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Organic Rankine-Cycle Power Systems Working Fluids Study: Topical Report No. 2 – Toluene

R. L. Cole, J. C. Demirgian, and J. W. Allen

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ABSTRACT

The U.S. Department of Energy initiated an investigation at Argonne National Laboratory in 1982 to experimentally determine the thermal stability limits and degradation rates of toluene as a function of maximum cycle temperature. Following the design and construction of a dynamic test loop capable of closely simulating the thermodynamic conditions of typical organic Rankine-cycle (ORC) power systems, four test runs, totaling about 3900 h of test time and covering a temperature range of 600-677°F, were completed. Both liquid and noncondensable-vapor (gaseous) samples were drawn periodically and analyzed using capillary-column gas chromatography, gas chromatography/mass spectrometry, and mass spectrometry. A computer program that can predict degradation in an ORC engine was developed. Experimental results indicate that, if oxygen can be excluded from the system, toluene is a stable fluid up to the maximum test temperature; the charge of toluene could be used for several years before replacement became necessary. (Additional data provided by Sundstrand Corp. from tests sponsored by the National Aeronautics and Space Administration indicate that toluene may be used at temperatures up to 750°F.) Degradation products are benign; the main liquid degradation products are bibenzyls, and the main gaseous degradation products are hydrogen and methane. A cold trap to remove gaseous degradation products from the condenser is necessary for extended operation.

1 INTRODUCTION

Recovery of waste heat in an economically acceptable manner from various industrial processes has been a vital facet of U.S. energy policy over the past several years. The Rankine-cycle power system (RCPS), using organic working fluids, has long been recognized as one of the viable technologies. (These systems operate on the Rankine cycle, in which a working fluid is alternately vaporized and condensed using waste heat and a low-temperature heat sink. The high-temperature, high-pressure fluid is expanded through an expansion engine to produce mechanical power or electricity.)

Typically, an organic Rankine-cycle power system (ORCPS) can use single-stage turbines and still achieve higher cycle efficiencies than systems using low-temperature steam, because of the relatively low enthalpy drop and full admission through the nozzle made possible by the high molecular weight of the organic fluid.

However, serious concerns over the long-term thermal stability of organic fluids and effects on system performance, reliability, and overall economic viability have impeded widespread development and deployment of this technology to date. Prototypical systems designed on the basis of thermal-stability results from static capsule testing have failed to operate entirely to the satisfaction of their developers. The working fluid must also be compatible with the common materials of construction at system design temperatures. Reliable data are required to establish the maximum possible temperature and the fluid replacement rate. Dynamic loop testing of working fluids of interest could provide such data for system designers. The findings presented here are part of a concerted effort under way to alleviate performance, reliability, and economic concerns and accelerate development and deployment of this technology in the industrial sector of the U.S. economy.

The thermal stability of an organic fluid is a general term used to describe the ability of the fluid to withstand a given temperature without significant decomposition. Above a certain temperature, an organic fluid will begin to decompose at a measurable rate, and this rate will increase with increasing temperature. In an "inert" environment, a fluid can decompose as a result of breakage of chemical bonds, without reacting with other materials. Stability against this type of decomposition is a measure of the inherent bond strength of the molecule and has been referred to as *thermal* stability.¹ The term *thermochemical* stability is used to describe the stability of a fluid in contact with the materials in the working environment. Thus, the thermochemical-stability temperature of the fluid provides a limitation on the maximum allowable operating temperature of a prototypical ORCPS. The thermochemical-stability temperature is typically less than the thermal-stability temperature by approximately 100-400°F.¹ In this report, the single term *thermal stability* will be used to indicate stability of a fluid in its working environment.

In a previous study, based on systems analysis covering performance and economic considerations (rate of return on investment, ROI), six organic fluids were identified to harness the energy content of a range of industrial waste-heat streams.² However, questions about long-term thermal-stability temperatures of these fluids for this application could not be explicitly answered on the basis of available information. In some cases, reliable data were either nonexistent or inconclusive. This uncertainty became the motivation for undertaking the current dynamic loop testing program.

The six fluids to be investigated under this program are as follows:

- Fluorinol 85 (a mixture of 85 mole percent [mol %] trifluoroethanol, $\text{CF}_3\text{CH}_2\text{OH}$, and 15 mol % water),
- 2-methylpyridine/water (a mixture of 35 mol % 2-methylpyridine, $\text{C}_6\text{H}_7\text{N}$, and 65 mol % water),

- R-113 (1,1,2-trichloro-1,2,2-trifluoroethane, $\text{CCl}_2\text{F}-\text{CClF}_2$),
- Toluene (methylbenzene, $\text{C}_6\text{H}_5-\text{CH}_3$),
- Methanol (CH_3OH), and
- R-11 (trichlorofluoromethane, CCl_3F).

Detailed thermodynamic properties and other characteristics of these fluids are listed in Ref. 2. In this report, discussion is limited to toluene.

Two approaches are used to obtain reliable data on thermal stability: static capsule tests and dynamic loop tests. Capsule tests of fluids serve as an important screening tool in qualitatively and, to some extent, quantitatively establishing the effects of pertinent variables on the fluid degradation rates. However, the results cannot be easily and reliably related to a prototypical system, because not all system operating conditions can be duplicated in the capsule tests. In comparison with a complete system test, small dynamic loop testing is a simpler, more cost-effective approach that still provides a fairly good simulation of the system operating conditions. This approach also allows testing over a wide range of operating temperatures and pressures, including those that could lead to some detrimental effects on an ORCPS due to fluid degradation. (It would be unwise to put an expensive, full-sized system at risk by using it to conduct thermal-stability experiments.)

A review of previous studies was undertaken to develop a viable testing approach for toluene. Table 1.1 lists the RCPS designs for which this fluid was chosen.³⁻¹¹ From these data, it can be seen that the temperature range for toluene is 240-450°C (465-842°F). Previous work associated with thermal-stability investigations is summarized in Table 1.2.^{1,5,7,8,12,13} Only two static capsule tests of toluene, and no dynamic loop tests, were found in the literature. Although the test reported by Miller et al.¹³ is too short in duration to be of much use, the capsule tests by Sundstrand⁵ are extensive.

On the basis of data from the cited work, we decided to examine toluene over a temperature range of 450 to approximately 700°F, the maximum design temperature of our dynamic loop, using a test-run time of about 1000 h at each temperature selected.

TABLE 1.1 Rankine-Cycle Systems Using Toluene

Organization	Application	Rated Power (kW)	Maximum Fluid Temperature (°F)	Reference
Sundstrand	Total energy plant	100	825	3-5
Sundstrand	Remote power	1	725	5-8
Sundstrand	25-passenger bus	67	700	5,9
Sundstrand	Bottoming cycle	600	465	5,7,8,10
Messerschmidt Bolkow-Blohm	Solar power	150	842	7,8
Sundstrand	Solar power	32	572	5,7,8
Sundstrand		1.5	-	5
Sundstrand	Solar irrigation	200	-	5
Ford Aerospace and Communica- tions	Solar power	25	750	11

TABLE 1.2 Thermal Stability and Materials Compatibility of Toluene

Type of Test	Test Duration (h)	Maximum Test Temp., $T_{\max}$ (°F)	Materials in Contact with Fluid at $T_{\max}$	Comments	Reference
Capsule	>336	720	1008 steel	-	1
Theoretical	-	700-730	-	Based on strength of molecular bonds	1
	-	750	-	No supporting data	12,13
	-	896	-	No supporting data	7,8
Capsule	1000	605	SS ^a	4.2×10^{-5} (see note b)	5
	1000	700	SS	6.85×10^{-4}	
	1000	800	SS	2.1×10^{-2}	
	1000	900	SS	9.9×10^{-1}	

^aSS = stainless steel.

^bDegradation rate in %/h.

2 EXPERIMENTAL APPARATUS

Dynamic test loops have previously been designed and constructed to study the thermal stability of organic working fluids for RCPS applications. Heat-addition methods have included direct electrical heating and indirect electrical heating by means of a salt bath or condensing heat-transfer fluid. Most of the loops built by others have used water-cooled condensers but have not included economizers. The types of pumps used have included diaphragm pumps, regenerative turbine pumps, and piston pumps. Fluid flow rates have varied from 10 to more than 250 lb_m/h. The fluid inventory circulation time has varied from 1.8 to 7.8 min for these systems. For comparison, the prototypical organic Rankine-cycle (ORC) engine systems that have been built and operated have a circulation time range of 2.1-12.6 min.

In our experiment, the basic loop consists of a vaporizer/superheater, a pressure reduction station, an economizer/preheater (regenerative heat exchanger), a condenser, and a diaphragm-type metering pump. Indirect heating using condensing Dowtherm®* A was selected for our system because of its low vapor pressure at relatively high temperature (14.7 psia at 495°F). Figure 2.1 shows a flow schematic of the dynamic test loop used in this investigation. Figure 2.2 shows an overall view of the test facility. Figure 2.3 shows the uninsulated evaporator/superheater section, and Fig. 2.4 shows the section containing pressure reduction valves, economizer, condenser, metering pump, pulsation damper, and liquid and noncondensable-vapor sampling stations. Figure 2.5 shows the system controls and data-acquisition center. Details of these components are given following a discussion of general design considerations.

FIGURE 2.1 Schematic Diagram of Organic Rankine-Cycle Test Loop

*"Dowtherm®" is a trademark of Dow Chemical USA, Midland, Mich.

FIGURE 2.2 Overall View of Test Facility

FIGURE 2.3 Evaporator Section during Assembly

FIGURE 2.4 Section Containing Pump, Condenser, Economizer, and Pressure-Reduction Valves

The dynamic test loops are designed to conduct long-term (approximately 1000 h per test) thermal-stability tests under simulated prototypical thermodynamic operational conditions. Stainless steel (SS, types 304 and 316) is used to minimize potential degradation resulting from interaction with loop materials. The system is designed to be leak-tight in order to prevent potential contamination of the fluids by in-leakage of air. Sampling stations are provided to periodically withdraw liquid and noncondensable-vapor samples for chemical analysis. Adequate instrumentation and controls are provided for monitoring the loops during steady-state operations, as well as to diagnose off-design operations. Safety controls are provided for automatic shutdown of the system in case of major system malfunctions.

FIGURE 2.5 Data-Acquisition and Control Center

The design conditions for the dynamic loop are listed in Table 2.1. The ideal design point test cycle is presented on the pressure-enthalpy (P-h) diagram of Fig. 2.6.

The system layout shown in Fig. 2.1 has four major subsystems: the working fluid loop; the fluid heating section; the heat rejection section; and the instrumentation, controls, and data-acquisition system.

2.1 WORKING FLUID LOOP

The working fluid loop consists of an evaporator/superheater, a set of pressure-reduction valves, a regenerative heat exchanger, a condenser, a metering pump, and a pulsation damper. A special procedure is used to remove dissolved air from the toluene before filling the loop.

2.1.1 Evaporator/Superheater

The evaporator/superheater is constructed of 0.375-in.-O.D., 0.035-in.-wall type 304 SS tubing. A tube approximately 79 ft long is formed in a coil of seven loops, each approximately 12 ft long, to provide a total internal heat-transfer surface area of 6.30 ft². This results in an average heat flux of 4310 Btu/h·ft² for toluene, operating at evaporator-outlet conditions of 700°F and 500 psia. Approximately 8 kW of power is required to heat the toluene.

As shown in Fig. 2.7, the evaporator/superheater is instrumented with 9 type-E thermocouples. Figure 2.7 also shows the locations of thermocouples near the pressure-reduction valve (equivalent to the turbine inlet) and in the regenerative heat exchanger.

2.1.2 Pressure-Reduction Station

The pressure-reduction section serves the function of an expansion turbine, though without producing any power. Two Nupro SS-6BW-STE bellows-sealed stainless-steel valves serve to reduce the pressure of superheated vapor emerging from the evaporator/superheater to the condenser pressure. These valves are manually operated and require only minor adjustments during operation.

2.1.3 Regenerative Heat Exchanger

The purpose of the regenerative heat exchanger (economizer) is to recover a substantial amount of heat from high-temperature, low-pressure vapor after the pressure-reduction station, rather than rejecting this heat through the condenser. In a prototypical system, this would result in increased system efficiency; in the dynamic loop, it reduces consumption of electricity.

TABLE 2.1 Design Conditions for Toluene Loop

Component and Location	Temp., T (°F)	Pressure, P (psia)	Fluid Flow Rate (lb _m /h) ^a
Evaporator			
Dowtherm® A vapor	720	<150	-
Working fluid, outlet	700	500	125
Pressure-reduction station outlet	698	18	125
Regenerative heat exchanger			
Vapor, outlet	470	18	125
Liquid, outlet	329	516	125
Condenser ^a			
Vapor	470	18	125
Liquid	120	18	125
Water, inlet	80	<5	[0.6]
Water, outlet	212 ^b	<5	[0.6]
Pump			
Inlet	120	18	125
Outlet	130	525	125

^aWith the exception of water flow rates (in square brackets), which are given in gal/min.

^bBoiling may occur.

The regenerative heat-exchanger shell is constructed of a 5-in.-O.D., 20-in.-long, schedule 40, type 304 SS pipe. The shell is fitted with a double coil made of a 42-ft-long, 0.5-in.-O.D., 0.035-in.-wall, type 304 SS tube. A tubular spacer baffle centers the coil in an annulus and provides cross-flow conditions across the tube bundle as the vapor moves downward toward the outlet. The coil inlet and outlet pass through the bottom end of the shell via vertical welded nozzles. The high-pressure, low-temperature liquid from the pump is heated by the low-pressure superheated vapor before it flows to the evaporator/superheater section.

2.1.4 Condenser

The condenser is a shell-and-tube heat exchanger similar in construction to the regenerative heat exchanger; the organic fluid condenses on the shell side, and laboratory

FIGURE 2.6 Ideal Test Cycle on a Pressure-Enthalpy Diagram

cooling water flows through the tube section. The vapor from the regenerative heat exchanger enters at the bottom of the condenser and flows upward in the shell, where it is condensed by contact with the cold tube. Noncondensable gaseous components tend to accumulate in the condenser, from which they are periodically removed for chemical analysis. The construction material is type 304 stainless steel. The shell is a 25-in.-long, 5-in.-O.D., schedule 40 pipe with spherical caps welded at both ends. The coil is fabricated from a 50-ft-long, 0.5-in.-O.D., 0.035-in.-wall tube. The coil is centered in the annulus by a baffle, which directs the vapor in a cross-flow pattern to the lower drain.

2.1.5 Diaphragm Metering Pump and Pulsation Damper

A Milton Roy FR 151-144 diaphragm metering pump with a rating of 27.9 gal/h and 600 psia at approximately 9-psia net positive suction head (NPSH) is used for pressurizing and circulating the fluids. This pump is constructed of stainless steel and uses a Teflon® diaphragm. Teflon® is inert at the design fluid temperature levels of the pump. The pump flow rate is easily adjusted over a range of 0.1-0.4 gal/min by changing the stroke length (using a micrometer adjustment screw).

FIGURE 2.7 Schematic Diagram of Evaporator, Showing Thermocouple Locations

A pulsation damper is included in the loop following the metering pump to smooth out the pressure fluctuations from the main metering pump. A Pulsatrol 38 damper constructed of stainless steel, it uses a Viton® diaphragm. Viton® has been found to be compatible with toluene in the temperature range of interest for this study. The damper has been modified to pressurize the diaphragm with toluene vapor. Inert gases used to pressurize the damper in previous experiments were found to permeate through the diaphragm and cause unacceptable increases in condenser pressure.

2.1.6 Liquid- and Vapor-Sampling Stations

Figure 2.8 shows schematic diagrams of the sampling stations for liquids and for noncondensable vapors. Periodic withdrawal of both liquid and noncondensable-vapor samples is required to monitor the fluid degradation during a test run. Precautions are taken to prevent contamination of the organic fluids during the sample withdrawal process. The liquid sampling station is located between the condenser and the pump. The liquid sampling procedure is simple and requires a 0.25-in.-O.D., 3-in.-long glass sample tube and a vacuum pump. After the tube is attached to the sample port and

*"Teflon®" and "Viton®" are trademarks of E.I. DuPont de Nemours and Co., Wilmington, Del.

a. Liquid Sampling Station

b. Noncondensable-Vapor Sampling Station

FIGURE 2.8 Schematic Diagrams of Sampling Stations

evacuated, the vacuum valve is closed and the sample valve opened to fill the sample tube. The sample valve is then closed, and the sample tube is removed from the sample port. Two such samples are taken from the loop at each sampling time; the first sample is discarded to remove any stagnant fluid that might be present in the lines leading to the sampling valve.

The noncondensable samples are drawn through the top of the condenser into a sample cooler. A 150-mL SS sampling cylinder is used to draw noncondensable samples from the sample cooler. To obtain a gas sample, a sampling cylinder is attached to the

sample cooler and evacuated. After the sample cooler has been filled with vapor and noncondensable gas from the condenser, the sample cooler is valved off and cooled for one hour to condense whatever vapor is present. The valve between the sample cooler and the sample cylinder is then opened briefly to obtain the sample for analysis.

2.1.7 Toluene Degasification Procedure

Before the toluene is loaded into the loop, dissolved air must be removed; otherwise, dissolved oxygen would react with the toluene during loop operation to produce benzaldehyde and give an unrealistic measurement of the degradation rate. The toluene is loaded into a vessel, where it is frozen with liquid nitrogen; the gas remaining in the vessel is removed with a vacuum pump. After valving off the vacuum pump, the toluene is allowed to melt and release the dissolved gases into the vessel. Three freeze-evacuate-thaw cycles are performed. The pressure in the vessel is monitored at the end of each thaw to ensure satisfactory removal of the dissolved air.

2.2 FLUID HEATING SECTION

Heat is added to the fluids in a pressure vessel enclosing the evaporator/superheater tube bundle. In this vessel, Dowtherm® A is heated and vaporized using three Watlow electrical immersion heaters. Heat is transferred to the working fluid by condensing the Dowtherm® vapor on the outside surfaces of the evaporator/superheater coil. The test loop uses three 480-V (alternating current), 7.5-kW heaters of the 2-in. by 11-1/2 National Pipe Thread (NPT) screw-plug type; each heater has three elements, for a total of nine heating elements. The heaters have a power density rating of 23 W/in.² with the steel sheath at 343°C (650°F). One heater is wired to provide 7.5 kW of heat in switchable, 2.5-kW increments. The other two heaters are connected to a Barber-Coleman 527Z temperature controller operating at 240 V (alternating current) to provide an additional 0-3.75 kW of heat.

The pressure vessel is constructed of 16-in.-diameter, 50-in.-long, type 304 SS pipe. It is designed to withstand 800°F at 150 psia. One end is closed by a welded cap, and the other end is closed by a flange through which the evaporator/superheater coils pass. The pressure vessel is fitted with a 150-psi rupture disk, a low-liquid-level alarm, a fluid drain, a fluid fill connection, and thermocouples. The system is designed to provide 700°F toluene vapor at the maximum allowable temperature of 720°F for Dowtherm® A.

2.3 HEAT REJECTION SYSTEM

The heat rejection system consists of a plastic coolant-storage tank, a heat-exchange coil, and a variable-speed gear pump. The cooling water is drawn from the storage tank by the pump, circulates through the condenser (where it removes heat from the loop), and returns to the storage tank. Chilled water circulating through the heat-exchange coil removes heat from the storage tank, maintaining its temperature at approximately 20°C. A potentiometer on the control panel adjusts the gear-pump speed to maintain the desired condenser pressure.

2.4 INSTRUMENTATION, CONTROLS, AND DATA-ACQUISITION SYSTEMS

Temperatures at various locations in the loop are measured by using type-E Chromel/Constantan thermocouples, and pressures are measured using Sensotech T- and Z-series strain-gauge pressure transducers. Mechanical gauges are installed to measure pressures at other locations and to verify the pressure transducer readings. The signals from the thermocouples and pressure transducers are recorded by a Digistrip II data logger and are monitored by the safety circuit, which automatically turns the loop off if a malfunction is detected.

Rough control of the heat input to the loop is achieved by manually switching three of the 480-V, 2.5-kW heater elements. Fine control is achieved by a Barber-Coleman 527Z proportional controller acting on the remaining six heater elements. The controller and its heater elements operate on 240-V, single-phase power to provide an additional 0-3.75 kW of heat. This arrangement provides four overlapping ranges of heat: 0-3.75 kW, 2.5-6.25 kW, 5.0-8.75 kW, and 7.5-11.25 kW. The temperature controller senses the toluene vapor temperature immediately upstream of the pressure-reduction valve with a type-K thermocouple.

The condenser pressure is controlled by manually adjusting the coolant flow rate. The evaporator pressure is controlled by manually adjusting the expansion valves. The toluene flow rate is controlled by manually adjusting the metering pump stroke.

A Digistrip II, 48-channel data logger is used to collect and record all temperature, pressure, and flow-rate data at half-hour intervals. In addition to its data-logging functions, the Digistrip II sends its measurements for each 20-s period to an Apple II+ computer by means of an RS-232C line. The computer was installed about 100 h after the second test began and continued recording data until the end of the last (fourth) test. The computer is programmed to (1) accept data from one or two Digistrip data loggers, each transmitting data for each 20-s period; (2) display all of the measurements on the screen; (3) transmit data at half-hour intervals to a digital tape recorder; (4) store up to eight sets of data (taken at half-hour intervals) in buffers; and (5) transmit the contents of those buffers to a dial-up modem, so that the loop can be monitored from a remote location.

A two-channel strip chart recorder (Lenseis 6502) continuously monitors the evaporator outlet temperature and condenser pressure.

3 ANALYTICAL TECHNIQUES AND PROCEDURES

In the present investigation, both liquid and gaseous (noncondensable-vapor) samples are periodically collected and analyzed. Components of the gaseous samples are identified and quantified by mass-spectrometric analysis. The approach used for the chemical analysis of liquid samples is to first identify the degradation products and then determine the concentration of each degradation product in each toluene sample; the sum of degradation-product concentrations is considered to be the total degradation for each sample. Qualitative identification is based upon gas chromatography/mass spectroscopy (GC/MS). Quantitative analysis of the concentration of degradation products is based upon gas chromatographic analysis using a flame ionization detector (FID). The data generated by the gas chromatograph were analyzed using custom-written software developed at Argonne for this project. The equipment used and the type of data obtained are described below.

3.1 MASS-SPECTROMETRIC GAS ANALYSIS

The gaseous (noncondensable-vapor) samples are collected from the condenser in 150-mL SS cylinders and analyzed with a modified Consolidated Engineering Co. CEC21-620/gas mass spectrometer. The sample cylinder is connected to the spectrometer, and the air between the two is pumped out. After a satisfactory vacuum is attained, the sample container is opened to the mass spectrometer. The sample is allowed to expand to 100 torr pressure in the inlet and a 3-mL calibrated volume; the 3 mL is further expanded in a 3000-mL reservoir and introduced into the spectrometer.

Gas molecules are ionized by bombardment with 70-eV electrons, and the ions are injected into a permanent magnetic field, where they are sorted according to mass number:

$$M/e = KH^2/V \quad (3.1)$$

where M is the mass of the ion, e is the charge of an electron, K is a calibration constant, H is the magnetic field, and V is the applied voltage. The focused ions impinge on a collector, where they give up their charge, and the signal thus generated is amplified and delivered to a strip-chart recorder. Every molecular species has a unique spectrum of fragments, isotopic ions, and multiply charged ions that identifies it. A tabulation of ion peak height at each mass number relative to the largest (base) peak is obtained from a spectrum of the pure gas. Since the height of the base peak is proportional to the pressure of the gas in the spectrometer, the spectrum is made at known pressure to determine the sensitivity coefficient (i.e., peak height/pressure).

In analyzing a gas mixture, therefore, the first step after reloading a spectrum and measuring the peak heights is to sort out the components by their patterns. The

second step is to ascertain the true height of the base peak of each component x and to calculate its partial pressure pp_x :

$$pp_x = h_x/s_x \quad (3.2)$$

where h_x is the peak height of component x and s_x is the instrument sensitivity for that component. The percentage of each component x is obtained by summing the partial pressures of all the components and normalizing:

$$f_x = \frac{pp_x}{\sum_i pp_i} \times 100 \quad (3.3)$$

where pp_i is the partial pressure of component i and f_x is the quantity of component x , expressed as a percentage of the total.

The accuracy of analysis depends on various factors, including the accuracy of pressure measurements for the standard, the stability of the electronic components, and the nature of the sample. If two or more components -- such as N_2 , CO , and C_2H_4 -- have the same base peaks, the sorting out by pattern becomes more difficult and less reliable. For relatively simple mixtures, an overall accuracy of about $\pm 2\%$ can be achieved. For complex mixtures, $\pm 5\%$ accuracy is more realistic.

3.2 GAS CHROMATOGRAPHY/MASS SPECTROSCOPY

Degraded samples of toluene are used to establish initial conditions applicable for both gas chromatography (GC) and GC/MS analysis. After conditions are optimized, GC/MS analysis is used to identify degradation products. We have found that the concentrations of the different degradation products vary substantially with temperature, but very few additional degradation products are formed at the higher temperatures compared with products formed at the lower temperatures.

