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THE PRODUCTS OF FLASH PYROLYSIS OF PHENOL-FORMALDEHYDE BY TIME-OF-FLIGHT MASS SPECTROSCOPY

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AEROPHYSICS OPERATION

(6) THE PRODUCTS OF FLASH PYROLYSIS OF PHENOL-FORMALDEHYDE BY TIME-OF-FLIGHT MASS SPECTROSCOPY*

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ABSTRACT

A sample of phenol-formaldehyde was flash pyrolyzed eleven times in the reaction chamber of a time-of-flight mass spectrometer. Quantitative analyses were determined for pyrolysis products which were present within ten seconds after each flash. A high degree of consistency between compositions of products from consecutive flashes resulted. A discussion of the results is presented.

I. INTRODUCTION

As plastics undergo ablation, pyrolysis products are injected into the boundary layer between the plastic and the environment. Material and energy transfer during ablation depends, in part, on the energy that is associated with the decomposition process, and the enthalpy of the decomposition gases. Many studies have been carried out wherein the decomposition products were analyzed by techniques which were suitable only for handling stable products. Under some conditions, it is quite probable that a considerable quantity of free radicals, atoms, or unstable complex molecules are evolved. Such species may have a marked influence on material and energy transfer.

Kistiakowsky and co-workers have used a time-of-flight mass spectrometer to study the intermediates of several reactions. Kistiakowsky and Kydd[1] flash photolyzed ketene and nitrogen dioxide in the reaction chamber of the mass spectrometer. Bradley and Kistiakowsky studied the thermal decomposition of nitrous oxide[2] and the polymerization and oxidation of acetylene[3] by shock waves, where the mass spectrometer leak was at the end of the driven section of the shock tube. Nelson and co-workers[4-6] have demonstrated that plastics may be heated to very high temperatures by radiation from xenon filled discharge tubes. Therefore, it seemed appropriate to attempt to study the products of flash pyrolysis of plastics in the reaction chamber of a time-of-flight mass spectrometer in order to observe compositions which might simulate those that are

present during ablation. While techniques for studying transitory products are still being developed, it has been possible to determine quantitative analyses for the more stable products which are present about ten seconds after the flash. This paper describes only the work which has been performed for the more stable products.

II. EXPERIMENTAL

The standard Model 14-100 Bendix Time-of-Flight Mass Spectrometer with several additions and modifications were used for these studies. The mass spectrometer is equipped with a Model 112 Bendix Two Channel Analog Output System. The Model 321 Scanner Channel was used as is, while the Model 311 Controller Channel was converted to a second scanner channel. The Model E-104 Bendix Trap Current Regulator was used to maintain a constant ionizing current. The high voltage supply to the multiplier field and dynode strips was regulated by the circuit that is shown in Figure 1*. The multiplier noise was reduced by placing a 0.1 microfarad capacitor between the multiplier scope anode and ground**. The D. C. voltages throughout the instrument were kept constant by use of a Model CU1 Sola Constant Voltage Transformer. A Model 450 Hewlett-Packard Amplifier was used to amplify the signal which was generated by the multiplier.

The flash lamp and sample geometry that was used for these experiments is described elsewhere[7]. A schematic diagram of the flash lamp and sample, as mounted in the reaction chamber of the mass spectrometer, are shown in Figure 2. In operation, the assembly is placed in the reaction chamber of the mass spectrometer with a solid sample in place. The glass lead from the hollow electrode is fastened to the external vacuum system, and the lamp is evacuated at 10^{-6} mm Hg for several hours. Meanwhile, the reaction chamber is evacuated through the mass spectrometer leak and

* This modification was recommended by Mr. Edward Younginger of the Bendix Corporation.

** This modification was recommended by Prof. Stig Claesson, Head of the Institute of Physical Chemistry, Uppsala, Sweden.

