Million KCal	2.11	<u>16.70</u>	<u>33,500</u>
Total Credit			\$ 48.10	\$ 96,500
Net Operating Cost			\$295.69	\$591,000

\* See footnotes to Table 6-4

FOOTNOTES TO TABLE 6-4

<u>Operating Labor</u>	<u>Annual Cost</u>
Pyrolyzer Feed System Operator 1 x 24 x 365 x 7.00 =	\$ 61,300
Pyrolyzer/Incinerator Operator 1 x 24 x 365 x 7.50 =	65,800
Helper 1 x 24 x 365 x 6.50 =	<u>56,900</u>
Direct Labor	\$184,000
Supervision (15% Direct Labor)	27,600
Supplies (20% Direct Labor)	36,800
Payroll Related Expense (35% Direct Labor)	<u>64,400</u>
Total Operating Labor	\$312,800

Electric Power

Forced Draft Blower 30 KW	$\left. \begin{array}{l} 75 \text{ Kwh/hr} \times \frac{1000}{250} = 300 \text{ Kwh/metric ton} \\ \text{Rubber Waste} \end{array} \right\}$
Extruder/Feeder 28 KW	
Rotary Hearth 12 KW	
Rubber Conveyor 5 KW	

TABLE 6-5  
CAPITAL INVESTMENT FOR PYROLYSIS, INCINERATION AND HEAT RECOVERY  
FOR 1000 METRIC TONS/YR OF RUBBER WASTE

Basis: 125 Kg/hr, 24 hrs/day, 330 days/yr

<u>Purchased Equipment</u>	<u>Size</u>	<u>Cost (March 1976\$)</u>
Forced Draft Blower	1,500 scfm	4,000
Rotary Hearth Pyrolyzer	4 ft diameter	113,000
Incinerator Burner	6 million Btu/hr	10,000
Instrumentation Package	-	11,000
Extruder/Feeder	300 lbs/hr	26,000
Feed Storage	500 cuft 5 days	2,000
Feed Conveyor (Belt)	100 ft	5,000
Heat Recovery Boiler	5 million Btu/hr	<u>27,000</u>
Purchased Equipment Cost		\$198,000
Installed Equipment Cost (IEC)		228,000
Piping (40% IEC)		91,000
Foundations (5% IEC)		11,000
Buildings and Structures (30% IEC)		68,000
Electrical (Including Instruments)		<u>50,000</u>
Total Physical Plant Cost (TPPC)		\$448,000
Engineering and Construction 30% TPPC		134,000
Contingency 20% TPPC		<u>90,000</u>
Total Capital Investment		\$672,000
Round to		\$670,000

TABLE 6-6

OPERATING COST FOR PYROLYSIS, INCINERATION AND HEAT RECOVERY  
FOR 1000 METRIC TONS/YR OF RUBBER WASTE

Basis: Fixed Capital Investment (FCI) \$670,000  
125 Kg/hr Rubber Waste  
Operation 24 hrs/day, 330 days/yr  
Rubber Waste Heat Value 5500 KCal/Kg (9800 Btu/lb) at 30% Water  
90% Conversion of Organics in Waste to Pyrolysis Gas

<u>Variable Costs</u>	<u>\$/Unit</u>	<u>Units per Metric Ton Waste</u>	<u>\$ per Metric Ton Waste</u>	<u>Annual Cost (\$)</u>
Operating Labor*			312.80	312,800
Utilities				
Oil or Gas	7.93/Million KCal (2.00/Million Btu)	2.66	21.11	21,100
Electricity*	.015 kwh	320	4.80	4,800
Maintenance (8% FCI)			53.60	53,600
Solid Waste	6.50/metric ton	0.12	<u>0.78</u>	<u>800</u>
Total Variable Costs			\$393.09	\$393,100
<u>Fixed Costs</u>				
Depreciation	(15% FCI)		100.50	100,500
Cost of Capital	(10% FCI)		67.00	67,000
Taxes and Ins.	(2% FCI)		<u>13.40</u>	<u>13,400</u>
Total Fixed Costs			\$180.90	\$180,900
Total Operating Costs			\$573.99	\$574,000
Credit for Recovered Heat (at 80% Boiler eff.)				
From Rubber Waste	7.93 Million KCal	3.96	31.40	31,300
From Auxiliary Fuel	7.93 Million KCal	2.11	<u>16.70</u>	<u>16,700</u>
Total Credit			\$48.10	\$48,000
Net Operating Cost			\$525.89	\$526,000

\*See Footnote to Table 6-6

FOOTNOTES TO TABLE 6-6

<u>Operating Labor</u>	<u>Annual Cost</u>
Pyrolyzer Feed System Operator 1 x 24 x 365 x 7.00 =	\$ 61,300
Pyrolyzer/Incinerator Operator 1 x 24 x 365 x 7.50 =	65,800
Helper 1 x 24 x 365 x 6.50 =	<u>56,900</u>
Direct Labor	\$184,000
Supervision (15% Direct Labor)	27,600
Supplies (20% Direct Labor)	36,800
Payroll Related Expense (35% Direct Labor)	<u>64,400</u>
Total Operating Labor	\$312,800

Electric Power

Forced Draft Blower 15 KW	} 40Kwh/hr x $\frac{1000}{125}$ = 320 Kwh/metric ton Rubber Waste
Extruder/Feeder 15 KW	
Rotary Hearth 5 KW	
Rubber Conveyor 5 KW	

TABLE 6-7

CAPITAL INVESTMENT FOR PYROLYSIS, INCINERATION AND HEAT RECOVERY  
300 METRIC TONS/YR OF API SEPARATOR BOTTOMS WASTE

Basis: 38 Kg/hr, 24 hrs/day, 330 days/yr

<u>Purchased Equipment</u>	<u>Size</u>	<u>Cost (March 1976\$)</u>
Forced Draft Blower	400 scfm at 2 psi	2,000
Rotary Hearth Pyrolyzer	2.3 ft diameter	83,000
Incinerator Burner		10,000
Instrumentation Package		11,000
Feed Pump		2,000
Feed Storage Tank	1,500 gal	1,500
Heat Recovery Boiler	1.2 million Btu/hr	<u>11,500</u>
Purchased Equipment Cost (PEC)		\$121,000
Installed Equipment Cost	(IEC)	\$140,000
Piping 40% IEC		56,000
Foundations 5% IEC		7,000
Building & Structures	(30% IEC)	42,000
Electrical (Including Instruments)		<u>50,000</u>
Total Physical Plant Cost (TPPC)		\$295,000
Engineering and Construction	30% TPPC	88,000
Contingency	20% TPPC	<u>59,000</u>
TOTAL CAPITAL INVESTMENT		\$442,000
Round to		\$440,000

TABLE 6-8

## OPERATING COST FOR PYROLYSIS, INCINERATION AND HEAT RECOVERY

## FOR 300 METRIC TONS/YR OF API SEPARATOR BOTTOMS WASTE

Basis: Fixed Capital Investment (FCI) \$440,000

38 Kg/hr Waste

Operation 24 hrs/day, 330 days/yr

API Waste Heat Value 1400 KCal/Kg (2500 Btu/lb) at 70% Water

75% Conversion of Organics in Waste to Pyrolysis Gas

<u>Variable Costs</u>	<u>\$/Unit</u>	<u>Units per Metric Ton Waste</u>	<u>\$ per Metric Ton Waste</u>	<u>Annual Cost (\$)</u>
Operating Labor*			373.33	112,000
Utilities				
Oil or Gas	7.93/Million KCal (2.00/Million Btu)	6.66	52.80	15,700
Electricity*	0.015/kwh	340	5.10	1,500
Maintenance (8% FCI/yr)			117.33	35,000
Solid Waste Disposal (@ 10% input)	6.50/metric ton	0.10	<u>0.65</u>	<u>200</u>
Total Variable Cost			\$549.21	\$164,400
<u>Fixed Costs</u>				
Depreciation	(15% FCI/yr)		220.00	66,000
Cost of Capital	(10 FCI/yr)		146.67	44,000
Taxes and Ins.	(2% FCI/yr)		<u>29.33</u>	<u>8,800</u>
Total Operating Cost			945.21	283,200
<u>Credit for Recovered Heat (80% Boiler Efficiency)</u>				
From API Waste	7.93/Million KCal	0.84	6.70	2,000
From Auxiliary Fuel	7.93/Million KCal	5.54	<u>44.00</u>	<u>13,200</u>
Total Credits			\$50.70	\$15,200
<u>Net Operating Costs</u>			\$894.51	\$268,000

\* See Footnotes to Table 6-8

# FOOTNOTES TO TABLE 6-8

<u>Operating Labor</u>	<u>Annual Cost</u>
Pyrolyzer System Operator 1 x 24 x 365 x 7.50	\$ 65,800
Supervision (15% Direct Labor)	9,900
Supplies (20% Direct Labor)	13,200
Payroll Related Expense (35% Direct Labor)	<u>23,100</u>
Total Operating Labor	\$112,000

## Electric Power

Forced Draft Blower 7 KW	}	$13 \frac{\text{kwh}}{\text{hr}} \times \frac{1000}{38} = 340 \text{ Kwh/Metric ton API Waste}$
Rotary Hearth 4 KW		
Feed Pumps 2 KW		

The labor requirements for the system would be the same for either 1000 or 6000 metric ton/yr of rubber waste (one full time operator for the feed system, one helper full time, and one operator full time for the pyrolyzer, incinerator and boiler). For the API bottoms waste, one operator (full time) should be able to handle the whole system.

The estimated auxiliary fuel and power requirements for the pyrolyzer were supplied by Surface Combustion. The additional power included in these estimates would be required for supplying compressed air to the pyrolyzer/incinerator and to drive the extruder/feeder or feed pump.

As indicated in each of these operating cost estimates, the credit for recovered heat is based on 80% recovery of the total heat input to the incinerator in a heat recovery boiler. The total heat input to the incinerator was taken as the total heat value of auxiliary fuel plus 90% of the gross heat value of the feed material in the case of rubber waste, and 75% of the gross heat value in the case of API waste. This assumed 90% conversion of the feed material organics to pyrolysis gas for the rubber waste and 75% conversion of the feed material organics to pyrolysis gas for the API waste.

As shown in Tables 1-1 and 5-11 the pyrolysis system was operated with the rubber waste to yield ash containing only 4-12% of the organics present in the feed. Although the material balance based on the analysis of the pyrolysis gas indicates that less than 90% of the organics in the rubber waste feed were converted to pyrolysis gas, only 80% of the organics in the feed were accounted for by the material balance for these test runs. Since there was no appreciable carbon (soot) formation in these test runs and since the organics in the ash (at 4-12%) could be more accurately measured than the weight of organics in the pyrolysis gas stream estimated, it is more likely that conversion ranged from 88-96% of organics to pyrolysis gas. For the purposes of these estimated operating costs 90% conversion of rubber waste organics to pyrolysis gas was assumed.

In the case of the API waste approximately 25% of the organics in the feed appeared in the ash (Table 5-3), so 75% conversion to pyrolysis gas was assumed.

## 7. CONCLUSIONS

### 7.1 GENERAL CONCLUSIONS ABOUT THE PYROLYSIS PROCESS

#### 7.1.1 Physical Characteristics of Suitable Wastes

The pyrolysis process is particularly well suited for destruction of solid or semi-solid wastes with high water or ash content.

#### 7.1.2 Chemical Characteristics of Suitable Wastes

The results of the tests indicate that an ideal waste, from the point of view of production of clean gaseous fuel for recovery, is highly aliphatic. For each of the wastes tested, the quantity of aliphatic component in the pyrolyzer effluent was correlated with the aliphatic content of the original waste. (In the case of styrene, the waste feed "aliphatic content" was in the form of alkyl substituents on aromatic compounds.) Aromatic waste feed components yield primarily aromatic effluent components, including substantial quantities of polynuclear aromatics. The aliphatic/aromatic content of the pyrolyzer effluent is of concern because aliphatics burn more cleanly in a subsequent heat recovery system.

#### 7.1.3 Operational Characteristics

The pyrolysis gases contain varying amounts of substances which condense at normal temperatures and pressures; consequently, these gases must be either combusted in a close-coupled heat recovery system or cleaned before they could be put into gas distribution systems. Because the chemical nature of the pyrolysis gas is similar to that from coking or gasification of coal, i.e., containing known carcinogens, the same occupational health and safety precautions are required. The operational characteristics of pyrolysis systems require the usual attention to controlling combustible and potentially explosive mixtures; however, these appear to be no more difficult to handle than similar problems in other processes.

#### 7.1.4 Economics

The capital investments and operating costs for a rotary hearth pyrolyzer are greater than a conventional incinerator of equivalent capacity. For this reason, the pyrolysis process is economically feasible only where energy recovery from waste materials cannot be effected in a less costly manner. Where thermal destruction of wastes containing high salt or ash content is required, or where difficult to control air pollution problems might result, the pyrolysis process may be the most economical.

### 7.2 API WASTE TESTS

#### 7.2.1 Resource Recovery

A total of about 70%-75% of the organic material in the waste was converted to a form which was combustible in the rich fume incinerator. Because the

original waste was largely aqueous, this corresponds to only 9% by weight of the total waste feed.

The pyrolysis gas was about 70% volatiles ( $\leq C_6$ ) and about 30% condensable aromatics at normal conditions.

#### 7.2.2 Solid Residue

The ASH, or solid residue, amounted to about 20% by weight of the total API waste feed, and was about 85% inorganic material.

#### 7.2.3 Potentially Hazardous Emissions

The API waste contains substantially higher levels of trace metals than typical high ash fuels such as coal. The major portions of these are found in the solid residue from the pyrolyzer. Less than 5% of the lead and zinc content of the waste is found in the pyrolyzer gas.

The sulfur content of the pyrolysis gas is  $136 \text{ mg/m}^3$  as sulfur.

The pyrolysis gas contains 3.2% of polynuclear aromatic hydrocarbons in the condensable fraction. This is equivalent to a pyrolysis emission rate of about  $350 \text{ mg/m}^3$ . Much of this material would probably be destroyed in a properly controlled heat recovery process.

#### 7.2.4 Engineering Considerations and Alternative Treatment Techniques

The high viscosity and ash content would make this waste unsuitable for a conventional liquid injection incinerator. This waste could be handled in a fluid bed incinerator, or, as these tests have shown, in a pyrolyzer. It would probably be more economical to dispose of this waste in a fluid bed incinerator, however, especially in view of the high water content and low heat value. In any case, the high viscosity of this waste would require a Moyno<sup>®</sup>, gear or other type of positive displacement pump for feeding the incinerator (or pyrolyzer).

#### 7.2.5 Economic Feasibility

The estimates indicate that construction of a pyrolysis facility to treat 300 metric tons per year of API waste would require a capital investment of \$444,000. The operating costs are estimated to be \$283,000 per year or \$945/ton of waste.

If allowance is made for recovered heat at \$7.93/million KCal (\$2.00/million Btu) operating cost is \$895/metric ton of waste.

### 7.3 STYRENE WASTE TESTS

#### 7.3.1 Resource Recovery

A total of about 57% of the organic material in the waste was converted to a form which was combustible in the rich fume incinerator. The pyrolysis gas was about 27% volatiles ( $\leq C_6$ ) and about 73% condensable aromatics at normal conditions.

### 7.3.2 Solid Residue

The ASH, or solid residue, amounted to about 0.5% by weight of the total waste feed, and was about 96% inorganic material.

### 7.3.3 Potentially Hazardous Emissions

The styrene waste contained only low levels of metals recognized as hazardous. The analyses indicate that none of these were present in the pyrolysis gas.

The sulfur content of the pyrolysis gas was  $753 \text{ mg/m}^3$  as sulfur. This was primarily carbon disulfide, carbonyl sulfide and sulfur dioxide.

The pyrolysis gas was found to contain 1.6% of polynuclear aromatic hydrocarbons, as pyrene in the condensable fraction. This is equivalent to an emission rate of  $400 \text{ mg/m}^3$ . Much of this material would probably be destroyed in an efficient heat recovery process.

### 7.3.4 Engineering Considerations and Alternative Treatment Techniques

Samples of this waste obtained before the test program indicated a relatively high viscosity. The waste actually obtained for the test was of much lower viscosity and could have been burned in a conventional liquid injection incinerator. Upon pyrolysis of the highly unsaturated chemical components considerable quantities of carbon particulates were generated which deposited in the off-gas duct work. This carbon particulate represents both a loss of fuel value and a potential handling problem.

### 7.3.5 Economic Feasibility

The economics of pyrolysis of this waste was not determined since the waste is not suitable for pyrolysis.

## 7.4 RUBBER WASTE TESTS

### 7.4.1 Resource Recovery

A total of about 80%-90% of the organic material in the waste was converted to a form which was deliverable to the rich fume incinerator. This corresponds to about 27% by weight of the original waste feed.

The pyrolysis gas was about 70% true volatiles ( $\leq C_6$ ) and about 30% condensable aromatics.

### 7.4.2 Solid Residue

The ASH, or solid residue, amounted to about 20% by weight of the total waste feed, and was about 80% inorganic material.

#### 7.4.3 Potentially Hazardous Emissions

The rubber waste contained significant concentrations of several metals recognized as potentially hazardous. The analyses indicated that these species are not present in the pyrolysis gas, but are concentrated in the ASH.

The sulfur content of the pyrolysis gas was  $189 \text{ mg/m}^3$  as sulfur.

The pyrolysis gas was found to contain 2.1% of polynuclear aromatic hydrocarbons in the condensable fraction. This is equivalent to an emission rate of  $490 \text{ mg/m}^3$ . Much of this material would probably be destroyed in an efficient heat recovery process.

#### 7.4.4 Engineering Considerations and Alternative Treatment Techniques

This waste is in a physical form (semi-solid lumps) which would make it very difficult to incinerate in virtually any other type of thermal destruction equipment. Even the destruction of this waste by pyrolysis requires that the waste be fed to the pyrolyzer in a thin enough layer on the hearth to allow complete pyrolysis. This can be accomplished by extruding the waste (in the proper thickness) directly onto the hearth.

An important factor in the thermal destruction of this waste by pyrolysis is the 80%-90% efficiency of conversion of the organic components in the waste to pyrolysis gas.

#### 7.4.5 Economic Feasibility

The estimates indicate the following costs for pyrolysis facilities to treat rubber waste:

<u>Capacity</u>	<u>Capital Investment</u>	<u>Operating Cost</u>	
		<u>Without Credit for Heat Recovery</u>	<u>With Credit for Heat Recovery</u>
1000 M.T./yr	\$670,000	\$574/M.T.	\$525/M.T.
2000 M.T./yr	\$920,000	\$344/M.T.	\$296/M.T.
6000 M.T./yr	\$1,500,000	\$165/M.T.	\$117/M.T.