Bibenzyl and six other classes of aromatic degradation products were identified. The classes are alkylbenzenes, multiring aromatics, substituted styrenes, dimethylbiphenyls (C2-BIP), C1-diphenylmethanes, and methyl and trimethyl biphenyls (C1-BIP, C3-BIP). In addition, biphenyl is present in much lower concentration. For the temperatures studied, bibenzyl is the major degradation product. Measurable but insignificant amounts of benzaldehyde were also formed from (1) air that remained in the loop after evacuation but before the initial charge of toluene was loaded, (2) air incursion into the loop during testing, (3) oxygen-bearing impurities present in the toluene before the tests began, and (4) small quantities of dissolved air remaining in the toluene after the triple freeze-thaw procedure for removing dissolved gases was completed.

A Hewlett-Packard (HP) 5984 GC/MS instrument is used with SPEED software. Mass spectra are obtained using electron impact (EI). The molecular ions were present for virtually all degradation products, which facilitated identification. Individual geometrical isomers could not be distinguished, because the mass spectra were virtually identical; hence, geometrical isomers are not reported (e.g., C1-biphenyl is used as the

name for all three geometrical isomers of the compound, 1-methylbiphenyl, 2-methylbiphenyl, and 3-methylbiphenyl).

Following GC/MS identification of the components, the retention indexes are calculated for each component by a computer program. A data file, consisting of the retention index and the name for each component, is entered into the PE 3600 laboratory computer. This file is updated for any new components identified in subsequent degradation samples. Customized software written for the program accesses the data file and makes preliminary identifications based on its contents. All identifications were verified before being included in this report.

3.3 GAS CHROMATOGRAPHY

All liquid samples are analyzed on an HP 5880A gas chromatograph equipped with an FID, a split/splitless injector, and a cartridge tape unit (CTU) to provide a quantitative measure of the nonvolatile organic degradation products formed. All injections are made split with a 30-mL/min split flow. The sample is injected undiluted. The GC conditions used for the analysis are shown in Table 3.1. Gas chromatograph run reports are stored on the CTU for further processing. The instrument has an interface with a Perkin Elmer (PE) Sigma 7500 chromatography data station, which is used to store the chromatogram. The software package used, Chromatographics 3, contains exceptional replotting capabilities.

The GC report, consisting of retention times and peak areas, is transmitted via data communication interface (DCI) on the 5880A to a PE 3600 laboratory computer, where it is stored on disk. Customized software was written to provide reports consisting of preliminary identification, retention index (based on an aromatic standard), and concentration (based on user-set response factors). These reports are both printed and stored on disk. The PE 3600 and 7500 laboratory computers both have an interface with the Argonne Vax computers, so results can be transferred to and stored on any other system. The Model 7500 computer can read Model 3600 disks, so the data generated on the older computer can be used on the newer system.

An analysis standard reflecting the nature of the degradation products was prepared (see Table 3.2). The standard consists of substituted benzenes, biphenyl,

TABLE 3.1 Conditions Employed for Gas Chromatography/Flame Ionization Detection and Gas Chromatography/Mass Spectroscopy Analysis^a

Item	Specified Value
Temperature (°C)	
Injector	270
Initial	40
Final	280
Time (min)	
Initial	2
Final	15
Ramp rate (°C/min)	4

^aColumn = 30 m x 0.32 mm
x 1 µm film (type DB5).

TABLE 3.2 Components of the Standard

Standard	Conc. (ppm)	Retention Time (min)	Response Factor	Time Range (min)
Benzene	22.2	2.64	RF(1)	<9
Ethylbenzene	16.5	7.79	RF(1)	<9
p-Xylene	7.9	8.09	RF(1)	<9
o-Xylene	22.8	8.97	RF(1)	<9
Nonane	39.8	9.24	Not used	Not used
Propylbenzene	10.6	11.37	RF(2)	9-20
Trimethyl- benzene	23.6	12.99	RF(2)	9-20
Naphthalene	61.8	20.69	RF(3)	20-30
Biphenyl	19.1	27.88	RF(3)	20-30
Bibenzyl	21.5	32.61	RF(4)	30-40
3,3'-Dimethyl- biphenyl	7.6	34.76	RF(4)	30-40
4,4'-Dimethyl- biphenyl	22.0	35.34	RF(4)	30-40
Phenanthrene	18.8	40.44	RF(5)	>40

bibenzyl, two substituted C2-biphenyls, and the retention index standards (benzene, naphthalene, and phenanthrene). An aromatic retention index is used because the degradation products are all aromatic, and as the column ages over the course of the project any retention-time drift would be equal for both the degradation products and the standard. That would not be the case if the Kovats alkane retention index system were used. In the aromatic system, the retention indexes of the three standards are benzene, 200; naphthalene, 400; and phenanthrene, 600.

The analysis standard is run each day a degraded toluene sample is analyzed. Customized software calibrates the standard immediately after the run and provides a check for instrument drift. Instrument problems are corrected immediately before samples are analyzed, and the correction is verified by rerunning the analysis standard. The response factors calculated by the computer are stored on disk and used in the calculation of the concentration of the components in the sample.

After each sample is run, the report is transferred to the PE 3600 laboratory computer, where the retention index (RI) program is run. This specially written software produces the report on the sample. It first calculates the retention indexes of all components in the sample. The program then reads the standard response-factor file and averages the response factors of standards in specified retention-time ranges. The concentrations of components in parts per million (ppm) are then calculated using the appropriate response factor. Finally, components are identified from the RI file; these results are printed and stored on disk. The toluene itself is used as the internal standard, and the peak areas are normalized for toluene, so the total concentration of toluene and

its degradation products is unity (1,000,000 ppm). This eliminates any problems with sample loading. All individual reports on samples are hand- verified, and corrections are made using the line editor.

4 SUMMARY OF DYNAMIC LOOP OPERATIONS

Approximately 3800 h of toluene testing in four test runs, ranging from 600 to 677°F, are summarized in this section. During these test runs, vapor temperatures and pressure at the evaporator/superheater exit, condenser pressure and temperatures of the condensate, and temperatures of vapor and liquids at the economizer inlet and exit ends were recorded every half hour. The maximum and minimum cycle operation temperatures and pressures were analyzed for each of the runs to verify relatively steady-state operation of the dynamic loop. A summary of these data for each of the runs is presented in Table 4.1, and the following subsections describe the operations. Appendix A provides detailed information on temperatures and pressures on a daily basis for each of the tests.

4.1 TEST RUN NO. 1 (840 h at 635°F)

The nominal operating conditions for this run were 635°F and 160 psia at the evaporator/superheater exit, 18 psia condenser pressure, and 120°F subcooled condensate temperature.* The actual average operating temperature was 635.3°F, with minimum and maximum values of 625.2 and 652.3°F, respectively. The minimum and maximum values of the pressure at the evaporator/superheater exit were 149.7 and 163.4 psia, respectively, with an overall average value of 158.9 psia. The condenser pressure was between 15.11 and 22.48 psia, with an overall average value of 17.42 psia.

Further insight into loop operations for this run can be gained by examining Table 4.2, which was derived from the operations log of the test. The most serious problems encountered were unplanned shutdowns due to coolant pump seizures at 483 and 485 h elapsed time. After the pump was repaired, samples were drawn at 504 h elapsed time to determine whether any air had entered the loop. An increase of benzaldehyde in the 504-h sample shows that a small quantity of air entered the loop during the downtime, but the amount is considered small enough that the test remains valid. The 168-h sample shows a smaller increase in benzaldehyde after a decrease of condenser pressure to 15.11 psia averaged over several pump strokes; during the pump suction stroke, the pressure was probably subatmospheric. One brief power outage, lasting less than one minute, occurred at 433.5 h elapsed time during a thunderstorm. Temperature and pressure data at 600.5 and 641 h elapsed time were unavailable, due to a data logger malfunction and running out of paper, respectively. The digital tape recorder was not installed as a backup to the paper printout until test no. 2 was under way. After completion of the test some key thermocouples were checked with an independent instrument, and the readings were found to disagree with the data logger readings. This discrepancy was traced to a stuck relay in the data logger and to the fact that all of the

*Results of the 635°F test run are presented first because this run preceded the 600, 650, and 677°F test runs chronologically. The 635°F run also differed substantially from the other runs with respect to evaporator pressure.

TABLE 4.1 Summary of Test Conditions

Run No.	Elapsed Time (h)	Temp. (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
		Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
1	840	635.3	625.2	652.3	158.89	149.70	163.40	17.42	15.11	22.48
2	1006	599.7	595.2	603.5	493.89	450.80	509.80	17.26	10.94	18.68
3	1006	649.7	635.8	652.0	497.94	482.80	516.60	17.82	14.73	19.01
4	1006	677.5	674.0	679.2	500.41	481.00	517.30	17.76	15.81	19.49

TABLE 4.2 Summary of Events for Test No. 1 (635°F)

Elapsed Time ^a (h)	Description of Event
Sample-Taking	
45	First samples
168	Second samples
338	Third samples
504	Fourth samples
649	Fifth samples
840	Sixth samples (test completed)
Other Events	
49	Adjust fluid pump and evaporator temperature.
51.5	Adjust condenser pressure.
67	Adjust condenser pressure and evaporator temperature.
99	Adjust condenser pressure.
164.5	Adjust fluid pump.
433.5	Power outage (<1 min) during thunderstorm.
483	Shutdown due to coolant pump seizure (pump repaired and loop restarted after 19 h downtime).
485	Shutdown due to coolant pump seizure (pump repaired and loop restarted after 2 h downtime).
572.5	Adjust condenser pressure.
600.5	Data logger malfunction due to air-conditioning failure (1.5 h of temperature data unavailable).
641	Data logger out of paper (5.5 h of temperature data unavailable).

^aElapsed time does not include downtime for coolant pump repairs.

thermocouples were the grounded type. After repairing the data logger, we replaced four of the grounded-type thermocouples with the ungrounded type for easier detection of the stuck-relay problem. The relay problem did not recur during the remaining tests. A means of correcting the temperature readings was found, and the corrected temperatures are used in this report.

Additional information can be gained from Table 4.3, which summarizes temperatures and pressures in the intervals between samples; two additional time intervals (terminated by the unplanned shutdowns for coolant pump repair) are shown in the table. The average temperatures for all but one time interval were within 5°F of the nominal temperature. Near the end of the 45-168-h time interval (at 164.5 h), the stroke of the fluid pump was adjusted to obtain better control of the temperature. Only minimum temperatures for the 45-168- and 482-484-h intervals and maximum temperatures for the 168-338- and 649-840-h intervals differed from the nominal

TABLE 4.3 Summary of Temperatures and Pressures in Intervals between Samples for Test No. 1 (635°F)

Interval (h)	Temp. (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
	Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
0-45	636.8	631.4	639.8	160.16	157.50	161.60	17.65	17.08	18.03
45-168	629.5	625.2	638.6	160.50	157.50	163.40	17.23	15.11	18.09
168-338	635.9	631.7	652.3	160.10	156.70	162.60	17.57	17.10	18.09
338-482 ^a	636.7	632.8	640.7	156.94	149.70	160.50	17.47	16.87	18.39
482-484 ^b	631.7	627.4	635.4	158.03	154.60	159.90	17.92	16.23	19.09
484-504	636.8	630.0	639.8	160.44	157.30	163.10	17.59	16.59	18.19
504-649 ^c	636.1	631.7	640.0	158.31	156.70	159.80	17.69	16.58	22.48
649-840	636.4	633.3	651.6	158.20	155.70	160.60	17.08	15.99	18.46
Overall	635.3	625.2	652.3	158.89	149.70	163.40	17.42	15.11	22.48

^aLoop down for 19 h at the end of this time period.

^bLoop down for 2 h at the end of this time period.

^cSeven hours of data unavailable in this time period.

temperature of 635°F by more than 5°F. Average evaporator pressures were within 5 psi of the nominal 160 psia (absolute pressure), and only the minimum pressure for 338-482 h differs from the nominal value by more than 5 psi. Average condenser pressures are all within 1 psi of the nominal 18 psia. Only the maximum pressure for 504-649 h differs from the nominal value by more than 3 psi.

4.2 TEST RUN NO. 2 (1006 h at 600°F)

For test run no. 2, the nominal conditions were 600°F evaporator temperature, 500 psia evaporator pressure, and 18 psia condenser pressure. The actual average evaporator temperature was 599.7°F, with a minimum of 595.2°F and a maximum of 603.5°F. The average evaporator pressure was 493.9 psia, with a minimum of 450.8 psia and a maximum of 509.8 psia. The average condenser pressure was 17.26 psia, with a minimum of 10.94 psia and a maximum of 18.68 psia.

Table 4.4 gives the elapsed times at which the samples were drawn and summarizes events that were recorded in the operations log. At 83 h elapsed time the data logger ran out of paper, making 10 h of temperature and pressure data unavailable; this occurred before the digital tape recorder was installed as a backup for the paper printouts. At 169.5 h a momentary power outage while some equipment was being installed near the loop caused an unplanned shutdown of less than one minute duration. The most serious event occurred at about 425 h, when the evaporator pressure reading began to drift because of a loose connection. Repairing the connection restored the accuracy of the pressure measurement.

TABLE 4.4 Summary of Events for Test No. 2 (600°F)

Elapsed Time (h)	Description of Event
Sample-Taking	
23	First samples
266	Second samples
455	Third samples
623	Fourth samples
766	Fifth samples
938	Sixth samples
1006	Seventh samples (test completed)
Other Events	
83	Data logger out of paper (10 h of temperature data unavailable).
169.5	Momentary power outage due to installation of equipment nearby (no loss of data).
262.5	Data logger overheated (no loss of data).
425	Evaporator pressure reading drifted for 4 h due to a loose connection; connection was repaired.

Table 4.5 summarizes the temperatures and pressures in the intervals between the taking of gaseous and liquid samples. All average, minimum, and maximum temperatures for all intervals were within 5°F of the nominal 600°F. Evaporator pressures tended to be somewhat lower than the nominal 500 psia, but all averages except those from the first 23 h were within 10 psi of the nominal value. All of the minima differed from the nominal value by more than 10 psi, but all of the maxima were within 10 psi of the nominal value. All of the condenser pressures (average, minimum, and maximum) for all intervals were within 3 psi of the nominal 18.0 psia, except for the minimum pressure at startup (0-23 h). This was a relatively uneventful test run.

4.3 TEST RUN NO. 3 (1006 h at 650°F)

For test run no. 3, the nominal conditions were 650°F evaporator temperature, 500 psia evaporator pressure, and 18 psia condenser pressure. These conditions differed from the nominal conditions of test no. 2 only in the evaporator temperature. For this run the actual average evaporator temperature was 649.7°F, with a minimum of 635.8°F and a maximum of 652.0°F. The average evaporator pressure was 497.9 psia, with a minimum of 482.8 psia and a maximum of 516.6 psia. The average condenser pressure was 17.82 psia, with a minimum of 14.73 psia and a maximum of 19.01 psia.

Table 4.6 gives the elapsed times at which the samples were drawn and summarizes events that were recorded in the operations log. As the table shows, the condenser and evaporator pressures required more frequent adjustment than were needed in the tests at lower temperatures. This is probably because more degradation occurs at the higher temperature, although the degradation rate is quite low. In several instances the data logger ran out of paper, but the tape recorder provided backup. At 551 h, the cooling system was temporarily switched to domestic water while the chilled water system was being serviced. At 616 h, the cooling system was switched back to the chilled water supply. The changes of the cooling system did not affect the test.

Table 4.7 summarizes the temperatures and pressures in the intervals between taking of gaseous and liquid samples. All temperatures except the minimum temperature at the end of the test are within 5°F of the nominal 650°F. Although the average evaporator pressure of 497.9 psia is close to the nominal pressure of 500 psia, all of the pressure minima and three of the pressure maxima (for 19-115, 115-282, and 619-786 h) differed from the nominal value by more than 10 psi. This situation is similar to that of test no. 2. All of the condenser pressures were within 3 psi of the nominal 18 psia.

4.4 TEST RUN NO. 4 (1006 h at 677°F)

Test run no. 4, with a nominal evaporator temperature of 677°F, nominal evaporator pressure of 500 psia, and nominal condenser pressure of 18 psia, differed from test runs nos. 2 and 3 only in the evaporator temperature. The actual average evaporator temperature was 677.5°F, with a minimum of 674.0°F and a maximum of 679.2°F. The average evaporator pressure was 500.4 psia, with a minimum of 481.0 psia and a

TABLE 4.5 Summary of Temperatures and Pressures in Intervals between Samples for Test No. 2 (600°F)

Interval (h)	Temp. (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
	Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
0-23	597.4	595.2	603.5	489.86	483.70	508.10	17.36	10.94	18.66
23-266 ^a	598.7	597.7	601.2	493.77	486.20	503.20	17.18	14.04	17.80
266-455	599.7	598.7	600.9	494.75	450.80	509.80	17.14	15.02	17.52
455-623	600.2	599.1	601.1	493.16	486.60	501.00	17.72	14.75	18.42
623-766	600.2	599.4	601.0	491.53	482.40	505.70	17.49	15.35	18.68
766-938	600.4	599.3	601.4	496.74	483.80	507.00	17.07	15.30	17.73
938-1006	600.1	599.4	600.7	492.78	470.50	501.70	16.78	15.82	17.13
Overall	599.7	595.2	603.5	493.89	450.80	509.80	17.26	10.94	18.68

^a10 h of data unavailable in this time period.

TABLE 4.6 Summary of Events for Test No. 3 (650°F)

Elapsed Time (h)	Description of Event
Sample-Taking	
19	First samples
115.5	Second samples
282.5	Third samples
450	Fourth samples
619	Fifth samples
786.5	Sixth samples
955.5	Seventh samples
1006	Eighth samples (test completed)
Other Events	
56	Data logger out of paper (tape recorder provided backup).
112	Adjust evaporator pressure.
141.5	Adjust condenser pressure.
161.5	Adjust evaporator pressure.
280	Adjust evaporator pressure.
280.5	Data logger out of paper (tape recorder provided backup).
329	Adjust condenser pressure.
376.5	Adjust evaporator pressure.
384	Adjust condenser pressure.
520.5	Adjust condenser pressure.
551	Switched to domestic water coolant while Plant Services modified chilled-water system.
616	Switched back to chilled-water system.
640.5	Adjust condenser pressure.
664.5	Adjust condenser pressure.
784.5	Adjust evaporator pressure.
833	Adjust condenser pressure.
952.5	Adjust evaporator pressure. Data logger out of paper (tape recorder provided backup).
976.5	Adjust evaporator pressure.
979.5	Adjust condenser pressure.

maximum of 517.3 psia. The average condenser pressure was 17.8 psia, with a minimum of 15.81 psia and a maximum of 19.49 psia. Although the original intent was to operate as close to 700°F as possible, the pressure limitation of 150 psi in the Dowtherm® A section of the evaporator prohibited a closer approach to 700°F.

Table 4.8 gives the elapsed times at which the samples were drawn and summarizes events that were recorded in the operations log. As the table shows, all of the events involve adjustment of temperature or pressure. Approximately the same number of adjustments were required for this test as for the test at 650°F.

TABLE 4.7 Summary of Temperatures and Pressures in Intervals between Samples for Test No. 3 (650°F)

Interval (h)	Temp. (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
	Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
0-19	647.0	646.1	648.9	496.03	482.80	505.80	18.29	17.93	18.74
19-115 ^a	649.3	648.0	650.5	502.35	487.20	515.80	17.15	15.90	17.75
115-282 ^b	649.5	648.1	650.7	497.98	485.30	516.60	17.91	16.12	18.63
282-450	649.7	648.5	651.3	498.13	488.30	506.80	18.03	15.79	18.58
450-619	649.9	648.3	652.0	497.75	484.00	507.50	17.73	15.71	18.38
619-786	649.9	648.5	650.9	498.46	486.10	512.60	17.90	16.02	18.70
786-955 ^b	650.1	648.0	651.2	495.81	485.00	506.60	17.75	15.51	19.01
955-1006	649.7	635.8	650.7	495.58	485.50	505.70	18.20	14.73	19.01
Overall	649.7	635.8	652.0	497.94	482.80	516.60	17.82	14.73	19.01

^aOne hour of data unavailable in this time period.

^b0.5 h of data unavailable in this time period.

TABLE 4.8 Summary of Events for Test No. 4 (677°F)

Elapsed Time (h)	Description of Event
Sample-Taking	
117.5	First samples
285	Second samples
452	Third samples
600	Fourth samples
789	Fifth samples
958	Sixth samples
1006	Seventh samples (test completed)
Other Events	
20	Adjust evaporator temperature.
92	Adjust evaporator temperature, evaporator pressure, and condenser pressure.
140	Adjust evaporator and condenser pressures.
259	Adjust condenser pressure.
427	Adjust evaporator pressure.
451	Adjust condenser and evaporator pressures.
477	Adjust condenser and evaporator pressures.
527.5	Adjust evaporator pressure.
596	Adjust evaporator temperature.
624.5	Adjust condenser pressure.
672	Adjust condenser pressure.
763.5	Adjust evaporator and condenser pressures.
817.5	Adjust condenser pressure.
843	Adjust evaporator pressure.

Table 4.9 summarizes the temperatures and pressures in the intervals between taking of gaseous and liquid samples. All temperatures were within 5°F of the nominal 677°F. Average evaporator pressures were within 10 psi of the nominal 500 psia, but several of the minima and maxima differed by more than 10 psi from the nominal value. All of the condenser pressures were within 3 psi of the nominal 18 psia.

TABLE 4.9 Summary of Temperatures and Pressures in Intervals between Samples for Test No. 4 (677°F)

Interval (h)	Temp. (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
	Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
0-117	676.6	674.0	678.7	498.84	481.00	516.70	17.76	16.80	18.54
117-285	677.9	676.3	679.2	501.03	487.20	517.30	17.58	16.35	18.18
285-452	678.1	677.0	679.2	503.12	494.90	513.10	17.96	16.55	18.76
452-600	677.1	675.9	678.4	500.07	493.80	510.70	17.95	17.30	18.53
600-789	677.6	675.9	679.1	502.95	490.40	515.30	17.98	16.69	19.49
789-958	677.3	675.8	678.9	496.54	490.70	509.40	17.38	16.10	18.71
958-1006	677.8	676.8	679.1	497.36	491.00	505.70	17.51	15.81	17.93
Overall	677.5	674.0	679.2	500.41	481.00	517.30	17.76	15.81	19.49

5 RESULTS

In this section, impurities and degradation products are identified and quantified for each sample. Chemical reactions leading to the major degradation products are identified. Degradation rates for each test are computed, and a method of predicting degradation, given temperatures and pressures in an ORC system, is developed for situations where one chemical reaction is dominant. A means of extending the prediction method to situations where two or more chemical reactions are important is shown.

5.1 IDENTIFICATION OF LIQUID-PHASE IMPURITIES AND DEGRADATION PRODUCTS

Impurities and degradation products in the liquid samples are identified by gas chromatography/mass spectroscopy (GC/MS), which produces a unique spectrum of molecular fragments for each component. Components are identified by comparing their spectra with catalogued spectra of known compounds. Spectra of components are shown in App. B. The retention time of the compound in the gas chromatograph is then correlated with the identification. When a sample is analyzed on the gas chromatograph with a flame ionization detector (FID), the chromatograms and reports shown in Apps. C and D, respectively, are produced.

The most important initial impurities in the toluene, in descending order of importance, are ethylcyclohexane, C2-cyclohexane isomers, xylene, ethylbenzene, benzene, dimethylcyclopentane isomers, C8-alkane isomers, and ethylmethylcyclopentane isomers. For the 635°F run, a small quantity of Dowtherm® A (consisting of biphenyl and diphenyl ether) is seen as a system contaminant. After operating the loop at temperature, the following degradation products are seen (in descending order of importance): bibenzyl, C2-biphenyl isomers, C1-diphenylmethane isomers, C1-biphenyl, diphenylmethane, C1-fluorene, C3-biphenyl, and benzaldehyde. Benzaldehyde, a partial oxidation product of toluene, could derive from four sources: (1) air remaining dissolved in the initial charge of toluene after the dissolved-gas-removal procedure; (2) air remaining in the loop after evacuation but before the initial charge of toluene was loaded, (3) oxygen-bearing impurities, such as ethers, that were present in the initial charge of toluene; and (4) in-leakage of air during the experimental runs. Whatever the source, oxygen reacts immediately with toluene to form benzaldehyde, as shown in Eq. 5.1.

Components seen in the gaseous samples included hydrogen, methane, carbon monoxide and nitrogen (which cannot be distinguished from one another, because both have a molecular weight of 28), carbon dioxide, argon and helium (which are used for purging the loop and for leak detection), toluene vapor, and other hydrocarbons. The most significant of these components are hydrogen and methane.

5.1.1 Impurities

Although the toluene purchased for these studies is the purest commercially available (Aldrich Gold label), two major classes of chemical impurities are still present at parts-per-million levels: saturated cycloalkanes and aromatics. These impurities elute immediately before or after the toluene.

5.1.1.1 Cycloalkanes

Substituted cycloalkanes are the predominant impurity in toluene. They are relatively stable compared with the toluene itself, and their concentrations vary by only a small amount throughout this study.