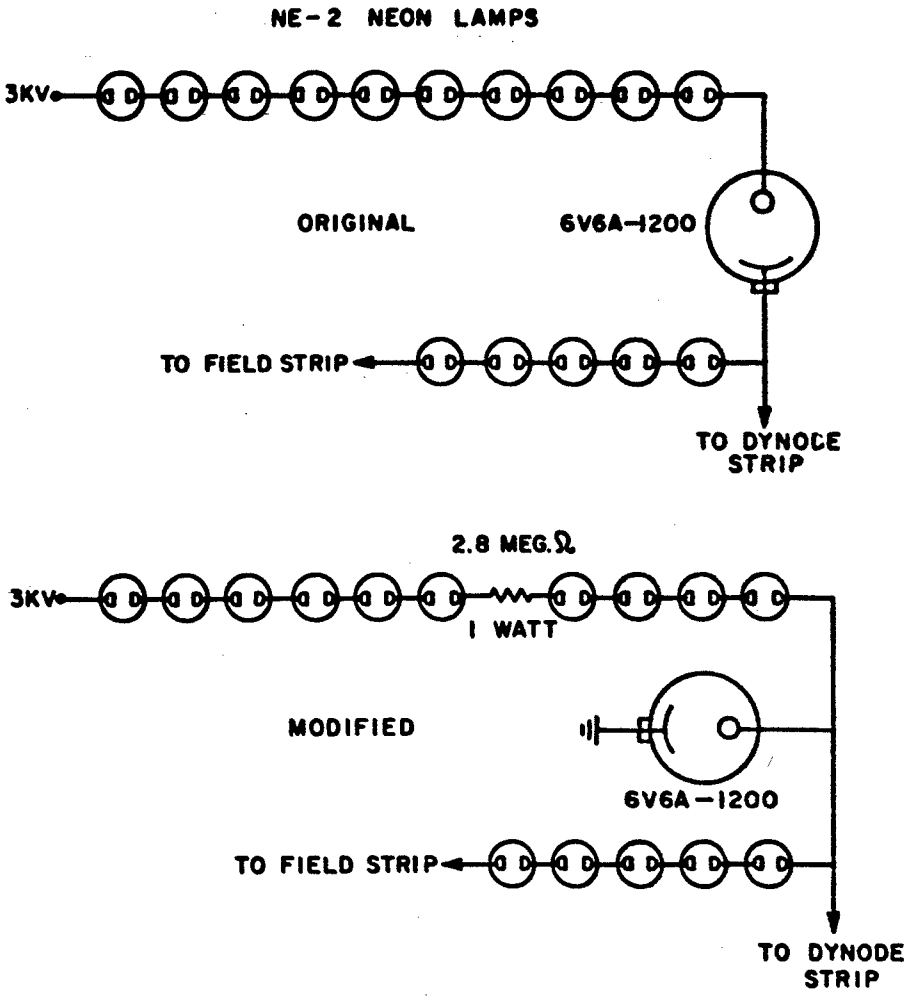

FIGURE 1. HIGH VOLTAGE REGULATION CIRCUITRY FOR ELECTRON MULTIPLIER

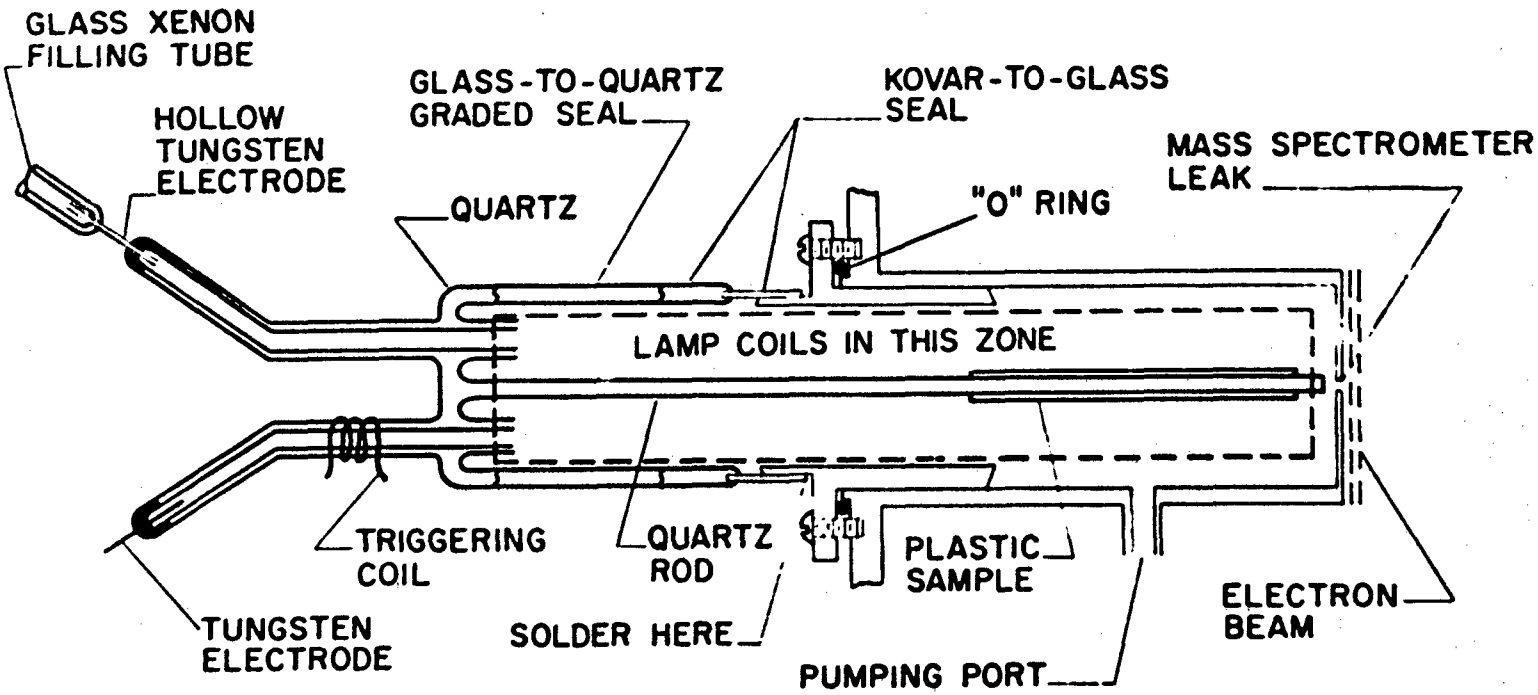

FIGURE 2 .Flash Lamp and Plastic Sample as Mounted in the Reaction Chamber of the Mass Spectrometer

through the reaction chamber pumping port by the external vacuum system. The lamp is then filled to about 40 mm Hg with xenon. A 220 microfarad capacitor bank at 5000 V is discharged through the lamp in order to produce the flash.

A single two mil diameter gold foil leak was used for these experiments. The decomposition products were ionized with 70 Volt electrons. All spectra were recorded with the ion lens of the mass spectrometer in operation. The liquid nitrogen trap of the mass spectrometer was filled as the lamp electrodes were being charged. The lamp was discharged two minutes after the trap was completely filled, in each case. The analog spectra were recorded by the following method.