## **APPENDIX A**

### **Techniques of Sample Preparation and Analysis**

- A. Extraction of Collected Samples**
- B. Analyses of Gaseous Effluents**
- C. Additional Analytical Techniques**
- D. Sample Identification Codes**
- E. Vendors for Outside Analyses**

APPENDIX A

TECHNIQUES OF SAMPLE PREPARATION AND ANALYSIS

A.1 EXTRACTION OF COLLECTED SAMPLES

A.1.1 Waste Feed Sample

A weighed aliquot of composited waste feed material was Soxhlet extracted with methylene chloride for 24 hours. The weights of residual and extractable material were determined by drying to constant weight at ambient temperature.

A.1.2 Pyrolysis Zone Sample Train Components

The contents of the knock-out impingers (Figure 4-1) and the pyrolysis zone probe washings (pentane plus acetone) were combined in the field. These samples were evaporated to dryness on a hot plate and the mass determined gravimetrically. The glass wool from the fourth impinger of the knock-out train was Soxhlet extracted for 24 hours with methylene chloride, then for 24 hours with methanol. The two extracts were combined and evaporated to dryness. The mass of extracted material was determined.

The pre-tared pyrolysis zone filter was dried to constant weight to determine the mass of collected material.

The filter, glass wool extract and dried knock-out trap samples were then combined in a Soxhlet thimble and extracted for 24 hours with methylene chloride. The extract was evaporated to dryness at ambient temperature and the total mass of extractable material determined. This is the fraction identified as GOO-SOL.

The sorbent trap was fitted into the specially designed extraction apparatus shown in Figure A-1 and extracted for 24 hours with pentane, then for 24 hours with methanol. The two extracts were individually evaporated to dryness at ambient temperature and the mass of material in each determined gravimetrically.

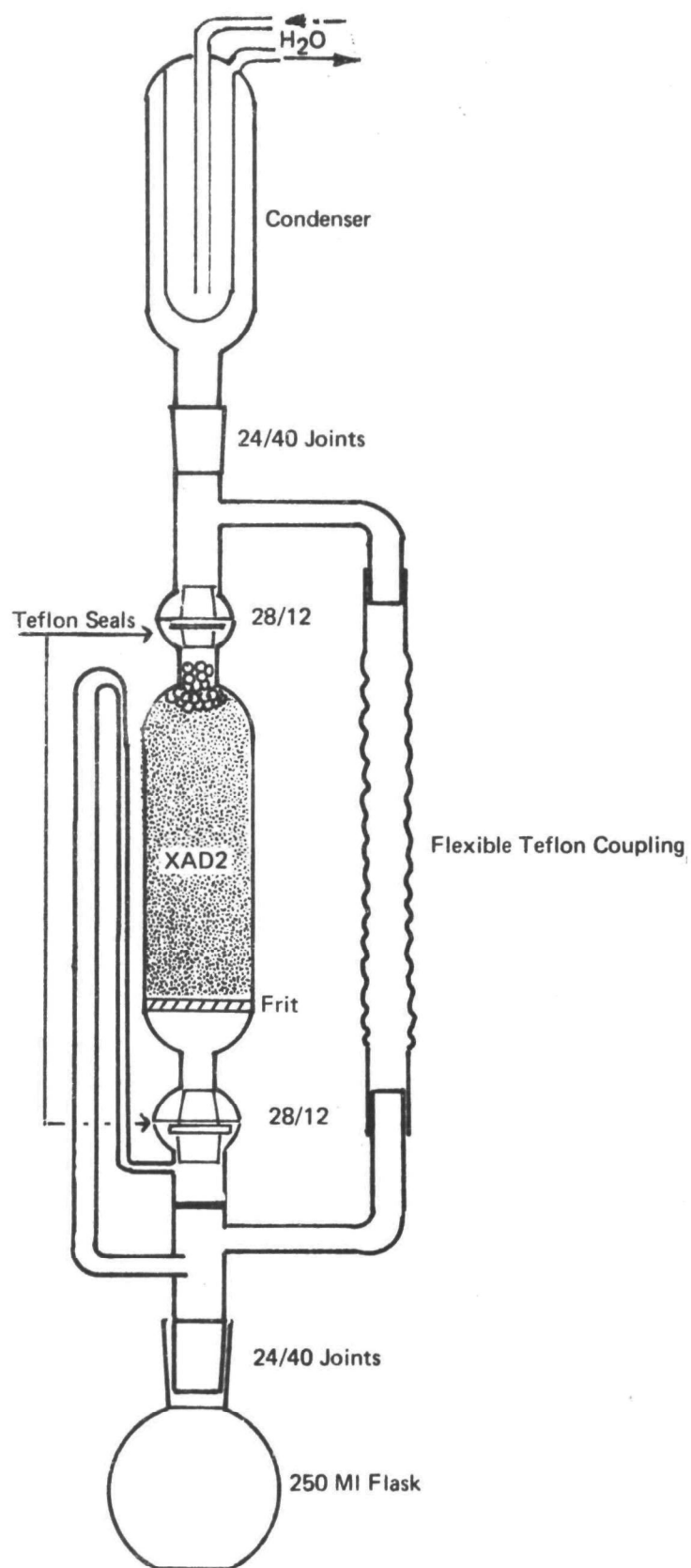


Figure A-1. Sorbent Trap Extractor

## A.2 ANALYSES OF GASEOUS EFFLUENTS

### A.2.1 On-Line Instruments

A continuous recording was made of the output of each of the five on-line instruments. In reducing the data, readings were made from the charts at 10 minute intervals and the values averaged. The range of values and the fluctuations in those values during the course of a run were:

Hydrocarbons	1.33 to 3.11%	$\pm 0.07$ to $0.37\%$
Carbon Monoxide	1436 to 2244 ppm	$\pm 8$ to $41$ ppm
Carbon Dioxide	10.1 to 11.1%	$\pm 0.06$ to $0.4\%$
Nitrous Oxide	64 to 100 ppm	$\pm 2$ to $10$ ppm
Oxygen	0.0%	$\pm 0.2\%$

The instruments were calibrated (zero and span) at least every two hours using the following gases (supplied with analyses by Matheson Gas Products Company).

<u>Analyzer</u>	<u>Zero Gas</u>	<u>Span Gas</u>	
Hydrocarbons	air	40 ppm C <sub>3</sub> H <sub>8</sub> in N <sub>2</sub>	} These were combined in one cylinder.
Carbon Monoxide	air	138 ppm CO	
Carbon Dioxide	air	12.4% CO <sub>2</sub>	
Nitrogen Oxides	air	432 ppm NO in N <sub>2</sub>	
Oxygen	CO, CO <sub>2</sub> , C <sub>3</sub> H <sub>8</sub> span gas	air	

The error introduced by use of span gas concentrations, very different from the measured sample gas concentrations (for hydrocarbons and carbon monoxide), would be expected to introduce an error of no more than 10%.

The NO<sub>x</sub> analyzer could not be operated in the NO<sub>x</sub> mode (which converts NO<sub>2</sub> to NO) for these sample streams. This is because the converter operates at a temperature of 750°C and NO is destroyed in the presence of large quantities of hydrocarbon and in the absence of oxygen.

### A.2.2 Gas Detecting Tubes

Bendix Gastec®\* tubes number 5 M, Sulfur Dioxide, Mid-Range

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\* Trademark of Bendix Environmental Science Division

(100-3600 ppm) and a Bendix hand sampling pump were used to monitor the stack effluent during API and styrene tests.

#### A.2.3 Gas Grab Samples

A metal bellows pump was used to transfer a portion of the pyrolysis zone gaseous effluent from the bypass line of the hydrocarbon analyzer to a 12 liter Saran gas sampling bag. The pumping rate was adjusted so that the sample was composited over a one hour period.

To eliminate losses due to diffusion, portions of the collected sample were transferred to glass bulbs with Teflon stopcocks. The 125 ml bulbs were evacuated and flushed with sample several times before filling.

The gas bulb samples were sent to an outside laboratory for qualitative and quantitative analysis. Unfortunately, the results of those analyses showed oxygen concentrations of 7% and higher, indicating that leakage occurred somewhere in the sampling procedure. The reported results were corrected to a zero oxygen concentration, but are inevitably less accurate than they should have been.

The results of these analyses were used primarily to determine an average molecular weight and carbon number for the very volatile portion of the pyrolyzer effluent. For this purpose it is only the relative abundances and not the absolute concentrations, of waste components which is important.

#### A.3 ADDITIONAL ANALYTICAL TECHNIQUES

A number of techniques were used for qualitative and quantitative analysis of waste feed and collected samples. These include:

##### Inorganic Species

X-Ray Fluorescence (XRF)

Atomic Absorption Spectroscopy (AAS)

Specific Ion Electrodes (SIE)

Gas-detecting tubes

##### Organic Species

Infrared Spectroscopy (IR)

Mass Spectroscopy (LRMS)

C,H,N,S Analysis

Gas Chromatography (GC)

Silica Gel Column Chromatography

These techniques were applied to the Surface Combustion samples where appropriate.

Analyses performed in ADL laboratories on the Surface Combustion samples included LRMS on a DuPont (CEC) 21-110B high resolution mass spectrometer using a glass inlet and solids probe for sample introduction, IR on a Perkin-Elmer 521 grating spectrophotometer, and gas chromatography using the system described below under boiling point distribution. Other analyses were performed by outside laboratories, listed in Section E of this appendix.

Because of the special features of the pyrolysis process investigated at Surface Combustion, some additional techniques were used to characterize the feed and effluent samples.

In a pyrolysis process, hydrocarbons are "cracked" to give organic species of lower molecular weight. In order to evaluate the Surface Combustion process, therefore, a number of methods were utilized which give an estimate of the molecular weight distribution in the analyzed sample. These methods, which are described briefly below, were Thermogravimetric Analysis (TGA), Boiling Point Distribution, and Gel Permeation Chromatography (GPC). It should be pointed out that these techniques do not provide a qualitative or quantitative determination of individual waste or effluent components; rather, they determine qualitative and quantitative changes in the distribution of sample components with respect to volatility and/or molecular weight. (Within a homologous series of organic compounds, volatility decreases monotonically with increasing molecular weight.)

#### A.3.1 Thermogravimetric Analysis

In a thermogravimetric analysis, the weight loss of a small (typically <50 mg) sample of material is recorded as the temperature of the sample is increased at a controlled rate.

In interpreting the TGA curves of the Surface Combustion samples, the criterion used was that a distinct change in the slope of the sample weight vs. sample temperature curve indicates the onset of a new "fraction" of the sample.

For the analyses a DuPont Model 950 system was used. The heating was performed in an inert (N<sub>2</sub>) atmosphere to minimize deterioration of the sample during analysis. The sample temperature was increased at a rate of 10-15°C/min. A typical curve is shown in Figure A-2.

#### A.3.2 Boiling Point Distribution

The boiling point distribution curve is an ASTM method for characterizing complex mixtures of hydrocarbons.\* In the procedure, a standard mixture of hydrocarbons is used to define a calibration curve of retention time vs. boiling point for a gas chromatographic analysis under carefully controlled conditions (e.g., carrier gas flow, temperature program).

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\*Standard Method of Test for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography, ASTM Designation: D2887-73.

PART NO. 950251

TGA (FURNACE C)



INSTRUMENTS

<b>SAMPLE:</b> O-STY-SOL-REP K. Thun SIZE <u>77.3</u> mg.	<b>X-AXIS</b>		<b>Y-AXIS</b>		RUN NO. _____ DATE <u>3/25/76</u>
	TEMP. SCALE <u>50</u> $^{\circ}\text{C}$ inch		SCALE <u>20</u> $\frac{\text{mg.}}{\text{inch}}$ (SCALE SETTING X2)		OPERATOR <u>LWG</u>
	SHIFT <u>0</u> inch		SUPPRESSION <u>0</u> mg.		HEATING RATE <u>10</u> $^{\circ}\text{C}$ min.
					ATM. <u>N<sub>2</sub> (dynamic v10)</u>
				TIME CONSTANT <u>2</u> sec.	

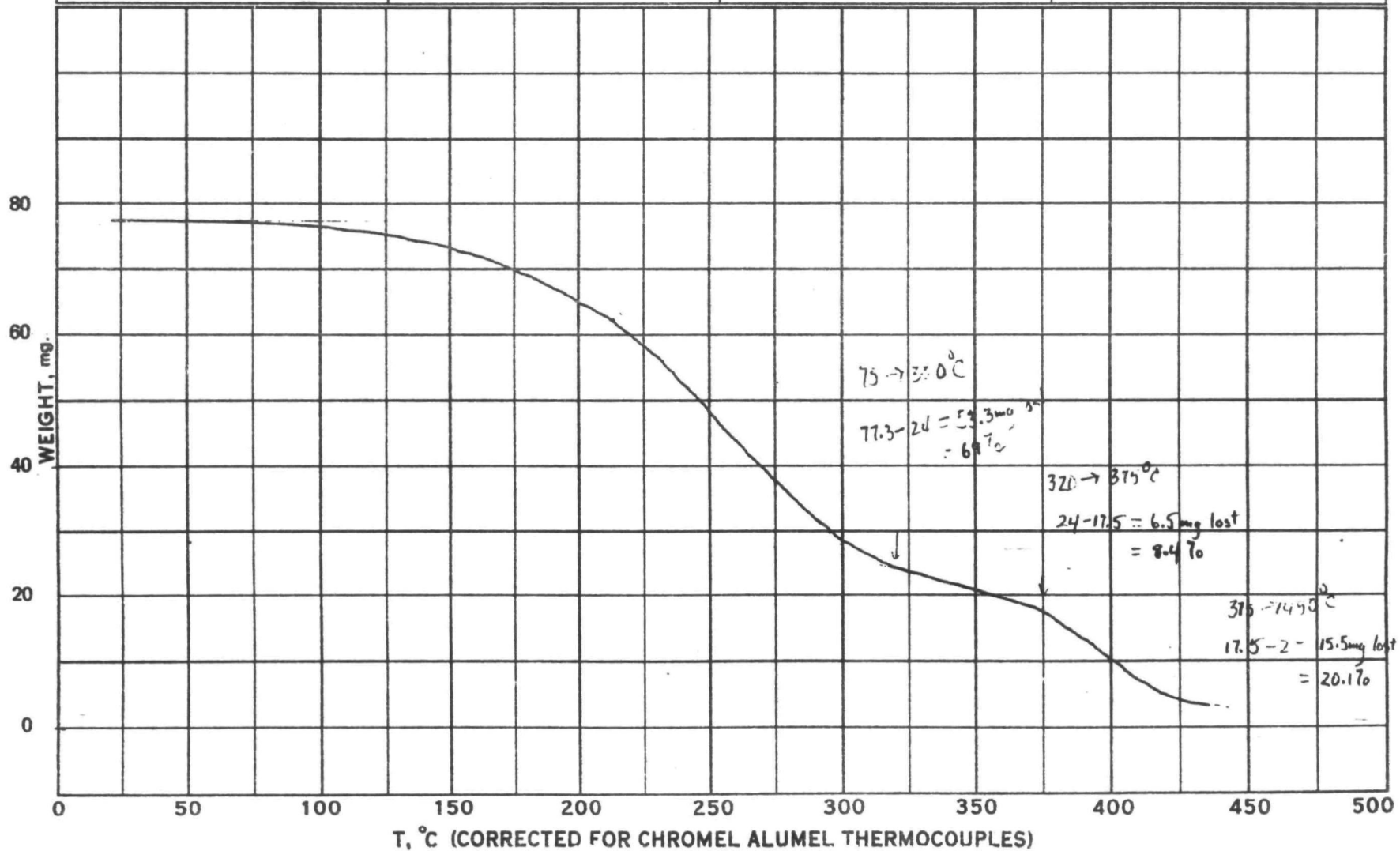


FIGURE A-2 TYPICAL TGA CURVE

The "unknown" sample is chromatographed under the same conditions and the integrated detector response for defined retention time intervals is determined. The retention time intervals are related to boiling point interval by use of the standard curve, and the cumulative amount of sample boiling at or below a given temperature is plotted against temperature. These analyses were done in ADL laboratories on a Varian Model 2700 gas chromatograph with a flame ionization detector. The column was 3% Dexsil®\* 400 on 100/120 mesh Supelcoport®\*, and the temperature was programmed from 60°C to 350°C at 10°/min. The detector response was integrated automatically with an Autolab System I integrator, which was specially modified to perform integrations over specific time intervals (rather than in response to changes in slope of detector output).

### A.3.3 Gel Permeation Chromatography (GPC)

This technique, in contrast to the previous two, relies on molecular size, rather than volatility, as an index of molecular weight. (Within a homologous series, molecular size varies monotonically with molecular weight.) The analysis is basically a chromatographic one, in which the stationary phase is a solid material with pores of defined size and the mobile phase is liquid. Molecules which are small enough to fill the pores of the stationary phase "see" a larger column volume than do those molecules which are too large to fit the pores. The retention time of smaller molecules in the column is therefore increased relative to that of large molecules. (This is the opposite of the situation in gas chromatographic methods, where retention time is longer for larger, higher molecular weight species.) To achieve adequate resolution, one customarily uses a series of columns of increasing pore size for a GPC analysis. The procedure is calibrated by use of polymers of known molecular weight.

In ADL laboratories, a Waters Model 6000A solvent delivery system, interfaced with a Model 440 Absorbance detector (256 nm) and Model R401 differential refractometer was used. The columns were Waters  $\mu$ -Styrogel®<sup>+</sup> of nominal pore size: 100 Å, 500Å, and 10<sup>4</sup>Å. Sample introduction was made with a Model U6K Universal Injector. The solvent was tetrahydrofuran and flow rate was 2.0 ml min<sup>-1</sup>.

A typical GPC output curve is shown in Figure A-3.

## A.4 SAMPLE IDENTIFICATION CODES

In the sections which follow, analytical results are reported for samples identified by codes which identify the source of the sample.

A.4.1 Each sample code begins with an Arabic numeral which identifies the run number (1-10).

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\*Trademark of Supelco, Inc.