By itself, GC/MS is not capable of identifying different isomers of each component. Hence, it is possible to identify an impurity as a C8-cycloalkane, but it is not always possible to positively determine whether the impurity is a C2-cyclohexane or a C3-cyclopentane. Even in cases where it is possible to differentiate the cyclohexanes from cyclopentanes, it is possible to distinguish the different alkylcyclohexane or cyclopentane isomers from each other only occasionally. An example of a typical mass spectrum for a C8-cycloalkane is shown in Fig. 5.1. As can be seen, the geometrical isomers have virtually identical mass spectra. Because these impurities play an insignificant role in degradation and because the identity of each individual isomer is not essential for this project, in most cases we limited the identification to the general form (i.e., C8-cycloalkane). The capability to show that the concentrations of individual cycloalkanes are constant throughout the temperature ranges studied is more important than specifying the identity of each impurity in terms of exact geometrical isomers. For a few of the components, where the identification is not ambiguous (e.g., ethylcyclohexane), we identify the component by its full name.

5.1.1.2 Aromatics

The aromatic impurities present in toluene are benzene, ethylbenzene, and the three xylenes. The benzene is readily discernible by GC/MS, and the ethylbenzene and xylenes (which have identical mass spectra) are identified by the use of standards. The aromatic components do participate in degradation reactions and form identifiable degradation products of their own. The concentrations of benzene, ethylbenzene, and xylene are noted in each of the degraded samples analyzed.

FIGURE 5.1 Mass Spectrum of C8-Cycloalkane Compared with Library Spectra

5.1.2 Degradation Products

Three major types and two minor types of degradation products are identified by GC/MS. The most important are dimers, which consist of bibenzyl, C2-biphenyls, and C1-diphenylmethanes. Alkylbenzenes and C1-fluorenes are present in low concentration.

Bibenzyl, which is easily identified by its unique mass spectrum, is the predominant dimer formed at the temperatures studied. It is formed from a benzyl radical ($\text{C}_6\text{H}_5\text{CH}_2^\bullet$) reacting with another benzyl radical.

C2-biphenyls and C1-diphenylmethane are also formed. In the case of C2-biphenyls, the radical is on a ring position and reacts with another ring radical. C1-diphenylmethane is formed by a benzyl radical reacting with a ring radical.

Two commercially available dimethylbiphenyls (3,3-dimethylbiphenyl and 4,4-dimethylbiphenyl) are used as calibration standards. Identifications in our samples are based upon the retention times of these two isomers and the four mass spectra available from the literature. In each case, the molecular ion at $M/e = 182$ is the base peak, and the M-13 and M-15 fragments are less than half the height of the molecular ion. All components for which this is the case are identified as C2-biphenyls. Only the 3,3 and 4,4 isomers are specifically identified, because their identification is based upon their retention time as established by the standard and their mass spectra. All others with the same mass spectra are identified only as C2-biphenyls. An example of a typical C2-biphenyl and its match with a library of spectra is shown in Fig. 5.2.

Isomers of C1-diphenylmethane are not commercially available, nor are they in mass spectra libraries. From their structure, we determined that their M-15 fragment is more stable than the molecular ion. This stability is due to the extension of aromaticity to the methane carbon (resonance hybrid), made possible by the loss of the methyl group. We anticipated that these components would have an M-15 fragment of greater intensity than the molecular ion. All components having a molecular fragment of $M/e = 167$ of greater intensity than the molecular ion at $M/e = 182$ are identified as C1-diphenylmethanes.

In addition to the above, small quantities of biphenyl, C1-biphenyl and C3-biphenyl, were identified. C1-biphenyls have a molecular ion at $M/e = 168$, and C3-biphenyls have a molecular ion at $M/e = 196$. No attempt was made to identify which isomers of each product are formed. The C1-biphenyl is the product of a benzyl radical and a phenyl radical, whereas the C3-biphenyl is the product of a benzyl radical and a xylenyl radical. These products demonstrate the reactivity of the toluene impurities, benzene and xylene.

As a group, the substituted benzenes are in low concentration. Because the mass spectra of geometrical isomers are identical, each geometrical isomer could not be specified; hence, identification is limited to C3-benzene. The identification of the length of the side chain is routine because of the increment of 14 mass units for each

FIGURE 5.2 Mass Spectrum of C2-Biphenyl Compared with Library Spectra

additional $-\text{CH}_2-$. The following list indicates the method used to identify each of these components:

<u>Name</u>	<u>Molecular Ion</u>
C3-benzene	120
C4-benzene	134
C5-benzene	148

C1-fluorenes were unexpected as degradation products. They are formed in low concentration by a benzyl radical reacting with a toluene radical. The formation of this type of compound indicates that at higher temperature other aromatics, such as phenanthrene and anthracene, would form. C1-fluorenes have molecular ions at $M/e = 180$, and identifications are based upon the presence of this ion.

A complete GC/MS run, including the total ion chromatogram and mass spectra for each component, is presented in App. B.

5.2 MECHANISMS OF DEGRADATION

Degradation appears to take place in three stages: (1) formation of radicals, (2) reaction of radicals with themselves and with toluene, and (3) complex reactions with degradation products, impurities, and their radicals. Formation of the benzyl radical as shown in Eq. 5.2 is the most likely first step, because such a reaction has a lower activation energy than other radical-formation reactions. The fact that bibenzyl is the most important degradation product, rather than C2-biphenyls or C1-diphenylmethane, supports this hypothesis. Removal of the methyl group (Eq. 5.3) or removal of a ring hydrogen (Eq. 5.4) are also possible, but these reactions have higher activation energies than that expressed in Eq. 5.2. At lower temperatures, Eq. 5.2 is expected to dominate, but at higher temperatures, Eq. 5.4 could dominate (because it has five possible activation sites, compared with only three for Eq. 5.2). Thus, at sufficiently high temperatures the probability of the reaction expressed in Eq. 5.4 occurring would be greater than the probability of that in Eq. 5.2 occurring. A fourth fundamental reaction, that of cleaving the ring to form alkenes, would have a higher activation energy than those of the reactions in either Eq. 5.3 or Eq. 5.4. Evidence of ring cleavage is found in the gas sample analysis.

The most likely reaction is that of a benzyl radical reacting with the methyl group of a toluene molecule to form bibenzyl (Eq. 5.5). For each bibenzyl molecule formed in this way, two hydrogen atoms are liberated, which can form a hydrogen molecule (Eq. 5.6). It is also possible for the benzyl radical to attack the ring of a toluene molecule to form C1-diphenylmethane (Eq. 5.7). Not much C1-diphenylmethane is formed by this mechanism, because more energy is required to remove a ring hydrogen from toluene than to remove a methyl-group hydrogen.

The phenyl radical and the methyl radical formed from the reaction in Eq. 5.3 can react in several ways. First, they can combine with hydrogen to form benzene (Eq. 5.8) or methane (Eq. 5.9). Benzene is a large impurity in the initial charge of toluene, so it is difficult to measure the change directly, but the formation of methane at the higher temperatures indicates that there is sufficient energy for these reactions to occur. Second, the methyl radical can react with the toluene molecule to form xylene, another large impurity (Eq. 5.10). Diphenyl methane and C1-biphenyl are seen at the higher temperatures and are evidence that the reactions expressed in Eqs. 5.11 and 5.12 occur.

The reaction expressed in Eq. 5.4 offers five times as many sites for radical formation as that expressed in Eq. 5.3, so the statistical probability for reaction 5.4 could be greater than that for reaction 5.3. Compounds that can be formed from the activated ring are C2-biphenyl (Eq. 5.13), C1-diphenylmethane (Eq. 5.14), and C1-fluorene (Eq. 5.15), all of which are seen at the higher temperatures. Formation of C1-diphenylmethane by reaction 5.14 appears to be more likely than by reaction 5.7. Equation 5.16 shows how xylene, a large impurity in the initial charge of toluene, can be formed.

Reactions of degradation products with toluene, initial impurities, and each other would be complex. Such reactions would lead to a large variety of high-molecular-weight compounds, as can be seen in the chromatograms for the higher-temperature runs. Such compounds are less volatile than toluene and would have a tendency to foul the evaporator; however, measurement of heat-transfer coefficients as a function of time was beyond the scope of the present work.

5.3 LIQUID-PHASE ANALYSIS

In this section, quantitative results of liquid-phase analysis are summarized. The various impurities present in the toluene before testing, as well as benzaldehyde, are shown to create no problems that would invalidate a test. Total degradation rates for each test temperature are computed, and the implications of different degradation products being formed by different mechanisms are discussed.

The gas chromatography reports shown in App. D are summarized in Tables 5.1-5.4. These tables show quantities of the most important impurities and degradation products in parts per million by weight for each sample that was analyzed, together with the totals of each. The most important impurities are benzene, ethylcyclohexane, and xylene. Test run no. 1 (635°F) shows a small quantity of diphenyl ether due to a leak of Dowtherm® A into the vacuum system. None of these impurities is present in a large enough quantity to invalidate the tests.

The major degradation products are bibenzyl, C1-biphenyl, C2-biphenyl, C1-fluorene, and diphenylmethane. Benzaldehyde is an indicator of air leakage into the loop. Benzaldehyde concentrations are plotted in Fig. 5.3. All but three points show benzaldehyde concentrations of less than 20 ppm. High values at 168 and 504 h for test run no. 1 (635°F) indicate sudden brief increases in the air leakage rate (due to low condenser pressure in the first case and 21 h of downtime in the second). All other points show a slow but steady increase in benzaldehyde concentrations, the highest rate being for test run no. 1. Between test runs nos. 1 and 2 and test runs nos. 2 and 3, several packed stem valves in the low-pressure areas of the loop were replaced with bellows-seal valves. Figure 5.3 shows that the later tests have a lower rate of benzaldehyde increase than the earlier tests have, but in no case is the quantity of benzaldehyde sufficient to indicate a catastrophic air leak.

TABLE 5.1 Degradation of Selected Components at 635°F (ppm by weight)

Component	0 h ^a	45 h	168 h	338 h	504 h	649 h	840 h
Benzene	114.2	112.4	73.2	63.4	79.3	67.6	65.6
Ethylcyclohexane	331.5	326.4	327.0	332.8	321.6	325.4	328.5
Xylene	263.9	253.1	254.8	267.1	248.4	252.9	256.5
Diphenyl ether	0	23.5	4.1	BT ^b	3.9	4.0	3.5
Benzaldehyde	0	32.4	22.4	12.6	44.0	18.6	17.3
Bibenzyl	0	9.8	16.4	26.5	32.7	45.1	48.9
C2-biphenyls	0	0	0	0	0	0	0
Cl-fluorene	0	0	0	8.1	8.3	5.9	8.2
Total impurities	1493.9	1502.1	1465.5	1565.6	1494.8	1487.7	1445.4
Total degradation products	0	48.3	40.8	47.1	85.0	69.5	76.3

^aBefore loading the loop.

^bBelow threshold.

TABLE 5.2 Degradation of Selected Components at 600°F (ppm by weight)

Component	Flush	23 h	266 h	455 h	623 h	766 h	938 h	1006 h
Benzene	127	109	107	100 ^a	91	94	93	93
Ethylcyclohexane	235	236	242	244	247	245	245	245
Xylene	138	145	160	164	171	168	167	168
Benzaldehyde	0	10.2	7.2	9.3	11.8	12.5	23.3	22.0
Dowtherm® and Cl-biphenyl	5.9	7.2	7.2	7.7	7.8	8.3	8.6	8.2
Bibenzyl	0	7.1	21.6	32.8	40.2	50.4	66.3	63.6
C2-biphenyl	0	0	0	0	2.4	0		3.8
Cl-fluorene	0	0	0	4.4	5.1	6.0	5.7	7.5
Total impurities	1177	1177	1139	1164	1164	1176	1269	1269
Total degradation products	5.9 ^b	24.5	36.0	54.2	70.2	80.0	103.9	105.4

^aValue calculated from PE 7500.

^bDowtherm® contaminant.

TABLE 5.3 Degradation of Selected Components at 650°F (ppm by weight)

Component	Flush	19 h	115 h	282 h	450 h	619 h	786 h	955 h	1006 h
Benzene	97.3	108.5	103.3	97.7	96.4	88.9	89.4	104.0	91.5
Ethylcyclohexane	201.6	196.3	194.4	198.3	195.7	187.0	195.7	194.3	190.9
Xylene	269.3	253.2	247.6	263.1	259.2	243.8	258.2	253.5	257.2
Benzaldehyde	0	2.0	2.1	4.9	5.4	5.6	7.4	8.3	12.0
1-biphenyl	0	0	0	0	4.0	4.2	6.0	7.6	7.7
Bibenzyl	0	7.6	19.0	63.2	85.5	89.3	128.5	153.2	165.5
C2-biphenyls	0	0	0	0	10.0	11.0	16.6	19.6	25.4
C1-fluorene	0	0	0	0	4.9	11.6	16.9	19.9	4.9
Total impurities	740	1189	1170	1267	1279	1231	1355	1402	1272
Total degradation products	0.0	9.6	21.1	71.1	115.9	126.0	194.6	231.9	228.0

TABLE 5.4 Degradation of Selected Components at 677°F (ppm by weight)

Component	0 h	117 h	285 h	600 h	789 h	958 h	1006 h
Benzene	83.9	82.0	82.5	75.9	73.8	76.9	72.4
Ethylcyclohexane	472.1	472.6	469.6	472.2	468.0	466.1	468.2
Xylene	89.5	92.0	91.5	90.3	90.7	90.8	93.3
Benzaldehyde	0	2.1	2.6	4.1	4.6	5.1	10.6
Diphenylmethane	0	0	3.0	6.5	9.1	12.0	9.6
Bibenzyl	0	37.1	60.6	120.7	163.2	202.8	178.8
C2-biphenyls	0	2.4	8.6	18.4	27.3	34.8	29.2
Cl-fluorene	0	0	3.8	4.7	8.5	10.6	-
Total impurities	1452.5	1449.0	1459.7	1480.0	1647.5	1696.0	1587.0
Total degradation products	0	41.6	78.6	161.3	237.7	302.9	246.0

FIGURE 5.3 Benzaldehyde Concentrations

Total degradation is plotted in Fig. 5.4 as a function of time and temperature. As one would expect, the figure shows a steady increase of degradation products with time, and the rate of increase increases with temperature. Linear least-square fits of the data for each test temperature are also shown in the figure. The slope of each line is the total degradation rate for that particular temperature. The total degradation rates are 0.108, 0.0726, 0.248, and 0.306 ppm/h for test temperatures of 600, 635, 650, and 677°F, respectively. The total degradation is the sum of individual products, each of which can have different formation mechanisms and formation rates.

The major degradation product is bibenzyl, which is formed through the processes indicated in Eqs. 5.2 and 5.5. Bibenzyl concentration as a function of time and test temperature is plotted in Fig. 5.5. The figure shows a steady increase in bibenzyl concentration with time, and the rate of increase increases with test temperature.

The second most important degradation product is C2-biphenyl, which is formed through the processes indicated in Eqs. 5.4 and 5.13. C2-biphenyl concentration as a function of time and test temperature are shown in Fig. 5.6. As with the bibenzyl concentration, the concentration of C2-biphenyl increases with time, and the rate of increase increases with temperature. At the lower test temperatures, 600 and 635°F, very little C2-biphenyl was found.

Two different mechanisms are required for formation of bibenzyl and C2-biphenyl. To form bibenzyl the methyl-group carbon must be activated, but to form C2-biphenyl a ring carbon must be activated. Since the concentration of bibenzyl greatly

FIGURE 5.4 Total Degradation, with a Least-Square Fit of Data for Each Test

FIGURE 5.5 Bibenzyl Concentrations

FIGURE 5.6 C2-Biphenyl Concentrations

exceeds that of C2-biphenyl, formation of bibenzyl must have a lower activation energy than formation of C2-biphenyl. At higher temperatures, however, the activation energy required for formation of C2-biphenyl is overcome, and the C2-biphenyl might become more prevalent than bibenzyl. To check this hypothesis, we have plotted the ratios of C2-biphenyl concentration to bibenzyl concentration in Fig. 5.7. This figure shows that formation of C2-biphenyl does become more significant at higher temperatures.

5.4 VAPOR-PHASE ANALYSIS

5.4.1 Toluene and Degradation Products

Table 5.5 lists the vapor-phase concentrations of toluene degradation products in mole percent (mol %). Because the total pressure in the capsules was not measured, absolute amounts of vapor-phase degradation products cannot be calculated. However, the data can be used to provide relative concentrations that can be compared under the different conditions studied. The data are presented at the four primary temperatures studied -- 600, 635, 650, and 677°F -- in ascending order of loop residence time. The main degradation products are hydrogen, methane, and C2 and C3 alkanes and alkenes. Because the 635°F test was run at a different evaporator pressure than the other tests, its vapor-phase analysis will be described after those of the other tests.

FIGURE 5.7 Ratios of C2-Biphenyl Concentration to Bibenzyl Concentration

The concentration of toluene in the vapor phase is also listed in Table 5.5. The vapor-phase concentration of toluene depends on the total pressure in the sample. The relative toluene concentration is highest when the total pressure is lowest, which occurs before degradation begins. Once degradation products are formed, the total vapor pressure in the sample increases, dramatically lowering the relative percent toluene in the vapor phase. For most samples, the toluene vapor-phase concentration is highest in the 600°F sample and decreases at each successively higher temperature. Three exceptions were observed, at 623 h (600°F) and at 955 and 1006 h (650°F). These samples show toluene concentrations of 23-32%. These high values are probably due to some liquid toluene adhering to the Swagelok fitting on the capsule and then entering the mass spectrometer, where it was vaporized by the high source vacuum.

The hydrogen concentration is observed to increase rapidly at 600°F and then level off at 12-13 mol %. At 650°F, this leveling off is also observed, but at 28-29 mol %; the higher concentration is due to the greater degradation at the higher temperature. At 677°F, no corresponding increase in hydrogen concentration is observed. The first sample, taken at 117 h, shows almost 26 mol % hydrogen and levels off at the same relative value (28-29 mol %) observed at 650°F, although total degradation in the vapor phase is greater at the higher temperature. Above 30 mol %, apparently, the hydrogen is consumed in subsequent reactions with degradation products. Undoubtedly, the hydrogen would react with C2 and C3 ring fragments (HC-CH and HC=CH-CH) to form ethylene and propylene. These might react in another hydrogenation to form ethane and propane, respectively. All four of these degradation products are observed.

TABLE 5.5 Toluene Degradation in the Vapor Phase

Temp. (°F)	Loop Time (h)	Toluene and Degradation Products (mol %)				
		Hydrogen	Methane	C ₂ H ₄ , C ₂ H ₆ , C ₃ H ₆ , C ₃ H ₈	Total Gaseous Degradation	Toluene
600	23	2.6	0.8	4.1	7.5	19.3
	266	9.7	3.0	6.4	19.1	12.0
	455	13.7	3.4	4.7	21.8	14.7
	623	17.8	2.3	3.2	23.3	25.7
	766	12.6	1.2	1.7	15.5	11.7
	938	11.8	1.4	1.0	14.2	9.9
	1006	12.1	1.6	1.9	15.6	14.7
635	45	7.2	1.3	2.2	10.7	6.9
	168	15.7	3.9	6.7	26.3	7.9
	338	22.1	6.5	6.2	34.8	17.6
	504	22.1	8.5	7.1	37.7	7.3
	649	28.4	8.2	5.5	42.1	8.0
	840	NA ^a	NA	NA	NA	NA
650	19	2.0	0.3	0.4	2.7	4.0
	115	14.8	4.2	3.3	22.3	8.3
	282	25.9	5.5	5.3	36.7	7.2
	450	27.8	5.9	6.2	39.9	10.6
	619	29.8	6.3	5.3	41.4	8.2
	786	33.1	6.2	4.4	43.7	9.3
	955	21.5	3.5	7.8	32.8	32.4
	1006	29.6	4.2	4.8	38.6	23.0
677	117	25.8	11.7	8.0	45.5	4.9
	285	28.6	17.1	11.9	57.6	7.1
	452	28.2	19.9	11.4	59.5	5.4
	600	28.8	16.4	13.2	58.4	7.1
	789	24.9	15.9	10.0	50.8	7.5
	958	28.3	16.9	10.9	56.1	8.0

^aNA = not analyzed.

The relative methane concentration is also constant at each degradation temperature. At 600°F, the relative concentration is 1-3 mol %. At 650°F, the concentration increases to 5-6 mol %, which further increases to 17-19 mol % at 677°F. Methane is formed when a methyl radical reacts with a hydrogen atom. Unlike the hydrogen concentration, the relative methane concentration at 677°F is greater than that at 650°F. This is expected, because methane is much less reactive than hydrogen and does not undergo the addition and reduction reactions that hydrogen does.

The C2 and C3 alkanes and alkenes -- ethane, ethylene, propane, and propylene -- would be formed by cleavage of the aromatic ring. The fact they are present at low concentration at 600°F proves that there are small amounts of ring cleavage even at this low temperature. They are also present at a level relative concentration at each temperature. As is the case for methane, at each temperature they are at a higher relative concentration level. At 600°F, their relative concentration is greater than the methane concentration, but the reverse is true at 677°F.

The ratio of the relative concentration of methane to ethanes and propanes is important. When the ring cleaves, two C3 or three C2 fragments can be formed in addition to methane. Alternatively, if no methane is formed, ring cleavage will produce two C2 and one C3 ring fragments. Both fragmentation patterns would be taking place; in both cases, however, the concentration of C2 and C3 products would be greater than that of methane, as is the case at 600°F. Hence, the greater relative methane concentration at 677°F must be due to the formation of methane by an additional mechanism, most probably by cleavage of the bond between the ring carbon and the methyl carbon.

The "total gaseous degradation" column in Table 5.5 sums the concentrations of hydrogen, methane, and C2 and C3 alkanes and alkenes. In each case this total relative concentration is greater at greater temperature. Degradation products consist of just under 60 mol % of the vapor phase at a loop temperature of 677°F.

Because degradation data at 635°F were obtained with a different evaporator pressure than in the other tests, these data are not directly comparable with the other data presented. However, the 635°F data show strong similarities to the data obtained at 650°F. About 40 mol % total degradation products occur in the vapor phase. The major degradation product is hydrogen, with the leveling off taking place at 22-28 mol % (a little lower than at 650°F). One major difference is that more than 8% methane was formed; at 650°F, relative methane concentration was 5-6 mol %. C2 and C3 alkane and alkene concentration was identical at both temperatures. It appears that reduced pressure slightly favors methane production over hydrogen. The data are presented in Table 5.5.

5.4.2 Other Vapor-Phase Components

The remaining 40 mol % of the vapor phase consists of carbon dioxide, nitrogen, carbon monoxide, oxygen, helium, and argon. Data on the relative concentrations of these vapor-phase components are presented in Table 5.6. Small quantities of argon are

TABLE 5.6 Relative Concentration of Background and Oxygen-Containing Components in the Vapor Phase

Temp. (°F)	Loop Time (h)	Components (mol %)					
		Carbon Dioxide	Nitrogen and Carbon Monoxide	Oxygen	Water	Helium	Argon
600	23	2.8	9.2	0.10	0.1	60.9	0.04
	266	5.7	23.0	0.05	0.1	39.9	0.17
	455	5.0	37.3	1.30	0.1	19.5	0.40
	623	3.1	30.4	0.40	0.1	16.7	0.40
	766	1.9	54.6	8.60	0.0	7.0	0.70
	938	4.0	65.6	1.20	0.0	4.2	0.80
	1006	3.1	58.2	5.00	0.1	2.7	0.60
635	45	5.7	71.2	1.20	0.2	0	4.20
	168	7.9	54.6	0.60	0.2	0	2.50
	338	7.6	38.1	0.60	0.1	0	1.50
	504	9.4	44.2	0.40	0.1	0	0.90
	649	8.0	41.2	0.80	0.2	0	0.40
	840	NA ^a	NA	NA	NA	NA	NA
650	19	2.4	6.9	0.05	0.0	83.9	0.05
	115	9.7	19.4	0.05	0.1	40.1	0.07
	282	9.0	29.9	0.10	0.0	16.7	0.18
	450	8.4	33.9	0.10	0.0	7.4	0.20
	619	7.0	38.9	0.20	0.0	4.0	0.30
	786	6.2	38.4	0.10	0.1	1.8	0.40
	955	4.2	27.2	2.00	0.3	0.7	0.40
	1006	3.4	33.3	0.60	0.1	0.8	0.30
677	117	8.4	19.1	0.01	0.1	21.9	0.06
	285	9.7	21.3	0.01	0.0	4.2	0.10
	452	9.5	23.1	0.05	0.0	2.4	0.10
	600	8.5	23.4	0.30	0.1	2.1	0.17
	789	7.4	26.6	0.50	0.1	7.0	0.12
	958	7.7	26.2	0.40	0.1	1.3	0.19

^aNA = not analyzed.

present because it is used to purge the system of air. Helium is used to check the loop for leaks, and some residual amount would be expected to remain in the loop after pump-down. The mole percent of helium is high in low-hour samples because the total pressure is low, and the low concentration of helium divided by its low molecular weight results in a high mole percent. As the pressure increases (due to the formation of degradation products), the relative concentration of helium decreases, although the mass of helium present in the loop is constant.

Because of the reactivity of oxygen with toluene at elevated temperatures, the concentration of oxygen in the vapor-phase sample should be very low, less than 1 mol %. This is the case for all but four samples. The presence of oxygen at greater than 1 mol % relative concentration in these samples indicates that the capsule leaked air after removal from the loop and before analysis.

