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AN ULTRAVIOLET PHOTOMETRIC DETECTOR FOR USE IN GAS CHROMATOGRAPH--ETC(U)
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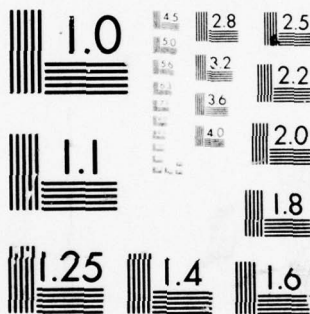
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AN ULTRAVIOLET PHOTOMETRIC DETECTOR FOR USE IN GAS CHROMATOGRAPHY

MONSANTO RESEARCH CORPORATION
DAYTON LABORATORY✓
DAYTON, OHIO 45407

NOVEMBER 1977

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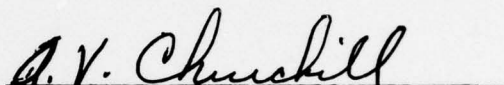
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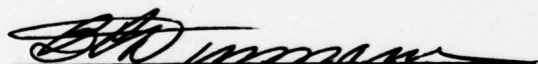
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Aromatic content of fuels	JP-4									
Gas chromatography	JP-8									
Gasoline	Jet-A									
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An ultraviolet photometric detector, which responds only to aromatic constituents in the vapor state, has been designed and constructed for use in gas chromatography. The unit exhibits appreciably greater sensitivity than the thermal conductivity detector. It was tested by chromatographing a variety of fuels including JP-4, JP-8, Jet-A, and regular gasoline. Tests indicate that the unit provides a viable means of determining the aromatic composition of fuels.										

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FOREWORD

This technical report was submitted by Monsanto Research Corporation on under Contract F33615-76-C-2014. The effort was sponsored by the Air Force Aero Propulsion Laboratory (AFAPL), Air Force Wright Aeronautical Laboratories (AFWAL), Air Force Systems Command (AFSC), Wright-Patterson Air Force Base, under Project 3048 (Fuels, Lubrication, and Fire Protection), Task 304805 (Aero Propulsion Fuels), Work Unit 30480574 (Analysis of Aircraft Fuels and Related Materials).

Mr. F. Neil Hodgson, Monsanto Research Corporation, is the Principal Investigator having primary responsibility for the work described in this report. Dr. Ronald D. Butler (AFAPL) is the Air Force project scientist.

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SECTION I

INTRODUCTION AND BACKGROUND

Gas chromatography has proven to be an invaluable tool for characterizing the complex hydrocarbon fuels required for military aircraft. Commonly-used JP-4, for example, contains a myriad of constituents ranging from C_4 to C_{16} in carbon number representing n-alkanes, branched alkanes, naphthenes and aromatic compound types. Even without complete resolution or identification of all components, properties such as distillation range, vapor pressure and heat of combustion can be calculated from GC data. Aromatic constituents generally cannot be distinguished from the large number of branched alkanes which surround and overlap them, thus conventional gas chromatography provides little insight into the aromatic makeup of a fuel. However, specific information on aromatics individually, or as a compound class, would be extremely useful, since these compounds in an aircraft fuel have a direct effect on engine power and performance, the kinds and amounts of exhaust emissions and other operational factors.

The use of an ultraviolet photometric gas chromatographic detector, which would respond only to aromatic constituents in the vapor state, has been suggested by one of the authors (Ref. 1). The proposed conceptual design incorporates an ultraviolet source, a flow-through absorption cell for vapors, and a UV radiation detector.

The feasibility of the proposed concept was tested (Ref. 2, 3) by interfacing a gas chromatograph having a thermal conductivity (TC) detector with a laboratory spectrophotometer. A flow-through cell having an internal volume of 150 μ l and a 1 cm optical path was connected in series with the TC detector. Absorption measurements were conducted at 260 nm. The preliminary investigation was directed primarily toward evaluating the specificity of the technique for aromatic compounds in the vapor state and the effect

of the dead volume of the cell and its interconnecting tubing on the chromatographic peak shape. Chromatograms of paraffin and alkylbenzene mixtures showed total specificity for the aromatic components at the absorption wavelength of 260 nm. Peaks were not significantly broadened despite the relatively large dead volume represented by connection between the TC detector and absorption cell. From these encouraging experiments, it was concluded that construction of a test model detector which would be independent of an existing spectrophotometer was warranted.