+Trademark of Waters Associates

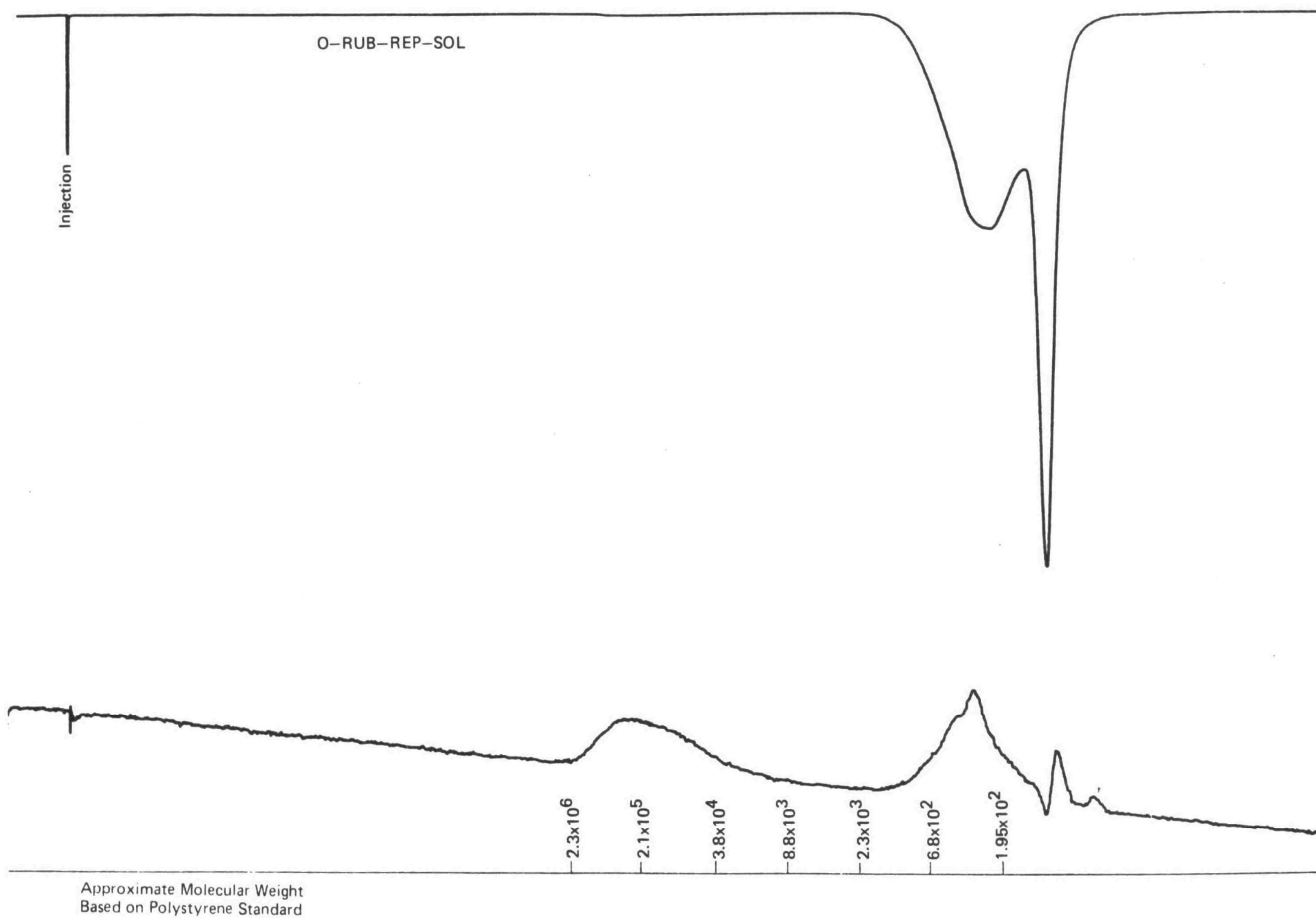


FIGURE A-3 TYPICAL GPC CURVE

A.4.2 The next syllable of the code identifies the waste tested: API - API Separator Bottoms, STY = styrene tars, SCB = Surface Combustion background, and RUB = rubber manufacturing wastes.

A.4.3 All effluent samples are coded with a -P- (pyrolysis zone sample) or -S- (stack sample) immediately following the waste designation.

A.4.4 The next syllable in the sample code indicates the specific source:

-REP-	composite of waste feed
-ASH-	residue from pyrolysis zone
-GB-	gas bulb
-PW-	probe wash
-KO-	knock-out trap
-GW-	glass wool from knock-out train
-F-	filter
-ST-	sorbent trap
-I-	impinger
-GOO-	combined KO + PW + F sample

A.4.5 The suffix -SOL- indicates that only the fraction of sample extractable with an organic solvent is included.

A.4.6 The suffixes -Pentane- and -Methanol- are used for the sorvent trap samples only, to identify the two organic extracts obtained from each trap.

## A.5 VENDORS FOR OUTSIDE ANALYSES

### A.5.1 Elemental analysis (C,H,N,S)

Galbraith Laboratories  
P.O. Box 4187 - Lonsdale  
2323 Sycamore Drive  
Knoxville, Tennessee 37921

### A.5.2 Spark Source Mass Spectrometry

Accu-Labs Research, Inc.  
11485 W. 48th Avenue  
Wheat Ridge, Colorado 80033

For these wastes only the REP samples were submitted for the extra-sensitive "spectrometric" analysis; others were submitted for the less exacting "geoscan" analysis.

### A.5.3 Mass Spectrometry of Gas Bulb Samples

Gollub Analytical Service Corporation  
47 Industrial Road  
Berkeley Heights, New Jersey 07922

## **APPENDIX B**

### **Sampling and Analytical Results**

- B.1 Sampling Data**
- B.2 Gravimetric Data**
- B.3 Selection of "Typical" Runs**
- B.4 Chemical Analyses of API Waste Samples**
- B.5 Chemical Analyses of Styrene Waste Samples**
- B.6 Chemical Analyses of Rubber Waste Samples**
- B.7 Chemical Analyses of Background Test Samples**

## APPENDIX B

### Sampling and Analysis Data

#### B.1 SAMPLING DATA

Table B-1 presents the data obtained by the EPA Method 5 procedure sampling of the stack gases. Table B-2 indicates the volumes of pyrolyzer effluent sampled by the comprehensive sampling train.

#### B.2 GRAVIMETRIC DATA

In Tables B-3 - B-6 are presented the absolute values of sample weights determined for all 10 tests at Surface Combustion. Examination of the data for solvent, sorbent trap and Soxhlet thimble controls in Table B-6 indicates that the values in B-3 through B-6 are uncertain by  $\pm 0.02$  g.

One striking feature of the data in Tables B-3 through B-5 is that, for some samples, the sum of soluble fraction and residual fraction is considerably less than the initial sample weight. This is particularly true for O-API-REP (74% lost), 1-API-P-ASH (16.9% lost), 3-API-P-ASH (38.3% lost) and O-RUB-REP (30.4% lost). This weight loss is primarily due to water in the sample, although some low boiling organic material is apparently also lost from the O-API-REP sample.

The significance of the ways in which the sample mass is distributed among the various effluent fractions is discussed elsewhere in this report.

#### B.3 SELECTION OF "TYPICAL" TESTS

Preliminary analyses of all of the waste effluent samples showed that the samples obtained from the three tests on each waste have similar composition. The degree of similarity can be illustrated by the elemental analysis of the three API-GOO samples:

	<u>% C</u>	<u>% H</u>	<u>% N</u>	<u>% S</u>
1-API-P-GOO-SOL	85.05	6.43	0.76	1.56
2-API-P-GOO-SOL	86.64	6.74	0.97	1.40
3-API-P-GOO-SOL	86.21	6.28	0.90	1.33

Another indication is the virtual identity of the gas chromatograms and IR spectra of corresponding samples for the three tests on a waste. For example, Figure B-1 shows the gas chromatograms for the ST-Pentane extracts for the three styrene burns. The similarity of chemical composition is not surprising, since the operational range of the pilot scale pyrolysis unit was found to be relatively restricted.

Date	Run Number	STACK PARAMETERS								SAMPLING PARAMETERS				Isokineti- city %	PARTICULATE DATA			
		Temp. °C	Press min Hg	O <sub>2</sub> %	CO <sub>2</sub> %	Excess Air %	H <sub>2</sub> O, %	Stack Gas Vel. m/sec	Volumetric Gas Velocity m <sup>3</sup> /sec	Avg. Nozzle Velocity m/sec	STP Dry Volume Sampled m <sup>3</sup>	STP Wet Volume Sampled m <sup>3</sup>	STP Total Volume Sampled m <sup>3</sup>		Probe Catch mg	Filter Catch mg	Total Catch mg	Normalized Catch mg/m <sup>3</sup>
1/18	1-API-S	355	744	15.6	1.0	243	1.8	29.8	2.18	30.1	1.491	0.028	1.519	101.0	41.0	*		
1/29	2-API-S	362	743	15.6	1.0	243	2.4	29.5	2.16	29.7	1.444	0.036	1.480	100.8	70.0	59.6	129.6	87.6
1/30	3-API-S	355	738	16.6	1.6	332	2.6	28.9	2.11	28.7	1.391	0.038	1.429	99.2	20.0	12.9	32.9	23.0
2/2	4-STY-S	360	744	16.0	2.0	283	2.7	29.1	2.12	29.2	1.412	0.040	1.452	100.2	20.5	*		
2/3	5-STY-S	364	746	17.0	2.0	388	2.8	29.1	2.12	29.8	1.441	0.041	1.482	102.5	25.2	15.5	40.7	27.5
2/4	6-STY-S	396	753	16.4	2.0	319	2.3	28.1	2.05	28.5	1.329	0.031	1.360	101.6	30.2	28.6	58.8	43.2
2/5	7-SCB-S	339	753	16.6	1.0	322	2.3	30.2	2.20	30.0	1.528	0.036	1.564	99.5	8.2	5.2	13.4	8.6
2/17	8-RUB-S	342	745	16.4	1.0	303	2.8	27.5	2.01	28.8	1.435	0.042	1.477	104.6	8.0	7.2	15.2	10.3
2/18	9-RUB-S	337	730	16.4	1.6	312	3.6	28.8	2.10	28.7	1.410	0.052	1.462	99.8	13.5	7.0	20.5	14.0
2/18	10-RUB-S	323	730	16.0	2.0	283	2.9	27.7	2.02	28.2	1.430	0.043	1.473	102.0	6.0	7.4	13.4	9.1

\* Sample Lost

TABLE B-1. Data Obtained by EPA Method 5 Procedure

Total Sample Volumes from Pyrolyzer at Surface Combustion					
Date of Sample	Sample Identifi- cation	Measured Dry Volume @ STP m <sup>3</sup>	Calculated Moisture Con- tent, %	Wet Volume @ STP	Total Sample Volume STP
1/28	1-API	0.895	23.9	0.281	1.176
1/29	2-API	0.900	21.7	0.249	1.149
1/30	3-API	0.890	25.3	0.301	1.191
2/2	4-STY	0.357	9.4	0.037	0.394
2/3	5-STY	0.808	8.9	0.079	0.887
2/4	6-STY	0.539	8.4	0.049	0.588
2/5	7-SCB	0.897	8.1	0.079	0.976
2/17	8-RUB	0.329	16.4	0.064	0.393
2/18	9-RUB	0.306	14.7	0.053	0.359
2/18	10-RUB	0.280	15.1	0.050	0.330

TABLE B-2. Volumes Sampled by Comprehensive Sampling Train

TABLE B-3

RESULTS OF GRAVIMETRIC ANALYSES ON API SAMPLES

Sample Source*	Weight in Grams			
	0-API	1-API	2-API	3-API
-REP-				
aliquot size	75.6330			
-SOL-	9.8184			
-RES-	9.8396			
net loss		74%		
-P-KO		2.1927	1.8148	1.7455
-P-F		.5607	.5341	.4823
-P-GW-SOL**		.0997	.7838	.4942
Total GOO		2.8531	3.1327	2.7220
-P-GOO-SOL		2.2207	2.4519	2.4519
-P-GOO-RES		.8615	.4851	.5308
-D-ST-Pentame		1.5227	1.4614	1.0571
-P-ST-Methanol		.0299	.0258	.0259
P-ASH				
aliquot size		34.9361	28.2394	28.8325
ASH-SOL		6.0018	4.3498	5.5473
ASH-RES***		23.0509	23.9021	12.2458

\* See SAMPLE IDENTIFICATION CODE, Appendix A.

\*\* There was no tare weight on the glass wool, so residue weight is unknown.

\*\*\* Residue was dried at 110°C for 1 hour.

**TABLE B-4**  
**RESULTS OF GRAVIMETRIC ANALYSIS ON STYRENE SAMPLES**

Sample Source*	Weight in Grams			
	0-STY	4-STY	5-STY	6-STY
-REP-				
aliquot size	58.6941			
-SOL-	57.5449			
-RES-	.3214			
net loss		1.4%		
-P-KO		3.5298	19.3956	13.0635
-P-F		.5062	.3388	4.7876
-P-GW-SOL**		2.2621	9.5498	1.6077
Total GOO		6.2981	29.2842	19.5688
-P-GOO-SOL		5.7364	25.6970	15.7280
-P-GOO-RES		.4487	3.5044	3.3529
-P-ST-Pentame		2.7158	***	5.6780
-P-ST-Methanol		.0610		.0383
-P-ASH				
aliquot size		34.6688	31.2235	20.6560
ASH-SOL		10.4547	4.273	.7630
ASH-REB		28.8343	30.8137	22.9033

\*See SAMPLE IDENTIFICATION CODE, APPENDIX A.

\*\*There was no tare weight on the glass wool, so residue weight is unknown.

\*\*\*The sorbent trap broke during overnight pentame extraction.

**TABLE B-5**  
**RESULTS OF GRAVIMETRIC ANALYSES ON RUBBER SAMPLES**

Sample Source*	Weight in Grams			
	<u>0-RUB</u>	<u>8-RUB</u>	<u>9-RUB</u>	<u>10-RUB</u>
-REP-				
Aliquot size	36.0786			
-SOL-	12.0133			
-RES-	13.0938			
net loss	30.4%			
-P-KO		1.2054	1.0062	1.5885
-P-F		.5423	.4431	.3404
-P-GW-SOL**		.1531	.2830	.0572
Total GOO		1.9008	1.7323	1.9861
-P-GOO-SOL		1.4266	1.2103	1.4613
-P-GOO-RES		.3779	.3159	.4737
-P-ST-Pentane		.3935	.6288	.3710
-P-ST-Methanol		.0278	.0231	.0324
-P-ASH-				
Aliquot size		15.3127	17.3026	16.6701
ASH-SOL		7.0555	3.1898	1.5962
ASH-RES		9.5249	14.7255	15.5166

\*See SAMPLE IDENTIFICATION CODE, APPENDIX A.

\*\*There was no tare weight on the glass wool, so residue weight is unknown.

**TABLE B-6**  
**RESULTS OF GRAVIMETRIC ANALYSES OF**  
**BACKGROUND SAMPLES AND CONTROLS**

Sample Source*	Weight in Grams			
	<u>7-SCB</u>	<u>SOXHLET THIMBLE CONTROL</u>	<u>SORBENT TRAP CONTROLS</u>	<u>SOLVENT BLANK</u>
-P-KO	.1207			
-P-F	.0291			
-P-GW-SOL*	.0349			
-P-GOO-SOL	.1378	-0.0185		- .0224
-P-GOO-RES	.1138	- .0204		
-P-ST-Pentane	.1229		.0145; .0052	
-P-ST-Methanol	.0211		.0087; .0198	
-P-ASH-SOL	****	+0.0544		
-P-ASH-RES		+0.0136		

\*See SAMPLE IDENTIFICATION CODE, APPENDIX A.

\*\*There was no tare weight on the glass wool, so residue weight is unknown.

\*\*\*There was no ash from the background burn.

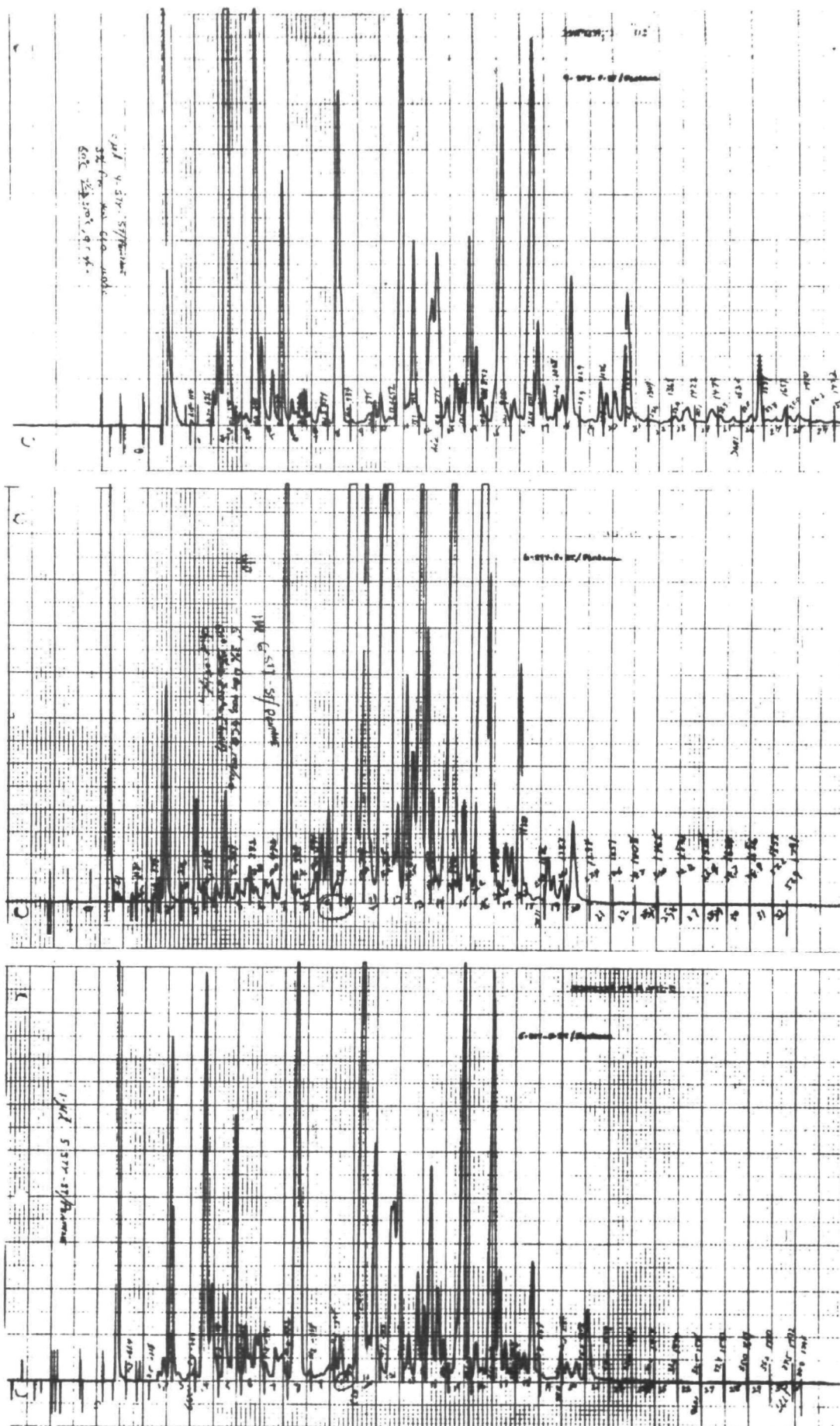


FIGURE B-1 GAS CHROMATOGRAPHS OF PENTANE EXTRACTS OF SORBENT TRAPS FOR 4-STY, 5-STY, AND 6-STY TESTS

Consequently, a set of samples corresponding to one test condition for each of the wastes was selected for detailed chemical analysis. The selection criteria were:

- The "typical" run should not be the first test on that waste. This eliminates memory effects in the pyrolysis unit and sample lines.
- No sample should have been lost for that run.
- The typical run should not correspond to the extremes of variations in feed rate, pyrolyzer temperature, etc.