Because the mass spectrometer cannot differentiate the slight difference in mass between molecular nitrogen and carbon monoxide, the concentrations of these components are combined in a single column. This complicates detection of air leakage into the loop, because the presence of nitrogen indicates air incursion into the loop, whereas carbon monoxide and carbon dioxide can be formed from oxygen-containing impurities in the toluene (especially ethers). If an air incursion into the loop occurred, the reaction of oxygen with toluene would produce oxidation products, such as benzaldehyde (which is observed), that could in turn degrade to form carbon monoxide and carbon dioxide.

The low concentration of benzaldehyde in the liquid phase implies that there must be some air incursion. However, the low concentration also indicates that the incursion is slight. Undoubtedly, some of the carbon monoxide and carbon dioxide can be attributed to this source. Some of the CO and CO₂ is also due to the oxygen-containing impurities that were present in the initial charge of toluene. The concentration of carbon dioxide is only 7-9 mol % of the total in most samples. Nitrogen and carbon monoxide concentrations combined are quite high, amounting to 20-35 mol % relative concentration in most samples. If an air incursion into the hydrogen-rich atmosphere of the loop occurs, water should be formed. In all cases, the concentration of water is exceptionally low (0.1 mol %), so air leakage into the loop is insignificant.

The relative concentration of background and oxygen-containing components for the 635°F test is similar to that of the other samples. Carbon dioxide concentration was 8-9 mol %. Oxygen concentration was higher by 25%. This extra oxygen probably resulted from air incursion, because the relative concentration of nitrogen and carbon monoxide was higher than that in all the other samples that had no vapor sample-capsule leak. Indeed, some of the packed-stem valves in the low-pressure areas of the loop were replaced with bellows-seal valves after the 635°F test, because a small air leak was suspected. The water concentration, though low, was double that of the other samples. From the data, it appears that the air incursion was at a low level. This is supported by slightly higher values for benzaldehyde concentration in the liquid phase. Data are presented in Table 5.6.

5.5 RELATION OF THE ARRHENIUS EQUATION TO DEGRADATION

The Arrhenius equation (Eq. 5.17) relates the reaction rate R for a chemical reaction to absolute temperature through the constants R_0 and K . Two problems arise in relating the Arrhenius equation to ORC system degradation: (1) The total degradation in the system is the sum of components from many different chemical reactions, and (2) the temperature in the system varies from point to point.

$$R = R_0 e^{-K/T_{abs}} \quad (5.17)$$

To be strictly correct, the Arrhenius equation can be applied only for individual chemical reactions, and many reactions are involved in toluene degradation. In the previous section we showed that formation of bibenzyl is by far the dominant reaction for the conditions of our tests. Therefore, we approximate the total degradation rate by a single Arrhenius equation with unknown constants R_0 and K . The object of these experiments is to determine the two constants and to relate the Arrhenius equation to degradation in an ORC system. The result will be valid if formation of bibenzyl is the dominant reaction. If higher operating temperatures -- where other reactions such as formation of C2-biphenyls become important -- are required, then it will be necessary to perform additional research, calculate a second set of constants, and sum the degradations from the two important reactions.

The fluid-dynamic continuity equation (Eq. 5.18) is used to determine a relationship between residence time and position in the system. Figure 5.8 shows a control volume having a mass flow $\dot{m}$ passing through it with cross-sectional area $A(x)$, fluid temperature $T(x)$, fluid pressure $p(x)$, and fluid density $\rho(p, T)$ at location x in the system. At x , the fluid velocity is $u(x)$.

$$\dot{m} = \rho(p, T)A(x)u(x) \quad (5.18)$$

The fact that $u(x) = dx/dt$ gives a relationship between time and position in the system:

$$dt = \frac{\rho(p, T)A(x)}{\dot{m}} dx \quad (5.19)$$

Equation 5.19 also imposes the mathematical constraint that the mass flow rate not be zero.

The total degradation is the integral of the rate of degradation with respect to time; using Eq. 5.19 in the integral gives the degradation for one circuit around the system D_1 , where L is the length of the circuit:

$$D_1 = \frac{R_0}{\dot{m}} \int_0^L e^{-K/T_{abs}} \rho(p, T)A(x) dx \quad (5.20)$$

The duration of system operation t_D and the total mass m of the fluid are known. Therefore, the number of circuits n made by the fluid during system operation is given by Eq. 5.21:

$$n = \frac{\dot{m} t_D}{m} \quad (5.21)$$

Finally, since the total degradation D_T is equal to nD_1 , one obtains the following expression:

$$D_T = \frac{R_o t_D}{m} \int_0^L e^{-K/T_{abs}} \rho(p, T) A(x) dx \quad (5.22)$$

The integral of Eq. 5.22 was evaluated from arbitrary location x_1 to x_2 in the system, assuming linear changes of temperature and density and constant cross-sectional area. The resulting degradation from point 1 to point 2 is

$$D_{1,2} = \frac{t_o R_o A}{m} \left(\frac{x_2 - x_1}{T_2 - T_1} \right) \left\{ \left[\rho_1 - \left(\frac{\rho_2 - \rho_1}{T_2 - T_1} \right) \left(T_1 + \frac{K}{2} \right) \right] \left[T_2 e^{-\frac{K}{T_2}} - T_1 e^{-\frac{K}{T_1}} \right] \right. \\ \left. - K \left(E_1 \left(\frac{K}{T_2} \right) - E_1 \left(\frac{K}{T_1} \right) \right) \right] + \frac{1}{2} \left(\frac{\rho_2 - \rho_1}{T_2 - T_1} \right) \left[T_2^2 e^{-\frac{K}{T_2}} - T_1^2 e^{-\frac{K}{T_1}} \right] \right\} \quad (5.23)$$

where $E_1(x)$ is defined and tabulated in Sec. 5 of *Handbook of Mathematical Functions*.¹⁴ Although the mass flow rate was used to derive Eq. 5.23, it does not appear in the equation; nevertheless, it cannot be zero. Physically, this makes sense, because a fraction of the fluid in the system occupies each small section of the system and degrades at a rate prescribed by the temperature of that section. It does not matter whether the flow rate is low or high, because the same fraction of fluid occupies that particular section regardless of the flow rate. However, if the flow rate were zero, the fluid in the hottest parts of the system would be totally degraded, while the fluid in the coolest parts of the system would have little degradation. Equation 5.23 is written as the short BASIC program shown in App. E.

5.6 DETERMINATION OF COEFFICIENTS FROM EXPERIMENTAL DATA

Coefficients of the Arrhenius equation are determined by adjusting the coefficients R_o and K to minimize the sum of the squares of the differences between measured and predicted degradations (i.e., the least-square error). A modified version of the computer program shown in App. E is used for the calculations. Data from the four

FIGURE 5.8 Definitions Used in Continuity Equation

tests performed at Argonne were fitted in this manner and used to predict the degradation for a test at 750°F performed by Sundstrand Corp. for the National Aeronautics and Space Administration (NASA).¹⁵

Both the computer program shown in App. E and the program that fits R_0 and K to the data require temperature and density data. The temperatures are determined from thermocouples located at various positions in the evaporator and other hot parts of the loop. These measured temperatures are plotted as a function of location in the loop in Figs. 5.9-5.13; the zero datum is at the inlet of the evaporator. Fluid densities are determined from the temperatures and the state (liquid or vapor) from tabulated thermodynamic-property data. The least-square fit gives values of $R_0 = 9.9917 \times 10^9$ ppm/h and $K = 23,056^\circ\text{R}$.

The predicted and measured degradations for the tests are compared in Table 5.7. Test run no. 1 (635°F) shows a 23% error, while the other Argonne tests show 6-7% errors. There are two reasons why the error for test run no. 1 should be larger than the error for the other Argonne tests:

1. Because of a problem with the data logger during test run no. 1, the measured temperatures are not as accurate as they are for the other tests.
2. Test run no. 1 was operated at a substantially different evaporator pressure than the other tests (160 psia vs. 500 psia).

FIGURE 5.9 Temperature versus Position for Test No. 1 (635°F)

FIGURE 5.10 Temperature versus Position for Test No. 2 (600°F)

FIGURE 5.11 Temperature versus Position for Test No. 3 (650°F)

FIGURE 5.12 Temperature versus Position for Test No. 4 (677°F)

FIGURE 5.13 Temperature versus Position for Sundstrand/NASA Test (750°F)

TABLE 5.7 Comparison of Predicted and Measured Degradation for Single-Reaction Model

Test	Nominal Temp. (°F)	Predicted Degradation (ppm)	Measured Degradation (ppm)	Error (%)
1	635	58.90	76.3	-23
2	600	111.82	105.4	+6
3	650	210.97	228.0	-7
4	677	260.94	246.0	+6
Sundstrand/ NASA	750	641.17	1212.9	-47

The Sundstrand/NASA test shows a much larger error of 47%. The main reason for this error is that a second chemical reaction leading to the formation of C2-biphenyl (Eqs. 5.4 and 5.13) is as important as the reaction leading to the formation of bibenzyl (Eqs. 5.2 and 5.5) on which the computation of the coefficients is based. Other possible reasons for the larger error include the following:

1. The Sundstrand/NASA test was operated at supercritical pressure, whereas the Argonne tests were operated at subcritical pressures.
2. The temperatures in the Sundstrand/NASA loop were not measured directly; instead, they were inferred from temperatures measured on the outside of the evaporator tube.

To determine whether the two-reaction hypothesis is correct, we determined the degradation due to methyl-group activation (Eq. 5.2) and ring activation (Eq. 5.4) for each of the tests, including the Sundstrand/NASA test, and determined the best fits of R_0 and K for each reaction separately. For the methyl-group activation reaction, we obtained $R_0 = 3.6442 \times 10^{11}$ ppm/h and $K = 27,163^\circ\text{R}$; for the ring activation reaction, we obtained $R_0 = 3.9485 \times 10^{30}$ ppm/h and $K = 79,215^\circ\text{R}$. Predictions based on these values are listed, together with measured values, in Table 5.8.

TABLE 5.8 Predicted and Measured Degradation for Two-Reaction Model

Test	Methyl-Group Activation			Ring Activation			Totals		
	Measured (ppm)	Predicted (ppm)	Error (%)	Measured (ppm)	Predicted (ppm)	Error (%)	Measured (ppm)	Predicted (ppm)	Error (%)
1	76.3	57.5	-25	0	8.4	-	76.3	65.9	-14
2	93.4	79.7	-15	12.0	0.3	-98	105.4	80.0	-24
3	194.9	186.9	-4	33.1	10.6	-68	228.0	197.5	-13
4	216.8	247.5	+14	29.2	37.2	+27	246.0	284.7	+16
Sund-strand/ NASA	677.4	668.5	-1	538.5	538.3	<1	1212.9	1206.8	<1

6 CONCLUSIONS AND RECOMMENDATIONS

The following important conclusions can be drawn from the results presented in this report:

1. A method of predicting degradation in ORC systems using toluene as the working fluid has been developed.
2. Air must be strictly excluded from any ORC system using toluene as a working fluid.
3. Provided that the toluene does not come in contact with oxygen, it is a very stable fluid at high temperatures.
4. Degradation products have been identified, and none is expected to cause serious problems in an ORC system.
5. Some impurities in toluene showed very little degradation in these experiments and could be potential ORC working fluids themselves.

The computer program shown in App. E can be used to predict degradation in ORC systems. With the constants $R_0 = 9.9917 \times 10^9$ ppm/h and $K = 23,056^\circ\text{R}$ in line 50010, the program gives good accuracy up to about 680°F , where a single chemical reaction resulting in formation of bibenzyl is dominant.* The input data required by the program are simple, and the program requires only about five seconds of running time on a personal computer. By performing repeated runs of the computer program for each major chemical reaction with the constants R_0 and K adjusted accordingly, then summing the degradations attributable to each chemical reaction, the temperature range of the program can be extended. Using $R_0 = 3.6442 \times 10^{11}$ ppm/h and $K = 27,163^\circ\text{R}$ for the methyl-group activation reaction (Eq. 5.2) and $R_0 = 3.9485 \times 10^{30}$ ppm/h and $K = 79,215^\circ\text{R}$ for the ring-carbon activation reaction (Eq. 5.4), the temperature range of the program can be extended at least to 750°F . Measured and predicted degradations for this two-activation-equation model are shown in Table 5.8.

Oxygen reacts almost immediately with toluene to form benzaldehyde in an ORC system. The sources of oxygen for the reaction are (1) air leakage into the system, (2) residual air not completely purged from the system before the toluene is loaded, (3) dissolved air in the toluene, and (4) oxygen-bearing impurities in the toluene. Therefore, it is imperative to design the ORC system so air cannot enter during operation or shutdown. We also recommend degassing the toluene before an ORC system is operated at temperature. This might be accomplished in the field by evacuating the system after loading the toluene or after a long period of shutdown and allowing some of the toluene to boil off along with the dissolved air.

*Because the computer program cannot include subscript symbols, the constant denoted by " R_0 " in the text is represented by "R0" in the program.

Toluene appears to be stable to at least 750°F, according to results from both the Sundstrand/NASA experiment and our own. If Sundstrand's result is extrapolated, the total degradation after one year of continuous operation would be less than 0.5% in their loop. (The actual degradation rate would decrease if the fluid inventory were increased.) If there were no temperature excursions, an ORC system could operate for several years at 750°F before a fluid change would become necessary.

In the absence of oxygen, no degradation products that would threaten the integrity of the system, such as strong acids, are formed. If there is more oxygen present than was the case in our tests, there is a potential for oxidizing the benzaldehyde into benzoic acid, but this is a relatively mild acid. The major liquid-phase degradation products are bibenzyl and, at higher temperatures, C2-biphenyl and C1-fluorene. Because these compounds are less volatile than toluene, they will have a tendency to collect in the evaporator and foul it. We recommend that evaporator fouling be investigated in the future.

Noncondensable degradation products are mainly hydrogen and methane, although some light alkanes and alkenes were found. All of these gases tend to accumulate in the condenser and blanket it, thus reducing its effective heat-transfer coefficient. In our tests, we observed that the coolant flow to the condenser had to be increased gradually during the experiment as noncondensable products accumulated. We recommend that ORC systems include a cold trap in their design to periodically or continuously remove noncondensable gases.

Some of the impurities in toluene, such as cyclopentanes and cyclohexanes, appear to be very stable themselves. Cyclohexane -- with a molecular weight of 84.16, a boiling point of 178.5°F, a critical temperature of 538°F, and a critical pressure of 59.6 psia -- might be useful for supercritical cycles. We recommend investigation of cyclopentanes and cyclohexanes (with or without added methyl or ethyl groups) as potential ORC fluids.

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APPENDIX A:

**DAILY AVERAGES, MINIMA, AND MAXIMA OF EVAPORATOR TEMPERATURE,
EVAPORATOR PRESSURE, AND CONDENSER PRESSURE**

APPENDIX A:**DAILY AVERAGES, MINIMA, AND MAXIMA OF EVAPORATOR TEMPERATURE,
EVAPORATOR PRESSURE, AND CONDENSER PRESSURE**

The data provided in this appendix are 24-hour averages, minima, and maxima of the evaporator temperature, evaporator pressure, and condenser pressure compiled from data taken each half hour during the test runs. The purpose of the data provided herein is to verify the accuracy with which the loop parameters were maintained. Since the evaporator temperature and pressure affect the temperature distribution in the hot parts of the loop, they are the most important indicators of how well the loop is controlled. Condenser pressure is included because, if it becomes subatmospheric, air could leak into the loop. Any oxygen in the loop reacts immediately with toluene to form benzaldehyde, a degradation product.

TABLE A.1 Daily Average, Minimum, and Maximum Pressures and Temperatures for Test Run No. 1 (635°F)

Interval (h)	Temperature (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
	Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
0-24	636.1	631.4	639.8	160.06	157.50	161.60	17.56	17.08	17.79
24-48	637.5	635.0	638.6	160.26	158.90	161.00	17.73	17.34	18.03
48-72	631.4	627.7	638.6	159.94	158.60	161.00	16.42	15.11	17.68
72-96	630.3	626.3	633.6	158.90	157.50	161.10	17.20	16.68	17.68
96-120	630.0	625.5	635.0	160.20	158.80	161.60	17.36	17.19	17.56
120-144	627.5	625.5	629.7	161.70	160.10	163.40	17.50	17.30	17.65
144-168	627.5	625.2	633.9	161.80	159.70	163.30	17.61	17.34	18.09
168-192	633.9	631.7	637.2	159.20	156.70	161.00	17.42	17.14	18.01
192-216	634.8	632.6	636.7	159.54	158.50	160.40	17.49	17.26	17.71
216-240	636.1	633.3	652.3	159.59	158.90	160.40	17.43	17.26	17.61
240-264	635.8	633.1	637.6	159.35	157.80	160.30	17.38	17.10	17.66
264-288	636.4	634.5	637.9	160.36	158.50	161.70	17.58	17.19	17.90
288-312	637.5	636.1	639.5	161.37	160.40	162.10	17.84	17.65	18.03
312-336	637.2	633.3	639.6	161.23	160.10	162.60	17.86	17.54	18.09
336-360	635.8	634.2	637.9	158.75	157.40	161.50	17.27	16.87	18.07
360-384	635.9	633.7	638.1	159.10	157.80	160.50	17.83	17.68	18.26
384-408	637.3	635.4	640.0	159.11	157.90	160.10	17.79	17.27	18.39
408-432	638.6	636.8	640.7	158.89	158.20	160.10	17.37	17.14	17.69
432-456	635.4	632.8	639.2	155.54	149.70	159.30	17.24	17.04	17.60
456-480	636.7	635.6	637.9	151.14	150.00	152.50	17.34	17.05	17.59
480-482 ^a	637.3	636.8	637.8	151.50	150.90	151.90	17.69	17.61	17.77
482-484 ^b	631.7	627.4	635.4	158.03	154.60	159.90	17.92	16.23	19.09
484-507	636.8	630.0	639.8	160.16	157.30	163.10	17.50	16.59	18.19
507-531	637.0	633.7	638.9	158.51	157.20	159.50	17.27	16.58	17.97
531-555	633.7	631.7	635.8	158.12	156.90	159.30	17.57	17.38	17.86
555-579	634.7	633.6	636.8	157.92	156.70	158.80	19.01	17.54	22.48
579-603 ^c	635.0	633.4	636.8	158.32	157.40	159.30	17.81	17.49	18.78
603-627	638.4	636.4	640.0	158.24	157.20	159.50	17.36	16.60	18.33
627-651 ^d	637.9	636.1	640.0	158.90	157.70	159.80	17.06	16.74	17.98
651-675	637.3	635.1	639.5	158.38	157.40	160.30	17.51	16.52	17.80
675-699	636.8	634.0	640.4	158.17	157.30	159.40	17.55	17.33	17.85
699-723	635.3	633.4	638.2	157.97	157.00	158.80	17.47	17.17	17.83
723-747	637.9	636.1	651.6	157.85	156.70	158.70	17.73	17.31	18.27
747-771	636.5	635.4	637.9	157.58	155.70	158.90	16.76	16.40	18.46
771-795	635.9	633.7	637.8	157.70	156.80	159.20	16.52	16.36	16.71
795-819	634.8	633.3	636.8	158.60	156.60	159.60	16.45	16.31	16.73
819-840	635.6	633.9	636.8	159.52	157.50	160.60	16.61	15.99	16.79
Overall	635.3	625.2	652.3	158.89	149.70	163.40	17.42	15.11	22.48

^aLoop down for 19 h at the end of this time period.^bLoop down for 2 h at the end of this time period.^c1.5 h of data unavailable in this time period.^d5.5 h of data unavailable in this time period.

TABLE A.2 Daily Average, Minimum, and Maximum Pressures and Temperatures for Test Run No. 2 (600°F)

Interval (h)	Temperature (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
	Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
0-24	597.4	595.2	603.5	489.93	483.70	508.10	17.35	10.94	18.66
24-48	598.7	597.7	599.4	495.59	489.70	502.90	16.83	14.04	17.16
48-72	599.0	598.4	599.6	498.77	492.30	503.20	17.31	17.04	17.57
72-96 ^a	598.8	598.5	599.1	499.19	490.90	502.60	17.48	17.28	17.80
96-120	598.8	598.3	599.3	490.79	486.80	493.70	17.65	17.50	17.78
120-144	598.7	598.0	599.1	491.67	488.00	496.20	17.48	17.26	17.72
144-168	598.8	598.1	599.3	491.95	486.20	497.20	17.37	17.19	17.57
168-192	598.8	598.1	601.2	493.66	490.40	499.00	17.26	17.06	17.34
192-216	598.7	598.2	599.1	492.76	487.20	497.70	16.98	16.75	17.14
216-240	598.5	598.0	599.1	492.47	487.40	497.20	16.83	16.70	16.92
240-264	598.4	597.9	599.1	492.65	488.90	498.10	16.79	16.73	16.86
264-288	599.8	599.1	600.5	495.97	488.00	501.80	17.04	15.02	17.52
288-312	599.4	598.7	600.3	492.98	488.00	509.80	17.16	16.78	17.43
312-336	599.8	599.2	600.2	495.36	489.10	500.80	17.31	17.20	17.40
336-360	599.6	599.1	600.3	495.82	492.30	500.10	17.27	17.10	17.37
360-384	599.7	599.1	600.2	494.91	490.80	500.30	17.16	17.04	17.26
384-408	599.7	599.1	600.3	494.74	490.70	500.70	17.15	17.00	17.26
408-432	599.4	598.9	600.2	488.61	450.80	499.90	17.14	17.02	17.26
432-456	600.2	599.5	600.9	499.92	490.40	505.60	16.81	15.60	17.19
456-480	600.5	600.1	601.1	493.83	488.00	500.00	17.41	14.75	18.42
480-504	600.3	599.5	601.0	493.76	489.50	498.50	17.50	17.22	18.16
504-528	600.5	599.9	600.9	494.55	489.70	498.20	18.05	17.93	18.21
528-552	600.2	599.6	600.7	491.32	486.60	496.50	18.03	17.81	18.20
552-576	600.2	599.4	600.8	492.84	488.40	497.40	17.91	17.72	18.09
576-600	599.5	599.1	599.9	491.01	486.90	495.30	17.59	17.34	17.72
600-624	600.1	599.5	601.0	495.02	487.30	505.70	17.51	15.35	17.77
624-648	600.0	599.4	600.5	491.21	484.60	498.60	17.00	16.07	17.84
648-672	600.4	600.1	601.0	490.80	482.40	498.10	18.12	17.62	18.58
672-696	600.3	599.6	600.9	491.51	486.10	497.10	17.60	17.23	18.68
696-720	600.2	599.7	600.8	490.48	486.80	495.40	17.51	17.35	17.63
720-744	600.1	599.5	600.8	492.58	487.70	498.60	17.52	17.37	17.64
744-768	600.2	599.7	600.8	492.42	489.10	498.00	17.29	17.11	17.73
768-792	600.6	599.9	601.2	493.56	488.10	499.70	17.10	15.30	17.39
792-816	600.3	599.7	600.8	491.38	487.30	498.00	17.34	17.22	17.46
816-840	600.4	600.0	600.8	495.42	490.20	502.60	17.42	17.25	17.56
840-864	600.8	600.4	601.4	499.34	494.00	503.70	17.38	17.15	17.57
864-888	600.5	599.8	601.2	499.08	494.10	503.60	16.94	16.73	17.12
888-912	600.1	599.3	600.9	499.24	495.10	503.80	16.70	16.45	16.84
912-936	600.1	599.7	600.5	500.17	488.20	507.00	16.56	16.31	17.13
936-960	600.0	599.4	600.6	490.30	470.50	501.70	16.63	15.82	17.09
960-984	600.0	599.5	600.6	493.10	486.70	497.70	16.77	16.67	16.92
984-1006	600.2	599.5	600.7	494.81	489.50	499.50	16.97	16.86	17.13
Overall	599.7	595.2	603.5	493.89	450.80	509.80	17.26	10.94	18.68

^a10 h of data unavailable during this time period.