In normal operation, the multiplier receives a new mass spectrum every 100 microseconds (10 KC). The analog gate is activated only for a fraction of a microsecond during each mass spectrum, and sweeps to either higher or lower mass numbers slowly from cycle to cycle. Thus, one may integrate a large number of events with the analog. The starting mass may be selected manually by choice of an initial voltage. The mass range is swept by continually increasing or decreasing this voltage. The entire process is controlled by the 10 KC master pulse of mass spectrometer. During flash pyrolysis experiments, it was necessary to start analog scan at the same time with respect to the flash for each flash experiment in order to obtain data that could be compared with a minimum of errors. The following procedure was found to give reproducible timing. The flash strikes a phototube, which generates a pulse. The pulse triggers the

Tektronix Model 543 Oscilloscope after a pre-set time delay (three seconds for these experiments). A rising voltage which is generated by the oscilloscope, at a predetermined rate, replaces a similar voltage source in the analog and drives the gate towards an increasing mass range. A photograph of the mass spectrum is obtained by opening the camera shutter with the oscilloscope set for single sweep before striking the flash, and closing the shutter after the desired mass range has been swept. It was desirable to use the second channel to study a greater mass range in shorter periods of time and to provide a wider range of sensitivity. Since the gate of the first channel is at the correct time relationship with respect to the master pulse, the gate signal of the first channel was used as a pulse to trigger the gate of the second channel. The starting mass of the second channel was set manually at the desired value. The gates of the two channels then began to sweep at the same predetermined time delay with respect to the flash and covered their respective mass ranges during the same interval of time. Calibration spectra were photographed in the same manner so that they could be compared with the unknowns more accurately. Analog channel 2, which covered the higher mass range, was set to a higher sensitivity than channel 1. Channel 1 covered m/e 1 to 100, while channel 2 covered m/e 12 to 130. Analog sweep was completed in seven seconds. Reference samples of n-butane were run periodically to standardize the instrument.

A sample of pure phenol-formaldehyde was flash pyrolyzed eleven times in the reaction chamber of the mass spectrometer. Each flash was

applied only after the products of the previous flash were pumped away, a process which took about ½ hour. The mass spectroscopy data were analyzed by the following method.

Calibration samples of pure Hydrogen, Methane, Carbon Monoxide, Ethane, Ethylene, Acetylene, Water, and Carbon Dioxide were run in order to obtain their mass spectra. Comparison of these spectra with the Mass Spectral Tables of the American Petroleum Institute showed that they were quite similar to spectra which were obtained at the National Bureau of Standards. Therefore, N.B.S. spectra were used for compounds which were not tested directly. Where N.B.S. spectra were not available, those spectra which were found to be most like N.B.S. spectra, for other compounds, were used. Note that the A.P.I. Mass Spectral Tables were used only for minor components, so that small discrepancies in the pattern and sensitivity would not be too important. Direct calibration spectra were obtained by a technique which simulated part of the flash pyrolysis experiment. Samples of known pressure were introduced to the reaction chamber from the three liter reservoir. At the appropriate moment, the valve which separates the reaction chamber from the reservoir was closed. The analog system was triggered manually at that instant, and the oscilloscope was triggered after a time delay of three seconds.

The first few analyses of data were performed by starting at the high mass end and assigning peaks to the compounds from which they probably originated. The lower mass fragment contributions which could be attributed to these compounds were then subtracted from the remaining mass spectrum

in order to see if the choices were reasonable. Changes were made when necessary. When a consistent set of compounds was obtained for several flash experiments, a matrix was derived for application to the other flash experiments. The matrix method was much faster than the earlier systematic elimination procedure.

III. RESULTS AND DISCUSSION

The results of these experiments are shown in different forms in Tables 1 and 2. Table 2 shows that a high degree of consistency is associated with the analysis. Absolute yields of the various products are plotted as a function of flash number in Figures 3 to 5. Examination of the individual yields shows several trends. H_2, CO, CH_4, C_4H_2, C_5H_6, C_6H_6, C_7H_8, and C_8H_8 show a maximum yield at about the fourth flash with comparatively smooth increases in the earlier flashes and decreases in the later experiments. C_4H_4, C_4H_6, and C_5H_8 showed the same general behavior as the above, with several small variations. H_2O, CO_2, C_2H_2, and C_3H_8 showed a more or less continual decrease in yield. C_2H_4, C_2H_6, C_3H_4, and C_3H_6 showed an early decrease in yield, followed by a leveling off and then a comparatively gradual decrease. C_4H_8 showed a rather different trend. The yield of C_4H_8 was very irregular with the maximum yield at the seventh flash. In general, however, H_2, CO, CH_4, and C_4 and heavier hydrocarbons showed one type of behavior, while H_2O, CO_2, C_2, and C_3 hydrocarbons showed a different type. This information could conceivably lead to understanding of the mechanism of high temperature degradation of phenol-formaldehyde and other complex plastics. For example, Ouchi and Honda[8] observed that H_2O was an early decomposition product of phenol-formaldehyde and suggested that it was probably produced by condensation of a pair of hydroxy groups to form an ether linkage and by reaction of a hydroxy group with a hydrogen of a bridge CH_2 group. CO_2 was also observed to be an early product. H_2, CO, and CH_4 formed later, but at about the same time. The following reactions were thought to be responsible for their formation.