The runs selected for most detailed analysis were: 2-API, 6-STY, 9-RUB and the background test, 7-SCB.

#### B.4 CHEMICAL ANALYSES OF API WASTE SAMPLES

##### B.4.1 Data From On-Line Analyzers

<u>Run</u>	<u>Hydrocarbons % (as CH<sub>4</sub>)</u>	<u>CO ppm</u>	<u>CO<sub>2</sub> %</u>	<u>O<sub>2</sub> %</u>	<u>NO ppm</u>
1-API	1.33 ± 0.07	1436 ± 13	10.8 ± 0.3	0.0 ± 0.2	100 ± 4
2-API	1.26 ± 0.13	1966 ± 32	11.1 ± 0.4	0.0 ± 0.2	94 ± 2
3-API	1.2 ± 0.1	2174 ± 35	11.1 ± 0.4	0.0 ± 0.2	95 ± 4

The error estimates are standard deviations of individual (10 minute interval) readings from the mean.

##### B.4.2 Gas Bulb Analyses

The results of analyses by Gollub Analytical Service Corp. corrected to zero oxygen concentration (see Appendix A-2) are shown below. The error in the tabulated values is estimated to be ± 100 ppm.

Concentration, ppm by volume

	<u>1-API</u>	<u>2-API</u>	<u>3-API</u>
Carbon Dioxide	6.2%	6.9%	7.7%
Carbon Disulfide	<80	<80	4100
Carbonyl Sulfide	<80	<80	380
Sulfur Dioxide	<80	<80	1100
Hydrogen	<30	<30	7900
Methane	2300	2400	4100
Ethane	360	390	380
C <sub>3</sub> -C <sub>5</sub> Hydrocarbon	660	680	1100
Benzene	670	590	690
Toluene	310	240	290
Xylene	<80	<80	<80
Acetylene	2800	2400	3500

From these data it was calculated that the average molecular weight of the hydrocarbon material in the gaseous pyrolyzer effluent is 32 and the average carbon number is 2.3. These estimates imply a C:H weight ratio in the volatile hydrocarbon fraction of 6.27.

B.4.3 Elemental Analysis of Major Constituents

The data obtained were:

	<u>% C</u>	<u>% H</u>	<u>% N</u>	<u>% S</u>
0-API-REP-SOL	84.72	11.87	0.16	1.24
2-API-P-GOO-SOL	86.64	6.74	0.97	1.40
2-API-API-P-ASH	19.70	2.42	0.67	3.38

These values have an estimated error of  $\pm 0.05$ .

These data show, first, that both GOO and ASH have a higher nitrogen content than the waste feed. The ash is also enriched in sulfur relative

to the feed. These observations suggest that the nitrogen and sulfur in the waste feed were present as nitrate and sulfate ions.

The data can also be used to calculate C:H weight ratios. These ratios are 7.14 for REP-SOL, 12.85 for GOO, and 8.14 for ASH. The values indicate that the GOO sample is much higher in unsaturates than the feed, while the organic content of the ASH is only slightly less saturated than the feed.

#### B.4.4 IR Spectra

##### O-API-REP-SOL

The curve was dominated by peaks corresponding to aliphatic hydrocarbons with a small percentage of aromatic bands.

<u>Absorption Maximum Frequency, <math>\text{cm}^{-1}</math></u>	<u>Assignment</u>
3100-3000 (w)	aromatic CH stretch
2960, 2930, 2880, 2860 (s)	aliphatic CH stretch
1600, shoulder at 1500 (w)	aromatic ring stretch
1460, 1380 (m)	aliphatic CH band
879, 810, 750, 730, 700 (w)	aromatic ring substitution patterns

##### 2-API-P-GOO

The IR spectrum for this sample was qualitatively different from that of the REP. The aromatic bands at 3100-3000, 1600 and 1500, and in the  $870-700\text{ cm}^{-1}$  range were all of moderate intensity. In addition there were new, weak bands at 1705 and in the  $1250-1150\text{ cm}^{-1}$  range which correspond to an oxygenated species. This may be an ester of an  $\alpha,\beta$ -unsaturated acid or, more probably, an aromatic ketone. There is also a very weak band at  $2230\text{ cm}^{-1}$  which corresponds to the  $\text{-C}\equiv\text{X}$  stretching region; this probably arises from an alkyne component of the sample.

##### 2-API-P-ASH-SOL

In contrast to the GOO, the soluble portion of the ASH sample has an IR spectrum virtually identical to that of the REP.

##### 2-API-P-ST-Pentane

The IR spectrum is similar to that of the GOO and indicates that this sample is more highly aromatic than the REP. There is also evidence of the presence of oxygenated material (weak bands at 1705 and  $250-1150\text{ cm}^{-1}$ ).

#### 2-API-P-ST-Methanol

The IR spectrum of this sample showed a broad absorption in the OH stretching region, indicating incomplete removal of solvent. In addition, the bands at 1705 and 1250-1150  $\text{cm}^{-1}$  were of much greater intensity than in any other sample.

In summary the IR data indicate that the gaseous effluent from the pyrolyzer (GOO and ST samples) is more highly aromatic than the waste feed. This effluent also is enriched in oxygenated species relative to the feed. The organic content of the ASH, appears to be mostly unpyrolyzed feed material.

#### B.4.5 Results of LRMS Analyses

In Table B-7 are the data obtained from LRMS analyses of the API representative waste sample and the effluent samples from Run 2 on this waste. The lower limit of detection was about 0.1% of the sample introduced to the instrument, but compounds present at or above this concentration accounted for >87% of the total volatilizable sample.

#### 0-API-REP

The data show that the representative waste feed sample was composed of 42.7% aliphatic hydrocarbons, of which 35% were unsaturated. The remainder of the major components of the REP sample were aromatics of up to 3 fused rings (benzene, naphthalene, phenanthrene, and anthracene), alkyl derivatives of these aromatics, and phenyl-substituted alkenes. None of the species usually referred to as polynuclear aromatics (pyrene, benzpyrene, etc.) were detected in the waste feed.

#### 2-API-P-ASH-SOL

The saturated and unsaturated aliphatics which were found in the REP sample are virtually absent from the ASH sample. This would seem to be consistent with the IR data. In fact, however, the ASH does appear to be substantially enriched in alkyl substituted aromatics. Three species, methyl-, dimethyl- and propyl- naphthalene, account for 31.7% of the ASH sample.

The ASH sample aromatics are distributed over roughly the same molecular weight range as are those of the REP.

#### A-API-P-GOO-SOL

Like the ASH, the GOO fraction of the effluent contains no purely aliphatic species but does contain alkyl substituted aromatics. The

TABLE B-7  
SPECIFIC COMPOUNDS IDENTIFIED IN FEED AND EFFLUENT SAMPLES FOR 2-API TEST

MW	COMPOUND	REP.	Concentration, %		
			ASH	GOO	ST-Pentane
	Aliphatics	42.7			
106	Ethyl Benzene	1.3			
116	Indene				6.5
118	Indane				2.6
120	Trimethyl Benzene	2.0	2.5		3.2
128	Napthalene				24.8
130	Methyl Indene				4.7
132	Methyl Indane/Dimethyl Styrene				2.3
134	Tetramethyl Benzene	1.7			2.6
142	Methyl Napthalene	2.3	4.5		11.2
152	Biphenylene/Acenapthylene				7.6
154	Biphenyl/Acenapthene				2.5
156	Dimethyl Napthalene		13.2		7.4
166	Fluorene			1.5	4.3
168	Diphenyl Methane	1.4			2.3
170	C <sub>13</sub> H <sub>14</sub> Propylnapthalene	4.0	14.0	1.2	3.0
178	Anthracene/Phenanthrene	1.4	4.5	5.4	4.5
180	Stilbene/Methyl Fluorene	1.8	3.7	2.8	2.0
182	Diphenyl Ethane		5.8	1.2	1.3
184	Butyl Napthalene	4.2	7.0	1.2	1.2
190	Methylene Phenanthrene			1.4	
192	Methyl Phenanthrene	2.7	3.7	4.9	1.5
194	Diphenyl Propene/Methyl Stilbene	1.6	2.5	1.9	
196	Diphenyl Propane	2.9	4.9	1.0	
198	C <sub>5</sub> Alkyl Napthalene	2.3	3.3	1.1	
202	Pyrene			10.9	
204	Phenyl Napthalene		2.1	2.3	
206	Dimethyl Phenanthrene	3.0	3.3	4.2	
208	Methyl Phenylindane/Hexahydropyrene	1.3	2.1	1.8	
210	Diphenyl Butane	2.0	2.9		
212		2.2	2.1		
216	Methyl Pyrene			5.2	
218				2.1	
220	Trimethyl Phenanthrene	2.1	1.6	2.6	
222		1.0	1.6	1.0	
224		1.2	1.6		
226	Benz Fluoranthene		1.2	4.7	
228	Chrysene/Napthacene/Benzanthracene			4.0	
230	Terphenyl			3.8	
232	C <sub>18</sub> H <sub>16</sub>			1.5	
234	Butyl Anthracene	1.0		1.7	
236	Diphenyl Thiophene	1.0	1.2	1.0	
238	Decahydro Benzanthracene		1.2		
240	Dodecahydro Bencanthracene/C <sub>8</sub> Napthalene		1.2	2.0	
242	Methyl Chrysene/Methyltriphenylene			2.0	
244	Triphenyl Methane			1.8	
252	Benzpyrene			4.2	
254	Binapthyl			1.9	
256	C <sub>2</sub> Benzanthracene, etc.			1.5	
258				1.1	
266				1.3	
276	Anthanthrene			1.2	
326	C <sub>21</sub> H <sub>26</sub> O <sub>3</sub>			1.9	
	TOTAL	87.1	91.7	89.3	95.5

$$\begin{aligned}
 \text{Aliphatics} &= 2n + 2 = 7.7\% \\
 2n &= 9.3 \\
 2n - 2 &= 6.5 \\
 2n - 4 &= 4.1 \\
 2n - 6 &= 6.3 \\
 2n - 8 &= 4.8 \\
 2n - 10 &= 4.0
 \end{aligned}$$

42.7

molecular weights of GOO constituents are shifted to a range about 50 amu units higher than that of the REP and ASH aromatics.

Of particular significance is the appearance in the GOO fraction of the higher polynuclear aromatics including 10.9% pyrene, and 4.2% benzpyrene, among others.

Also of interest is the tentative identification of the mwt 326 peak as hydroxy octoxybenzophenone in the GOO. This compound is possibly responsible for the carbonyl peak observed in the IR spectrum of the GOO sample.

#### 2-API-P-ST-Pentane

Again, no aliphatics are found. The aromatic species identified are all of molecular weight <200. This is a definite shift to lower molecular weight compared to REP.

#### B.4.6 TGA Data

As noted in Appendix A, the TGA data are reported as the percentage of original sample mass lost in temperature intervals defined by distinct changes in slope of the sample weight versus sample temperature curve.

O-API-REP-SOL	25-100°C	0 %
	100-355	85.7
	355-450	<u>6.7</u>
	Total	92.4
2-API-P-GOO-SOL	25-100°C	0 %
	100-580	71.7
	580-750	<u>2.0</u>
	Total	73.7
2-API-P-ST-Pentane	25-250°C	<u>97.5</u>
	Total	97.5
2-API-P-ASH-SOL	20-275°C	62 %
	275-500	<u>35.2</u>
	Total	97.2

It is difficult to interpret these data to yield quantitative comparisons of the feed and effluent samples. Qualitatively, it is clear that the GOO sample is less volatile than the REP, with 26% of the sample remaining after heating to 750°C. The sorbent trap sample, on the other hand, is much more volatile than the REP. Finally, the ASH-SOL sample is slightly more volatile than the waste feed.

#### B.4.7 Boiling Point Distribution Curves

The boiling point distribution curves for the feed and effluent samples for the 2-API test are shown in Figure B-2.

These data confirm the results of the TGA experiments. The sorbent trap curve is shifted to lower boiling points than the REP-curve. The ASH-SOL curve is slightly displaced and the GOO-SOL curve more markedly displaced towards higher boiling points. (The apparent shifts in volatility are less dramatic in the boiling point data than in the TGA data, because the former are normalized in a way which excludes the totally non-volatile portion of the sample.)

#### B.4.8 SSMS Analyses for Trace Constituents

A portion of the O-API-REP sample was subjected to spectrometric SSMS analysis. This procedure, which has a detection limit of 0.01 ppm and a precision of  $\pm 100\%$ , identified a total of 63 elements in the waste. In addition, mercury was found to be present at a concentration of 1.7 ppm. In Table B-8 are the SSMS data for all elements found at concentrations  $>5$  ppm.

Also in Table B-8 are data obtained by a less sensitive SSMS technique (detection limit 1 ppm and precision  $\pm 500\%$ ) for two effluent samples: 2-API-P-ASH and 2-API-S-F. These data indicate that most of the trace elements in the feed are emitted from the pyrolyzer in the ASH. Detectable levels of a few elements, however, appear in the stack filter sample.

#### B.4.9 Gastec® Analysis

Analysis of the stack effluent with Gastec® tubes showed 30-50 ppm of  $\text{SO}_2$  for all three tests.

#### B.4.10 Analyses of Impinger Solutions

Aliquots of the 2-API-P-I and 2-API-S-I impinger samples were oxidized with hydrogen peroxide, boiled to destroy excess oxidant, then analyzed for sulfate by the barium chloranilate method. The results were:

<u>Sample</u>	<u>Concentration as <math>\text{SO}_4^{=}</math>, ppm</u>	<u>Total Sulfur in Impingers, as S, mg</u>
2-API-P-I	910	156
2-API-S-I	495	71

The amount of sulfur detected in the pyrolysis zone impinger sample is 8.5% of the quantity which would have been expected if all sulfur in the waste feed had been converted to gaseous acidic sulfur species

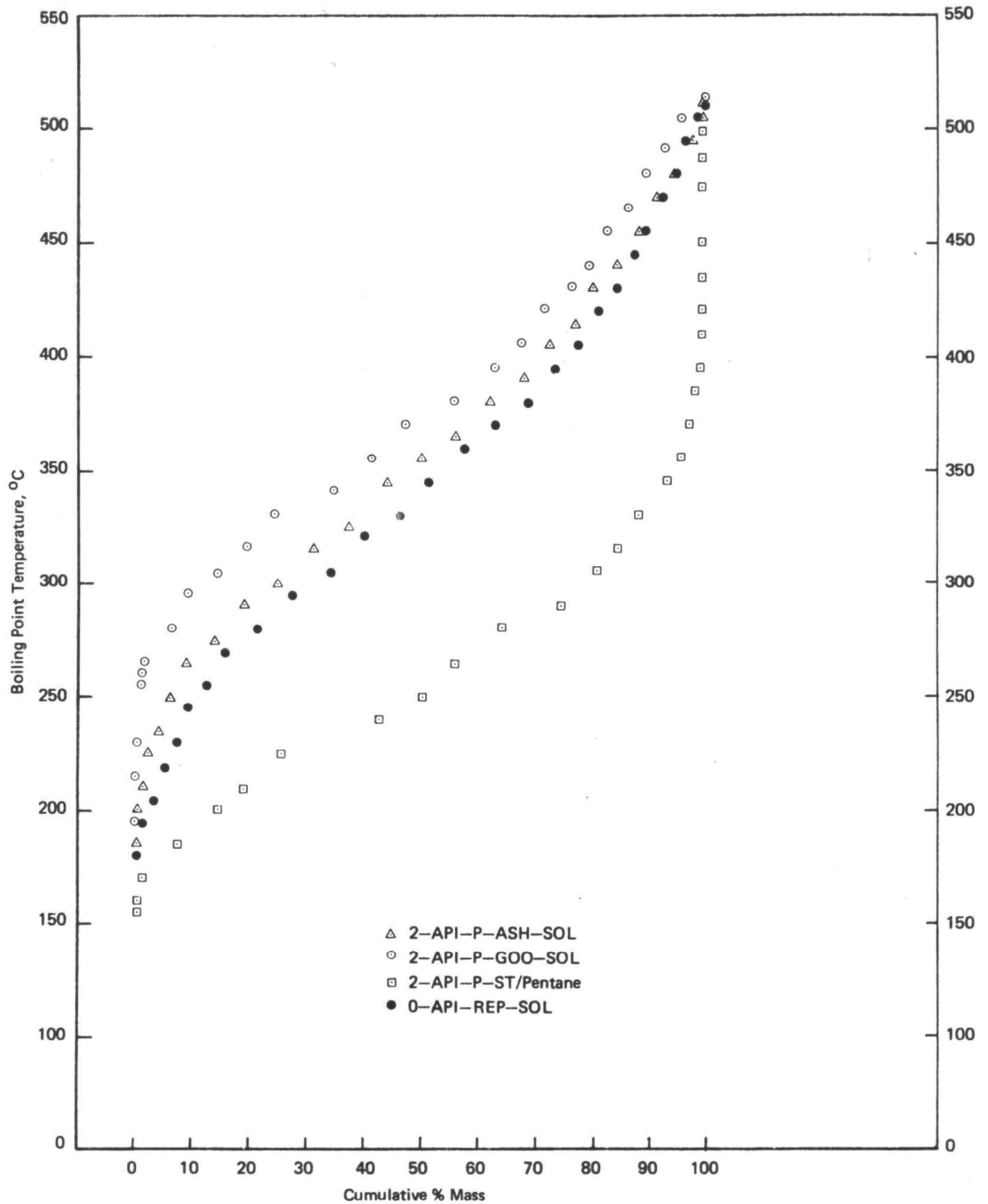


FIGURE B-2 BOILING POINT DISTRIBUTION CURVES FOR  
SAMPLES FROM 2-API TEST

**TABLE B-8. SSMS Data for API Feed and Effluent Samples**

	<u>0-API- REP</u>	<u>2-API-P ASH</u>	<u>2-API- S-F*</u>
Aluminum	> 1%	> 1%	
Calcium	> 1%	> 1%	
Silicon	> 1%	> 1%	
Sulfur	> 1%	> 1%	
Magnesium	> 0.5%	> 1%	
Phosphorus	> 0.5%	~ 1%	
Iron	~4600ppm	> 1%	
Sodium	~2500	~ 0.5%	
Potassium	~1000	~ .3%	
Zinc	~1000	> 1%	93
Strontium	810	~1000ppm	
Barium	740	~1000	
Titanium	540	~3000	
Chromium	420	~3000	
Copper	410	~ 0.5%	
Fluorine	240	700ppm	
Lead	210	~3000	99
Manganese	170	300	
Lanthanum	96	~1000	
Vanadium	92	700	
Neodymium	84	700	
Nickel	58	300	
Praesodymium	44	100	
Cerium	43	300	
Chlorine	31	~1000	
Zirconium	22	100	
Tin	18	100	
Rubidium	14	70	
Cobalt	9.3	100	
Samarium	9.1	30	
Yttrium	8.6	30	
Lithium	7.4	10	
Molybdenum	6.7	10	
Bromine	6.2	30	

\* Entire filter analyzed 7-SCB-SF used as blank

(H<sub>2</sub>S, SO<sub>2</sub>, etc.). This is consistent with the SSMS data showing substantial amounts of sulfur in the ash.