TABLE A.3 Daily Average, Minimum and Maximum Pressures and Temperatures for Test Run No. 3 (650°F)

Interval (h)	Temperature (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
	Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
0-24	647.5	646.1	650.1	495.50	482.80	505.80	17.95	15.90	18.74
24-48	649.7	649.1	650.3	497.21	489.60	504.00	17.07	16.56	17.27
48-72	649.7	649.1	650.5	501.71	494.40	507.80	17.16	16.96	17.37
72-96 ^a	649.1	648.6	649.7	505.06	499.60	515.00	17.15	16.91	17.39
96-120 ^a	648.6	648.0	649.9	505.71	488.30	515.80	17.28	16.12	17.89
120-144	650.0	649.3	650.7	494.18	488.10	501.80	18.21	17.50	18.63
144-168	649.8	649.2	650.6	491.23	485.30	496.70	17.55	17.31	17.92
168-192	649.8	649.0	650.5	492.91	488.80	498.50	17.91	17.62	18.12
192-216	649.8	649.1	650.5	494.85	489.90	501.40	17.90	17.52	18.10
216-240	649.1	648.1	649.8	501.51	496.60	510.40	17.99	17.62	18.18
240-264	648.9	648.5	649.3	505.78	500.80	511.00	18.04	17.86	18.22
264-288 ^a	649.1	648.1	651.0	505.40	486.40	516.60	17.75	15.79	18.29
288-312	650.6	649.7	651.3	494.78	490.90	499.90	18.14	17.73	18.58
312-336	650.1	649.5	650.6	498.06	492.90	503.50	18.16	17.69	18.54
336-360	649.8	649.3	650.7	499.72	494.20	506.80	17.78	17.63	17.90
360-384	649.8	649.3	650.3	499.32	489.70	506.60	17.90	17.66	18.19
384-408	649.0	648.5	650.0	497.76	494.10	502.20	18.14	17.97	18.41
408-432	649.2	648.7	649.6	498.92	491.40	505.30	18.15	17.91	18.32
432-456	649.0	648.3	650.0	498.45	484.00	504.80	17.89	15.71	18.45
456-480	650.2	649.5	650.8	494.26	489.10	500.20	18.15	17.79	18.38
480-504	650.4	649.8	651.2	494.90	486.70	502.30	18.10	17.84	18.37
504-528	650.7	649.9	652.0	497.39	488.40	502.90	17.83	16.96	18.31
528-552	650.7	649.0	651.9	495.86	489.40	504.90	17.30	17.07	17.53
552-576	649.1	648.6	649.6	500.68	495.40	507.50	17.56	17.35	17.75
576-600	649.0	648.5	649.4	501.43	495.20	506.30	17.70	17.54	17.88
600-624	649.1	648.5	650.5	499.92	486.00	506.90	17.56	16.02	17.96
624-648	650.1	649.5	650.9	497.20	493.20	502.50	18.39	17.71	18.70
648-672	650.1	649.5	650.7	496.71	491.50	501.80	18.24	17.49	18.66
672-696	650.0	649.4	650.8	495.87	490.80	500.80	17.76	17.35	17.97
696-720	650.1	649.5	650.6	496.77	491.60	502.40	17.74	17.53	17.97
720-744	650.1	649.5	650.7	496.80	493.70	502.20	17.66	17.46	17.93
744-768	649.5	648.5	650.6	503.25	494.20	509.40	17.79	17.61	17.94
768-792	649.4	648.0	649.9	502.23	486.80	512.60	17.72	15.51	18.23
792-816	650.3	649.4	650.8	496.36	489.20	501.00	18.49	17.64	18.81
816-840	650.2	649.6	650.8	493.61	485.00	500.10	18.48	17.24	19.01
840-864	650.2	649.5	650.8	497.19	489.90	501.50	17.59	17.27	17.78
864-888	650.5	649.8	650.9	494.67	489.80	500.70	17.51	17.24	17.77
888-912	650.3	649.6	651.2	492.26	486.20	497.70	17.34	17.09	17.52
912-936	650.0	649.0	650.9	498.12	491.00	505.40	17.44	17.21	17.65
936-960 ^a	649.5	648.8	650.4	498.90	489.30	506.60	17.37	14.73	17.93
960-984	650.2	649.6	650.7	499.07	488.40	505.70	18.16	17.44	19.01
984-1006	649.1	635.8	650.3	491.98	485.50	501.80	18.55	18.27	18.75
Overall	649.7	635.8	652.0	497.94	482.80	516.60	17.82	14.73	19.01

^a0.5 h of data unavailable in this time period.

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TABLE A.4 Daily Average, Minimum, and Maximum Pressures and Temperatures for Test Run No. 4 (677°F)

Interval (h)	Temperature (°F)			Evaporator Pressure (psia)			Condenser Pressure (psia)		
	Avg.	Min.	Max.	Avg.	Min.	Max.	Avg.	Min.	Max.
0-24	676.3	674.0	677.3	495.87	481.00	501.90	17.67	17.37	17.96
24-48	676.3	675.2	677.8	494.62	490.90	497.30	17.51	17.21	17.73
48-72	676.5	675.4	677.2	496.57	493.50	501.80	17.56	17.30	17.86
72-96	676.6	675.3	678.1	507.86	494.60	516.70	18.18	17.87	18.54
96-120	677.4	676.3	678.7	498.76	489.70	502.60	17.75	16.35	18.17
120-144	677.6	676.6	678.7	497.39	487.20	502.30	17.07	16.61	17.36
144-168	677.9	677.0	678.7	498.02	493.40	501.80	17.64	17.28	17.99
168-192	677.7	676.6	678.6	499.26	491.10	503.70	17.90	17.60	18.18
192-216	677.6	676.6	678.3	495.13	489.70	498.80	17.61	17.44	17.81
216-240	678.4	677.5	679.2	506.83	497.00	515.10	17.50	17.16	17.80
240-264	678.1	677.3	679.2	511.23	498.70	517.30	17.72	17.36	17.96
264-288	678.3	677.3	679.1	500.24	495.10	506.20	17.77	17.59	17.93
288-312	678.1	677.5	679.2	500.49	497.30	505.30	17.59	16.55	17.98
312-336	678.2	677.0	679.2	500.02	496.20	504.60	17.71	17.30	18.17
336-360	678.0	677.3	678.7	502.04	498.80	505.70	18.08	17.92	18.29
360-384	678.2	677.3	679.1	502.42	498.20	506.70	18.03	17.88	18.24
384-408	677.9	677.0	678.6	505.23	501.50	509.50	18.14	18.00	18.37
408-432	678.1	677.4	678.9	507.01	503.30	511.40	17.99	17.83	18.14
432-456	677.9	676.2	679.1	504.63	494.90	513.10	18.22	17.49	18.76
456-480	676.9	676.0	678.1	502.35	496.60	508.60	18.27	17.77	18.53
480-504	677.3	676.3	678.2	500.71	497.60	506.30	17.68	17.54	17.91
504-528	677.3	676.2	678.4	503.15	495.50	510.70	17.72	17.30	18.07
528-552	676.9	675.9	678.0	497.45	493.80	502.30	18.05	17.63	18.42
552-576	677.1	675.9	678.0	497.48	494.20	501.20	17.90	17.52	18.24
576-600	677.0	676.1	678.1	498.91	494.80	503.50	18.06	17.45	18.41
600-624	678.3	677.0	679.1	497.01	494.10	501.40	18.74	16.69	19.49
624-648	677.1	676.4	678.0	499.73	495.20	504.40	17.89	17.69	18.03
648-672	677.8	676.8	678.7	503.87	500.40	507.00	17.62	17.34	17.80
672-696	678.3	677.0	679.0	502.91	497.20	506.80	17.76	17.41	18.07
696-720	677.6	676.4	678.6	500.89	498.20	507.90	17.99	17.77	18.17
720-744	677.7	676.6	678.6	510.35	504.70	513.20	17.98	17.76	18.36
744-768	677.4	675.9	678.8	508.38	494.80	515.30	18.03	17.71	18.55
768-792	677.0	676.0	678.2	499.98	490.40	503.80	17.70	16.24	18.71
792-816	678.0	677.1	678.9	498.17	493.50	501.40	17.92	17.76	18.22
816-840	677.6	676.3	678.8	498.64	494.40	509.40	17.43	17.11	18.22
840-864	677.1	675.9	678.1	495.52	492.20	507.10	17.45	17.19	17.60
864-888	676.8	675.8	677.9	493.95	490.70	496.80	17.23	17.04	17.45
888-912	677.3	675.9	678.1	494.07	491.50	500.00	17.20	16.99	17.40
912-936	677.3	676.0	678.3	497.48	493.40	500.30	17.21	16.89	17.42
936-960	677.2	676.3	678.2	497.59	492.60	501.90	17.19	15.81	17.71
960-984	677.9	677.2	678.7	495.05	491.10	499.80	17.47	17.23	17.93
984-1006	677.7	676.8	679.1	500.04	496.30	505.70	17.66	17.46	17.87
Overall	677.5	674.0	679.2	500.41	481.00	517.30	17.76	15.81	19.49

APPENDIX B:
IDENTIFICATION OF LIQUID-PHASE IMPURITIES
AND DEGRADATION PRODUCTS

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IDENTIFICATION OF LIQUID-PHASE IMPURITIES
AND DEGRADATION PRODUCTS

Figures B.1-B.32 are the ion-chromatograms and mass spectra used to identify liquid-phase impurities in and degradation products of toluene samples obtained in ORC tests.

FIGURE B.1 Total Ion Chromatogram Showing 5-11 min of Toluene Degradation for 956 h at 677°F

FIGURE B.2 Total Ion Chromatogram Showing 8-11 min of Toluene Degradation for 956 h at 677°F

FIGURE B.3 Total Ion Chromatogram Showing 5-17 min of Toluene Degradation for 956 h at 677°F

FIGURE B.4 Total Ion Chromatogram Showing 17-30 min of Toluene Degradation for 956 h at 677°F

FIGURE B.5 Total Ion Chromatogram Showing 30-40 min of Toluene Degradation for 956 h at 677°F

FIGURE B.6 Peak at 5.4 min: Benzene

FIGURE B.7 Peak at 6.4 min: Heptane

FIGURE B.8 Peak at 7.0 min: Dimethylcyclopentane Isomer

FIGURE B.9 Peak at 7.5 min: Methylbenzene

FIGURE B.10 Peak at 9.0 min: Dimethylhexane Isomer

FIGURE B.11 Peak at 9.1 min: Dimethylcyclohexane Isomer

FIGURE B.12 Peak at 9.2 min: Dimethylcyclohexane Isomer

FIGURE B.13 Peak at 9.3 min: A C9 Cycloalkane

FIGURE B.14 Peak at 9.4 min: A C9 Cycloalkane

FIGURE B.15 Peak at 9.5 min: A C8 Cycloalkane

FIGURE B.16 Peak at 9.6 min: A C8 Cycloalkane

FIGURE B.17 Peak at 9.6 min: Dimethylcyclohexane Isomer

FIGURE B.18 Peak at 9.7 min: Ethylcyclohexane

FIGURE B.19 Peak at 9.8 min: A C9 Cycloalkane

FIGURE B.20 Peak at 10.1 min: Ethylbenzene

FIGURE B.21 Peak at 10.2 min: Xylene Isomer

FIGURE B.22 Peak at 14.5 min: C4-Benzene Isomer

FIGURE B.23 Peak at 22.0 min: Methylbiphenyl Isomer

FIGURE B.24 Peak at 23.0 min: C1-Diphenylmethane Isomer

FIGURE B.25 Peak at 23.3 min: C1-Diphenylmethane Isomer

FIGURE B.26 Peak at 23.6 min: Bibenzyl

FIGURE B.27 Peak at 23.8 min: C1-Diphenylmethane Isomer

FIGURE B.28 Peak at 24.7 min: Dimethylbiphenyl Isomer

FIGURE B.29 Peak at 24.9 min: Dimethylbiphenyl Isomer

FIGURE B.30 Peak at 25.0 min: Dimethylbiphenyl Isomer

FIGURE B.31 Peak at 25.7 min: C3-Biphenyl Isomer

FIGURE B.32 Peak at 26.6 min: Methylfluorene Isomer

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APPENDIX C:
GAS CHROMATOGRAMS OF
LIQUID SAMPLES

APPENDIX C:
GAS CHROMATOGRAMS OF
LIQUID SAMPLES

Figures C.1-C.19 are gas chromatograms obtained from liquid samples.

FIGURE C.1 Replotted Chromatogram of Toluene Standard

FIGURE C.2a Replotted Chromatograms for 635°F, 0 h, Time Segment = 0-40 min — Major Components

FIGURE C.2b Replotted Chromatograms for 635°F, 0 h, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.3a Replotted Chromatograms for 635°F, 45 h, Runs 330 and 319, Time Segment = 0-40 min — Major Components

FIGURE C.3b Replotted Chromatograms for 635°F, 45 h, Runs 330 and 319, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.3c Replotted Chromatograms for 635°F, 45 h, Runs 330 and 319, Time Segment = 27-40 min — Degradation Products

FIGURE C.4a Replotted Chromatograms for 635°F, 45 h, Runs 343 and 341, Time Segment = 0-40 min — Major Components

FIGURE C.4b Replotted Chromatograms for 635°F, 45 h, Runs 343 and 341, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.4c Replotted Chromatograms for 635°F, 45 h, Runs 343 and 341, Time Segment = 27-40 min — Degradation Products

FIGURE C.5a Replotted Chromatograms for 635°F, 168 and 45 h, Runs 331 and 341, Time Segment = 0-40 min — Major Components

FIGURE C.5b Replotted Chromatograms for 635°F, 168 and 45 h, Runs 331 and 341, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.5c Replotted Chromatograms for 635°F, 168 and 45 h, Runs 331 and 341, Time Segment = 27-40 min — Degradation Products

FIGURE C.6a Replotted Chromatograms for 635°F, 504 and 338 h, Runs 335 and 332, Time Segment = 0-40 min — Major Components

FIGURE C.6b Replotted Chromatograms for 635°F, 504 and 338 h, Runs 335 and 332, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.6c Replotted Chromatograms for 635°F, 504 and 338 h, Runs 335 and 332, Time Segment = 27-40 min — Degradation Products

FIGURE C.7a Replotted Chromatograms for 635°F, 504 h, Runs 335 and 327, Time Segment = 0-40 min — Total Degradation

FIGURE C.7b Replotted Chromatograms for 635°F, 504 h, Runs 335 and 327, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.7c Replotted Chromatograms for 635°F, 504 h, Runs 335 and 327, Time Segment = 27-40 min — Degraded Toluene Products

FIGURE C.8a Replotted Chromatograms for 635°F, 840 and 649 h, Runs 324 and 336, Time Segment = 0-40 min — Major Components

FIGURE C.8b Replotted Chromatograms for 635°F, 840 and 649 h, Runs 324 and 336, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.8c Replotted Chromatograms for 635°F, 840 and 649 h, Runs 324 and 336, Time Segment = 27-40 min — Degradation Products

FIGURE C.9a Replotted Chromatograms for 600°F, 23 and 0 h, Runs 239 and 313, Time Segment = 0-40 min — Major Components

FIGURE C.9b Replotted Chromatograms for 600°F, 23 and 0 h, Runs 239 and 313, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.9c Replotted Chromatograms for 600°F, 23 and 0 h, Runs 239 and 313, Time Segment = 27-40 min — Degradation Products

FIGURE C.10a Replotted Chromatograms for 600°F, 455 and 266 h, Runs 232 and 230, Time Segment = 0-40 min — Major Components

FIGURE C.10b Replotted Chromatograms for 600°F, 455 and 266 h, Runs 232 and 230, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.10c Replotted Chromatograms for 600°F, 455 and 266 h, Runs 232 and 230, Time Segment = 27-40 min — Degradation Products

FIGURE C.11a Replotted Chromatograms for 600°F, 1006 and 766 h, Runs 234 and 229, Time Segment = 0-40 min — Major Components

FIGURE C.11b Replotted Chromatograms for 600°F, 1006 and 766 h, Runs 234 and 229, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.11c Replotted Chromatograms for 600°F, 1006 and 766 h, Runs 234 and 229, Time Segment = 27-40 min — Degradation Products

FIGURE C.12a Replotted Chromatograms for 650°F, 19 and 0 h, Time Segment = 0-40 min — Major Components

FIGURE C.12b Replotted Chromatograms for 650°F, 19 and 0 h, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.12c Replotted Chromatograms for 650°F, 19 and 0 h, Time Segment = 27-40 min — Degradation Products

FIGURE C.13a Replotted Chromatograms for 650°F, 282 and 115 h, Runs 312 and 222, Time Segment = 0-40 min — Major Components

FIGURE C.13b Replotted Chromatograms for 650°F, 282 and 115 h, Runs 312 and 222, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.13c Replotted Chromatograms for 650°F, 282 and 115 h, Runs 312 and 222, Time Segment = 27-40 min — Degradation Products

FIGURE C.14a Replotted Chromatograms for 650°F, 450 and 282 h, Runs 240 and 226, Time Segment = 0-40 min — Major Components

FIGURE C.14b Replotted Chromatograms for 650°F, 450 and 282 h, Runs 240 and 226, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.14c Replotted Chromatograms for 650°F, 450 and 282 h, Runs 240 and 226, Time Segment = 27-40 min — Degradation Products

FIGURE C.15a Replotted Chromatograms for 650°F, 619 and 450 h, Runs 311 and 240, Time Segment = 0-40 min — Major Components

FIGURE C.15b Replotted Chromatograms for 650°F, 619 and 450 h, Runs 311 and 240, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.15c Replotted Chromatograms for 650°F, 619 and 450 h, Runs 311 and 240, Time Segment = 27-40 min — Degradation Products

FIGURE C.16a Replotted Chromatograms for 650°F, 955 and 786 h, Runs 334 and 323, Time Segment = 0-40 min — Major Components

FIGURE C.16b Replotted Chromatograms for 650°F, 955 and 786 h, Runs 334 and 323, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.16c Replotted Chromatograms for 650°F, 955 and 786 h, Runs 334 and 323, Time Segment = 27-40 min — Degradation Products

FIGURE C.17a Replotted Chromatograms for 677°F, 117 and 0 h, Runs 352 and 351, Time Segment = 0-40 min — Major Components

FIGURE C.17b Replotted Chromatograms for 677°F, 117 and 0 h, Runs 352 and 351, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.17c Replotted Chromatograms for 677°F, 117 and 0 h, Runs 352 and 351, Time Segment = 27-40 min — Degradation Products

FIGURE C.18a Replotted Chromatograms for 677°F, 600 and 285 h, Runs 363 and 353, Time Segment = 0-40 min — Major Components

FIGURE C.18b Replotted Chromatograms for 677°F, 600 and 285 h, Runs 363 and 353, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.18c Replotted Chromatograms for 677°F, 600 and 285 h, Runs 363 and 353, Time Segment = 27-40 min — Degradation Products

FIGURE C.19a Replotted Chromatograms for 677°F, 958 and 789 h, Runs 364 and 362, Time Segment = 0-40 min — Major Components

FIGURE C.19b Replotted Chromatograms for 677°F, 958 and 789 h, Runs 364 and 362, Time Segment = 0-15 min — Toluene Impurities

FIGURE C.19c Replotted Chromatograms for 677°F, 958 and 789 h, Runs 364 and 362, Time Segment = 27-40 min — Degradation Products

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APPENDIX D:
TOLUENE DEGRADATION REPORTS

APPENDIX D:
TOLUENE DEGRADATION REPORTS

TABLE D.1 Toluene Degradation Report — 635°F, 0 h, Run 318

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		135.7	5.8	1.66	23
2	BENZENE	200.0	115.4	2.44	467
3		206.8	19.9	3.06	81
4	METHYLCYCLOHEXANE	212.3	29.2	3.57	118
5	C2-CYCLOPENTANE	215.0	1.0	3.82	4
6	C2-CYCLOPENTANE	215.5	0.4	3.86	1
7	C2-CYCLOPENTANE	216.4	2.3	3.95	9
10	C8-ALKANE	237.0	33.8	5.83	137
11	C8-ALKANE	237.5	48.2	5.87	195
12	C2-CYCLOHEXANE	238.8	168.3	5.99	681
13	C2-CYCLOHEXANE	240.4	137.4	6.14	556
14	ISOPROPYLCYCLOPENTANE	242.0	28.4	6.28	115
15		243.0	5.7	6.37	23
16	ETHYLMETHYCYCLOPENTANE	243.7	15.8	6.44	64
17	ETHYLMETHYCYCLOPENTANE	245.2	57.4	6.57	232
18	DIMETHYLCYCLOHEXANE	246.5	16.3	6.70	66
19	DIMETHYLCYCLOHEXANE	247.4	72.4	6.78	293
20	ETHYLCYCLOHEXANE	249.0	334.9	6.93	1355
21	ETHYLBENZENE	259.2	126.0	7.86	510
22	XYLENE	262.3	268.3	8.14	1085
23		271.7	23.0	9.00	81

RRI REPORT

SAMPLE:	FD635FT3	TOTAL CONCENTRATION = 1509.6
DISK FILE:	31802	NO. OF INTERPRETED PEAKS = 22
GC RUN:	4 17 86	TOTAL NO. OF PEAKS = 23

TOLUENE DATA:

RT = 5.76	AREA = 4039750
ARI = 236%	CONC = 998490

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.2 Toluene Degradation Report — 635°F, 0 h, Run 329

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		101.1	1.8	1.24	9
2		135.1	5.6	1.65	27
3	BENZENE	200.0	112.9	2.44	543
4		206.7	19.7	3.05	95
5	METHYLCYCLOHEXANE	212.3	33.6	3.57	161
6	C2-CYCLOPENTANE	214.8	4.1	3.79	20
9	C8-ALKANE	237.7	27.1	5.87	130
10	C8-ALKANE	238.1	49.4	5.91	238
11	C2-CYCLOHEXANE	239.4	164.4	6.03	791
12	ISOPROPYLCYCLOPENTANE	240.9	134.5	6.16	647
13		242.5	27.6	6.30	133
14		243.4	5.4	6.39	26
15	ETHYLMETHYCYCLOPENTANE	244.2	15.4	6.46	74
16		245.6	56.1	6.59	270
17	DIMETHYLCYCLOHEXANE	246.9	16.0	6.71	77
18		247.8	71.0	6.79	342
19	ETHYLCYCLOHEXANE	249.4	328.0	6.94	1578
20	ETHYLBENZENE	259.6	122.1	7.87	587
21	XYLENE	262.7	259.4	8.15	1248
22		272.1	24.3	9.00	97

RRI REPORT

SAMPLE: FD635FOHT3 TOTAL CONCENTRATION = 1478.1
 DISK FILE: 32902 NO. OF INTERPRETED PEAKS = 21
 GC RUN: 4 24 86 TOTAL NO. OF PEAKS = 22

TOLUENE DATA:

RT = 5.80 AREA = 4805160
 ARI = 237% CONC = 998522

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.3 Toluene Degradation Report — 635°F, 45 h, Run 319

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		115.4	2.3	1.43	15
2		134.7	6.2	1.67	41
3	BENZENE	200.0	115.1	2.48	755
4	C6H14	206.3	6.2	3.06	41
5	METHYLCYCLOHEXANE	212.0	1.2	3.57	8
6	C2-CYCLOPENTANE	214.5	1.1	3.79	7
10	C2-CYCLOHEXANE	240.0	29.2	6.11	192
11	C2-CYCLOHEXANE	240.3	45.4	6.14	298
12	ISOPROPYLCYCLOPENTANE	241.2	162.9	6.22	1069
13		242.6	136.0	6.34	893
14	ETHYLMETHYCYCLOPENTANE	243.9	27.9	6.47	183
15	ETHYLMETHYCYCLOPENTANE	244.7	5.2	6.54	34
16		245.5	15.4	6.61	101
17	DIMETHYLCYCLOHEXANE	246.8	56.2	6.72	369
18		248.0	16.1	6.84	106
19	ETHYLCYCLOHEXANE	248.9	71.0	6.92	466
20	ETHYLCYCLOHEXANE	250.4	327.0	7.06	2146
21	ETHYLBENZENE	260.3	118.9	7.95	780
22	XYLENE	263.3	253.1	8.23	1661
23		272.5	23.4	9.06	134
24	BENZALDEHYDE	300.7	30.9	11.62	177
25	C1-STYRENE	333.4	5.0	14.59	29
26	BIPHENYL	473.0	9.1	27.85	58
27	DIPHENYLETHER	481.0	22.8	28.64	145
28	BIBENZYL	520.9	9.0	32.59	58

RRI REPORT

SAMPLE: FD635F51HT3 TOTAL CONCENTRATION = 1496.9
 DISK FILE: 31902 NO. OF INTERPRETED PEAKS = 26
 GC RUN: 4 17 86 TOTAL NO. OF PEAKS = 28

TOLUENE DATA:

RT = 6.06 AREA = 6551450
 ARI = 240% CONC = 998503

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.4 Toluene Degradation Report — 635°F, 45 h, Run 330

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		116.0	1.6	1.42	9
2		135.5	5.9	1.66	35
3	BENZENE	200.0	110.5	2.45	647
4		206.7	20.2	3.06	118
5	METHYLCYCLOHEXANE	212.4	34.5	3.58	202
6	C2-CYCLOPENTANE	214.7	8.7	3.79	51
7	C2-CYCLOPENTANE	216.2	9.4	3.93	55
11	C2-CYCLOHEXANE	239.3	75.1	6.02	440
12	C2-CYCLOHEXANE	240.4	162.3	6.12	151
13	ISOPROPYLCYCLOPENTANE	241.8	133.9	6.25	784
14		243.2	27.4	6.38	160
15	ETHYLMETHYCYCLOPENTANE	244.1	5.3	6.46	31
16	ETHYLMETHYCYCLOPENTANE	244.9	15.3	6.53	90
17		246.2	55.4	6.65	325
18		247.5	15.9	6.77	93
19		248.4	70.1	6.85	411
20	ETHYLCYCLOHEXANE	250.0	322.1	6.99	1886
21	ETHYLBENZENE	260.0	117.1	7.90	686
22	XYLENE	263.0	249.1	8.18	1459
23		272.3	24.2	9.02	118
24	BENZALDEHYDE	300.7	36.6	11.60	178
25	C1-STYRENE	333.5	5.0	14.58	24
26	BIPHENYL	473.0	9.7	27.83	53
27	DIPHENYLETHER	481.0	23.9	28.62	130
28	BIBENZYL	520.9	9.8	32.57	53

RRIREPORT

SAMPLE: FD635F51HT3 TOTAL CONCENTRATION = 1548.8
 DISK FILE: 33002 NO. OF INTERPRETED PEAKS = 26
 GC RUN: 4 24 86 TOTAL NO. OF PEAKS = 28

TOLUENE DATA:

RT = 5.93 AREA = 5848720
 ARI = 238% CONC = 998451

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.5 Toluene Degradation Report — 635°F, 45 h, Run 340