TABLE 1

DEGRADATION PRODUCTS FROM FLASH PYROLYSIS OF PURE PHENOL-FORMALDEHYDE SAMPLE
AS DETERMINED BY THE BENDIX TIME-OF-FLIGHT MASS SPECTROMETER

Component	Flash No. ⟶	Yield in Arbitrary Units, Proportional to Moles										
		1	2	3	4	5	6	7	8	9	10	11
H_2		149.7	250.0	291.9	309.5	283.1	261.1	221.3	190.3	172.7	170.3	181.9
CH_4		33.8	52.4	61.3	73.6	61.7	50.9	40.9	34.8	33.7	32.6	32.5
H_2O		176.9	131.3	82.7	86.5	67.5	54.2	33.9	33.3	29.5	23.8	32.3
CO		94.2	153.0	152.6	175.3	138.8	124.8	100.0	67.8	55.4	54.6	61.0
C_2H_2		22.9	15.1	13.1	10.9	9.0	10.1	10.1	6.7	4.6	5.4	3.3
C_2H_4		21.9	11.1	13.7	12.7	11.6	6.3	6.6	12.2	7.3	9.4	6.8
C_2H_6		11.8	7.8	8.6	8.7	7.9	9.0	8.0	7.0	6.2	6.4	4.9
C_3H_4		4.2	1.5	2.5	2.8	2.1	2.9	2.5	2.2	1.7	1.7	1.3
C_3H_6		9.7	5.8	4.2	4.5	6.0	5.4	6.4	5.5	3.9	4.7	2.9
C_3H_8		9.9	5.4	4.8	4.8	4.4	4.1	3.9	2.8	1.8	2.1	1.9
CO_2		23.6	16.2	11.3	10.3	8.0	7.1	5.9	4.7	3.9	4.3	5.1
C_4H_2		0.4	0.9	1.2	1.2	1.0	1.0	0.8	0.7	0.4	0.4	0.5
C_4H_4		0.3	1.4	1.2	1.5	0.9	0.9	1.0	0.3	0.4	0.6	0.5
C_4H_6		0.6	1.1	0.8	1.6	1.0	1.4	0.8	0.6	0.6	0.5	0.6
C_4H_8		1.8	1.8	0.7	1.6	1.3	2.0	2.5	1.8	1.4	1.6	1.2

TABLE 1 (Continued)

Component	Flash No. ⟶	Yield in Arbitrary Units, Proportional to Moles										
		1	2	3	4	5	6	7	8	9	10	11
C_5H_6		0.3	2.2	3.1	3.0	2.7	2.2	2.0	1.6	1.3	1.1	1.4
C_5H_8		0.9	1.7	1.8	2.1	1.9	0.9	1.2	0.8	1.0	0.8	1.3
C_6H_6		1.2	2.5	3.6	4.0	3.2	2.6	1.9	1.6	1.2	1.1	1.3
C_7H_8		0.6	1.0	1.2	1.3	1.0	0.8	0.6	0.6	0.4	0.4	0.4
C_8H_8		0.6	0.8	1.0	1.0	0.8	0.6	0.5	0.4	0.3	0.3	0.2
C_9H_{12}		-	-	trace	trace	trace	trace	trace	-	-	-	-

TABLE 2

DEGRADATION PRODUCTS FROM FLASH PYROLYSIS OF PURE PHENOL-FORMALDEHYDE SAMPLE
AS DETERMINED BY THE BENDIX TIME-OF-FLIGHT MASS SPECTROMETER

		Mole Percent										
Component	Flash No. ⟶	1	2	3	4	5	6	7	8	9	10	11
H_2		26.5	37.7	44.1	43.2	46.1	47.6	49.2	50.7	52.7	52.9	53.3
CH_4		6.0	7.9	9.3	10.3	10.1	9.3	9.1	9.3	10.3	10.1	9.5
H_2O		31.3	19.8	12.5	12.1	11.0	9.9	7.4	8.8	9.0	7.4	9.5
CO		16.7	23.1	23.1	24.4	22.6	22.8	22.2	18.1	16.9	17.0	17.9
C_2H_2		4.0	2.3	2.0	1.5	1.5	1.8	2.2	1.8	1.4	1.7	1.0
C_2H_4		3.9	1.7	2.1	1.8	1.9	1.1	1.5	3.1	2.2	2.9	2.0
C_2H_6		2.1	1.2	1.3	1.2	1.3	1.6	1.8	1.9	1.9	2.0	1.4
C_3H_4		0.7	0.2	0.4	0.4	0.3	0.5	0.5	0.6	0.5	0.5	0.4
C_3H_6		1.7	0.9	0.6	0.6	1.0	1.0	1.4	1.5	1.2	1.5	0.8
C_3H_8		1.7	0.8	0.7	0.7	0.7	0.8	0.9	0.8	0.6	0.7	0.5
CO_2		4.2	2.4	1.7	1.4	1.3	1.3	1.3	1.2	1.2	1.3	1.5
C_4H_2		0.1	0.1	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.1	0.1
C_4H_4		0.1	0.2	0.2	0.2	0.1	0.2	0.2	0.1	0.1	0.2	0.1
C_4H_6		0.1	0.2	0.1	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.2
C_4H_8		0.3	0.3	0.1	0.2	0.2	0.4	0.6	0.5	0.4	0.5	0.3

TABLE 2 (Continued)

Component	Flash No. ⟶	1	2	3	4	5	6	7	8	9	10	11
		Mole Percent										
C_5H_6		0.1	0.3	0.5	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
C_5H_8		0.1	0.3	0.3	0.3	0.3	0.2	0.3	0.2	0.3	0.2	0.4
C_6H_6		0.2	0.4	0.5	0.6	0.5	0.5	0.4	0.4	0.4	0.4	0.4
C_7H_8		0.1	0.1	0.2	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1
C_8H_8		0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
C_9H_{12}		-	-	trace	trace	trace	trace	trace	-	-	-	-
		Average pressure in reaction chamber during duration of analog sweep in mm Hg relative to room temperature										
		3.54	4.15	4.14	4.48	3.84	3.43	2.81	2.35	2.05	2.01	2.14