#### B.4.11 Water Content of API Waste

During the separation of the O-API-REP sample into organic soluble and residual fractions a 74% loss of the original sample mass was noted. It was presumed that most of this loss was due to water, which evaporated when the fractions were dried.

In an attempt to accurately determine the water content of the waste, an aliquot was placed in an oven at 110°C, and the weight loss recorded at intervals. Drying to constant weight required 3.5 hours and indicated a water content of 70.4%. A TGA analysis of a separate aliquot indicated a water content of 65.3% (weight lost up to 225°C),

The discrepancies among the three estimates of water content (75%, 70.4%, 65.3%) appear to be due to the fact that the waste is an oil-water emulsion which is difficult to break.

It is concluded that the water content is 70 $\pm$  5%.

### B.5 CHEMICAL ANALYSIS OF STYRENE WASTE SAMPLES

#### B.5.1 Data from On-Line Analyzers

<u>Run</u>	<u>Hydrocarbons</u> <u>% (as CH<sub>4</sub>)</u>	<u>CO</u> <u>ppm</u>	<u>CO<sub>2</sub></u> <u>%</u>	<u>O<sub>2</sub></u> <u>%</u>	<u>NO</u> <u>ppm</u>
4-STY	2.53 $\pm$ 0.12	2240 $\pm$ 23	10.7 $\pm$ 0.16	0.0 $\pm$ 0.2	64 $\pm$ 10
5-STY	2.38 $\pm$ 0.37	2095 $\pm$ 41	11.0 $\pm$ 0.4	0.0 $\pm$ 0.2	78 $\pm$ 10
6-STY*	2.4	2150	6.9	0.0	75

The error estimates are standard deviations of individual (10 minute interval) readings from the mean.

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\* During the 6-STY run the sampling line plugged frequently and a Saran bag grab sample was taken. The sample was then fed from the bag to each analyzer in turn.

### B.5.2 Gas Bulk Analyses

The results of analyses by Gollub Analytical Service Corp. corrected to zero oxygen concentration (see Appendix A.2) are shown below. The error in the tabulated values is estimated to be  $\pm 100$  ppm.

	<u>Concentration, ppm by volume except as noted</u>		
	<u>4-STY</u>	<u>5-STY</u>	<u>6-STY</u>
Carbon Dioxide	6.6%	8.7%	8.7%
Carbon Disulfide	600	1320	1333
Carbonyl Sulfide	150	329	400
Sulfur Dioxide	60	260	200
Hydrogen	2850	9630	9600
Methane	680	1700	2800
Ethane	210	356	480
C <sub>3</sub> -C <sub>5</sub> Hydrocarbon	<150	<150	<130
Benzene	1650	2770	4933
Toluene	900	870	920
Xylene	<70	105	130
Acetylene	2100	1715	1866

From these data it is calculated that the average molecular weight of the hydrocarbon material in the gaseous pyrolyzer effluent is 51 and the average carbon number is 4.0. These estimates imply a C:H ration in the volatile hydrocarbon fraction of 16. If the hydrogen found in the analyses is included, the C:H ratio in the gaseous effluent drops to 9.

It should also be noted that these samples show relatively high levels of carbon disulfide and carbonyl sulfide.

### B.5.3 Elemental Analysis of Major Constituents

The data obtained were:

	<u>% C</u>	<u>% H</u>	<u>% N</u>	<u>% S</u>
0-STY-REP-SOL	84.46	6.96	.02	7.86
6-STY-P-GOO	87.76	5.93		2.92
6-STY-P-ASH	90.59	2.37		4.65

These data imply C:H weight ratios of 12.1 in the REP, 14.8 in the GOO and 38.2 in the ASH. The ASH sample is therefore highly unsaturated with respect to the feed.

In contrast to the API results, these data do not show enrichment of sulfur in the GOO and ASH samples. This is not surprising, since the sulfur in the styrene tar was identified as free sulfur (in the survey analysis) which might be readily volatilized.

#### B.5.4 IR Spectra

##### O-STY-REP-SOL

The IR spectrum of this sample resembled that of styrene-butadiene rubber with the addition of extra bands at 865-740  $\text{cm}^{-1}$  substituted aromatics.

<u>Adsorption Maximum Frequency in <math>\text{cm}^{-1}</math></u>	<u>Assignment</u>
3100 - 3000 (m/s)	Aromatic CH Stretch
3000 - 2850 (m/s)	Aliphatic CH Stretch
1600 (m)	Aromatic C=C
1595 (m/s)	"
1455 (m/s)	"
1370	Aliphatic C-H Bend
1300 - 1000 (multiple, w)	Aromatic substitution patterns
960, 980 (w)	C=C Stretch
865 (w), 815 (w), 740 (s)	Aromatic substitution other than mono-
760, 700 (s)	Monosubstituted aromatic

##### 6-STY-P-GOO

The spectrum of this sample was almost the same as that of the REP, with the exception that the intensity of aromatic bands was somewhat increased relative to the aliphatic.

##### 5-STY-P-ASH-SOL

This sample, again, was similar to the REP but appeared to have a higher aliphatic content. The polysubstituted aromatic bands at 865, 815 and 740  $\text{cm}^{-1}$  were relatively weaker in the ASH-SOL sample. Two new, weak bands at 88p and 1415  $\text{cm}^{-1}$  are possibly due to C-H deformation vibrations of alkenes.

#### 6-STY-P-ST-Pentane

The IR spectrum of this sample was very much like that of the G00. There were two peaks, at  $1490\text{ cm}^{-1}(\text{m})$ , and  $790\text{ cm}^{-1}(\text{m})$  which were not readily assignable. These peaks were not present in the REP, G00 or ASH samples.

#### 6-STY-P-ST-Methanol

The IR spectrum of the methanol sample indicated substantial quantities of oxygenated material. Evidence includes a peak at  $1705\text{ cm}^{-1}(\text{m})$ , carbonyl, and a number of peaks in the C-O stretching region (around  $1200\text{ cm}^{-1}$ ). There was also indication of residual solvent.

In summary, the IR spectra of the gaseous pyrolyzer effluent fractions were similar to those of the waste feed, although slightly enriched in aromatics. The ASH-SOL fraction showed some enrichment in aliphatics relative to the REP. Some oxygenated material was found in the methanol extract of the sorbent trap.

### B.5.5 Results of LRMS Analyses

Table B-9 presents the data obtained from the LRMS analyses of the styrene representative waste sample and the effluent samplers from Run 6 on this waste.

The data show a remarkable similarity among the feed and effluent samples. None of the samples has any significant contribution from purely aliphatic compounds. The G00 sample is shifted slightly to higher, and the ST sample to lower, molecular weight ranges, but the differences are not dramatic. Some of the higher polynuclear aromatics are found in the G00 sample.

### B.5.6 TGA Data

The TGA data are reported as the percentage of original sample mass lost in temperature intervals defined by distinct changes in slope of the sample weight versus sample temperature curve.

#### O-STY-REP-SOL

20 - 75°C	0 %
75 - 300	69.0
320 - 450	<u>20.1</u>
Total	97.5

#### 6-STY-P-G00-SOL

20 - 100	0 %
100 - 580	89.5
580 - 700	<u>0.4</u>
Total	89.9

TABLE B-9

SPECIFIC COMPOUNDS IDENTIFIED IN FEED AND  
EFFLUENT SAMPLES FOR 6-STY TEST

<u>MW</u>	<u>COMPOUND</u>	<u>Concentration, %</u>			
		<u>REP</u>	<u>ASH</u>	<u>GOO</u>	<u>ST</u>
92	Toluene		2.1		
104	Styrene	1.8	5.6		1.4
128	Napthalene				5.6
134	Butyl Benzene				2.3
142	Methyl Napthalene				1.4
154	Biphenyl/Acenapthene	6.0	4.2	4.2	15.5
160	C <sub>12</sub> H <sub>16</sub>				1.9
166	Fluorene			1.2	2.3
168	Diphenyl Methane	5.5	4.2	3.6	10.3
178	Anthracene/Phenanthrene	21.8	21.1	23.3	23.5
180	Stilbene/Methyl Fluorene	15.5	21.8	12.3	17.8
182	Diphenyl Ethane	10.4	9.2	1.7	
190	Methylene Phenanthrene	2.5			
192	Methyl Phenanthrene	1.8	2.8	5.2	3.3
194	Diphenyl Propene/Methyl Stilbene	7.6	4.9	1.9	2.8
196	Diphenyl Propane	7.3	2.8		
202	Pyrene			3.1	
204	Phenyl Napthalene	3.5	5.6	10.6	5.2
206	Dimethyl Phenanthrene	1.1		1.2	
208	Methylphenyl Indane/Hexahydro Pyrene	1.8	1.4		
210	Diphenyl Butane	2.9	2.1	1.2	
218				2.0	
230	Terphenyl			2.1	
236	C <sub>16</sub> H <sub>12</sub> S Diphenyl Thiophene	1.1	4.9	9.2	1.4
242	Methyl Chrysene/Methyltriphenylene	1.6			
306	Quarterphenyl	—	2.1	—	—
	TOTAL	92.2	94.8	82.8	94.7

#### 6-STY-P-ST-Pentane

25 - 218	<u>98.2</u>
Total	98.2

#### 6-STY-P-ASH-SOL

25 - 295	50.6
295 - 530	42.7
530 - 970	<u>8.4</u>
Total	91.7

It is difficult to make quantitative comparisons on the basis of these data. Qualitatively, however, it is clear that the sample volatility decreases in the order: ST > REP > ASH ~ GOO.

#### B.5.7 Boiling Point Distribution

The boiling point distribution curves for the feed and effluent samples for the 6-STY test are shown in Figure B-3.

These data are consistent with the results found by TGA. The sorbent trap sample curve is shifted to lower boiling points and the ASH and GOO curves to higher boiling points than the REP sample. The difference between ASH and GOO is more pronounced in the boiling point curves because they are normalized in a way that excludes from consideration the extremely non-volatile material.

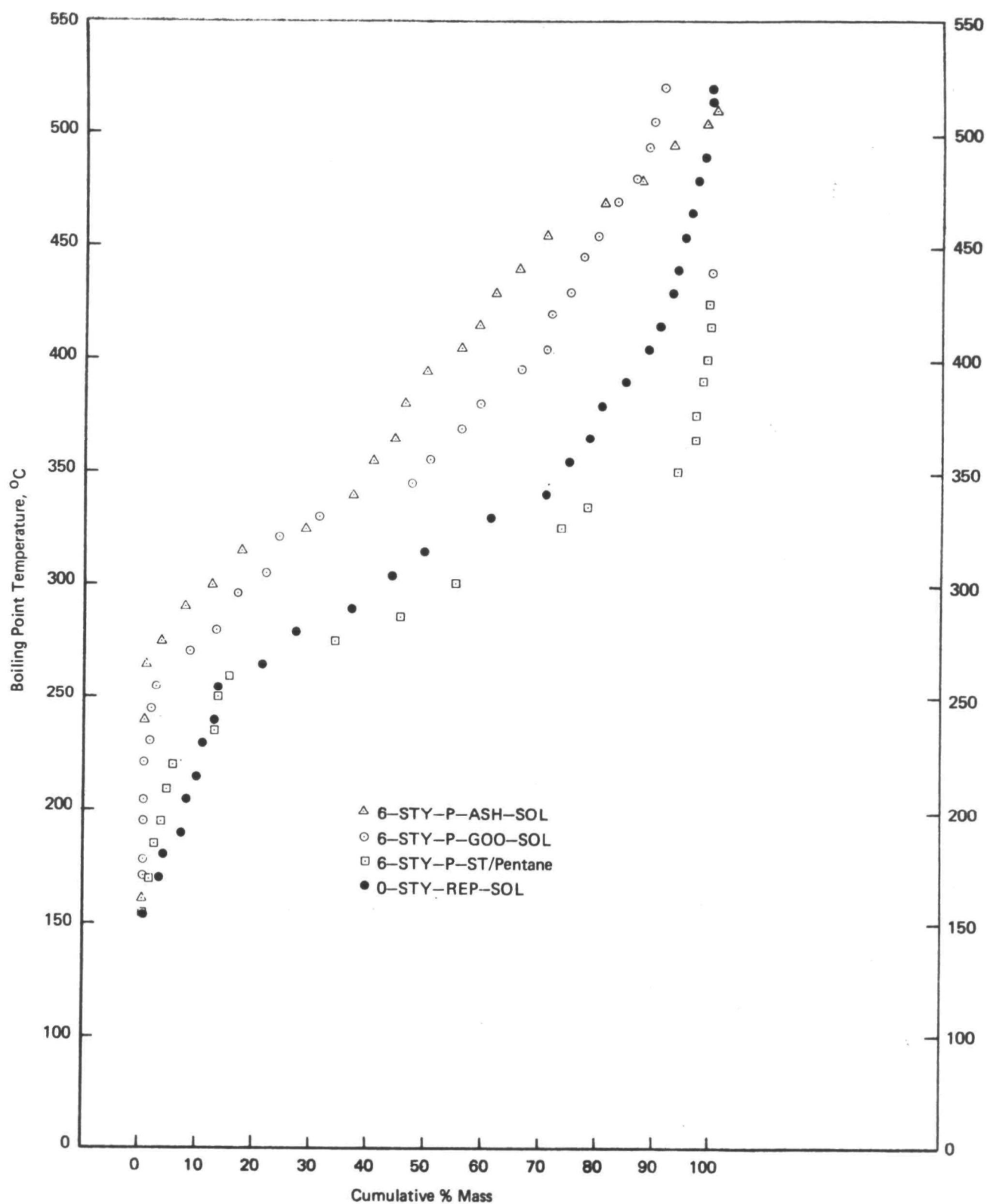
#### B.5.8 SSMS Analyses for Trace Constituents

A portion of the O-STY-REP sample was subjected to spectrometric SSMS analysis. This procedure, which has a detection limit of 0.01 ppm and a precision of  $\pm 100\%$ , identified a total of 32 elements in the waste. In a separate analysis, mercury was found to be present at 0.02 ppm. In Table B-10 are the SSMS data for all elements found at concentrations >1 ppm. It is interesting to note that SSMS shows a very low sulfur concentration, while a combustion technique (above) indicated >7% sulfur. The sulfur is added to this waste as the free element and is apparently lost in the SSMS ashing technique.

Also in Table B-10 are data obtained by a less sensitive SSMS technique (detection limit 1 ppm and precision  $\pm 500\%$ ) for two effluent samples; 6-STY-P-ASH and 6-STY-S-F. These data indicate that most of the trace elements in the feed are emitted from the pyrolyzer in the ASH.

#### B.5.9 Gastec® Analyses

Analysis of the stack effluent with Gastec® tubes showed 100-200 ppm of SO<sub>2</sub> for all three tests.



**FIGURE B-3 BOILING POINT DISTRIBUTION CURVES FOR SAMPLES FROM 6-STY TEST**

TABLE B-10

SSMS DATA FOR STYRENE FEED AND EFFLUENT

<u>Element</u>	<u>Samples</u>		
	<u>0-STY- REP</u>	<u>6-STY-P- ASH</u>	<u>6-STY- S-F</u>
Silicon	35ppm	5000ppm	
Aluminum	32	1000	
Sodium	32	3000	
Iron	31	> 1%	
Phosphorus	18	1000ppm	
Magnesium	13	700	
Sulfur	9	~3000	2900
Calcium	6.2	~3000	
Zinc	1.7	> 0.5%	
Potassium	1.4	300	
Copper	0.54	1000ppm	
Chromium	0.19	300	
Manganese	0.37	300	
Barium	0.25	100	
Strontium	0.15	100	
Titanium	0.16	100	
Lead	0.11	70	
Fluorine	0.20	30	
Nickel	0.03	30	
Cobalt	0.010		

\* Entire filter analyzed. 7-SCB-S-F used as blank.

#### B.5.10 Analyses of Impinger Solutions

Aliquots of the 6-STY-P-I and 6-STY-S-I impinger samples were oxidized with hydrogen peroxide, boiled to destroy excess oxygen, then analyzed for sulfate by the barium chloranilate method. The results were:

	Concentration as <u>SO<sub>4</sub><sup>=</sup>, ppm</u>	Total Sulfur in Impingers <u>as S, mg</u>
6-STY-P-I	2575	443
6-STY-S-I	1150	172

The amount of sulfur detected in the pyrolysis zone impingers is 11% of the quantity which would have been expected if all sulfur in the waste feed had been converted to acidic sulfur gases (H<sub>2</sub>S, SO<sub>2</sub>, etc.). This is consistent with the SSMS data which indicate that substantial amounts of sulfur are found in the ASH.

#### **B.6 CHEMICAL ANALYSES OF RUBBER WASTE SAMPLES**

##### B.6.1 Data From On-Line Analyzers

	Hydrocarbons <u>% (as CH<sub>4</sub>)</u>	CO <u>ppm</u>	CO <sub>2</sub> <u>%</u>	O <sub>2</sub> <u>%</u>	NO <u>ppm</u>
8-RUB	3.09 ± 0.21	2083 ± 10	10.1 ± 0.1	0.0 ± 0.2	75 ± 4
9-RUB	3.11 ± 0.08	1947 ± 14	9.97 ± 0.06	0.0 ± 0.2	75 ± 3
10-RUB	2.45 ± 0.18	2125 ± 8	9.89 ± 0.06	0.0 ± 0.2	66 ± 3

The error estimates are standard deviations of individual (10 minute interval) readings from the mean.

##### B.6.2 Gas Bulb Analyses

The gas bulb samples from the rubber samples showed oxygen concentrations of >20%. It was impossible to correct these samples back to zero oxygen concentration. The concentrations reported by Gollub are tabulated below.