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		114.8	1.8	1.42	10
2		134.5	6.1	1.66	35
3	BENZENE	200.0	110.5	2.47	636
4		206.8	20.7	3.08	119
8	C2-CYCLOHEXANE	239.1	30.6	6.01	176
9	C2-CYCLOHEXANE	239.5	45.9	6.04	264
10	ISOPROPYLCYCLOPENTANE	240.5	164.0	6.13	943
11	ISOPROPYLCYCLOPENTANE	241.9	135.6	6.26	780
12		243.3	27.8	6.39	160
13	ETHYLMETHYCYCLOPENTANE	244.2	5.4	6.46	31
14	ETHYLMETHYCYCLOPENTANE	245.0	15.5	6.53	89
15		246.3	56.5	6.65	325
16		247.6	16.2	6.77	93
17		248.4	71.3	6.85	411
18	ETHYLCYCLOHEXANE	250.0	327.5	6.99	1884
19	ETHYLBENZENE	260.0	120.3	7.89	692
20	XYLENE	263.0	257.4	8.16	1481
24	BIPHENYL	473.0	9.7	27.74	54
25	DIPHENYLETHER	481.0	23.8	28.53	134
26	BIBENZYL	520.9	10.1	32.46	56

RRI REPORT

SAMPLE: FD635F51HT3 TOTAL CONCENTRATION = 1515.9
 DISK FILE: 34002 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 5 6 86 TOTAL NO. OF PEAKS = 26

TOLUENE DATA:

RT = 5.95 AREA = 5743420
 ARI = 239% CONC = 998484

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.6 Toluene Degradation Report - 635°F, 45 h, Run 341

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		115.7	1.8	1.40	10
2		135.0	6.0	1.63	33
3	BENZENE	200.0	113.5	2.42	617
4		206.7	21.0	3.02	114
5	METHYLCYCLOHEXANE	212.5	36.9	3.54	201
6	C2-CYCLOPENTANE	216.2	11.8	3.87	64
10	C2-CYCLOHEXANE	240.1	131.3	6.03	715
11	ISOPROPYLCYCLOPENTANE	241.5	129.7	6.16	706
12		243.0	26.0	6.29	142
13	ETHYLMETHYCYCLOPENTANE	244.6	19.1	6.44	104
14		246.0	56.3	6.56	307
15		248.1	87.8	6.75	478
16	ETHYLCYCLOHEXANE	249.7	328.6	6.90	1788
17	ETHYLBENZENE	259.8	119.0	7.80	648
18	XYLENE	262.8	252.9	8.08	1376
19		272.1	20.7	8.91	112
20	BENZALDEHYDE	300.5	34.9	11.47	168
21	C1-STYRENE	333.3	6.1	14.43	29
22	BIPHENYL	473.0	9.6	27.64	51
23	DIPHENYLETHER	481.1	23.3	28.43	123
24	BIBENZYL	520.9	10.2	32.36	54

RRI REPORT

SAMPLE: FD635F51HT3 TOTAL CONCENTRATION = 1446.7
 DISK FILE: 34102 NO. OF INTERPRETED PEAKS = 22
 GC RUN: 5 6 86 TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.83 AREA = 5434100
 ARI = 238% CONC = 998553

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.7 Toluene Degradation Report — 635°F, 168 h, Run 320

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		135.9	3.2	1.66	17
2	BENZENE	200.0	79.3	2.44	415
3		206.7	19.5	3.05	102
4	METHYLCYCLOHEXANE	212.6	34.4	3.59	180
5	C2-CYCLOPENTANE	214.9	8.6	3.79	45
6	C2-CYCLOPENTANE	216.5	9.5	3.95	50
10	C2-CYCLOHEXANE	239.2	78.8	6.00	412
11	C2-CYCLOHEXANE	240.3	166.2	6.10	868
12	ISOPROPYLCYCLOPENTANE	241.7	137.0	6.24	716
13		243.2	28.0	6.37	146
14	ETHYLMETHYCYCLOPENTANE	244.1	5.3	6.45	28
15	ETHYLMETHYCYCLOPENTANE	244.9	15.7	6.52	82
16		246.2	56.6	6.65	296
17		247.6	16.2	6.77	85
18		248.4	71.9	6.85	376
19	ETHYLCYCLOHEXANE	250.0	330.6	6.99	1727
20	ETHYLBENZENE	260.1	120.9	7.91	632
21	XYLENE	263.1	258.1	8.18	1349
22		272.4	24.2	9.03	110
23	BENZALDEHYDE	300.8	23.6	11.61	107
24	DIPHENYLETHER	481.1	4.1	28.65	21
25	BIBENZYL	520.9	19.7	32.58	101

RRI REPORT

SAMPLE: FD635F176HT3 TOTAL CONCENTRATION = 1511.4
 DISK FILE: 32002 NO. OF INTERPRETED PEAKS = 23
 GC RUN: 4 17 86 TOTAL NO. OF PEAKS = 25

TOLUENE DATA:

RT = 5.91 AREA = 5217830
 ARI = 238% CONC = 998489

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.8 Toluene Degradation Report — 635°F, 168 h, Run 331

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	67.1	2.43	306
2	METHYLCYCLOHEXANE	212.6	32.0	3.58	146
3	C2-CYCLOPENTANE	215.1	4.0	3.80	19
4	C2-CYCLOPENTANE	216.5	1.3	3.93	6
9	C8-ALKANE	237.6	33.1	5.85	151
10	C8-ALKANE	238.1	47.0	5.89	215
11	C2-CYCLOHEXANE	239.3	164.3	6.01	751
12	ISOPROPYLCYCLOPENTANE	240.9	134.4	6.15	614
13		242.5	27.5	6.29	125
14	ETHYLMETHYCYCLOPENTANE	243.5	5.5	6.38	25
15	ETHYLMETHYCYCLOPENTANE	244.2	15.4	6.45	70
16		245.6	55.6	6.58	254
17	DIMETHYLCYCLOHEXANE	247.0	16.2	6.70	74
18		247.9	70.2	6.78	321
19	ETHYLCYCLOHEXANE	249.5	323.4	6.93	1478
20	ETHYLBENZENE	259.7	117.0	7.86	534
21	XYLENE	262.8	251.5	8.14	1149
22		272.2	20.0	8.99	92
23	BENZALDEHYDE	300.7	21.1	11.59	80
24	BIBENZYL	520.9	13.0	32.57	55

RRI REPORT

SAMPLE:	FD635F176HT3	TOTAL CONCENTRATION = 1419.5
DISK FILE:	33102	NO. OF INTERPRETED PEAKS = 21
GC RUN:	4 24 86	TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.78	AREA = 4561510
ARI = 237%	CONC = 998581

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.9 Toluene Degradation Report — 635°F, 338 h, Run 321

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		135.5	7.2	1.66	34
2	BENZENE	200.0	62.3	2.45	292
3		206.9	17.1	3.08	80
4	METHYLCYCLOHEXANE	212.4	27.5	3.58	129
5	C2-CYCLOPENTANE	214.8	2.6	3.80	12
6	C2-CYCLOPENTANE	216.3	.5	3.93	2
12	C8-ALKANE	238.1	32.7	5.91	154
13	C2-CYCLOHEXANE	238.5	47.4	5.95	223
14	C2-CYCLOHEXANE	239.7	167.2	6.06	786
15	ISOPROPYLCYCLOPENTANE	241.2	137.9	6.19	648
16		242.7	28.3	6.33	133
17	ETHYLMETHYCYCLOPENTANE	243.7	5.6	6.42	26
18	ETHYLMETHYCYCLOPENTANE	244.4	16.0	6.49	75
19		245.8	57.4	6.62	269
20	DIMETHYLCYCLOHEXANE	247.1	16.7	6.74	78
21		248.0	72.8	6.82	342
22	ETHYLCYCLOHEXANE	249.6	335.0	6.96	1574
23	ETHYLBENZENE	259.8	123.7	7.89	581
24	XYLENE	262.9	267.4	8.17	1256
25		272.2	25.7	9.02	105
26	BENZALDEHYDE	300.7	12.0	11.61	49
27	BIBENZYL	520.9	24.7	32.59	115
28	C1-FLUORENE	578.8	7.4	38.32	34

RRI REPORT

SAMPLE: FD635F344T3 TOTAL CONCENTRATION = 1594.9
 DISK FILE: 32102 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 4 17 86 TOTAL NO. OF PEAKS = 28

TOLUENE DATA:

RT = 5.84 AREA = 4690700
 ARI = 2387 CONC = 998505

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.10 Toluene Degradation Report — 635°F, 338 h, Run 332

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		135.6	7.2	1.66	32
2	BENZENE	200.0	64.4	2.44	287
3		206.7	17.1	3.05	76
4	METHYLCYCLOHEXANE	212.3	33.3	3.56	149
5	C2-CYCLOPENTANE	215.0	7.2	3.80	32
11	C8-ALKANE	237.3	32.4	5.83	144
12	C8-ALKANE	237.8	45.5	5.87	203
13	C2-CYCLOHEXANE	239.1	164.8	5.99	734
14	ISOPROPYLCYCLOPENTANE	240.6	135.5	6.14	604
15	ISOPROPYLCYCLOPENTANE	242.2	27.7	6.28	123
16		243.2	5.4	6.37	24
17	ETHYLMETHYCYCLOPENTANE	244.0	15.6	6.44	70
18	ETHYLMETHYCYCLOPENTANE	245.4	56.5	6.57	252
19	DIMETHYLCYCLOHEXANE	246.8	16.3	6.69	73
20		247.7	71.8	6.77	320
21	ETHYLCYCLOHEXANE	249.3	330.6	6.92	1473
22	ETHYLBENZENE	259.5	124.8	7.85	556
24	XYLENE	262.6	266.8	8.13	1189
25		272.0	22.6	8.99	101
26	BENZALDEHYDE	300.6	13.1	11.58	49
27	BIBENZYL	520.9	28.3	32.56	117
28	C1-FLUORENE	578.8	8.7	38.29	36

RRI REPORT

SAMPLE: FD635F344HT3 TOTAL CONCENTRATION = 1567.2
 DISK FILE: 33202 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 4 24 86 TOTAL NO. OF PEAKS = 34

TOLUENE DATA:

RT = 5.76 AREA = 4449570
 ARI = 237% CONC = 998504

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.11 Toluene Degradation Report — 635°F, 504 h, Run 327

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		135.8	5.9	1.66	31
2	BENZENE	200.0	77.3	2.44	404
3		206.7	20.8	3.04	108
4	METHYLCYCLOHEXANE	212.3	32.5	3.56	170
5	C2-CYCLOPENTANE	214.9	4.4	3.79	23
6	C2-CYCLOPENTANE	216.5	2.0	3.94	10
11	C8-ALKANE	238.4	32.0	5.93	167
12	C2-CYCLOHEXANE	238.8	46.4	5.96	242
13	C2-CYCLOHEXANE	239.9	163.6	6.07	855
14	ISOPROPYLCYCLOPENTANE	241.4	133.9	6.21	699
15		242.9	27.2	6.34	142
16	ETHYLMETHYCYCLOPENTANE	243.9	5.2	6.43	27
17	ETHYLMETHYCYCLOPENTANE	244.6	15.3	6.50	80
18		246.0	55.2	6.62	288
19	DIMETHYLCYCLOHEXANE	247.3	16.0	6.74	83
20		248.2	69.6	6.82	364
21	ETHYLCYCLOHEXANE	249.8	321.0	6.97	1677
22	ETHYLBENZENE	259.9	115.4	7.89	603
23	XYLENE	263.0	247.1	8.16	1291
24		272.3	22.7	9.01	101
25	BENZALDEHYDE	300.7	39.4	11.60	175
26	BIBENZYL	520.9	30.8	32.57	154
27	C1-FLUORENE	578.9	7.6	38.30	38

RRI REPORT

SAMPLE: FD635F512HT3 TOTAL CONCENTRATION = 1491.4
 DISK FILE: 32702 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 4 24 86 TOTAL NO. OF PEAKS = 27

TOLUENE DATA:

RT = 5.72 AREA = 5216190
 ARI = 236% CONC = 998509

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.12 Toluene Degradation Report — 635°F, 504 h, Run 335

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		136.2	6.0	1.66	29
2	BENZENE	200.0	81.3	2.43	392
3		206.7	1.3	3.05	6
4	METHYLCYCLOHEXANE	212.4	8.8	3.56	42
5	METHYLCYCLOHEXANE	212.8	23.8	3.60	115
6	C2-CYCLOPENTANE	215.0	4.3	3.80	21
7	C2-CYCLOPENTANE	216.5	2.0	3.93	9
12	C8-ALKANE	237.9	32.6	5.88	158
13	C8-ALKANE	238.3	46.8	5.92	225
14	C2-CYCLOHEXANE	239.5	163.9	6.03	789
15	ISOPROPYLCYCLOPENTANE	241.1	133.8	6.17	645
16		242.6	27.2	6.31	131
17	ETHYLMETHYCYCLOPENTANE	243.6	5.2	6.40	25
18	ETHYLMETHYCYCLOPENTANE	244.3	15.3	6.47	74
19		245.7	55.3	6.59	266
20	DIMETHYLCYCLOHEXANE	247.1	15.9	6.72	77
21		248.0	70.0	6.80	337
22	ETHYLCYCLOHEXANE	249.6	322.1	6.94	1552
23	ETHYLBENZENE	259.7	116.7	7.87	562
24	XYLENE	262.8	249.6	8.15	1202
25		272.2	20.4	9.00	98
26	BENZALDEHYDE	300.7	48.6	11.59	194
27	DIPHENYLETHER	481.1	3.9	28.64	17
28	BIBENZYL	520.9	34.5	32.57	151
29	C1-FLUORENE	578.8	9.0	38.30	40

RRI REPORT

SAMPLE: FD635F512HT3 TOTAL CONCENTRATION = 1498.2
 DISK FILE: 33502 NO. OF INTERPRETED PEAKS = 26
 GC RUN: 4 29 86 TOTAL NO. OF PEAKS = 29

TOLUENE DATA:

RT = 5.81 AREA = 4809310
 ARI = 237% CONC = 998502

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.13 Toluene Degradation Report — 635°F, 649 h, Run 325

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		136.0	3.1	1.66	13
2	BENZENE	200.0	70.0	2.44	299
3		206.8	19.4	3.05	83
4	METHYLCYCLOHEXANE	212.4	30.9	3.56	132
5	C2-CYCLOPENTANE	215.1	2.8	3.81	12
6	C2-CYCLOPENTANE	216.5	1.0	3.94	4
10	C8-ALKANE	237.2	33.7	5.82	144
11	C8-ALKANE	237.7	48.0	5.87	205
12	C2-CYCLOHEXANE	239.0	165.8	5.99	709
13	ISOPROPYLCYCLOPENTANE	240.6	135.4	6.13	579
14	ISOPROPYLCYCLOPENTANE	242.2	27.6	6.28	118
15		243.3	5.2	6.37	22
16	ETHYLMETHYLCYCLOPENTANE	244.0	15.5	6.44	66
17		245.5	55.8	6.57	239
18	DIMETHYLCYCLOHEXANE	246.8	16.0	6.69	69
19		247.7	70.6	6.77	302
20	ETHYLCYCLOHEXANE	249.3	326.4	6.92	1395
21	ETHYLBENZENE	259.6	118.4	7.86	506
22	XYLENE	262.7	253.7	8.14	1085
23		272.1	20.7	9.00	88
24	BENZALDEHYDE	300.7	18.1	11.59	66
25	BIBENZYL	520.9	44.6	32.57	179
26	C1-FLUORENE	578.9	5.8	38.31	23

RRI REPORT

SAMPLE: FD635F657HT3 TOTAL CONCENTRATION = 1488.5
 DISK FILE: 32502 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 4 23 86 TOTAL NO. OF PEAKS = 26

TOLUENE DATA:

RT = 5.74 AREA = 4269080
 ARI = 236% CONC = 998512

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.14 Toluene Degradation Report — 635°F, 649 h, Run 336

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		135.5	3.3	1.66	16
2	BENZENE	200.0	65.2	2.45	323
3		206.6	15.5	3.05	204
7	C2-CYCLOPENTANE	214.8	5.2	3.80	26
6	C2-CYCLOPENTANE	216.3	3.3	3.93	16
10	C8-ALKANE	237.9	32.5	5.90	161
11	C8-ALKANE	238.3	46.7	5.93	231
12	C2-CYCLOHEXANE	239.5	164.1	6.04	813
13	ISOPROPYLCYCLOPENTANE	241.1	134.5	6.18	667
14		242.6	27.3	6.32	135
15	ETHYLMETHYCYCLOPENTANE	243.6	5.2	6.41	26
16	ETHYLMETHYCYCLOPENTANE	244.3	15.4	6.48	76
17		245.7	55.5	6.60	275
18	DIMETHYLCYCLOHEXANE	247.0	16.1	6.72	80
19		247.9	70.3	6.81	349
20	ETHYLCYCLOHEXANE	249.5	324.3	6.95	1607
21	ETHYLBENZENE	259.7	117.5	7.87	582
22	XYLENE	262.8	252.0	8.15	1248
23		272.1	23.8	9.00	97
24	BENZALDEHYDE	300.6	19.0	11.59	78
25	DIPHENYLETHER	481.1	4.0	28.64	18
26	BIBENZYL	520.9	45.5	32.57	204
27	C1-FLUORENE	578.9	5.9	38.30	27

RRI REPORT

SAMPLE: FD635F657HT3 TOTAL CONCENTRATION = 1486.9
 DISK FILE: 33602 NO. OF INTERPRETED PEAKS = 25
 GC RUN: 4 29 86 TOTAL NO. OF PEAKS = 27

TOLUENE DATA:

RT = 5.83 AREA = 4946760
 ARI = 237% CONC = 998513

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.15 Toluene Degradation Report — 635°F, 840 h, Run 324

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	64.5	2.45	322
2	C2-CYCLOPENTANE	214.9	1.0	3.81	5
3	C2-CYCLOPENTANE	216.4	0.6	3.94	3
7	C8-ALKANE	238.0	32.4	5.90	162
8	C8-ALKANE	238.4	47.0	5.94	235
9	C2-CYCLOHEXANE	239.6	164.7	6.05	822
10	ISOPROPYLCYCLOPENTANE	241.1	135.2	6.19	674
11		242.6	27.5	6.33	137
12	ETHYLMETHYCYCLOPENTANE	243.6	5.2	6.41	26
13	ETHYLMETHYCYCLOPENTANE	244.4	15.5	6.48	77
14		245.8	55.6	6.61	277
15	DIMETHYLCYCLOHEXANE	247.1	16.1	6.73	80
16		248.0	70.6	6.81	352
17	ETHYLCYCLOHEXANE	249.6	325.7	6.96	1625
18	ETHYLBENZENE	259.7	117.9	7.88	588
19	XYLENE	262.8	253.2	8.16	1263
20		272.2	24.1	9.01	102
21	BENZALDEHYDE	300.6	16.0	11.60	68
22	DIPHENYLETHER	481.1	3.5	28.64	17
23	BIBENZYL	520.9	48.8	32.58	229
24	C1-FLUORENE	578.9	8.1	38.31	38

RRI REPORT

SAMPLE: FD635F847HT3 TOTAL CONCENTRATION = 1433.6
 DISK FILE: 32402 NO. OF INTERPRETED PEAKS = 22
 GC RUN: 4 23 86 TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.83 AREA = 4980520
 ARI = 237% CONC = 998566

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.16 Toluene Degradation Report — 635°F, 840 h, Run 346

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	66.6	2.41	367
2		206.8	2.5	3.03	14
3	C2-CYCLOPENTANE	214.6	0.5	3.73	3
7	C2-CYCLOHEXANE	238.9	31.3	5.92	173
8	C2-CYCLOHEXANE	239.3	46.6	5.96	257
9	C2-CYCLOHEXANE	240.4	165.7	6.05	913
10	ISOPROPYLCYCLOPENTANE	241.8	136.8	6.18	754
11		243.2	27.9	6.31	153
12	ETHYLMETHYCYCLOPENTANE	244.1	5.2	6.39	29
13	ETHYLMETHYCYCLOPENTANE	244.9	15.8	6.46	87
14		246.2	56.8	6.58	313
15		247.5	16.4	6.70	90
16		248.4	72.2	6.78	398
17	ETHYLCYCLOHEXANE	249.9	331.4	6.92	1826
18	ETHYLBENZENE	260.0	120.4	7.82	663
19	XYLENE	263.0	259.7	8.10	1431
20		272.3	21.8	8.93	120
21	BENZALDEHYDE	300.6	18.6	11.49	91
22	DIPHENYLETHER	481.0	3.8	28.44	20
23	BIBENZYL	520.9	49.1	32.36	265
24	C1-FLUORENE	578.8	8.2	38.07	44

RRI REPORT

SAMPLE: FD635F847HT3 TOTAL CONCENTRATION = 1457.2
 DISK FILE: 34602 NO. OF INTERPRETED PEAKS = 22
 GC RUN: 5 29 86 TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.87 AREA = 5502150
 ARI = 238% CONC = 998543

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.17 Toluene Degradation Report — 600°F, 0 h, Run 313

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
3	PROPYLETHYLETHER	149.0	13.8	1.82	55
4	THF	162.7	4.8	1.98	19
5	BENZENE	200.0	126.8	2.44	505
6	C6H14	201.7	23.8	2.59	95
7		206.9	15.0	3.06	60
8	METHYLCYCLOHEXANE	212.6	32.2	3.58	128
9	C2-CYCLOPENTANE	215.1	8.5	3.81	34
11	C8-ALKANE	237.2	23.5	5.82	94
12	C8-ALKANE	237.6	43.5	5.86	173
13	C2-CYCLOHEXANE	239.0	139.3	5.99	555
14	ISOPROPYLCYCLOPENTANE	240.6	107.2	6.13	427
15	ISOPROPYLCYCLOPENTANE	242.2	21.5	6.27	86
16		243.2	5.1	6.36	20
17	ETHYLMETHYCYCLOPENTANE	244.0	11.6	6.43	46
18	ETHYLMETHYCYCLOPENTANE	245.4	48.3	6.57	193
19	DIMETHYLCYCLOHEXANE	246.8	10.7	6.69	43
20		247.7	53.4	6.77	213
21	ETHYLCYCLOHEXANE	249.3	235.5	6.92	938
22	ETHYLBENZENE	259.6	98.7	7.85	393
23	XYLENE	262.7	138.6	8.13	552
24		272.1	9.4	8.99	37
25	DOWTHERM/C1-BIPHENYL	481.2	5.9	28.65	23

RRI REPORT

SAMPLE:	FD600FOHT5	TOTAL CONCENTRATION = 1177.4
DISK FILE:	31302	NO. OF INTERPRETED PEAKS = 23
GC RUN:	4 15 86	TOTAL NO. OF PEAKS = 25

TOLUENE DATA:

RT = 5.74	AREA = 3979470
ARI = 236%	CONC = 998823

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.18 Toluene Degradation Report — 600°F, 23 h, Run 239

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		101.7	7.4	1.24	25
2	PROPYLETHYLETHER	148.6	10.1	1.82	34
3	BENZENE	200.0	109.4	2.44	374
4	C6H14	201.7	17.1	2.60	59
5		206.8	13.5	3.06	46
6	METHYLCYCLOHEXANE	212.4	30.8	3.58	106
7	C2-CYCLOPENTANE	215.1	8.6	3.82	30
10	C8-ALKANE	236.6	27.2	5.78	93
11	C8-ALKANE	237.1	42.3	5.82	145
12	C8-ALKANE	237.8	10.1	5.88	35
13	C2-CYCLOHEXANE	238.6	128.9	5.95	441
14	C2-CYCLOHEXANE	240.1	106.1	6.10	363
15	ISOPROPYLCYCLOPENTANE	241.8	21.2	6.24	73
16		242.8	5.2	6.34	18
26	DOWTHERM/C1-BIPHENYL	480.9	7.2	28.65	23
27	BIBENZYL	520.8	7.1	32.60	23

RRI REPORT

SAMPLE: FD600F46HT5 TOTAL CONCENTRATION = 1177.2
 DISK FILE: 23902 NO. OF INTERPRETED PEAKS = 26
 GC RUN: 4 8 86 TOTAL NO. OF PEAKS = 27

TOLUENE DATA:

RT = 5.69 AREA = 3417050
 ARI = 236% CONC = 998823

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.19 Toluene Degradation Report — 600°F, 266 h, Run 230

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		148.1	5.2	1.82	27
2	BENZENE	200.0	106.6	2.45	565
3	C2-CYCLOPENTANE	214.8	.2	3.80	1
6	C2-CYCLOHEXANE	238.7	26.3	5.97	139
7	C2-CYCLOHEXANE	239.0	42.5	6.00	225
8	C2-CYCLOHEXANE	240.1	142.6	6.11	756
9	ISOPROPYLCYCLOPENTANE	241.6	108.1	6.24	573
10		243.1	22.1	6.37	117
11	ETHYLMETHYCYCLOPENTANE	244.0	4.9	6.45	26
12	ETHYLMETHYCYCLOPENTANE	244.7	11.7	6.52	62
13		246.1	48.9	6.65	259
14	DIMETHYLCYCLOHEXANE	247.4	11.1	6.77	59
15		248.3	55.1	6.84	292
16	ETHYLCYCLOHEXANE	249.8	241.8	6.99	1282
17	ETHYLBENZENE	259.9	102.3	7.90	543
18	XYLENE	263.0	160.0	8.18	848
19		272.3	13.2	9.03	61
20	BENZALDEHYDE	300.7	7.2	11.62	33
21	DOWTHERM/C1-BIPHENYL	481.1	7.2	28.66	37
22	BIBENZYL	520.8	21.6	32.59	109