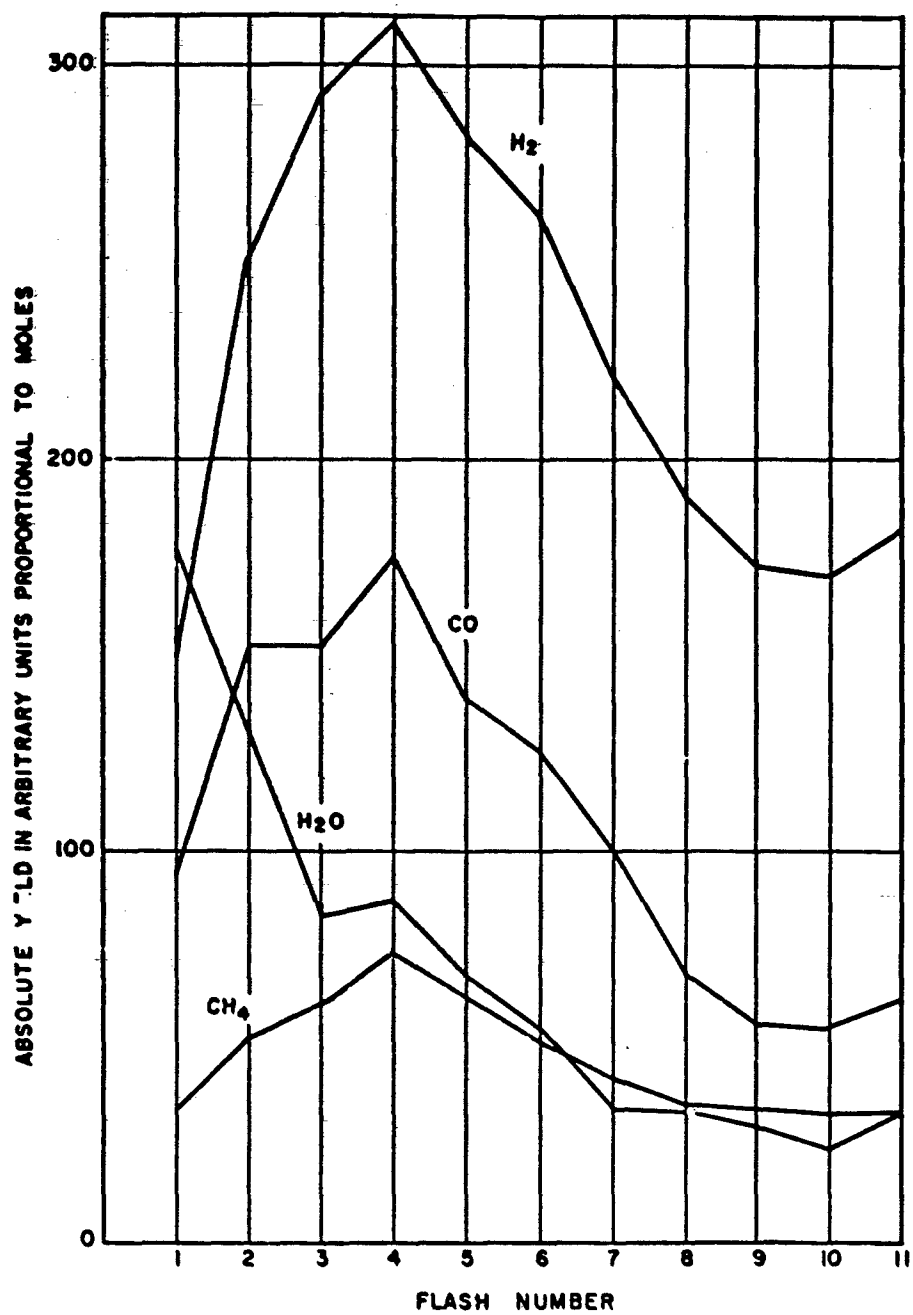

FIGURE 3 PRODUCT YIELDS FROM FLASH PYROLYSIS OF PHENOL-FORMALDEHYDE (H_2, H_2O, CO, CH_4)