	<u>Concentration, % volume:volume</u>		
	<u>8-RUB</u>	<u>9-RUB</u>	<u>10-RUB</u>
Nitrogen	77+	77+	77+
Oxygen	20.6	21.4	20.7
Argon	0.97	0.97	0.99
Carbon Dioxide	0.60	0.18	0.78
Hydrogen	<0.002	<0.002	<0.002
Carbon Monoxide	0.21	0.064	0.35
Methane	0.014	0.0028	0.015
Benzene	0.017	0.0067	0.030

If using the reported relative concentrations of methane and benzene, an average molecular weight of 55 and average carbon number of 4.2 are estimated. These imply a C:H weight ratio of 11.0. If the mass spectrometric analyses are corrected to correspond to the on-line instrument total hydrocarbon concentration, and assume that hydrogen is present at the detection limit, the values are mw = 41, C no = 3.1, and C:H ratio = 9.

#### B.6.3 Elemental Analysis of Major Components

The data obtained were:

	<u>% C</u>	<u>% H</u>	<u>% N</u>	<u>% S</u>
0-RUB-REP-SOL	79.53	10.10	0.08	0.48
9-RUB-P-GOO	89.09	6.41	0.57	0.84
9-RUB-P-ASH	79.93	4.64	--	1.12

As for the API waste, the sulfur in the feed appears to be enriched in the GOO and ASH. The nitrogen also appears to be enriched in the GOO; the corresponding analysis was not performed on the ash.

The calculated C:H ratios are: REP, 7.87; GOO, 13.90; ASH, 17.23. The GOO and ASH are clearly less saturated than the waste feed.

#### B.6.4 IR Spectra

##### 0-RUB-REP-SOL

Overall, the infrared curve resembles those of butadiene-styrene polymers, with some additional bands (marked with an asterisk in the following list).

<u>Absorption Maximum Frequency in <math>\text{cm}^{-1}</math></u>	<u>Assignment</u>
3100 - 3000 (w)	Aromatic C-H Stretch
300 - 2850 (s)	Aliphatic C-H Stretch
1705* (m)	Carbonyl of conjugated ester or aromatic ketone
1640 (w)	Unconjugated alkene
1600 (w), 1495 (w)	Aromatic C-C
1450 (w), 1380 (w), 1365 (w)	Aliphatic C-H bend
1260 (w)*	C-O stretch of conjugated ester, or aromatic ketone
965, 910 (s, m)	Terminal vinyl group CH bend
880, 820, 720 (w)	Trisubstituted Aromatic
760, 700 (m, s)	Monosubstituted Aromatic

#### 9-RUB-P-GOO-SOL

The spectrum of this sample is remarkably like that of the REP except that the aromatic band intensities are somewhat increased and that the butadiene-like bands at 950 and 910  $\text{cm}^{-1}$  are absent. The carbonyl peak is of greatly reduced intensity. There is a new band at 750  $\text{cm}^{-1}$  (s), which represents a disubstituted aromatic.

#### 9-RUB-P-ASH-SOL

The IR spectrum of this sample shows very weak butadiene and carbonyl bands. The intensity of the aromatic C-H stretching bands is also greatly reduced compared to the REP. The aliphatic C-H stretching intensity is still high.

#### 9-RUB-P-ST-Pentane

In this sample the carbonyl and butadiene bands were very weak. The aromatic substitution pattern was very different from that of the GOO and REP samples, with strong bands at 815, 760 and 740  $\text{cm}^{-1}$ .

#### 9-RUB-P-ST-Methanol

The strong carbonyl peak and C-O stretching bands observed in the REP sample reappear in this spectrum. The aromatic stretching and bending bands are all of moderate intensity but somewhat shifted from those in the REP. The butadiene peaks are absent.

In summary, the infrared spectra for the rubber waste samples indicate that the gaseous pyrolyzer effluent is more highly aromatic and the ASH more highly aliphatic than the representative waste. The terminal vinyl functional group ( $-HC=CH$ ) observed in the waste feed does not seem to be present in any of the effluent samples. The carbonyl compound(s) in the feed are found only in the sorbent trap methanol extract.

#### B.6.5 Results of LRMS Analyses

The data obtained from LRMS analyses of rubber feed and effluent samples are present in Table B-11.

##### O-RUB-REP

The data show that, of the material which is volatile in the LRMS inlet, 48.7% is primarily unsaturated aliphatic compounds. Only a small number of individual aromatic compounds were present in concentrations high enough for identification. Nonyl phenol accounts for 6.2% of the sample and 3 other, unidentified, oxygenated species of molecular weight >300 account for an additional 19.8%.

##### O-RUB-P-ASH-SOL

No aliphatic material was detected in this sample. The number of aromatic compounds detected was much larger than in the REP sample, but the molecular weight range was comparable.

##### O-RUB-P-GOO-SOL

Again, no aliphatics were detected. The GOO sample shows a shift to slightly higher molecular weight in the distribution of aromatic species. In particular, detectable concentrations of the higher polynuclear aromatics are found in this sample.

It is interesting that the material tentatively identified as hydroxyoctoxy benzophenone appears in this sample as well as in the 2-API-P-GOO-SOL sample.

##### 9-RUB-P-ST-Pentane

The sorbent trap sample again shows no purely aliphatic compounds, but it does show a shift to lower molecular weight.

#### B.6.6 TGA Data

The data are reported as the percentage of original sample mass lost in temperature intervals defined by distinct changes in slope of the sample weight versus sample temperature curve.

TABLE 8-11

## COMPOUNDS IDENTIFIED IN FEED AND EFFLUENT SAMPLES FOR 9-RUB TEST

MW	COMPOUND	Concentration, %			
		REP	ASH	GOO	ST
	Aliphatics	48.7			
92	Toluene		2.9		
104	Styrene				2.4
116	Indene				9.4
118	Methnyl Styrene/Indane				1.8
128	Napthalene		4.7		33.0
134	Butyl Benzene	1.9			
142	Methyl Napthalene		4.7		10.0
144	C <sub>11</sub> H <sub>12</sub>				1.0
146	C <sub>11</sub> H <sub>14</sub>	2.3			
152	Biphenylene/Acenaphthylene			2.6	9.4
154	Biphenyl/Acenaphthene		7.6	1.3	5.9
156	Dimethyl Napthalene		4.7		3.0
166	Fluorene			3.0	4.8
168	Diphenyl Methane		5.3	1.2	2.2
170	Propyl Napthalene		4.1		
178	Anthracene/Phenanthrene		14.6	9.8	4.9
180	Stilbene/Methyl Fluorene		10.5	3.2	2.4
182	Diphenyl Methane		6.4		
184	Butyl Napthalene		1.7		
190	Methylene Phenanthrene			1.8	
192	Methnyl Phenanthrene		4.7	6.2	2.5
194	Diphenyl Propene/Methnyl Stilbene		3.5		
196	Diphenyl Propane		3.5		
202	Pyrene		3.5	11.1	
204	Phenyl Napthalene		4.1	4.9	
206	Dimethyl Phenanthrene		2.9	3.0	
210	Diphenyl Butane	1.0	2.9		
216	Methyl Pyrene			3.8	
218			1.8	3.5	
220	Nonyl Phenol	6.2			
220	Trimethnyl Phenanthrene			1.4	
226	Benzfluoranthene			2.9	
228	Chrysene/Naphtacene/Benzanthracene			6.1	
230	Terphenyl			3.9	
232	C <sub>18</sub> H <sub>16</sub>			1.5	
236	Diphenyl Thiophene		1.8		
238	Decahydro Benzanthracene		1.8		
240	Dodecahydro Benzanthracene/C <sub>8</sub> Napthalene			1.9	
242	Methyl Chrysene/Methyl Triphenylene			3.2	
244	Triphenyl Methane			1.4	
246	Octadecahydrochrysene	1.2			
252	Benzpyrene			3.3	
254	Binaphthyl			2.0	
256	C <sub>2</sub> Benzanthracene, etc.			1.2	
264		2.3			
266				1.0	
300	C <sub>28</sub> H <sub>28</sub> O <sub>2</sub>	9.4			
302	C <sub>20</sub> H <sub>30</sub> O <sub>2</sub>	5.7			
304	C <sub>20</sub> H <sub>32</sub> O <sub>2</sub>	4.7			
326	C <sub>21</sub> H <sub>26</sub> O <sub>3</sub>			1.4	
	TOTAL	84.6	97.7	86.6	92.7

$$\begin{aligned}
 \text{Aliphatics} &= 2n + 2 = 5.5\% \\
 2n &= 6.7 \\
 2n - 2 &= 6.7 \\
 2n - 4 &= 7.8 \\
 2n - 6 &= 8.6 \\
 2n - 8 &= 8.1 \\
 2n - 10 &= 5.3 \\
 &= 48.7\%
 \end{aligned}$$

O-RUB-REP-SOL

30 - 250°C	19.1%
250 - 350	25.9
350 - 450	<u>18.8</u>
Total	63.8

P-RUB-P-GOO-SOL

20 - 100°C	0 %
100 - 500	70.3
500 - 850	<u>8.0</u>
Total	78.3

P-RUB-P-ST-Pentane

20 - 250°C	<u>95.5%</u>
Total	95.5

9-RUB-P-ASH-SOL

25 - 438°C	56.5%
438 - 515	31.2
515 - 670	<u>9.5</u>
Total	97.2

For the rubber waste, in contrast to API and styrene wastes, all of the pyrolyzer effluent samples were more volatile than the REP waste feed sample. As observed previously, the sorbent trap sample is the most volatile of the effluent samples. For the rubber waste, the ASH contains appreciably more volatile components than does the GOO.

B.6.7 Gel Permeation Chromatography (GPC)

The rubber samples were not suitable for gas chromatographic analysis because they contained very non-volatile components. For these samples, (GPC) provided a measure of molecular weight distribution. The data obtained are given below, with molecular weights assigned based on polystyrene standards. These molecular weights may not be absolutely correct, but do give an accurate indication of changes in the molecular weight distribution.

	<u>MW</u>	<u>% of Total</u>
O-RUB-REP-SOL	$10^6 - 5 \times 10^4$	34
	$\sim 10^3$	27
	$\sim 10^2$	38

	<u>MW</u>	<u>% of Total</u>
9-RUB-P-GOO-SOL	$5 \times 10^3 - 10^3$	11
	$10^3 - 10^2$	21
	$\sim 10^2$	68
9-RUB-P-ST-Pentane	$\sim 10^2$	100
9-RUB-P-ASH-SOL	$2 \times 10^6 - 10^3$	39
	$10^3 - 10^2$	49
	$\sim 10^2$	11

#### B.6.8 SSMS Analyses for Trace Constituents

A portion of the O-RUB-REP sample was subjected to spectrometric SSMS analysis. This procedure, which has a detection limit of 0.01 ppm and a precision of  $\pm 100\%$ , identified a total of 61 elements in the waste. In a separate analysis, mercury was found to be present at 0.3 ppm. In Table B-12 are the SSMS data for all elements found at concentrations  $>5$  ppm.

Also in Table B-12 are data obtained by a less sensitive SSMS technique (detection limit 1 ppm and precision  $\pm 500\%$ ) for two effluent samples: 9-RUB-O-ASH and 9-RUB-S-F. These data indicate that most of the trace elements in the feed are emitted from the pyrolyzer in the ASH.

#### B.6.9 Analyses of Impinger Solutions

Aliquots of the 9-RUB-P-I and 9-RUB-S-I impinger solutions were oxidized with hydrogen peroxide, boiled to destroy excess oxidant, and analyzed for sulfate by the barium chloranilate method. The results were:

	<u>Concentration as <math>\text{SO}_4^{=}</math>, ppm</u>	<u>Total Sulfur as S, mg</u>
9-RUB-P-I	380	68
9-RUB-S-I	405	58

The amount of sulfur detected in the pyrolysis zone impinger is 39% of the total which would be expected if all of the sulfur in the waste feed were converted to acidic sulfur gases ( $\text{H}_2\text{S}$ ,  $\text{SO}_2$ , etc.). This is consistent with the SSMS data, showing substantial sulfur in the ASH.

TABLE B-12

SSMS DATA ON FEED AND EFFLUENT SAMPLES FOR 9-RUB TESTS

<u>Element</u>	<u>0-RUB- REP</u>	<u>9-RUB-P- ASH</u>	<u>9-RUB-S- F*</u>
Calcium	>1%	>1%	
Sulfur	>1%	>1%	
Silicon	>0.5%	>1%	
Iron	~2800ppm	>1%	
Aluminum	1500	~ 5000ppm	
Sodium	760	~ 3000	
Phosphorus	750	~ 1000	
Potassium	710	~ 1000	
Magnesium	440	~ 3000	
Chlorine	430	~ 3000	
Nickel	160	~ 300	
Chromium	130	~ 700	
Titanium	66	~300	
Lead	62	300	
Zinc	53	300	
Barium	42	100	
Strontium	41	100	
Fluorine	20	100	
Manganese	16	30	
Bismuth	15	10	
Bromine	12	10	
Molybdenum	12	30	
Copper	11	100	
Cobalt	5.3	30	

\* Entire filter analyzed. 7-SCB-S-F used as blank.

## B.7 CHEMICAL ANALYSES OF BACKGROUND TEST SAMPLES

### B.7.1 Data From On-Line Analyzers:

	Hydrocarbons, <u>% (as CH<sub>4</sub>)</u>	CO <u>ppm</u>	CO <sub>2</sub> <u>%</u>	O <sub>2</sub> <u>%</u>	NO <u>ppm</u>
7-SCB	0.06 ± 0.01	1766 ± 10	11.5 ± 01	0.0 ± 0.2	38 ± 2

The error estimates are standard deviations of individual (10 minute interval) readings from the mean.

### B.7.2 Gas Bulb Analysis

The results of analyses by Gollub Analytical Service Corp., corrected to zero oxygen concentration, are:

Carbon dioxide	5.89	Ethane	0.016
Carbon disulfide	<.009	C <sub>3</sub> -C <sub>5</sub> Hydrocarbon	<0.019
Carbonyl sulfide	<.009	Benzene	0.020
Sulfur dioxide	<.009	Toluene	<0.009
Hydrogen	<.004	Xylene	<0.009
Methane	0.006	Acetylene	<0.009

### B.7.3 Elemental Analyses for Major Constituents

<u>Sample</u>	<u>% C</u>	<u>% H</u>	<u>% S</u>
7-SCB-P-GOO-SOL	85.04	7.46	2.99

These are very similar to the results for the 6-STY-P-GOO-SOL sample.

### B.7.4 IR Spectra

#### 7-SCB-P-GOO-SOL

The IR spectrum of this sample is qualitatively very similar to that of the 6-STY-P-GOO-SOL sample. The background sample has a higher ratio of aliphatic to aromatic stretching intensities. The spectrum of the background sample also has a carbonyl peak [1735 cm<sup>-1</sup>, (w)] which is missing in the corresponding styrene sample.

#### 7-SCB-P-ST-Pentane

This sample has an IR spectrum which matches, peak for peak, the spectrum of the 6-STY-P-ST-Pentane sample.

In summary, the IR data imply that the material found in the gaseous pyrolyzer effluent from the background test was primarily due to residues from the preceding styrene test.

#### B.7.5 Results of LRMS Analyses

Since all of the other evidence indicated that these samples resembled those for the styrene tests, a detailed LRMS analysis on the background sample was not performed. Major components identified in the LRMS spectra of the background GOO and effluent samples are listed in Table B-13.

#### B.7.6 Analyses of Impinger Solutions

Aliquots of the 7-SCB-P-I and 7-SCB-S-I impinger samples were oxidized with hydrogen peroxide, boiled to destroy excess oxidant, then analyzed for sulfate by the barium chloronilate method. The results were:

	<u>Concentration,</u> <u>as SO<sub>4</sub><sup>=</sup>, ppm</u>	<u>Total Sulfur</u> <u>as S, mg</u>
7-SCB-P-I	310	54
7-SCB-S-I	550	86

These values are unexpectedly high, since the unit was operating with natural gas. It seems probable that the sulfur is due to carry-over from the styrene burn immediately preceding.

#### B.7.7 Other

Because all of the preliminary analyses indicated that the collected 7-SCB effluent samples represented carry-over from the 6-STY test immediately preceding, no further analyses of the 7-SCB samples was done. [The 7-SCB-S-F (stack filter) was analyzed by SSMS and the results used to make corrections for the elements present in the filter medium.]

TABLE B-13

SPECIFIC COMPOUNDS IDENTIFIED IN EFFLUENT SAMPLES FOR 7-SCB TEST

		Concentration %	
		<u>7-SCB-P-G00-SOL</u>	<u>7-SCB-P-ST-Pentene</u>
92	Toluene		
104	Styrene		
128	Napthalene		>10%
134	Butyl Benzene		tr.
142	Methyl Napthalene		> 1%
152	Biphenylene/Acenapthylene	> 1%	
154	Biphenyl/Acenapthene		>10%
160	C <sub>12</sub> H <sub>16</sub>		>10%
166	Fluorene		
168	Diphenyl Methane		>10%
178	Anthracene/Phenanthrene	>10%	>10%
180	Stilbene/Methyl Fluorene		> 1%
182	Diphenyl Ethane		
190	Methylene Phenanthrene		
192	Methyl Phenanthrene	> 1%	tr.
194	Diphenyl Propene/Methyl Stilbene		> 1%
196	Diphenyl Propane		
202	Pyrene	>10%	
204	Phenyl Napthalene	tr.	> 1%
206	Dimethyl Phenanthrene	tr.	
208	Methyl Phenyl Indene/Hexahydro Pyrene	tr.	
210	Diphenyl Butane	> 1%	
218		> 1%	tr.
230	Terphenyl	>10%	tr.
236	Diphenyl Thiophene	>10%	> 1%
242	Methyl Chrysene/Methyl Triphenylene	tr.	
306	Quaterphenyl	tr.	

## **APPENDIX C**

### **OPERATING DATA**

TABLE C-1

## PROCESS DATA FOR RUN NO. -1

WASTE - API SEPARATOR BOTTOM

DATE - 1.28.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp	Effluent Gas Temp	$\text{ZnO}_2$
	$\Delta P$ *	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	°F	°F	
10:43	2.35	1750	8.5	182	0.9	1430	0.54	3250	.04	1400	960	0.0
11:15	2.8	1940	8.5	182	0.9	1430	0.83	3950	.06	1400	1080	0.0
11:30	2.6	1840	8.5	182	0.9	1430	0.76	3760	.05	1400	1090	0.0
11:46	2.6	1840	8.5	182	0.9	1430	0.77	3800	.05	1400	1090	0.0
12:00	2.5	1800	8.5	182	0.9	1430	0.77	3800	.06	1400	1090	0.0
1:30	2.6	1840	8.6	185	1.0	1500	0.82	3900	.05	1400	1105	0.0
1:45	2.7	1850	8.5	182	1.0	1500	0.80	3875	.04	1400	1100	0.0
2:00	2.7	1850	8.6	185	1.0	1500	0.81	3880	.06	1400	1105	0.0
2:15	2.6	1840	8.5	182	1.0	1500	0.79	3850	.04	1400	1100	0.0
2:30	2.4	1750	8.5	182	1.0	1500	0.78	3825	.04	1400	1090	0.2
3:00	2.4	1750	8.5	182	1.0	1500	0.82	3900	.05	1400	1090	0.2
3:30	2.3	1730	8.6	185	1.0	1500	0.78	3815	.03	1400	1080	0.2

NOTE: Feed Started at 11:00 a.m. and stopped at 3:43 p.m.