SECTION II

CONSTRUCTION OF THE PROTOTYPE

A. Calculation of Detector Response

The detector response which might reasonably be expected can be estimated by considering a typical aromatic species of molecular weight about 100 (toluene for example), which is present at a concentration of 1% in an inert solvent having a density of 0.80 gms/ml. These figures are typical of JP-4. In one microliter of sample, the actual quantity of aromatic to be detected is

$$\begin{aligned} 10^{-3} \text{ ml} \times 0.8 \text{ gm/ml} \times 0.01 \text{ gm/gm} &= 8 \times 10^{-6} \text{ gm} \\ &= 8 \times 10^{-8} \text{ mole} \end{aligned}$$

For the purpose of further calculations, the detector cell volume can be arbitrarily assumed to be 10 microliters and the optical path to be 1 centimeter. Typical operating conditions might be a carrier gas flow of 10 ml/min and a peak width of 30 sec.

During the 30 seconds that the sample is being swept through the detector by the carrier gas, a total of 5 ml of carrier and sample passes through the cell. If the 8×10^{-8} mole of aromatic species were uniformly distributed throughout the 5 ml of carrier, its concentration would be

$$8 \times 10^{-8} \text{ mole} / 0.05 \text{ liter} = 1.6 \times 10^{-6} \text{ mole/liter}$$

Typically, the molar absorptivity coefficient of a strong ultraviolet absorber, such as an aromatic, is on the order of 5×10^3 at the wavelength of maximum absorption. For a 1.0-cm path length, then, the equivalent absorbance would be

$$\begin{aligned} A &= 5 \times 10^3 \times 1.0 \times 1.6 \times 10^{-6} \\ &= 0.008 \end{aligned}$$

This calculation assumes a "square wave" type of response with time (because the absorbing species was assumed to be uniformly distributed throughout a finite volume), which of course is not the case in actual practice. The response is actually nearly gaussian in shape, and the gaussian curve can be reasonably approximated by an isosceles triangle. An equivalent triangular response, having the same integrated area as the square-wave approximation, would have a maximum reading twice the calculated value. Therefore, the model sample would produce a maximum absorbance of about 0.016 with the basic detector geometry that we have assumed. This maximum response falls within the capabilities of modern solid-state photometers.

Two factors can be used to increase the response. First, a longer optical path can be used without seriously creating dead-volume problems. Second, consider that the absorbance values were calculated from molar absorption coefficients observed in solution chemistry. The absorbance characteristics of aromatic molecules in the gas phase are quite different; in particular, the absorption bands are much sharper, and consequently more intense. It is, therefore, reasonable to expect the molar absorbances to be several times higher than the value of 5×10^3 used above. The detector response would be amplified accordingly.

These calculations demonstrate that a conventional photometric detector, properly modified and using a 10-cm light path, should be sensitive enough to monitor low levels of aromatic compounds in a flowing gas stream.

B. Design and Fabrication

The opposing sample cell requirements of long optical path, small volume and high radiation through-put make careful optimization of cell dimensions essential. A 100-mm cell path is considered

necessary for adequate sensitivity. This requirement dictates a cell diameter in the order of 1 mm in order to maintain a volume of approximately 100 microliters. Since the cell will be of the flow-through type, and the light intensity will be monitored axially, careful design is necessary to ensure uniform gas flow with no hold-up. That is, there can be no pockets where materials can be entrapped. Therefore, the inlet and exit ports must be positioned as closely to the end windows as possible.

An absorption cell was constructed to meet these requirements using a 100-mm section of 16-gauge stainless steel hypodermic needle as the optical path. The cell was constructed to have a T-shaped gas flow path which allowed sample and carrier gas to enter at the center of the 100 mm length and to flow in both directions, exiting at either end of the cell. This design was employed to eliminate the need for cell windows which are a potential source of radiation loss due to reflection and absorption. The cell in its mounting is shown in Figure 1. The thin-wall stainless steel tubing was made rigid by jacketing it in a length of heavy-wall tubing having an inside diameter exactly the same as the outside diameter of the smaller tubing. The aluminum block upon which the cell was mounted contained a well to accommodate a thermistor for temperature measurement. The center port was fitted with a stainless steel Crawford fitting to accept 1/16" O.D. tubing. The entire unit was heated by a bar heater which was mounted in contact with the aluminum block. This assembly was mounted in a metal box filled with fibrous insulation. A mercury-filled ozone generating lamp, GE No. G4S11, was positioned at one end of the optical path of the cell. A Baird S-17 band-pass filter, passing the mercury 254 nm line, and PIN type UV sensitive photodiode were placed on the opposite end of the cell. A