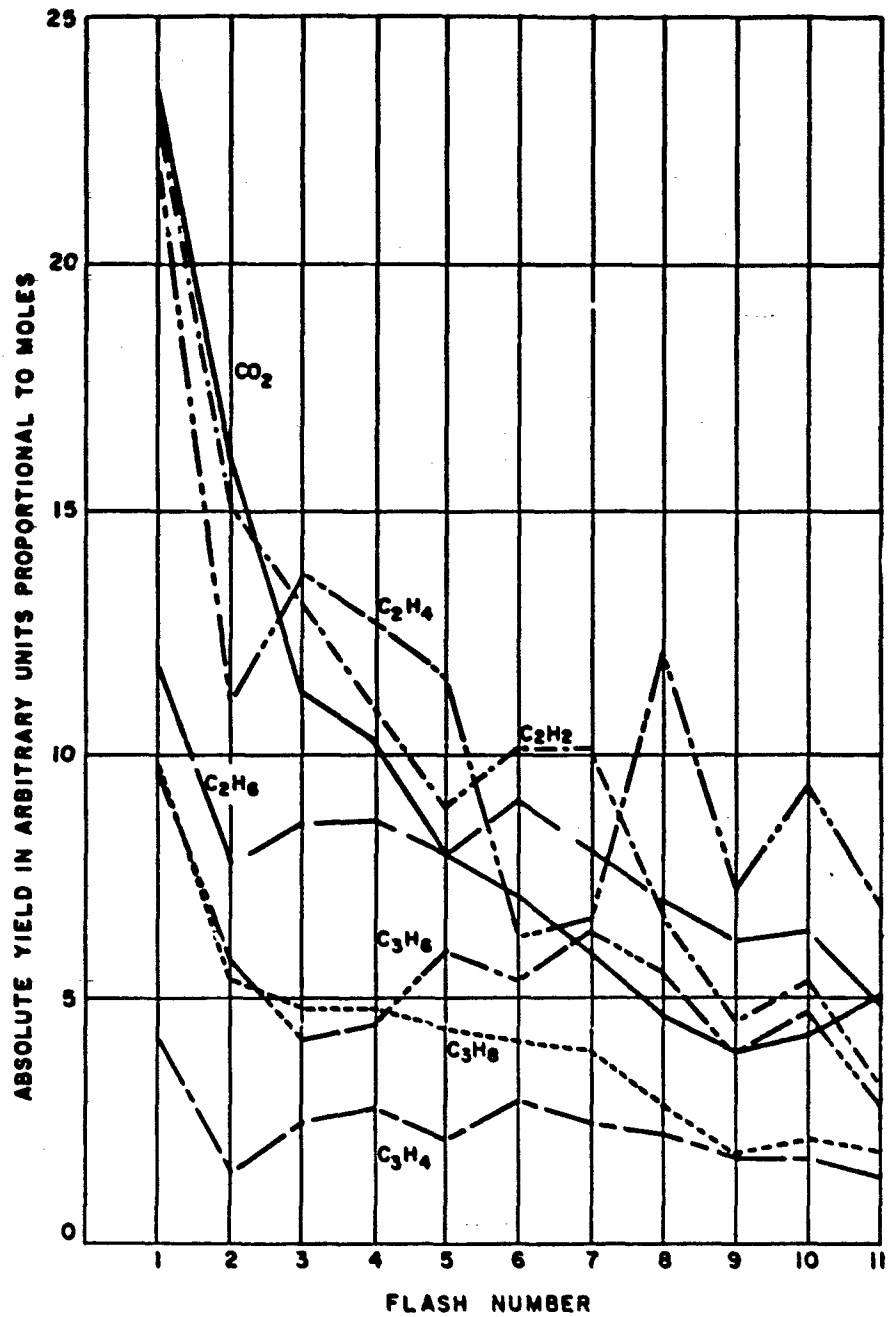

FIGURE 4 PRODUCT YIELDS FROM FLASH PYROLYSIS OF PHENOL-FORMALDEHYDE (CO_2,C_2 & C_3 HYDROCARBONS)

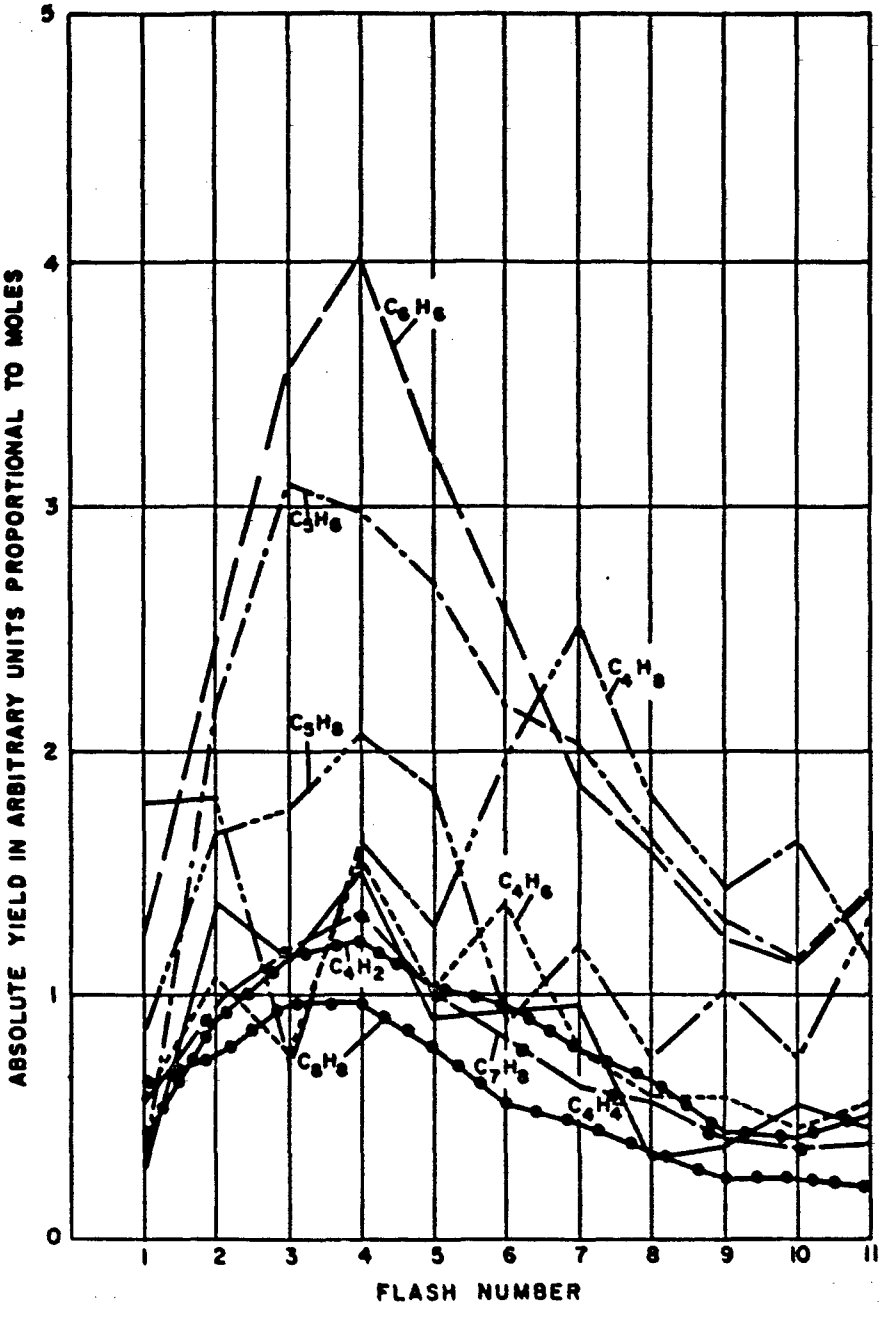

FIGURE 5 PRODUCT YIELDS FROM FLASH PYROLYSIS OF PHENOL-FORMALDEHYDE (C_4 TO C_8 HYDROCARBONS)