\* Pressure Differential - inches of water

\*\* Flow rate - standard cubic feet per hour.

TABLE C-2

PROCESS DATA FOR RUN NO. -1

WASTE - API SEPARATOR BOTTOM

DATE - 1.28.76

Time	Incinerator Burner Gas Burner #1      Burner #2				Incinerator Air		Incinerator Auxiliary Gas Burner #1      Burner #2				Incin. Temp.	Vapor Inlet	Stack Temp.
	$\Delta P^*$	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	°F	°F	°F
10:43	.8	290	.8	290	19.5	34,000	2.75	445	2.75	445	1520	860	650
11:15	.7	270	.7	270	23.5	37,500	2.5	425	2.5	425	1520	920	680
11:30	.7	270	.7	270	23.5	37,500	2.5	425	2.5	425	1510	950	670
11:46	.7	270	.7	270	24.5	38,500	2.5	425	2.5	425	1520	960	670
12:00	.7	270	.7	270	24.0	38,000	2.5	425	2.5	425	1520	1000	675
1:30	.6	255	.6	255	25.5	39,000	2.1	390	2.1	390	1520	1020	670
1:45	.6	255	.6	255	25.5	39,000	2.3	410	2.3	410	1520	1015	670
2:00	.6	255	.6	255	24.5	38,500	2.2	400	2.2	400	1520	1035	665
2:15	.6	255	.6	255	25.5	39,000	2.1	390	2.1	390	1520	1000	670
2:30	.6	255	.6	255	25.5	39,000	2.1	390	2.1	390	1520	1000	670
3:00	.6	255	.6	255	25.5	39,000	2.2	400	2.2	400	1515	1020	680
3:30	.6	255	.6	255	25.0	38,750	2.3	410	2.3	410	1520	960	670

\* Pressure Differential - inches of water

\*\* Flow rate - standard cubic feet per hour.

TABLE C-3

PROCESS DATA FOR RUN NO. -1

WASTE - API SEPARATOR BOTTOM

DATE - 1.28.76

HEARTH CYCLE TIME	3 PER HOUR
RESIDENCE TIME IN HOT ZONE	12.5 MINS
FEEDING RATE	36.7 LBS/HR
TOTAL AMOUNT FED	136.5 LBS
RESIDUE COLLECTED	26.5 LBS
LAYER THICKNESS	1 INCH
FEEDER	1/8" x 6" NOZZLE, MOYNO PUMP

COMMENTS - FEED NOZZLE CLOSER TO HOT ZONE, NO SCRAPER

TABLE C-4

## PROCESS DATA FOR RUN NO. -2

WASTE - API SEPARATOR BOTTOM

DATE - 1.29.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp °F	Effluent Gas Temp °F	%O <sub>2</sub>
	ΔP*	SCFH**	ΔP	SCFH	ΔP	SCFH	ΔP	SCFH				
12:45	1.9	1575	7.0	167	1.0	1500	.57	3350	.06	1400	1010	0.0
1:03	2.1	1650	7.2	170	1.0	1500	.74	3800	.08	1400	1050	0.0
1:15	2.4	1760	8.5	183	1.0	1500	.86	4020	.07	1400	1070	0.0
1:30	2.4	1760	8.5	183	1.0	1500	.8	3900	.06	1400	1080	0.0
1:45	2.4	1760	8.5	183	1.0	1500	.83	3950	.07	1400	1085	0.0
2:15	2.5	1820	8.5	183	1.0	1500	.86	4000	.07	1400	1090	0.0
2:45	2.5	1800	8.5	183	1.0	1500	.92	4150	.07	1400	1090	0.3
3:15	2.6	1840	8.5	183	1.0	1500	.93	4170	.07	1400	1090	0.0

NOTES: Feed Started at 1:00 p.m. and stopped at 3:30 p.m.

\* Pressure Differential - inches of water

\*\* Flow rate - standard cubic feet per hour.

TABLE C-5

## PROCESS DATA FOR RUN NO. -2

WASTE - SEPARATOR BOTTOM

DATE - 1.29.76

Time	Incinerator Burner Gas Burner #1      Burner #2				Incinerator Air		Incinerator Auxiliary Gas Burner #1      Burner #2				Incin. Temp.	Vapor Inlet	Stack Temp.
	$\Delta P$ *	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	°F	°F	°F
12:45	.7	270	.7	270	21.0	35,500	2.8	450	2.8	450	1520	890	650
1:03	.7	270	.7	270	25.0	38,750	2.3	412	2.3	412	1520	980	680
1:15	.7	270	.7	270	25.0	38,750	2.3	412	2.3	412	1520	1000	680
1:30	.65	265	.65	265	25.0	38,750	2.6	435	2.6	435	1520	995	680
1:45	.6	255	.6	255	24.5	38,500	2.5	425	2.5	425	1520	1000	710
2:15	.65	265	.65	265	25.3	38,600	2.5	425	2.5	425	1515	1000	685
2:45	.6	255	.6	255	25.5	39,000	2.5	425	2.5	425	1520	980	700
3:15	.65	265	.65	265	26.0	39,500	2.2	400	2.2	400	1515	1010	680

\* Pressure Differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-6  
PROCESS DATA FOR RUN NO. -2

WASTE - API SEPARATOR BOTTOM

DATE - 1.29.76

HEARTH CYCLE TIME	3 PER HOUR
RESIDENCE TIME IN HOT ZONE	12.5 MINS
FEEDING RATE	32.4 LBS/HR
TOTAL AMOUNT FED	81.0 LBS
RESIDUE COLLECTED	16.0 LBS
LAYER THICKNESS	1/2 INCH
FEEDER	1/8" x 8" NOZZLE, MOYNO PUMP

COMMENTS - FEED NOZZLE LOCATED AWAY FROM HOT ZONE AND A SCRAPER  
ATTACHED TO FEED NOZZLE

TABLE C-7

PROCESS DATA FOR RUN NO. -3

WASTE - API SEPARATOR BOTTOM

DATE - 1.30.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp.	Effluent Gas Temp	%O <sub>2</sub>
	$\Delta P$ *	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	°F	°F	
9.57	3.2	2040	8.6	185	1.0	1500	.81	4150	.05	1400	900	0.2
10:25	2.3	1720	8.4	182	1.0	1500	.65	3600	.06	1400	1010	0.2
10:45	2.8	1900	8.5	183	1.0	1500	1.1	4550	.06	1400	1075	0.2
11:15	2.8	1900	8.6	185	1.0	1500	.98	4300	.05	1400	1110	0.0
11:34	2.8	1900	8.5	183	1.0	1500	.99	4320	.05	1400	1110	0.0
11:45	2.8	1900	8.5	183	1.0	1500	.99	4320	.05	1400	1110	0.0
12:00	2.8	1900	8.5	183	1.0	1500	.99	4320	.05	1400	1110	0.0
12:30	2.8	1900	8.5	183	1.0	1500	1.1	4550	.06	1400	1105	0.0
1:00	2.9	1940	8.5	183	1.0	1500	1.0	4340	.05	1400	1120	0.0
1:30	2.8	1900	8.5	183	1.0	1500	1.0	4340	.05	1400	1120	0.0

NOTES: Feed Started at 10:30 a.m. and stopped at 1:40 p.m.

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-8

## PROCESS DATA FOR RUN NO. -3

WASTE - API SEPARATOR BOTTOM

DATE - 1.30.76

Time	Incinerator Burner Gas				Incinerator Air		Incinerator Auxiliary Gas				Incin. Temp.	Vapor Inlet	Stack Temp.
	Burner #1	Burner #2	Burner #1	Burner #2	Air	Air	Burner #1	Burner #2	Burner #1	Burner #2			
	$\Delta P$ *	SCFH **	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	°F	°F	°F
9:57	.7	270	.7	270	17.5	32,400	2.7	440	2.7	440	1520	750	600
10:25	.7	270	.7	270	20.5	35,000	2.8	450	2.8	450	1520	860	630
10:45	.75	280	.75	280	23.5	37,500	2.55	430	2.55	430	1515	920	700
11:15	.7	270	.7	270	23.5	37,500	2.5	425	2.5	425	1515	975	690
11:34	.7	270	.7	270	24.5	38,400	2.4	415	2.4	415	1520	990	715
11:45	.7	270	.7	270	25.5	39,000	2.4	415	2.4	415	1520	990	695
12:00	.7	270	.7	270	25.5	39,000	2.3	410	2.3	410	1520	1000	680
12:30	.7	270	.7	270	25.5	39,000	2.3	410	2.3	410	1520	1000	680
1:00	.65	260	.65	260	25.5	39,000	2.3	410	2.3	410	1520	1010	680
1:30	.7	270	.7	270	25.5	39,000	2.25	405	2.25	405	1520	1010	680

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-9

PROCESS DATA FOR RUN NO. -3

WASTE - API SEPARATOR BOTTOM

DATE - 1.30.76

HEARTH CYCLE TIME	3 PER HOUR
RESIDENCE TIME IN HOT ZONE	12.5 MINS
FEEDING RATE	55.6 LBS/HR
TOTAL AMOUNT FED	176.0 LBS
RESIDUE COLLECTED	66.0 LBS
LAYER THICKNESS	3/4 INCH
FEEDER	1/8" x 8" NOZZLE, MOYNO PUMP

COMMENTS - FEED NOZZLE AWAY FROM HOT ZONE AND A SCRAPER  
ATTACHED TO IT.

TABLE C-10

PROCESS DATA FOR RUN NO. -4

WASTE - STYRENE TAR WASTE

DATE - 2.2.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp.	Effluent Gas Temp	%O <sub>2</sub>
	$\Delta P$ *	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	°F	°F	
11:15	2.4	1750	8.5	183	1.1	1580	.78	3900	.05	1400	1010	0.1
11:45	2.4	1750	8.4	181	1.1	1580	.87	4100	.06	1400	1040	0.1
12:00	2.3	1725	8.4	181	1.1	1580	.84	4020	.06	1400	1040	0.1
12:30	2.1	1650	6.9	165	1.1	1580	.79	3900	.06	1400	1030	0.1
1:00	1.85	1550	6.7	162	1.1	1580	.76	3850	.06	1400	1060	0.1
1:30	1.8	1530	6.6	161	1.1	1580	.75	3800	.06	1400	1070	0.1
2:00	1.6	1440	5.7	150	1.1	1580	.72	3700	.06	1400	1070	0.1
2:30	1.7	1480	6.2	157	1.1	1580	.78	3820	.05	1400	1070	0.1

NOTES: Feeded Started at 11:30 a.m. and stopped at 2:30 p.m.

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-11

PROCESS DATA FOR RUN NO. -4

WASTE - STYRENE TAR WASTE

DATE - 2.2.76

Time	Incinerator Burner Gas				Incinerator Air		Incinerator Auxiliary Gas				Incin. Temp.	Vapor Inlet	Stack Temp.
	Burner #1	Burner #2					Burner #1	Burner #2					
	ΔP *	SCFH **	ΔP	SCFH	ΔP	SCFH	ΔP	SCFH	ΔP	SCFH	°F	°F	°F
11:15	.85	300	.85	300	19.0	33,800	2.8	450	2.8	450	1500	800	620
11:45	.7	270	.7	270	25.5	39,000	2.4	415	2.4	415	1510	1080	700
12:00	.7	270	.7	270	25.0	38,600	2.4	415	2.4	415	1510	1110	730
12:30	.7	270	.7	270	26.0	39,500	2.4	415	2.4	415	1520	1120	700
1:00	.7	270	.7	270	26.0	39,500	2.4	415	2.4	415	1520	1100	700
1:30	.7	270	.7	270	26.0	39,500	2.4	415	2.4	415	1515	1110	690
2:00	.7	270	.7	270	26.5	40,000	2.4	415	2.4	415	1520	1160	700
2:30	.7	270	.7	270	26.5	40,000	2.3	410	2.3	410	1515	1100	680

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-12

PROCESS DATA FOR RUN NO. -4

WASTE - STYRENE TAR

DATE - 2.2.76

HEARTH CYCLE TIME	3 PER HOUR
RESIDENCE TIME IN HOT ZONE	12.5 MINS
FEEDING RATE	11.67 LBS/HR
TOTAL AMOUNT FED	35.0 LBS
RESIDUE COLLECTED	0.5 LBS
LAYER THICKNESS	-
FEEDER	1/8" x 8" NOZZLE, MOYNO PUMP

COMMENTS - THE SCREEN AT THE BOTTOM OF THE TANK PLUGGED UP PARTIALLY  
AND FEED RATE HAD DECREASED FROM INITIAL FEEDING RATE.  
SCREEN REPLACED BY LARGER SIZE SCREEN FOR THE REMAINING  
TEST RUNS.

TABLE C-13

## PROCESS DATA FOR RUN NO. -5

WASTE - STYRENE TAR

DATE - 2.3.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp.	Effluent Gas Temp	%O <sub>2</sub>
	$\Delta P$ *	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	°F	°F	
10:15	1.9	1570	7.0	166	1.0	1500	.71	3900	.03	1200	920	0.1
10:45	1.85	1550	7.0	166	1.0	1500	.75	3850	.04	1200	1000	0.1
11:00	1.75	1500	6.0	154	1.0	1500	.7	3700	.04	1200	1020	0.1
11:30	1.5	1400	5.6	148	1.0	1500	-	-	.05	1200	1050	0.1
12:00	1.0	1140	2.7	104	1.0	1500	-	-	.03	1200	1020	0.1
12:30	0.9	1080	2.9	107	1.0	1500	.56	3300	.06	1200	1010	0.1

NOTE: Feed Started at 10:30 a.m. and stopped at 12:45 p.m.

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-14

## PROCESS DATA FOR RUN NO.-5

WASTE - STYRENE TAR

DATE - 2.3.76

Time	Incinerator Burner #1		Incinerator Gas Burner #2		Incinerator Air		Incinerator Burner #1		Incinerator Auxiliary Gas Burner #2		Incin. Temp.	Vapor Inlet	Stack Temp.
	$\Delta P^*$	SCFH **	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$^{\circ}F$	$^{\circ}F$	$^{\circ}F$
10:15	.8	290	.8	290	18.5	33,300	2.8	450	2.8	450	1510	790	610
10:45	.7	270	.7	270	24.5	38,300	2.6	435	2.6	435	1515	1030	700
11:00	.7	270	.7	270	25.0	38,800	2.4	415	2.4	415	1510	1160	690
11:30	.6	250	.6	250	27.0	40,200	1.6	330	1.6	330	1510	1190	720
12:00	.65	250	.65	250	26.0	39,500	2.2	400	2.2	400	1520	1220	690
12:30	.65	250	.65	250	27.0	40,200	2.3	407	2.3	407	1520	1280	700

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-15

PROCESS DATA FOR RUN NO. -5

WASTE - STYRENE TAR

DATE - 2.3.76

HEARTH CYCLE TIME	3 PER HOUR
RESIDENCE TIME IN HOT ZONE	12.5 MINS
FEEDING RATE	16.3 LBS/HR
TOTAL AMOUNT FED	35 LBS
RESIDUE COLLECTED	1 LB
LAYER THICKNESS	-
FEEDER	1/8" x 8" NOZZLE, MOYNO PUMP

COMMENTS - PROBLEMS WITH MEASUREMENT OF EFFLUENT GAS FLOW AS  
THE PRESSURE TAPS FOR MANOMETER WERE PLUGGING UP DUE  
TO SOOT IN THE EFFLUENT GAS

TABLE C-16

## PROCESS DATA FOR RUN NO. -6

WASTE - STYRENE TAR

DATE - 2.4.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp.	Effluent Gas Temp	$\text{XO}_2$
	$\Delta P^*$	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	$^{\circ}\text{F}$	$^{\circ}\text{F}$	
10:00	2.3	1730	8.4	182	1.0	1500	.85	4100	.08	1400	1020	0.1
10:15	1.85	1550	6.6	162	1.0	1500	.75	3800	.05	1400	1060	0.1
10:30	2.3	1730	8.4	182	1.0	1500	1.0	-	.08	1400	1100	0.1
10:50	2.0	1610	7.5	172	1.0	1500	.84	3950	.09	1400	1100	0.1
11:25	1.6	1440	5.5	147	1.0	1500	.84	3950	.07	1400	1100	0.1
12:00	1.55	1420	5.3	145	1.0	1500	.88	4050	.09	1400	1105	0.1
12:30	1.9	1570	6.6	163	1.0	1500	1.2	-	.15	1400	1160	0.1
1:00	2.0	1610	7.3	170	1.0	1500	1.4	-	.19	1400	1180	0.1

NOTE: Feed Started at 10:25 a.m. and stopped at 1:25 p.m.