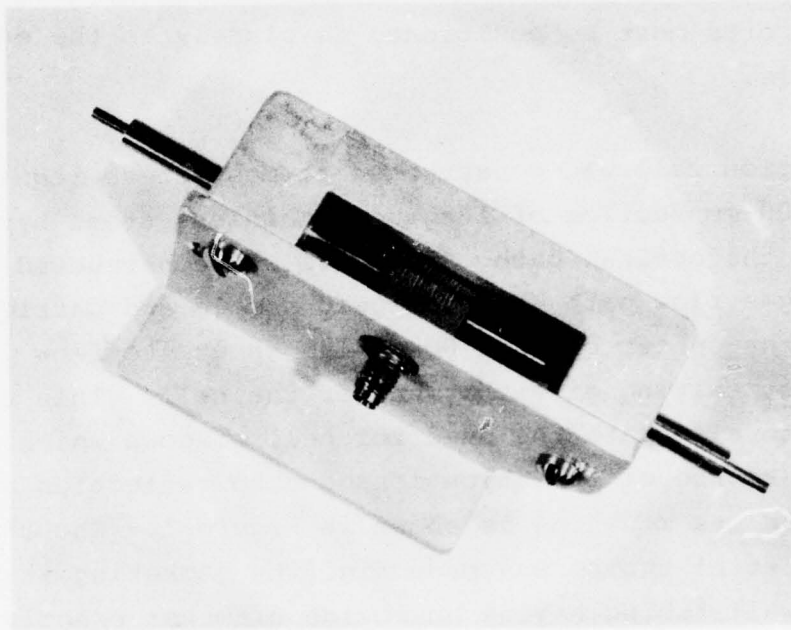


Figure 1. Detector Cell Installed in Mounting.

schematic diagram of the device is shown in Figure 2. The signal from the photodiode detector is fed into an electrometer, through a binary attenuator, and is then displayed on a stripchart recorder.

Initial tests were conducted using quartz lenses to focus the UV radiation prior to its entering the cell and again as it exited the cell. It was found, however, that little gain in sensitivity was obtained by this procedure. The reflective interior of the metal capillary minimized the need for highly collimated radiation. Thus, for the sake of simplicity, lenses were not used.

C. Performance Evaluation and Modifications

To test the performance of the device, it was connected in series with a thermal conductivity (TC) detector, taking care to minimize the dead volume of the connecting tubing. Chromatographic effluent first passed through the TC detector and then to the UV absorption cell. Outputs from the two detectors were recorded on a single chart by a dual-channel recorder. The physical arrangement of the unit is shown in Figure 3.

Trial chromatographic runs on a synthetic blend of alkanes and alkyl benzenes showed excellent response to the aromatics while, as anticipated, the alkanes remained undetected. The UV detector was found to have approximately the same sensitivity for aromatic compounds as the thermal conductivity detector. Several undesirable performance characteristics were observed:

- (a) a high noise level at maximum sensitivity
- (b) a drifting or unstable baseline

Since this behavior was not observed when the UV lamp was off but with the detector and electrometer on, it was concluded that the problem was not of an electronic nature.