$$H_2O + \quad >CH_2 \longrightarrow CO + 2\ H_2$$

$$H_2 \ + \quad >CH_2 \longrightarrow CH_4$$

$$-O- + \quad >CH_2 \longrightarrow CO + H_2$$

$>CH_2$ and -O- represent bridge methylene and ether groups, respectively. The observations of the current experiment do not disagree with these findings of Ouchi and Honda.

Although the analytical data show great consistency, there are several possible sources of error. One type of error is normal instrumental error. Another source of error may be chemical reactions which occur among the products during the time of analog scan. A further source of error may be caused by continued generation of gases from the hot surface long after the flash is extinguished. Another type of error is due to the fact that the lighter gases will be pumped out of the 100 cc reaction chamber through the two mil diameter leak more rapidly than the heavier gases. This error is not very large and is rendered less serious than otherwise because the lighter masses are scanned earliest. In fact, it is possible to correct for this error to some extent, and some experiments have been carried out to that end. Samples of hydrogen, methane, and n-butane were admitted into the mass spectrometer at initial pressure of 67 microns. In each case, the gate of one analog channel was focussed at the parent peak. At the appropriate time, the stopcock, which isolates the reaction chamber from the reservoir, was closed and ion current was monitored as a function of time. Figure 6 shows plots of the logarithm of ion current as a function of time. Kinetic theory shows that for effusive flow

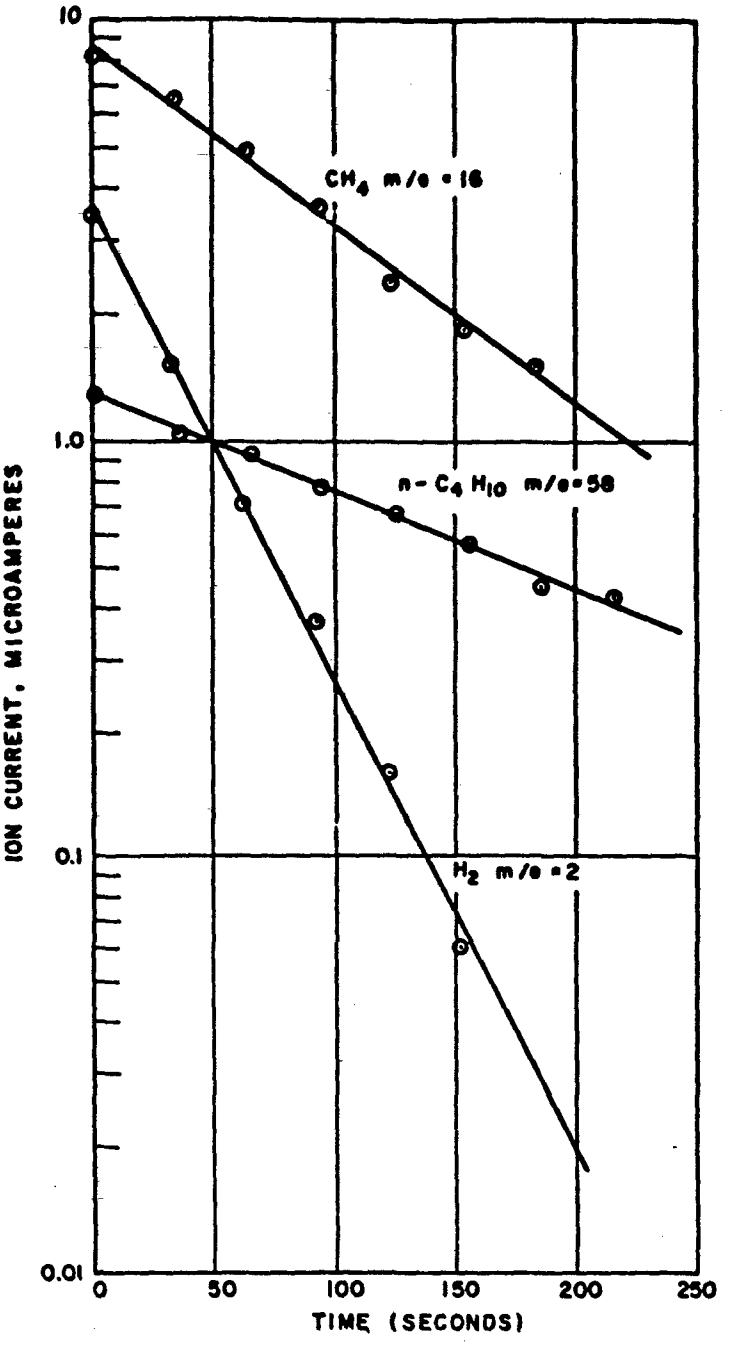

FIGURE 6 FLOW OF PURE GASES FROM FLASH PYROLYSIS REACTION CHAMBER INTO THE MASS SPECTROMETER

$$\ln I = \ln I_o + \frac{\alpha}{\sqrt{M}} t$$

where

I = ion current

I_o = initial ion current

α = a constant

M = molecular weight

t = time

so it is reasonable that the plots are linear. The interrelationships between the slopes are shown in Table 3. Note that the experimental and theoretical $\sqrt{M_2/M_1}$'s are quite close to each other, but the experimental values are somewhat lower. This could mean that the pressure is slightly too high for effusive flow through a two mil hole in some cases. A study with n-butane at a variety of higher pressures showed that the slope increased (smaller negative number) with increasing pressure which also indicated that flow was not effusive, at least at the higher pressures. Reference to Table 2 shows that the pressures were too high for this type of correction to be applied. Future experiments will be carried out with smaller samples.