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-17

## PROCESS DATA FOR RUN NO.-6

WASTE - STYRENE TAR

DATE - 2.4.76

Time	Incinerator Burner Gas				Incinerator		Incinerator Auxiliary Gas				Incin. Temp.	Vapor Inlet	Stack Temp.
	Burner #1	Burner #2			Air		Burner #1	Burner #2					
	ΔP*	SCFH**	ΔP	SCFH	ΔP	SCFH	ΔP	SCFH	ΔP	SCFH	°F	°F	°F
10:00	.8	290	.8	290	16.5	31,500	2.8	450	2.8	450	1610	850	625
10:15	.8	290	.8	290	17.5	32,400	2.8	450	2.8	450	1610	895	625
10:30	.65	260	.65	260	25.0	38,800	2.2	400	2.2	400	1610	1225	790
10:50	.7	270	.7	270	26.0	39,500	2.1	390	2.1	390	1610	1160	770
11:25	.7	270	.7	270	26.5	39,800	2.1	390	2.1	390	1610	1280	770
12:00	.7	270	.7	270	26.0	39,500	2.3	410	2.3	410	1610	1220	750
12:30	.65	260	.65	260	27.0	40,200	1.8	360	1.8	360	1610	1300	780
1:00	.6	250	.6	250	28.0	41,000	1.7	350	1.7	350	1610	1330	780

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-18

PROCESS DATA FOR RUN NO. -6

WASTE - STYRENE TAR

DATE - 2.4.76

HEARTH CYCLE TIME	3 PER HOUR
RESIDENCE TIME IN HOT ZONE	12.5 MINS
FEEDING RATE	22 LBS/HR
TOTAL AMOUNT FED	66 LBS
RESIDUE COLLECTED	0.31 LBS
LAYER THICKNESS	-
FEEDER	1/8" x 8" NOZZLE, MOYNO PUMP

COMMENTS - PROBLEMS WITH PLUGGING OF PRESSURE TAPS FOR ORIFICE METER  
IN THE EFFLUENT GAS DUCT FROM PYROLYZER

TABLE C-19

## PROCESS DATA FOR RUN NO. -7

WASTE - NO FEED (BACKGROUND DATA)

DATE - 2.5.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp.	Effluent Gas Temp	$\%O_2$
	$\Delta P^{**}$	SCFH <sup>***</sup>	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P^*$	SCFH	$\Delta P^*$	$^{\circ}F$	$^{\circ}F$	
10:00	2.35	1750	8.25	180	1.0	1500	1.3	4800	0.15	1400	1160	0.1
10:30	1.70	1500	6.0	154	1.0	1500	1.1	4400	0.13	1400	1190	0.1
11:00	1.50	1400	5.4	145	1.0	1500	1.0	4200	0.12	1400	1210	0.1
11:30	1.50	1400	5.3	144	1.0	1500	1.0	4200	0.13	1400	1210	0.1
12:00	1.60	1440	5.2	142	1.0	1500	0.99	4150	0.11	1400	1220	0.1
12:30	1.30	1300	4.6	135	1.0	1500	0.93	4040	0.12	1400	1200	0.1
1:00	1.40	1350	4.7	137	1.0	1500	0.96	4100	0.11	1400	1200	0.1

\* Higher  $\Delta P$  readings are due to the plug-up of pressure taps from carbon soot formed in prior runs with styrene tar waste

\*\* Pressure differential - inches of water

\*\*\*Flow rate - standard cubic feet per hour

TABLE C-20

## PROCESS DATA FOR RUN NO. -7

WASTE - NO FEED (BACKGROUND DATA)

DATE - 2.5.76

Time	Incinerator Burner Gas Burner #1                  Burner #2				Incinerator Air		Incinerator Auxiliary Gas Burner #1                  Burner #2				Incin. Temp.  °F	Vapor Inlet  °F	Stack Temp.  °F
	$\Delta P^*$	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH			
10:00	0.7	270	0.7	270	24.5	38,400	2.8	450	2.8	450	1520	1000	650
10:30	0.7	270	0.7	270	22.5	36,600	2.8	450	2.8	450	1510	1025	650
11:00	0.7	270	0.7	270	23.5	37,500	2.8	450	2.8	450	1510	1040	650
11:30	0.7	270	0.7	270	24.0	38,000	2.8	450	2.8	450	1510	1040	650
12:00	0.6	252	0.6	252	24.0	38,000	2.7	442	2.7	442	1515	1060	650
12:30	0.7	270	0.7	270	24.0	38,000	2.8	450	2.8	450	1515	1040	660
1:00	0.7	270	0.7	270	24.0	38,000	2.7	442	2.7	442	1510	1030	660

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-21

PROCESS DATA FOR RUN NO. -8

WASTE - RUBBER WASTE

DATE - 2.17.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp.	Effluent Gas Temp	%O <sub>2</sub>
	$\Delta P^*$	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	°F	°F	
10:00	2.5	1800	8.5	183	1.0	1500	.61	3350	.06	1400	1120	0.1
10:30	2.2	1675	8.5	183	1.0	1500	.61	3350	.06	1400	1160	0.1
10:45	2.2	1675	8.5	183	1.0	1500	.6	3300	.06	1400	1170	0.2
11:00	2.2	1675	8.5	183	1.0	1500	.63	3450	.06	1400	1180	0.2
11:30	2.3	1725	8.5	183	1.0	1500	.73	3700	.09	1400	1190	0.2
12:10	2.2	1675	8.5	183	1.0	1500	.89	4100	1.	1400	1190	0.2

NOTE: Feed started at 10:15 a.m. and stopped at 12:15 p.m.

\* Pressure differential - inches of water

\*\*Flow rate - standard cubic feet per hour

TABLE C-22

## PROCESS DATA FOR RUN NO. -8

WASTE - RUBBER WASTE

DATE - 2.17.76

Time	Incinerator Burner Gas				Incinerator		Incinerator Auxiliary Gas				Incin. Temp.	Vapor Inlet	Stack Temp.
	Burner #1	Burner #2		Air		Burner #1	Burner #2						
	ΔP *	SCFH**	ΔP	SCFH	ΔP	SCFH	ΔP	SCFH	ΔP	SCFH	°F	°F	°F
10:00	.75	280	.75	280	20.0	34,750	2.85	455	2.85	455	1510	940	620
10:30	.7	270	.7	270	23.5	37,500	2.4	415	2.4	415	1510	1070	660
10:45	.7	270	.7	270	23.5	37,500	2.4	415	2.4	415	1510	1105	660
11:00	.6	250	.6	250	24.0	38,000	2.2	400	2.2	400	1520	1105	670
11:30	.65	260	.65	260	25.0	38,800	2.0	380	2.0	380	1515	1130	665
12:10	.55	240	.55	260	25.0	38,800	2.0	390	2.0	380	1515	1150	665

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-23  
PROCESS DATA FOR RUN NO. -8

WASTE - RUBBER WASTE

DATE - 2.17.76

HEARTH CYCLE TIME	2.5 PER HOUR
RESIDENCE TIME IN HOT ZONE	15 MINS
FEEDING RATE	26.75 LBS/HR
TOTAL AMOUNT FED	53.5 LBS
RESIDUE COLLECTED	16 LBS
LAYER THICKNESS	5/8" to 3/4"
FEEDER	1/2" x 7-1/2" NOZZLE, PISTON

COMMENTS - 90 TO 95% OF THE RESIDUE WAS IN THE LUMP FORM. THESE LUMPS WERE CHARRED ON THE OUTSIDE, BUT THE CORE WAS NOT PYROLYZED. THE SPEED OF THE PISTON TRAVEL WAS FAST AND IT WAS IN THE MAGNITUDE OF 2 TO 3 SECONDS. THE NUMBER OF STROKES WERE 3 PER MINUTE.

TABLE C-24

## PROCESS DATA FOR RUN NO. -9

WASTE - RUBBER WASTE

DATE - 2.18.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp.	Effluent Gas Temp	%O <sub>2</sub>
	$\Delta P$ *	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	°F	°F	
8:15	2.8	1900	7.0	166	0.7	1250	.63	3400	.04	1400	1120	0.4
8:30	1.6	1440	6.0	154	0.7	1250	.44	2600	.03	1400	1140	0.4
8:45	2.0	1610	7.4	170	0.7	1250	.6	3300	.06	1400	1140	0.3
9:00	2.0	1610	7.5	172	0.7	1250	-	-	.07	1400	-	0.2
9:35	1.7	1480	6.4	160	0.7	1250	-	-	.07	1400	1160	0.2
10:00	2.0	1610	7.5	172	0.7	1250	-	-	.08	1400	1170	0.2

NOTE: Feed started at 8:30 a.m. and stopped at 10:20 a.m.

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-25

## PROCESS DATA FOR RUN NO.-9

WASTE - RUBBER WASTE

DATE - 2.18.76

Time	Incinerator Burner Gas				Incinerator		Incinerator Auxiliary Gas				Incin. Temp.	Vapor Inlet	Stack Temp.
	Burner #1	Burner #2			Air		Burner #1	Burner #2					
	$\Delta P$ *	SCFH **	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	°F	°F	°F
8:15	.75	280	.75	280	17.5	32,400	2.8	450	2.8	450	1500	950	590
8:30	.75	280	.75	280	20.0	34,750	2.8	450	2.8	450	1510	965	600
8:45	.75	280	.75	280	23.0	37,000	2.3	410	2.3	410	1510	1040	650
9:00	.6	250	.6	250	22.5	36,800	2.2	400	2.2	400	1515	1100	650
9:35	.65	260	.65	260	22.0	36,250	2.4	415	2.4	415	1510	1000	620
10:00	.6	250	.6	250	23.0	37,000	2.0	380	2.0	380	1510	1160	660

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-26

PROCESS DATA FOR RUN NO. -9

WASTE - RUBBER WASTE

DATE - 2.18.76 (A.M.)

HEARTH CYCLE TIME	2.5 PER HOUR
RESIDENCE TIME IN HOT ZONE	15 MIN
FEEDING RATE	20.7 LBS/HR
TOTAL AMOUNT FED	34.5 LBS
RESIDUE COLLECTED	6 LBS
LAYER THICKNESS	1/2" to 5/8"
FEEDER	3/8" x 7-1/2" NOZZLE, PISTON

COMMENTS - 60 to 70% OF THE RESIDUE WAS IN THE FORM OF LUMPS WHICH WERE PYROLYZED ONLY FROM THE OUTSIDE. THE REMAINING RESIDUE WAS IN THE FORM OF SMALL PARTICLES AND WAS COMPLETELY PYROLYZED. THE PISTON TRAVEL SPEED WAS FAST ( 2 to 3 SECONDS) AND NUMBER OF STROKES WERE 2/MIN.

TABLE C-27

## PROCESS DATA FOR RUN NO. -10

WASTE - RUBBER WASTE

DATE - 2.18.76

Time	Pyro. Burner Air		Pyro. Burner Gas		Inert Gas		Effluent Gas		Pyro. Press	Pyro. Temp.	Effluent Gas Temp	%O <sub>2</sub>
	$\Delta P^*$	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	°F	°F	
12:55	1.5	1400	5.2	145	.65	1225	.47	2900	.05	1390	1140	0.35
1:15	1.45	1375	5.2	145	.65	1225	.52	3050	.07	1395	1170	0.4
1:45	1.85	1535	6.8	165	.65	1225	.64	3400	.07	1400	1170	0.3
2:00	1.6	1440	5.6	148	.65	1225	.58	3250	.07	1400	1180	0.3
2:35	1.9	1575	7.1	168	.65	1225	-	-	.06	1400	1190	0.3

NOTE: Feed started at 1:15 p.m. and stopped at 3:00 p.m.

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-28

PROCESS DATA FOR RUN NO. -10

WASTE - RUBBER WASTE

DATE - 2.18.76

Time	Incinerator Burner Gas Burner #1                  Burner #2				Incinerator Air		Incinerator Auxiliary Gas Burner #1                  Burner #2				Incin. Temp.	Vapor Inlet	Stack Temp.
	$\Delta P^*$	SCFH**	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	$\Delta P$	SCFH	°F	°F	°F
12:15	.6	250	.6	250	22.0	36,250	2.3	410	2.3	410	1510	1000	600
1:15	.55	240	.55	240	23.5	37,500	1.7	350	1.7	350	1520	1100	620
1:45	.55	240	.55	240	24.0	38,000	1.65	345	1.65	345	1510	1240	630
2:00	.55	240	.55	240	25.5	39,000	1.9	370	1.9	370	1510	1110	630
2:35	.5	230	.5	230	23.5	37,500	1.5	330	1.5	330	1410	1225	630

\* Pressure differential - inches of water

\*\* Flow rate - standard cubic feet per hour

TABLE C-29

PROCESS DATA FOR RUN NO. -10

WASTE - RUBBER WASTE

DATE - 2.18.76 (P.M.)

HEARTH CYCLE TIME	2.5 PER HOUR
RESIDENCE TIME IN HOT ZONE	15 MINS
FEEDING RATE	16 LBS/HR
TOTAL AMOUNT FED	28 LBS
RESIDUE COLLECTED	3.5 LBS
LAYER THICKNESS	3/8" to 1/2"
FEEDER	1/4" x 7-1/2" NOZZLE, PISTON

COMMENTS - RESIDUE CONTAINED ONLY ABOUT 5-10% LUMPS AND REST OF IT WAS  
IN THE PARTICLE FORM WHICH WAS ALMOST COMPLETELY PYROLYZED.

## APPENDIX D

### ASSESSMENT OF ENVIRONMENTAL IMPACT OF DESTROYING CHEMICAL WASTES

at

SURFACE COMBUSTION DIVISION  
MIDLAND-ROSS CORPORATION  
2375 DORR STREET  
TOLEDO, OHIO 43691

The rotary hearth pyrolyzer will be evaluated for its capability of destroying the following chemical wastes:

Styrene Tars  
Rubber Wastes  
API Separator Bottoms (petroleum wastes)

The pilot size pyrolyzer is estimated to have a maximum capacity of 45 kilograms per hour. It is equipped with a rich fume incinerator for combustion of the off-gases from the pyrolyzer. The incinerator exhausts to the atmosphere through a short stack (approximately 8 meters high) at a temperature of approximately 870°C. There is no water used in the pyrolyzers; consequently, the emissions to the environment will be stack gases and solid wastes such as the waste shipping containers and char from the pyrolyzer.

The pyrolyzer is located in a building within the extensive manufacturing complex of Surface Combustion. It is estimated to be approximately 0.1 kilometers from the edge of their property. The surrounding area is industrial/residential. On one side of the Surface Combustion property, furthest from the location of the pyrolyzer, is a high concentration of homes and apartment buildings. Other residences are scattered among the various industrial properties and the closest of these is approximately 0.2 kilometers away. A cemetery and a vacant food storage warehouse are the closest properties to the location of the pyrolyzer. In addition, an asphalt blending plant is located adjacent to the Surface Combustion properties and other manufacturing or research development facilities are located in the immediate vicinity. The University of Toledo Campus is within one kilometer of the site. The vegetation in the immediate vicinity of the plant is urban in nature, i.e., trees and lawns. The only apparent wildlife in the immediate vicinity is the usual birdlife found in such urban developments and, probably, the normal rodent population. A major motor vehicle artery lies on one side of the property and there is heavy traffic within less than 0.2 kilometers of the pyrolyzer. The traffic density has been so heavy in the past as to effect the carbon monoxide readings on sensitive instruments being used to monitor combustion

processes. Operation of the rich fume incinerator is moderately noisy (estimated to be between 85 and 90 db at the unit) which should not present any impact on the neighborhood noise level above that of the vehicular traffic.

The most severe potential environmental impacts are expected to be from (1) storage and handling of the wastes prior to testing, (2) the emissions that occur during the test and (3) the disposal of the shipping containers, undegraded wastes and the residue remaining from pyrolysis. Before discussing the unique aspects of each area of concern, it is well to recognize that the components identified in the wastes are not exceptionally toxic. Information taken from the Toxics Substances List of 1974, list the following pertinent information for the identified constituents.

Styrene -	Range of lowest level of reported toxicity to man is from 376 - 600 ppm: inhalation effects are principally irritation and nervous system. OSHA standards for time weighted average exposure in air is 100 ppm with ceiling of 200 ppm and peak exposures of 600 ppm.
Butadiene -	OSHA standard is time weighted average exposure in air of 1000 ppm.
Nonylphenol -	(mixed isomers) reported LD50 in rats is 1620 mg/kg.
Methylnaphthalene -	Oral LD50 in rates is 4360 mg/kg.
Dimethyl Naphthalene -	Not reported in Toxic Substances List
Sulfur -	Not included in Toxic Substances List.

Consequently, the most significant problem expected from these wastes is hazardous in nature such as the possibilities of explosive mixtures occurring in tightly enclosed spaces, fire, etc., since they are not apparently very toxic to human or animal life.

#### Storage and Handling

Upon receipt, the waste shipments will be inspected by the Receiving Dock personnel at the Surface Combustion and the Senior Research and Development Engineer in charge of the program. Storage of the 12 drums of each waste will be either on an outdoor concrete pad adjacent to the pyrolyzer building or in an appropriate storage building. Since none of the wastes are highly fluid, diking around the storage area is not considered necessary. Any leakage or spillage will be absorbed with sawdust and put into containers for subsequent treatment or disposal. There will be a characteristic hydrocarbon odor in the immediate vicinity when drums are opened prior to sampling and feeding into the pyrolyzer.

Odor detection beyond the boundaries of the property should not be apparent especially because of the high density of vehicular traffic in the area.

### Test Runs

The greatest potential environmental impact foreseen during the test would occur if the rich fume incinerator failed and the hot gases from the pyrolyzer vented to the stack. Because the stack refractory will be hot, there is an excellent possibility that ignition of these gases would occur. However, the conditions for combustion will be less than optimum and it is expected that a smoke plume would occur. The design of the system is such as to make this an unlikely occurrence and furthermore, if such a failure did occur, it is not likely to be of long duration. A less obvious environmental impact would occur if all of the sulfur contained in the styrene tar wastes reported to the off-gases and was burned to sulfur oxides in the rich fume incinerator. Dispersion calculations based on the assumption that all of the sulfur was emitted as oxides from the stack during peak feed rates indicated that ground-level conditions might reach a value of 117 micrograms per cubic meter at a distance of 0.3 kilometers from the stack when the wind velocity is under 3 meters/second and 153 micrograms per cubic meter at a distance of 0.17 kilometers from the stack and a wind velocity of 7 meters per second. These concentrations are above the annual arithmetic standards for primary ambient air quality of 80 micrograms per cubic meter but below the maximum 24 hour concentration of 365 micrograms per cubic meter permitted once per year.

This information will be reviewed with the Toledo Pollution Control Agency, 26 Main Street, Toledo, Ohio 43605, by Surface Combustion for purposes of ascertaining if such conditions are permitted under the applicable codes. Because of the proximity of the asphalt blending plant, it is doubtful that if the worst conditions prognosticated above occurred, there will be any significant additional environmental impact. Because the maximum concentration level estimated above is approximately 1/8 of the threshold odor of concentration (0.47 ppm) for SO<sub>2</sub> it is highly unlikely that the ground-level SO<sub>2</sub> concentrations will be detectable except by ambient air monitoring equipment. To prevent these conditions from occurring, it is proposed to periodically monitor these sulfur dioxide emissions from the stack and establish a maximum level at which operations would be curtailed.

### Disposal of Containers and Residues

The anticipated method for disposal of emptied shipping containers char residue from the tests, and any excess wastes not used in the test program will be via Glass City Disposal Company into a landfill at Bryan, Ohio, which is operated by H&H Industry. This landfill is reportedly approved by the State of Ohio for drummed chemical wastes including those with high heating values. In the eventuality that approval for landfill

of any excess wastes is not forthcoming, it is expected that the excess wastes will be pyrolyzed and only the empty drums and excess char would go to landfill. No material will be sent to landfill until the results of analyses on wastes and residues have been obtained and examined to insure that they are compatible with landfilling regulations.

## APPENDIX E

### METRIC TO ENGLISH UNIT CONVERSION

<u>Metric Units</u>	<u>Equivalent English Units</u>
1 KCal	3.966 Btu
1 m <sup>3</sup>	35.3 CuFt

μσ1467a  
SW-122c.2