RRI REPORT

SAMPLE: FD600F266HT5 TOTAL CONCENTRATION = 1138.7
 DISK FILE: 23002 NO. OF INTERPRETED PEAKS = 21
 GC RUN: 4 3 84 TOTAL NO. OF PEAKS = 22

TOLUENE DATA:

RT = 5.91 AREA = 5295500
 ARI = 238% CONC = 998861

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.20 Toluene Degradation Report — 600°F, 455 h, Run 232

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	99.8	2.44	628
2	METHYLCYCLOHEXANE	212.4	6.4	3.56	41
3	C2-CYCLOPENTANE	214.9	9.7	3.79	61
6	C2-CYCLOHEXANE	240.0	24.7	6.08	155
7	C2-CYCLOHEXANE	240.3	41.6	6.11	261
8	ISOPROPYLCYCLOPENTANE	241.3	141.6	6.20	890
9		242.7	107.8	6.32	678
10	ETHYLMETHYCYCLOPENTANE	244.0	22.1	6.45	139
11	ETHYLMETHYCYCLOPENTANE	244.9	4.7	6.52	29
12		245.6	11.7	6.59	74
13	DIMETHYLCYCLOHEXANE	246.9	48.6	6.71	306
14		248.2	11.0	6.83	69
15		249.0	55.5	6.90	349
16	ETHYLCYCLOHEXANE	250.6	243.7	7.04	1533
17	ETHYLBENZENE	260.5	102.7	7.94	646
18	XYLENE	263.5	164.1	8.21	1032
19		272.7	13.8	9.06	75
20	BENZALDEHYDE	300.9	9.3	11.63	51
21	DOWTHERM/C1-BIPHENYL	481.0	7.7	28.65	47
22	BIBENZYL	520.8	32.8	32.60	196
23	C1-FLUORENE	578.8	4.4	38.33	27

RRI REPORT

SAMPLE: FD600F455HT5 TOTAL CONCENTRATION = 1164
 DISK FILE: 23202 NO. OF INTERPRETED PEAKS = 22
 GC RUN: 4 3 86 TOTAL NO. OF PEAKS = 23

TOLUENE DATA:

RT = 5.97 AREA = 6280080
 ARI = 239% CONC = 998836

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.21 Toluene Degradation Report — 600°F, 623 h, Run 236

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	90.6	2.43	707
2	C2-CYCLOPENTANE	215.0	.8	3.80	7
5	ISOPROPYLCYCLOPENTANE	241.9	62.5	6.25	489
6		242.7	140.0	6.33	1094
7	ETHYLMETHYCYCLOPENTANE	244.0	106.9	6.44	835
8	ETHYLMETHYCYCLOPENTANE	245.2	22.1	6.55	173
9		245.9	4.3	6.61	33
10	DIMETHYLCYCLOHEXANE	246.7	11.5	6.68	90
11		247.9	48.6	6.80	380
12		249.2	10.4	6.91	82
13	ETHYLCYCLOHEXANE	250.0	56.4	6.98	441
14	ETHYLCYCLOHEXANE	251.4	247.0	7.12	1931
15	ETHYLBENZENE	261.1	106.8	7.99	835
16	XYLENE	264.0	170.6	8.26	1334
17		273.1	14.6	9.09	100
18	BENZALDEHYDE	301.2	11.8	11.64	80
19	BIPHENYL	473.0	2.9	27.86	22
20	DOWTHERM/C1-BIPHENYL	481.0	7.8	28.66	60
21	BIBENZYL	520.9	40.2	32.60	309
22	C2-BIPHENYL	542.8	2.4	34.77	18
23	C1-FLUORENE	578.8	5.1	38.33	39

RRI REPORT

SAMPLE: FD600F622HT5 TOTAL CONCENTRATION = 1163.5
 DISK FILE: 23602 NO. OF INTERPRETED PEAKS = 22
 GC RUN: 4 4 84 TOTAL NO. OF PEAKS = 23

TOLUENE DATA:

RT = 5.75 AREA = 7806880
 ARI = 236% CONC = 998837

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.22 Toluene Degradation Report — 600°F, 766 h, Run 229

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	93.7	2.45	591
2		206.7	0.9	3.06	6
3	METHYLCYCLOHEXANE	212.3	0.5	3.57	3
4	C2-CYCLOPENTANE	214.8	0.5	3.79	3
7	C2-CYCLOHEXANE	239.9	24.6	6.08	155
8	C2-CYCLOHEXANE	240.2	42.3	6.11	267
9	ISOPROPYLCYCLOPENTANE	241.2	142.2	6.20	897
10		242.6	107.3	6.32	677
11	ETHYLMETHYCYCLOPENTANE	243.9	22.2	6.45	140
12	ETHYLMETHYCYCLOPENTANE	244.8	4.4	6.52	28
13		245.5	11.8	6.59	74
14	DIMETHYLCYCLOHEXANE	246.8	48.3	6.71	305
15		248.1	11.0	6.83	70
16		248.9	55.4	6.90	350
17	ETHYLCYCLOHEXANE	250.5	244.8	7.04	1544
18	ETHYLBENZENE	260.4	104.8	7.94	661
19	XYLENE	263.4	167.5	8.22	1056
20		272.6	14.2	9.05	78
21	BENZALDEHYDE	300.8	12.5	11.62	68
22	BIPHENYL	473.0	2.8	27.86	17
23	DOWTHERM/C1-BIPHENYL	481.0	8.3	28.65	50
24	BIBENZYL	520.8	50.4	32.59	303
25	C1-FLUORENE	578.8	6.0	38.32	36

RRI REPORT

SAMPLE: FD600F768HT5 TOTAL CONCENTRATION = 1176
 DISK FILE: 22902 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 4 3 84 TOTAL NO. OF PEAKS = 25

TOLUENE DATA:

RT = 6.03 AREA = 6301120
 ARI = 239% CONC = 998824

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.23 Toluene Degradation Report — 600°F, 938 h, Run 228

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	92.5	2.44	419
2		206.9	12.9	3.07	58
3	MEHTYLCYCLOHEXANE	212.6	29.9	3.59	135
4	C2-CYCLOPENTANE	215.1	8.3	3.81	38
5	C2-CYCLOPENTANE	216.4	11.0	3.93	49
8	C8-ALKANE	238.2	73.5	5.92	333
9	C2-CYCLOHEXANE	239.4	144.5	6.03	654
10	ISOPROPYLCYCLOPENTANE	240.9	107.5	6.17	487
11		242.5	22.3	6.31	101
12	ETHYLMETHYCYCLOPENTANE	243.5	4.5	6.40	21
13	ETHYLMETHYCYCLOPENTANE	244.2	11.9	6.47	54
14		245.6	48.0	6.60	217
15	DIMETHYLCYCLOHEXANE	247.0	11.2	6.72	51
16		247.9	55.2	6.80	250
17	ETHYLCYCLOHEXANE	249.5	244.8	6.94	1108
18	ETHYLBENZENE	259.7	105.1	7.87	476
19	XYLENE	262.8	167.3	8.15	757
20		272.2	14.1	9.01	55
21	BENZALDEHYDE	300.7	23.3	11.60	92
22	DOWTHERM/C1-BIPHENYL	481.0	8.6	28.66	37
23	BIBENZYL	520.8	66.3	32.59	286
24	C1-FLUORENE	578.9	5.7	38.34	25

RRI REPORT

SAMPLE: FD600F938HT5 TOTAL CONCENTRATION = 1268.7
 DISK FILE: 22802 NO. OF INTERPRETED PEAKS = 23
 GC RUN: 4 3 84 TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.80 AREA = 4519340
 ARI = 237% CONC = 998731

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.24 Toluene Degradation Report — 600°F, 1006 h, Run 234

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	92.9	2.46	505
2		206.7	13.0	3.07	71
3	METHYLCYCLOHEXANE	212.4	28.2	3.59	153
4	C2-CYCLOPENTANE	214.7	8.2	3.79	45
5	C2-CYCLOPENTANE	216.3	15.2	3.94	82
7	C2-CYCLOHEXANE	239.1	71.2	6.02	387
8	C2-CYCLOHEXANE	240.3	142.5	6.12	774
9	ISOPROPYLCYCLOPENTANE	241.7	106.9	6.25	581
10		243.2	22.1	6.38	120
11	ETHYLMETHYCYCLOPENTANE	244.1	4.4	6.46	24
12	ETHYLMETHYCYCLOPENTANE	244.8	11.8	6.53	64
13		246.2	48.0	6.65	261
14		247.5	11.0	6.77	60
15		248.3	55.4	6.85	301
16	ETHYLCYCLOHEXANE	249.9	245.2	6.99	1332
17	ETHYLBENZENE	260.0	105.4	7.91	573
18	XYLENE	263.0	167.8	8.18	912
19		272.3	14.3	9.03	67
20	BENZALDEHYDE	300.7	22.3	11.61	105
21	DOWTHERM/C1-BIPHENYL	481.1	8.2	28.65	44
22	BIBENZYL	520.9	63.6	32.58	340
23	C2-BIPHENYL	542.9	3.8	34.75	20
24	C1-FLUORENE	578.9	7.5	38.32	40

RRI REPORT

SAMPLE: FD600F100HT5 TOTAL CONCENTRATION = 1269
 DISK FILE: 23402 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 4 4 84 TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.92 AREA = 5425390
 ARI = 238% CONC = 998731

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.25 Toluene Degradation Report — 650°F, 0 h, Run 224

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	PROPYLETHYLETHER	148.9	6.2	1.86	27
2	BENZENE	200.0	97.3	2.50	419
3	C6H14	201.8	4.9	2.66	21
4	METHYLCYCLOHEXANE	212.7	17.4	3.64	75
7	C8-ALKANE	238.1	32.4	5.93	139
8	C2-CYCLOHEXANE	238.5	51.8	5.97	222
9	C2-CYCLOHEXANE	239.9	156.4	6.09	672
10	ISOPROPYLCYCLOPENTANE	241.4	91.9	6.23	395
11		243.0	22.5	6.37	97
12	ETHYLMETHYCYCLOPENTANE	244.0	6.2	6.46	27
13	ETHYLMETHYCYCLOPENTANE	244.7	8.1	6.53	34
14		246.2	45.7	6.66	197
15		247.5	8.9	6.78	38
16		248.4	51.6	6.86	222
17	ETHYLCYCLOHEXANE	250.0	201.6	7.00	866
18	ETHYLBENZENE	260.4	129.1	7.94	554
19	XYLENE	263.5	269.3	8.22	1157
20		272.9	14.5	9.07	99

RRI REPORT

SAMPLE: FD650F
 DISK FILE: 22402
 GC RUN: 3 25 84

TOTAL CONCENTRATION = 1216
 NO. OF INTERPRETED PEAKS = 19
 TOTAL NO. OF PEAKS = 20

TOLUENE DATA:

RT = 5.69 AREA = 4292290
 ARI = 235% CONC = 998784

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.26 Toluene Degradation Report — 650°F, 19 h, Run 223

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	PROPYLETHYLETHER	149.4	4.8	1.85	27
2	BENZENE	200.0	108.5	2.48	601
3	C2-CYCLOPENTANE	215.0	1.1	3.84	6
8	C2-CYCLOHEXANE	239.2	30.3	6.04	168
9	C2-CYCLOHEXANE	239.5	50.3	6.07	279
10	ISOPROPYLCYCLOPENTANE	240.6	155.5	6.17	862
11	ISOPROPYLCYCLOPENTANE	242.0	103.7	6.30	575
12		243.4	22.4	6.43	124
13	ETHYLMETHYCYCLOPENTANE	244.3	5.9	6.51	33
14	ETHYLMETHYCYCLOPENTANE	245.1	8.0	6.58	44
15		246.4	45.0	6.70	250
16		247.7	8.5	6.82	47
17	ETHYLCYCLOHEXANE	248.6	50.9	6.90	282
18	ETHYLCYCLOHEXANE	250.1	196.3	7.04	1089
19	ETHYLBENZENE	260.3	121.5	7.96	674
20	XYLENE	263.3	253.2	8.24	1404
21		272.6	13.6	9.08	120
22	BENZALDEHYDE	301.0	2.0	11.67	18
23	BIBENZYL	520.8	7.6	32.61	57

RRI REPORT

SAMPLE: FD650F26HT5 TOTAL CONCENTRATION = 1189.4
 DISK FILE: 22302 NO. OF INTERPRETED PEAKS = 20
 GC RUN: 3 25 84 TOTAL NO. OF PEAKS = 23

TOLUENE DATA:

RT = 5.98 AREA = 5537600
 ARI = 239% CONC = 998811

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.27 Toluene Degradation Report — 650°F, 115 h, Run 222

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	199.9	103.3	2.46	664
2	METHYLCYCLOHEXANE	212.6	0.9	3.61	6
3	C2-CYCLOPENTANE	215.0	1.0	3.83	6
7	C2-CYCLOHEXANE	240.3	27.6	6.13	178
8	ISOPROPYLCYCLOPENTANE	240.7	48.3	6.16	311
9	ISOPROPYLCYCLOPENTANE	241.6	156.7	6.25	1007
10		243.0	97.8	6.37	629
11	ETHYLMETHYLCYCLOPENTANE	244.3	22.2	6.49	143
12	ETHYLMETHYLCYCLOPENTANE	245.2	5.2	6.57	33
13		245.9	7.9	6.64	51
14	DIMETHYLCYCLOHEXANE	247.1	44.4	6.75	285
15	ETHYLCYCLOHEXANE	248.5	8.4	6.87	54
16	ETHYLCYCLOHEXANE	249.3	50.4	6.95	323
17		250.8	194.4	7.08	1249
18	ETHYLBENZENE	260.8	118.8	7.99	764
19	XYLENE	263.7	247.6	8.26	1591
20		273.0	13.4	9.10	136
21	BENZALDEHYDE	301.2	2.1	11.67	21
22	BIBENZYL	520.8	19.0	32.59	163

RRI REPORT

SAMPLE: FD650F123HT6 TOTAL CONCENTRATION = 1169.5
 DISK FILE: 22202 NO. OF INTERPRETED PEAKS = 20
 GC RUN: 3 25 84 TOTAL NO. OF PEAKS = 22

TOLUENE DATA:

RT = 6.08 AREA = 6419530
 ARI = 240% CONC = 998830

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.28 Toluene Degradation Report — 650°F, 282 h, Run 312

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		101.7	1.9	1.24	8
2	BENZENE	200.0	97.7	2.43	392
3	METHYLCYCLOHEXANE	212.5	13.7	3.56	55
4	C2-CYCLOPENTANE	215.2	1.9	3.82	8
8	C8-ALKANE	237.3	33.1	5.82	133
9	C8-ALKANE	237.7	53.0	5.86	213
10	C8-ALKANE	238.4	10.1	5.92	40
11	C2-CYCLOHEXANE	239.1	145.8	5.99	585
12	ISOPROPYLCYCLOPENTANE	240.6	91.3	6.13	366
13	ISOPROPYLCYCLOPENTANE	242.2	22.4	6.27	90
14		243.2	5.2	6.37	21
15	ETHYLMETHYLCYCLOPENTANE	244.0	8.1	6.44	32
16	ETHYLMETHYLCYCLOPENTANE	245.4	44.6	6.57	179
17	DIMETHYLCYCLOHEXANE	246.8	8.9	6.69	35
18		247.7	50.7	6.77	203
19	ETHYLCYCLOHEXANE	249.3	198.3	6.92	796
20	ETHYLBENZENE	259.6	125.1	7.86	502
21	XYLENE	262.7	263.1	8.14	1056
22		272.1	22.7	9.00	91
23	BENZALDEHYDE	300.7	4.9	11.60	17
24	BIBENZYL	520.9	65.2	32.59	249

RRI REPORT

SAMPLE: FD650F290HT6 TOTAL CONCENTRATION = 1267.5
 DISK FILE: 31202 NO. OF INTERPRETED PEAKS = 22
 GC RUN: 4 15 84 TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.75 AREA = 4006900
 ARI = 236% CONC = 998733

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.29 Toluene Degradation Report — 650°F, 282 h, Run 226

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	99.4	2.44	552
2	METHYLCYCLOHEXANE	212.5	11.7	3.58	65
3	C2-CYCLOPENTANE	215.0	2.8	3.80	16
7	C2-CYCLOHEXANE	239.1	30.6	6.00	170
8	C2-CYCLOHEXANE	239.4	50.9	6.03	283
9	ISOPROPYLCYCLOPENTANE	240.5	155.6	6.13	864
10	ISOPROPYLCYCLOPENTANE	241.9	91.5	6.26	508
11		243.4	22.4	6.39	125
12	ETHYLMETHYLCYCLOPENTANE	244.3	5.0	6.47	28
13	ETHYLMETHYLCYCLOPENTANE	245.0	8.1	6.54	45
14		246.4	44.9	6.66	249
15		247.7	8.6	6.78	48
16	ETHYLCYCLOHEXANE	248.5	51.1	6.86	284
17	ETHYLCYCLOHEXANE	250.1	198.8	7.00	1103
18	ETHYLBENZENE	260.2	123.2	7.91	683
19	XYLENE	263.2	258.1	8.19	1432
20		272.5	25.1	9.04	123
21	BENZALDEHYDE	301.0	4.6	11.62	23
22	BIBENZYL	520.9	52.0	32.58	287
23		712.5	6.4	49.75	37

RRI REPORT

SAMPLE: FD600F290HT6 TOTAL CONCENTRATION = 1251
 DISK FILE: 22602 NO. OF INTERPRETED PEAKS = 21
 GC RUN: 4 1 86 TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.73 AREA = 5541780
 ARI = 236% CONC = 998749

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.30 Toluene Degradation Report — 650°F, 450 h, Run 240

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	96.4	2.44	462
2	C2-CYCLOPENTANE	215.0	1.0	3.81	5
6	C2-CYCLOHEXANE	238.6	30.7	5.96	147
7	C2-CYCLOHEXANE	239.0	50.6	5.99	242
8	C2-CYCLOHEXANE	240.2	153.6	6.10	735
9	ISOPROPYLCYCLOPENTANE	241.6	89.5	6.23	428
10		243.1	22.0	6.36	105
11	ETHYLMETHYCYCLOPENTANE	244.0	4.9	6.45	24
12	ETHYLMETHYCYCLOPENTANE	244.8	7.9	6.52	38
13		246.1	43.5	6.64	208
14		247.5	8.5	6.76	41
15		248.3	49.8	6.84	239
16	ETHYLCYCLOHEXANE	249.9	195.7	6.98	937
17	ETHYLBENZENE	260.0	123.1	7.90	589
18	XYLENE	263.1	259.2	8.18	1241
19		272.4	26.4	9.03	107
20	BENZALDEHYDE	300.8	5.4	11.61	22
21	C1-BIPHENYL	512.2	4.0	31.72	18
22	BIBENZYL	520.8	85.5	32.58	386
23	BIBENZYL	521.9	6.1	32.68	27
24	C2-BIPHENYL	542.7	5.2	34.74	23
25	C2-BIPHENYL	546.1	4.8	35.08	22
26	C1-FLUORENE	578.8	4.9	38.31	22

RRI REPORT

SAMPLE: FD650F457HT6 TOTAL CONCENTRATION = 1278.7
 DISK FILE: 24002 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 4 8 84 TOTAL NO. OF PEAKS = 26

TOLUENE DATA:

RT = 5.75 AREA = 4780850
 ARI = 236% CONC = 998721

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.31 Toluene Degradation Report — 650°F, 619 h, Run 311

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	93.5	2.45	487
2	C2-CYCLOPENTANE	214.9	0.5	3.81	2
3	C2-CYCLOPENTANE	216.5	1.3	3.95	7
7	C2-CYCLOHEXANE	238.7	30.7	5.97	160
8	C2-CYCLOHEXANE	239.0	51.3	6.00	268
9	C2-CYCLOHEXANE	240.2	155.0	6.10	808
10	ISOPROPYLCYCLOPENTANE	241.6	89.8	6.23	469
11		243.1	22.2	6.37	116
12	ETHYLMETHYCYCLOPENTANE	244.0	4.9	6.45	26
13	ETHYLMETHYCYCLOPENTANE	244.7	8.0	6.52	42
14		246.1	43.7	6.64	228
15	DIMETHYLCYCLOHEXANE	247.4	8.6	6.76	45
16		248.3	50.2	6.84	261
17	ETHYLCYCLOHEXANE	249.8	196.7	6.98	1026
18	ETHYLBENZENE	259.9	122.1	7.90	636
19	XYLENE	263.0	256.5	8.18	1337
20		272.3	25.7	9.03	115
21	BENZALDEHYDE	300.8	5.9	11.61	27
22	DIPHENYLMETHANE	491.5	4.5	29.68	23
23	C1-BIPHENYL	512.1	4.4	31.72	22
24	BIBENZYL	520.9	93.9	32.59	466
25	C2-BIPHENYL	542.7	5.9	34.75	29
26	C2-BIPHENYL	546.0	5.7	35.08	28
27	C1-FLUORENE	578.7	12.2	38.31	61

RRI REPORT

SAMPLE: FD650F627HT6 TOTAL CONCENTRATION = 1293.5
 DISK FILE: 31102 NO. OF INTERPRETED PEAKS = 25
 GC RUN: 4 15 84 TOTAL NO. OF PEAKS = 27

TOLUENE DATA:

RT = 5.88 AREA = 5205750
 ARI = 238% CONC = 998705

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.32 Toluene Degradation Report — 650°F, 786 h, Run 323

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	89.4	2.45	489
2	METHYLCYCLOHEXANE	212.4	7.1	3.57	39
3	C2-CYCLOPENTANE	214.8	0.7	3.79	4
6	C2-CYCLOHEXANE	238.6	30.8	5.95	168
7	C2-CYCLOHEXANE	238.9	51.3	5.99	280
8	C2-CYCLOHEXANE	240.1	153.9	6.09	842
9	ISOPROPYLCYCLOPENTANE	241.5	88.7	6.22	486
10		243.0	22.0	6.36	120
11	ETHYLMETHYLCYCLOPENTANE	243.9	4.9	6.44	27
12	ETHYLMETHYLCYCLOPENTANE	244.7	8.0	6.51	44
13		246.1	43.1	6.63	235
14	DIMETHYLCYCLOHEXANE	247.4	8.5	6.75	47
15		248.2	49.7	6.83	272
16	ETHYLCYCLOHEXANE	249.8	195.7	6.97	1071
17	ETHYLBENZENE	259.9	122.4	7.89	670
18	XYLENE	263.0	258.2	8.17	1413
19		272.3	26.2	9.02	122
20	BENZALDEHYDE	300.8	7.4	11.60	35
21	DIPHENYLMETHANE	491.5	6.3	29.67	33
22	C1-BIPHENYL	512.2	6.0	31.71	31
23	BIBENZYL	520.9	128.5	32.57	661
24	C1-DIPHENYLMETHANE	523.0	4.6	32.78	24
25	C1-DIPHENYLMETHANE	526.8	3.6	33.16	19
26	C2-BIPHENYL	542.7	8.4	34.73	43
27	C2-BIPHENYL	546.0	8.2	35.06	42
28	C3-BIPHENYL	561.9	4.7	36.63	24
29	C1-FLUORENE	578.8	16.9	38.30	87

RRI REPORT

SAMPLE: FD650F794HT6 TOTAL CONCENTRATION = 1355.1
 DISK FILE: 32302 NO. OF INTERPRETED PEAKS = 28
 GC RUN: 4 23 86 TOTAL NO. OF PEAKS = 29

TOLUENE DATA:

RT = 5.89 AREA = 5465480
 ARI = 238% CONC = 998645

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.33 Toluene Degradation Report — 650°F, 955 h, Run 334

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	104.0	2.45	473
2	METHYLCYCLOHEXANE	212.4	13.2	3.57	60
3	C2-CYCLOPENTANE	215.0	1.8	3.81	8
4	C2-CYCLOPENTANE	216.3	0.7	3.93	3
7	C8-ALKANE	237.4	32.6	5.85	149
8	C8-ALKANE	237.8	52.4	5.89	239
9	C2-CYCLOHEXANE	238.5	9.8	5.94	45
10	C2-CYCLOHEXANE	239.2	143.6	6.01	654
11	ISOPROPYLCYCLOPENTANE	240.7	88.0	6.14	401
12	ISOPROPYLCYCLOPENTANE	242.2	21.7	6.29	99
13		243.3	5.0	6.38	23
14	ETHYLMETHYLCYCLOPENTANE	244.0	7.8	6.45	36
15	ETHYLMETHYLCYCLOPENTANE	245.4	42.4	6.57	193
16	DIMETHYLCYCLOHEXANE	246.8	8.7	6.70	39
17		247.7	49.1	6.78	224
18	ETHYLCYCLOHEXANE	249.2	194.3	6.92	885
19	ETHYLBENZENE	259.5	119.2	7.86	543
20	XYLENE	262.6	253.5	8.14	1155
21		272.0	21.5	8.99	98
22	BENZALDEHYDE	300.6	8.3	11.59	31
23	DIPHENYLMETHANE	491.5	7.7	29.67	32
24	C1-BIPHENYL	512.1	7.6	31.71	31
25	BIBENZYL	520.9	153.2	32.57	635
26	C1-DIPHENYLMETHANE	523.1	5.6	32.79	24
27	C1-DIPHENYLMETHANE	526.8	4.5	33.16	19
28	C2-BIPHENYL	542.8	10.0	34.74	42
29	C2-BIPHENYL	546.1	9.6	35.06	40
30	C3-BIPHENYL	561.9	5.5	36.63	23
31	C1-FLUORENE	578.8	19.9	38.30	83