A sample of phenol-formaldehyde was pyrolyzed in the Space Sciences Laboratory Arc-Image Furnace Facility according to the procedures which are described in References 9 and 10. A partial qualitative analysis disclosed the presence of H_2, CO, CH_4, N_2 (traces), CO_2, C_2H_2, C_2H_4, C_2H_6, H_2O, benzene, styrene, toluene, and xylene. Quantitative comparisons of some of the products with those of the flash pyrolysis

TABLE 3

FLOW OF PURE GASES FROM THE REACTION CHAMBER INTO THE MASS SPECTROMETER

(Initial Pressure - 67 microns)

Gas	m/e	$\alpha \sqrt{1/i}$
H_2	2	$-(1.13 \pm 0.02) \times 10^{-2}$
CH_4	16	$-(4.25 \pm 0.12) \times 10^{-3}$
$n\text{-}C_4H_{10}$	58	$-(2.33 \pm 0.05) \times 10^{-3}$

Gases compared	Theory $\sqrt{M_2/M_1}$	Experiment
H_2/CH_4	2.828	2.7 ± 0.1
H_2/C_4H_{10}	5.385	4.9 ± 0.2
CH_4/C_4H_{10}	1.904	1.8 ± 0.1

experiments are listed in Table 4. The agreement of results is very good, considering the differences in the experiments. Because of the pumping error associated with the analog readings, the relative yields of the flash experiments are probably too low.

Examinations of plastic samples after flash pyrolysis have disclosed that the finish of the surface influences the absorption of energy. In some cases, the surface appears to be blackened rather uniformly, while in others, some parts are hardly blackened. Machining notches, which are readily discerned by the eye, appear to be readily blackened. It would certainly be helpful to have a technique to produce a standard and uniform surface finish on samples if one desires to compare different samples of the same material. In addition, the energy flux is not uniform over the surface of the sample. Still, it would be helpful to know an average energy input and/or surface temperature. Knowledge of the pressure in the reaction chamber would also be helpful. It is planned that such measurements will be carried out in the future.

Since H_2, H_2O, CO, and CO_2 were major decomposition products for the eleven flash experiments, temperatures were calculated based on the possibility that the water gas equilibrium was important. The water gas equilibrium temperature would be valid if these four products came to equilibrium on the surface and then were cooled rapidly upon leaving the surface. Equilibrium constants were calculated from Table 1 and temperatures were determined from Reference 11. They are listed in Table 5. The temperatures and trend are thought to be quite reasonable. One might expect that, during earlier flashes, the sample would start to blacken and

TABLE 4

COMPARISON OF ARC-IMAGE AND FLASH PYROLYSIS DATA

Component	Arc Image Furnace	Flash No. ⟶	Relative Yield, moles/mole H_2 1	2	3	4	5	6	7	8	9	10	11
H_2	1		1	1	1	1	1	1	1	1	1	1	1
CO	.377		.629	.612	.523	.566	.490	.478	.451	.356	.321	.320	.336
CH_4	.244		.266	.210	.210	.238	.218	.195	.185	.183	.195	.191	.179
fraction 2*	.198		.714	.272	.212	.196	.188	.192	.219	.234	.186	.217	.158
			Relative Yield, grams/mole H_2										
fraction 3**	38.6		23.3	11.9	7.9	7.9	6.9	5.9	4.9	5.1	4.9	4.1	5.1

* Fraction 2 in arc image furnace experiment consists of components which are not volatile at liquid nitrogen temperatures and distill from a CO_2 ice cooled trap. For the flash pyrolysis experiments, this fraction is assumed to contain all C_2, C_3, and C_4 hydrocarbons and CO_2.

** Fraction 3 in arc image furnace experiment consists of components which are not volatile at CO_2 ice temperatures and distill from a trap at room temperature. For flash pyrolysis experiments, this fraction is assumed to contain all C_5 and heavier hydrocarbons and H_2O.

TABLE 5

SURFACE TEMPERATURE CALCULATED FROM WATER GAS EQUILIBRIUM

Flash Number	Temperature, ^{o}K
1	2025
2	2090
3	1790
4	2030
5	1880
6	1755
7	1495
8	1480
9	1450
10	1305
11	1385

might become a better receptor of radiant energy. However, with each flash, solids which are driven off from the plastic surface could coat the lamp and may thus reduce its energy output. A portion of the decomposition gases could come from the lamp surface and other surfaces of the reaction chamber.

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TECHNICAL INFORMATION SERIES

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Henry L. Friedman	Plastic Degradation	R62SD81
		DATE September 1962

TITLE THE PRODUCTS OF FLASH PYROLYSIS OF PHENOL-FORMALDEHYDE BY TIME-OF-FLIGHT MASS SPECTROSCOPY

ABSTRACT A sample of phenol-formaldehyde was flash pyrolyzed eleven times in the reaction chamber of a time-of-flight mass spectrometer. Quantitative analyses were determined for pyrolysis products which were present within ten seconds after each flash. A high degree of consistency between compositions of products from consecutive flashes resulted. A discussion of the results is presented.

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