RRI REPORT

SAMPLE: FD650962HT6 TOTAL CONCENTRATION = 1401.5
 DISK FILE: 33402 NO. OF INTERPRETED PEAKS = 30
 GC RUN: 4 29 86 TOTAL NO. OF PEAKS = 31

TOLUENE DATA:

RT = 5.77 AREA = 4548180
 ARI = 237% CONC = 998598

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.34 Toluene Degradation Report — 650°F, 1006 h, Run 378

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		85.3	12.8	1.02	69
2	BENZENE	200.0	91.5	2.39	494
3	METHYLCYCLOHEXANE	212.5	15.7	3.52	85
4	C2-CYCLOPENTANE	214.7	6.9	3.72	37
6	ISOPROPYLCYCLOPENTANE	240.8	120.1	6.08	648
7	ISOPROPYLCYCLOPENTANE	242.1	79.6	6.20	430
8	ETHYLMETHYLCYCLOPENTANE	243.5	17.9	6.32	96
9	ETHYLMETHYLCYCLOPENTANE	245.1	9.9	6.47	53
10		246.4	41.2	6.58	223
11	ETHYLCYCLOHEXANE	248.5	56.3	6.78	304
12	ETHYLCYCLOHEXANE	250.0	190.9	6.91	1031
13	ETHYLBENZENE	260.0	121.2	7.81	654
14	XYLENE	263.0	257.2	8.09	1388
15		272.2	22.6	8.92	122
16	BENZALDEHYDE	300.6	12.0	11.48	55
17	DIPHENYLMETHANE	491.5	7.5	29.50	37
18	C1-BIPHENYL	512.1	7.7	31.55	35
19	BIBENZYL	520.9	165.5	32.41	758
20	C1-DIPHENYLMETHANE	526.7	5.0	32.99	23
21	C2-BIPHENYL	542.7	10.2	34.57	47
22	C2-BIPHENYL	546.0	9.8	34.90	45
23	C3-BIPHENYL	561.9	5.4	36.46	25
24	C1-FLUORENE	578.8	4.9	38.14	23

RRI REPORT

SAMPLE: FD650F1013HT6 TOTAL CONCENTRATION = 1272
 DISK FILE: 37801 NO. OF INTERPRETED PEAKS = 24
 GC RUN: 12 3 86 TOTAL NO. OF PEAKS = 24

TOLUENE DATA:

RT = 5.89 AREA = 5391270
 ARI = 239% CONC = 998728

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.35 Toluene Degradation Report — 677°F, 0 h, Run 351

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	83.9	2.40	426
2		201.5	85.2	2.53	433
3	C6H14	205.3	15.6	2.88	79
4	C6H14	206.4	11.9	2.98	60
7	C2-CYCLOHEXANE	238.7	15.4	5.87	78
8	C2-CYCLOHEXANE	239.0	20.9	5.90	106
9	C2-CYCLOHEXANE	240.2	128.5	6.00	653
10	ISOPROPYLCYCLOPENTANE	241.7	185.2	6.14	941
11		243.1	28.2	6.27	143
12	ETHYLMETHYCYCLOPENTANE	244.1	4.1	6.35	21
13	ETHYLMETHYCYCLOPENTANE	244.8	26.0	6.42	132
14		246.2	72.6	6.54	369
15		247.5	22.9	6.66	116
16		248.4	89.4	6.74	454
17	ETHYLCYCLOHEXANE	250.0	472.1	6.89	2399
18	ETHYLBENZENE	260.1	101.1	7.79	514
19	XYLENE	263.2	89.5	8.07	455

RRI REPORT

SAMPLE: FD677FOHT7 TOTAL CONCENTRATION = 1452.5
 DISK FILE: 35102 NO. OF INTERPRETED PEAKS = 18
 GC RUN: 6 12 86 TOTAL NO. OF PEAKS = 19

TOLUENE DATA:

RT = 5.81 AREA = 5073450
 ARI = 238% CONC = 998547

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.36 Toluene Degradation Report — 677°F, 117 h, Run 352

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		99.7	2.5	1.21	17
2		108.7	5.1	1.32	35
3	BENZENE	200.0	82.0	2.43	551
4	C6H14	203.3	0.6	2.73	4
5		206.7	37.6	3.03	252
6	METHYLCYCLOHEXANE	212.1	16.2	3.52	109
13	C2-CYCLOHEXANE	240.2	12.9	6.05	87
14	C2-CYCLOHEXANE	240.4	21.6	6.07	145
15	ISOPROPYLCYCLOPENTANE	241.4	126.3	6.16	849
16		242.7	183.1	6.28	1231
17	ETHYLMETHYCYCLOPENTANE	244.0	27.9	6.39	188
18	ETHYLMETHYCYCLOPENTANE	244.8	3.9	6.47	26
19		245.5	25.6	6.53	172
20	DIMETHYLCYCLOHEXANE	246.8	71.2	6.65	478
21		248.1	22.6	6.76	152
22		248.9	88.6	6.84	595
23	ETHYLCYCLOHEXANE	250.5	472.6	6.98	3176
24		256.5	2.1	7.52	14
25		258.2	3.1	7.67	21
26	ETHYLBENZENE	260.2	106.8	7.85	718
27	XYLENE	263.2	92.0	8.12	618
28		272.4	3.0	8.95	21
29	BENZALDEHYDE	300.7	2.1	11.50	14
30	BIBENZYL	520.9	37.1	32.35	273
31	C2-BIPHENYL	542.8	2.4	34.51	18

RRI REPORT

SAMPLE: FD677F117HT7 TOTAL CONCENTRATION = 1449
 DISK FILE: 35202 NO. OF INTERPRETED PEAKS = 26
 GC RUN: 6 12 86 TOTAL NO. OF PEAKS = 31

TOLUENE DATA:

RT = 6.00 AREA = 6711240
 ARI = 240% CONC = 998550

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.37 Toluene Degradation Report — 677°F, 285 h, Run 353

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1		110.8	3.4	1.34	25
2		135.5	3.6	1.63	27
3	BENZENE	200.0	82.5	2.41	618
4		206.6	18.7	3.01	139
5	METHYLCYCLOHEXANE	212.3	21.5	3.52	161
14	ISOPROPYLCYCLOPENTANE	241.1	14.2	6.12	106
15	ISOPROPYLCYCLOPENTANE	241.4	20.4	6.14	153
16	ISOPROPYLCYCLOPENTANE	242.2	125.1	6.22	936
17		243.4	180.7	6.33	1353
18	ETHYLMETHYLCYCLOPENTANE	244.7	27.8	6.44	208
19		245.5	3.5	6.51	26
20		246.2	25.3	6.58	189
21	DIMETHYLCYCLOHEXANE	247.4	69.6	6.69	521
22		248.7	22.3	6.80	167
23		249.5	87.1	6.87	652
24	ETHYLCYCLOHEXANE	251.0	469.6	7.01	3517
25		256.9	1.9	7.54	15
26	ETHYLBENZENE	258.6	3.0	7.70	22
27	ETHYLBENZENE	260.6	103.4	7.88	774
28	XYLENE	261.8	2.9	7.98	22
29	XYLENE	263.6	91.5	8.14	685
30		272.7	3.3	8.97	25
31	BENZALDEHYDE	300.8	2.6	11.50	19
32	DIPHENYLMETHANE	491.4	3.0	29.46	25
33	BIBENZYL	520.9	60.6	32.36	496
34	C2-BIPHENYL	542.8	4.4	34.52	35
35	C2-BIPHENYL	546.1	4.2	34.84	34
36	C1-FLUORENE	578.8	3.8	38.07	31

RRI REPORT

SAMPLE: FD677F283HT7 TOTAL CONCENTRATION = 1459.7
 DISK FILE: 35302 NO. OF INTERPRETED PEAKS = 29
 GC RUN: 6 12 86 TOTAL NO. OF PEAKS = 36

TOLUENE DATA:

RT = 6.08 AREA = 7476760
 ARI = 241% CONC = 998540

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.38 Toluene Degradation Report — 677°F, 600 h, Run 363

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	75.9	2.43	416
5	C2-CYCLOHEXANE	239.7	14.8	6.01	81
6	C2-CYCLOHEXANE	240.1	23.0	6.04	126
7	ISOPROPYLCYCLOPENTANE	241.1	127.2	6.13	697
8	ISOPROPYLCYCLOPENTANE	242.4	176.5	6.26	968
9	ETHYLMETHYLCYCLOPENTANE	243.8	27.5	6.38	151
10	ETHYLMETHYLCYCLOPENTANE	244.7	3.6	6.46	20
11	ETHYLMETHYLCYCLOPENTANE	245.4	25.4	6.52	139
12	DIMETHYLCYCLOHEXANE	246.7	67.4	6.64	369
13		248.0	22.3	6.76	122
14		248.9	86.0	6.84	471
15	ETHYLCYCLOHEXANE	250.5	472.2	6.98	2589
16	ETHYLBENZENE	260.3	103.3	7.87	567
17	XYLENE	263.3	90.3	8.14	495
18		272.5	3.5	8.97	19
19	BENZALDEHYDE	300.8	4.1	11.52	20
20	DIPHENYLMETHANE	491.5	6.5	29.48	36
21	C1-BIPHENYL	512.2	6.9	31.52	38
22	BIBENZYL	521.0	120.7	32.39	673
23	C2-BIPHENYL	542.8	9.4	34.54	52
24	C2-BIPHENYL	546.1	9.0	34.86	50
25	C1-FLUORENE	578.8	4.7	38.09	26

RRI REPORT

SAMPLE: FD677F599HT7 TOTAL CONCENTRATION = 1480
 DISK FILE: 36302 NO. OF INTERPRETED PEAKS = 23
 GC RUN: 7 8 86 TOTAL NO. OF PEAKS = 25

TOLUENE DATA:

RT = 5.62 AREA = 5474350
 ARI = 235% CONC = 998520

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.39 Toluene Degradation Report — 677°F, 789 h, Run 362

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	73.8	2.43	313
2		206.9	40.2	3.05	170
3	METHYLCYCLOHEXANE	212.7	62.1	3.57	263
7	C8-ALKANE	237.9	16.0	5.85	68
8	C8-ALKANE	238.3	23.8	5.88	101
9	C2-CYCLOHEXANE	239.5	128.8	5.99	546
10	ISOPROPYLCYCLOPENTANE	241.1	174.7	6.13	741
11		242.6	27.5	6.27	117
12	ETHYLMETHYLCYCLOPENTANE	243.6	3.5	6.36	15
13	ETHYLMETHYLCYCLOPENTANE	244.3	25.5	6.42	108
14		245.7	65.9	6.55	280
15	DIMETHYLCYCLOHEXANE	247.0	21.7	6.67	92
16		247.9	84.3	6.75	358
17	ETHYLCYCLOHEXANE	249.6	468.0	6.90	1985
18	ETHYLBENZENE	259.7	103.6	7.81	440
19	XYLENE	262.7	90.7	8.08	385
20	BENZALDEHYDE	300.6	4.6	11.49	17
21	DIPHENYLMETHANE	491.5	9.1	29.48	38
22	C1-BIPHENYL	512.2	9.9	31.52	43
23	C1-DIPHENYLMETHANE	516.6	4.3	31.95	19
24	BIBENZYL	521.0	163.2	32.38	704
25	C1-DIPHENYLMETHANE	523.1	5.4	32.59	23
26	C2-BIPHENYL	542.8	14.0	34.53	60
27	C2-BIPHENYL	546.1	13.3	34.86	58
28	C3-BIPHENYL	561.9	5.4	36.42	23
29	C1-FLUORENE	578.8	8.5	38.08	36

RRI REPORT

SAMPLE: FD677F787HT7 TOTAL CONCENTRATION = 1647.5
 DISK FILE: 36202 NO. OF INTERPRETED PEAKS = 27
 GC RUN: 7 8 86 TOTAL NO. OF PEAKS = 29

TOLUENE DATA:

RT = 5.78 AREA = 4235120
 ARI = 237% CONC = 998352

^aArea is in a unit that is calibrated when the standard sample is analyzed.

TABLE D.40 Toluene Degradation Report — 677°F, 958 h, Run 364

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	76.9	2.39	305
2		206.8	39.6	3.01	157
3	METHYLCYCLOHEXANE	212.4	46.9	3.51	186
7	C8-ALKANE	237.4	16.4	5.77	65
8	C8-ALKANE	237.9	24.4	5.81	97
9	C2-CYCLOHEXANE	239.1	129.1	5.92	513
10	ISOPROPYLCYCLOPENTANE	240.7	173.3	6.07	688
11	ISOPROPYLCYCLOPENTANE	242.3	27.4	6.21	109
12		243.3	3.5	6.30	14
13	ETHYLMETHYCYCLOPENTANE	244.0	25.5	6.36	101
14	ETHYLMETHYCYCLOPENTANE	245.4	64.9	6.49	258
15	DIMETHYLCYCLOHEXANE	246.7	21.6	6.61	86
16		247.6	83.5	6.69	331
17	ETHYLCYCLOHEXANE	249.3	466.1	6.84	1850
18	ETHYLBENZENE	259.5	102.9	7.76	409
19	XYLENE	262.5	90.8	8.04	361
20	BENZALDEHYDE	300.5	5.1	11.46	18
21	DIPHENYLMETHANE	491.6	12.0	29.47	48
22	C1-BIPHENYL	512.3	12.9	31.51	52
23	C1-DIPHENYLMETHANE	516.6	5.5	31.94	22
24	BIBENZYL	521.0	202.8	32.38	818
25	C1-DIPHENYLMETHANE	523.1	7.0	32.58	28
26	C1-DIPHENYLMETHANE	526.9	5.4	32.95	22
27	C2-BIPHENYL	542.8	17.8	34.53	72
28	C2-BIPHENYL	546.1	17.0	34.85	68
29	C3-BIPHENYL	562.0	6.8	36.42	27
30	C1-FLUORENE	578.8	10.6	38.08	43

RRI REPORT

SAMPLE: FD677F956HT7 TOTAL CONCENTRATION = 1696
 DISK FILE: 36402 NO. OF INTERPRETED PEAKS = 28
 GC RUN: 7 8 86 TOTAL NO. OF PEAKS = 30

TOLUENE DATA:

RT = 5.70 AREA = 3963260
 ARI = 237% CONC = 998304

^aArea is in a unit that is calibrated when the standard sample is analyzed.

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TABLE D.41 Toluene Degradation Report — 677°F, 1006 h, Run 376

Peak No.	Name	Aromatic Retention Index	Conc. (ppm)	Ret. Time (min)	Area ^a
1	BENZENE	200.0	72.4	2.36	320
2		206.8	37.5	2.97	166
3	METHYLCYCLOHEXANE	212.6	60.1	3.50	266
6	C2-CYCLOHEXANE	239.8	108.9	5.97	482
7	ISOPROPYLCYCLOPENTANE	241.3	166.1	6.10	735
8		242.8	24.5	6.24	108
9	ETHYLMETHYCYCLOPENTANE	244.5	26.9	6.39	119
10		245.9	65.5	6.51	290
11		248.1	107.3	6.71	475
12	ETHYLCYCLOHEXANE	249.7	468.2	6.86	2072
13	ETHYLBENZENE	259.8	105.9	7.77	469
14	XYLENE	262.8	93.5	8.04	413
15		272.2	4.3	8.89	19
16	BENZALDEHYDE	300.7	10.6	11.47	40
17	DIPHENYLMETHANE	491.5	9.6	29.51	39
18	C1-BIPHENYL	512.2	11.6	31.55	43
19	BIBENZYL	520.9	178.8	32.41	670
20	C1-DIPHENYLMETHANE	523.0	6.2	32.62	23
21	C2-BIPHENYL	542.8	15.0	34.57	56
22	C2-BIPHENYL	546.1	14.2	34.90	53

RRI REPORT

SAMPLE: FD677F1005HT7 TOTAL CONCENTRATION = 1587
 DISK FILE: 37601 NO. OF INTERPRETED PEAKS = 21
 GC RUN: 12 3 86 TOTAL NO. OF PEAKS = 22

TOLUENE DATA:

RT = 5.76 AREA = 4418300
 ARI = 238% CONC = 998413

^aArea is in a unit that is calibrated when the standard sample is analyzed.

APPENDIX E:
COMPUTER PROGRAM AND PREDICTED DEGRADATION

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COMPUTER PROGRAM AND PREDICTED DEGRADATION

The computer program that follows runs on an Apple II+ computer. The constants R_0 and K in line 50010 were determined empirically.* The ONERR GOTO 1000 instruction in line 40 causes control to be transferred to line 1000 when the end of the input file (or any other error) is detected. The POKE 216,0 instruction in line 1000 re-establishes normal error-detection procedures. An input data file consists of the following lines:

Line 1: Informational header

Line 2: Duration of operation (h), cross-sectional area of the fluid passageways (in.^2), fluid inventory (lb)

Lines 3...n: Distance from datum (in.), fluid temperature ($^{\circ}\text{R}$), fluid density ($\text{lb}_m/\text{in.}^3$)

```

10  GOSUB 50000: PRINT D$;"PR#1": PRINT D$;"OPEN";FI$: PRINT D$;"READ";FI$
20  INPUT H$: PRINT H$: PRINT
30  INPUT TE,A,M:C4 = TE * R0 * A / M: PRINT "DURATION = ";TE: PRINT
    "CROSS-SECTIONAL AREA = ";A: PRINT "FLUID WEIGHT = ";M: PRINT
35  PRINT "          X          TEMP          DENSITY          INTEGRAL"
40  INPUT X1,T1,D1:Z = X1: GOSUB 40000:Z = T1: GOSUB 40000:Z = D1:
    GOSUB 40000: PRINT : ONERR GOTO 1000
50  INPUT X2,T2,D2:Z = X2: GOSUB 40000:Z = T2: GOSUB 40000:Z = D2: GOSUB
    40000
60  IF T1 > T2 THEN 100
70  DI = C4 * EXP ( - K / T2) * (D1 + D2) * (X2 - X1) / 2: PRINT " ";DI
80  GOTO 150
100 C1 = K * (D1 - ((D2 - D1) / (T2 - T1)) * T1):C2 = K * K * (D2 - D1) /
    (T2 - T1) / 2:C3 = C4 * (X2 - X1) / (T2 - T1)
110 DI = C3 * ( FN F(K / T2) - FN F(K / T1)): PRINT " ";DI
150 II = II + DI:X1 = X2:T1 = T2:D1 = D2: GOTO 50
1000 POKE 216,0: PRINT "          INTEGRAL SUM ";:
    PRINT " ";II: PRINT D$;"CLOSE": PRINT D$;"PR#0": END
39999 REM PRINT FORMATTING
40000 PRINT RIGHT$ ("          " + LEFT$ ( STR$ (Z),10),12);: RETURN
49999 REM INITIALIZATION
50000 D$ = CHR$ (4): INPUT "FILE NAME: ";FI$
50010 R0 = 9.9917E9:K = 23056:II = 0

```

*Because the computer program cannot include subscript symbols, the constant denoted by " R_0 " in the text is represented by "R0" in the program.

```

50020  A1 = .99997:A2 = 3.03962:B1 = 5.03637:B2 = 4.19160: DEF  FN XE(X) =
      (A1 * X + A2) / ((X + B1) * X + B2): REM FUNCTION IS
      1.0-X*EXP(X)*E1(X)
50021  REM SEE ABRAMOWITZ & STEGUN, PAGE 231, EQ'N 5.1.55.
50030  DEF  FN F(X) = ((C1 - C2) * FN XE(X) + C2 / X) * EXP ( - X) / X
50040  RETURN

```

The following variables are used in the program:

A	Cross-sectional area of the fluid passageway
A1, A2, B1, B2	Constants used to compute the exponential integral. (See Abramowitz and Stegun, p. 231.)
C1, C2, C3, C4	Constants computed from input data
D\$	Control-D, used by Apple computer to issue disk commands
D1, D2	Fluid density at points 1 and 2
DI	Integral of predicted degradation from point 1 to point 2
FI\$	Input file name
H\$	Informational heading
II	Total predicted degradation
K	Constant of the Arrhenius equation
M	Mass inventory of fluid in the system
R0	Constant of the Arrhenius equation
T1, T2	Temperature at points 1 and 2
TE	Duration of operation
X	Dummy variable used to define functions
X1, X2	Locations of points 1 and 2 relative to datum
Z	Temporary variable

Computations of predicted degradation are shown below.

635 DEGREE TEST

DURATION = 840
 CROSS-SECTIONAL AREA = 0.0731
 FLUID WEIGHT = 11.5

X	TEMP	DENSITY	INTEGRAL
0	943.5	9.968E-04	
98	1038.6	8.561E-04	0.445023739
148	1078.9	8.118E-04	0.793262208
257	1135.8	7.57E-04	4.29078119
307	1138.9	7.54300001	3.16663015
518	1143.5	7.502E-04	14.246291
564	1141.4	7.519E-04	3.16964631
662	1141	7.524E-04	6.6145264
708	1140	7.533E-04	3.06937864
963	1140	7.533E-04	16.874501
1095	1095.4	7.948E-04	6.12679581
1096	1085.8	8.206E-05	0.0157792727
1130	1085.8	8.206E-05	0.0893080269
		INTEGRAL SUM	58.9019238

Measured degradation is 76.3 ppm. Error in the prediction is -23%.

600 DEGREE TEST

DURATION = 1006

CROSS-SECTIONAL AREA = 0.0731

FLUID WEIGHT = 11.4

X	TEMP	DENSITY	INTEGRAL
0	809.2	0.02596	
98	970.6	0.02107	1.56853069
148	1008.8	0.01926	5.08047858
190	1037.4	0.0169	8.05885903
495	1037.4	4.262E-03	46.3416608
518	1046.1	4.055E-03	1.50795385
564	1053.4	3.889E-03	3.40928182
662	1060.5	3.132E-03	7.44225191
708	1063.5	3.695E-03	3.77325898
963	1062	3.714E-03	23.050653
1095	1059.7	3.745E-03	11.5552069
1096	977.2	8.895E-05	0.0262217618
1130	977.2	8.895E-05	0.0110447528
INTEGRAL SUM			111.825402

Measured degradation is 105.4 ppm. Error in the prediction is +6%.

650 DEGREE TEST

DURATION = 1006

CROSS-SECTIONAL AREA = 0.0731

FLUID WEIGHT = 11

X	TEMP	DENSITY	INTEGRAL
0	845.9	0.02503	
98	1029.7	0.01773	5.45126037
100	1038.6	0.01677	0.479086155
220	1038.6	4.342E-03	19.3414882
257	1063.5	3.749E-03	2.98472557
307	1090.3	3.418E-03	6.06538151
518	1119.4	3.106E-03	40.0612632
564	1119.8	3.104E-03	10.8676496
662	1121.2	3.094E-03	23.4940566
708	1121.3	3.093E-03	11.1607982
963	1121	3.096E-03	61.7761491
1095	1109.7	3.178E-03	29.1640439
1096	1044.2	8.571E-05	0.0701111439
1130	1044.2	8.571E-05	0.0501214574
INTEGRAL SUM			210.966135

Measured degradation is 228.0 ppm. Error in the prediction is -7%.

677 DEGREE TEST

DURATION = 1006

CROSS-SECTIONAL AREA = 0.0731

FLUID WEIGHT = 11.5

X	TEMP	DENSITY	INTEGRAL
0	857.5	0.02472	
98	1020.4	0.01845	4.84511975
106	1039.3	0.01668	1.7047221
228	1039.3	4.392E-03	19.0560628
257	1058.3	3.873E-03	2.17632502
307	1084.5	3.52E-03	5.35991704
518	1136.1	3.008E-03	43.1309723
564	1139.8	2.981E-03	13.9753493
662	1114	2.95E-03	24.3785347
708	1146	2.935E-03	12.0533928
963	1150	2.905E-03	90.2068855
1095	1137.5	2.998E-03	43.8539656
1096	1076.4	8.281E-05	0.109463765
1130	1076.4	8.281E-05	0.0896659795
INTEGRAL SUM			260.940377

Measured degradation is 246.0 ppm. Error in the prediction is +6%.

SUNDSTRAND/NASA 750-DEGREE LOOP TEST

DURATION = 2296

CROSS-SECTIONAL AREA = 0.1452

FLUID WEIGHT = 36

X	TEMP	DENSITY	INTEGRAL
0	760	0.02718	
34	840	0.02525	0.0332614046
137	990	0.02052	4.09920671
272	1067	0.01405	41.8336167
413	1100	6.859E-03	76.5689867
565	1155	3.915E-03	98.8517053
721	1178	3.666E-03	143.143812
820	1193	3.529E-03	118.1031
884	1203	3.438E-03	90.4842306
927	1210	3.374E-03	68.050629
INTEGRAL SUM			641.168548

Measured degradation is 1212.9 ppm. Error in the prediction is -47%.