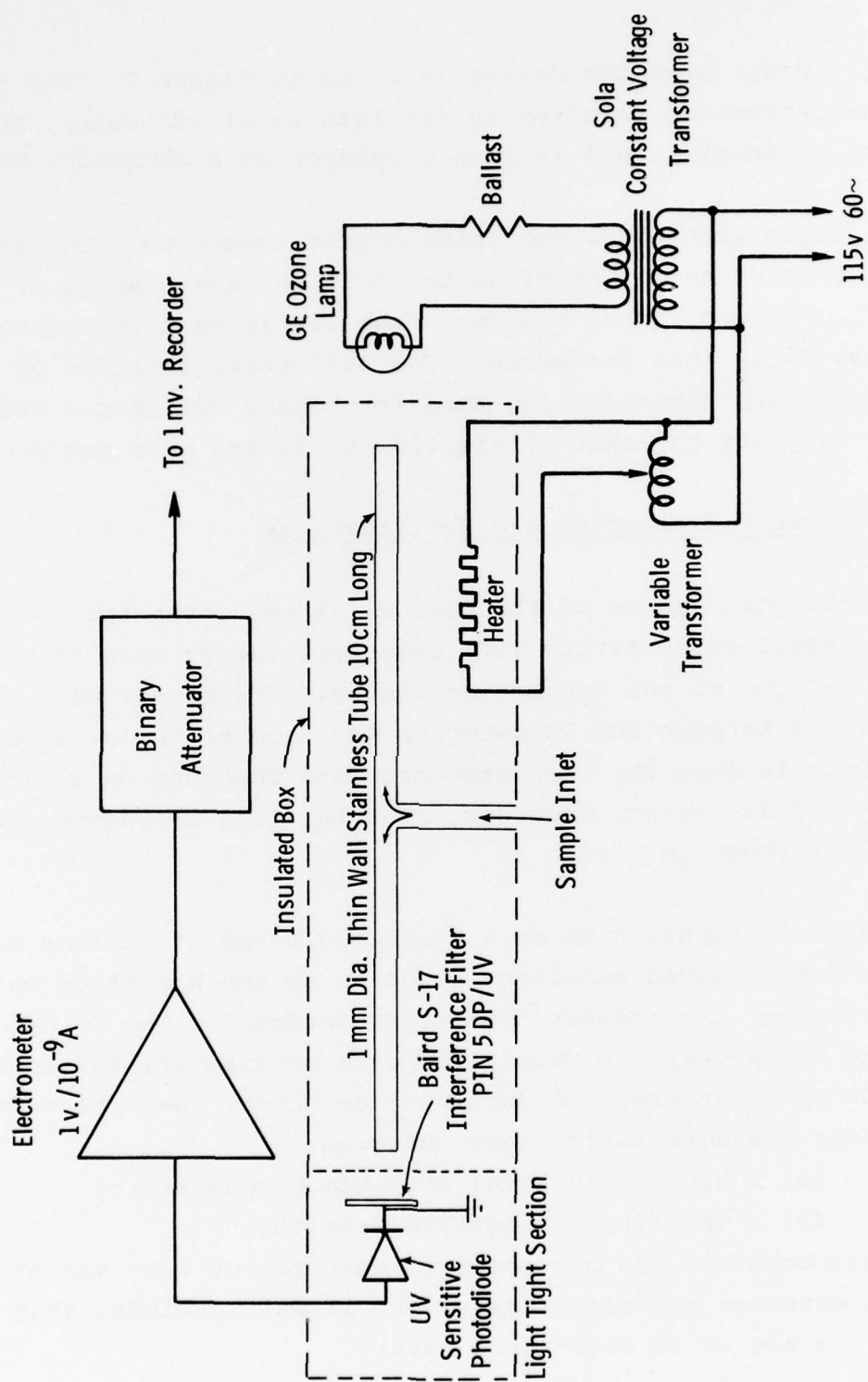


Figure 2. Schematic Diagram of UV Photometric Detector.

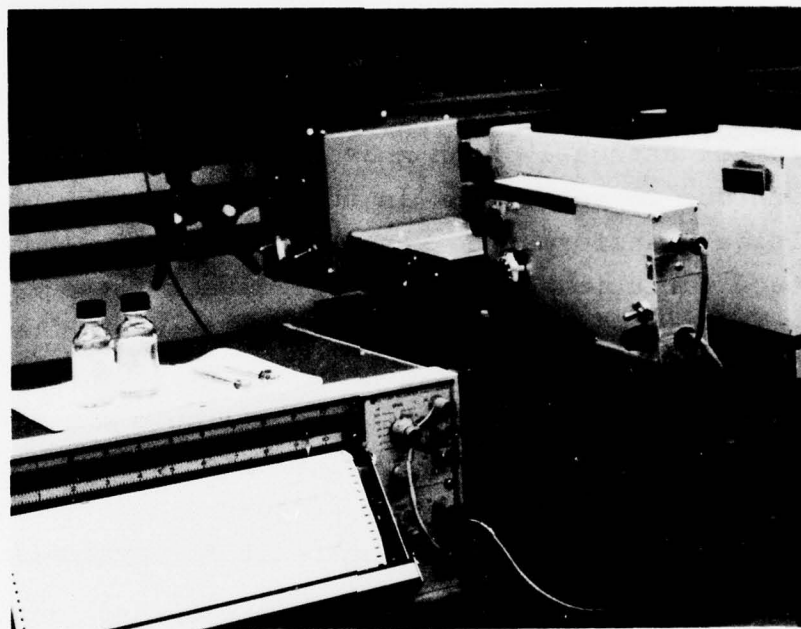


Figure 3. Prototype Detector Installed in Gas Chromatograph.

Varying the cell temperature was observed to produce reversible and reproducible changes in the baseline. Increased temperature moved the baseline in the direction of greater UV absorption. These variations were ultimately traced to a slight distortion in the optical path as a result of heating. Potential thermal distortion of the cell was anticipated and methods of rigid mounting and reinforcing were used to minimize this effect. Subsequent efforts to eliminate thermal distortion from this cell were not completely successful and construction of a new cell was planned.

The problem of unusually high noise at high amplifier gain was shown to be due to variations in the UV lamp. The use of a constant voltage transformer to supply power to the lamp eliminated line voltage variations as a possible cause of the fluctuations. The GE No. G4S11 lamp originally used was replaced by a GE No. G4T4 lamp. This latter lamp unit, consisting of lamp, ballast and starter, was temporarily removed from a liquid chromatograph detector. The lamp, having a U-configuration as opposed to the global shape of the previous lamp, required construction of a temporary lamp housing to accommodate it for evaluation.

A new absorption cell of heavy stainless steel construction was fabricated. Dimensions of the optical path were essentially unchanged from the previous cell. Since the amount of radiation which could be passed through the cell was more than adequate, quartz windows were used at either end of this cell. The stainless steel block was heated by means of a cartridge heater which was inserted into a cavity that paralleled the optical path. A drawing of the cell is presented in Figure 4.

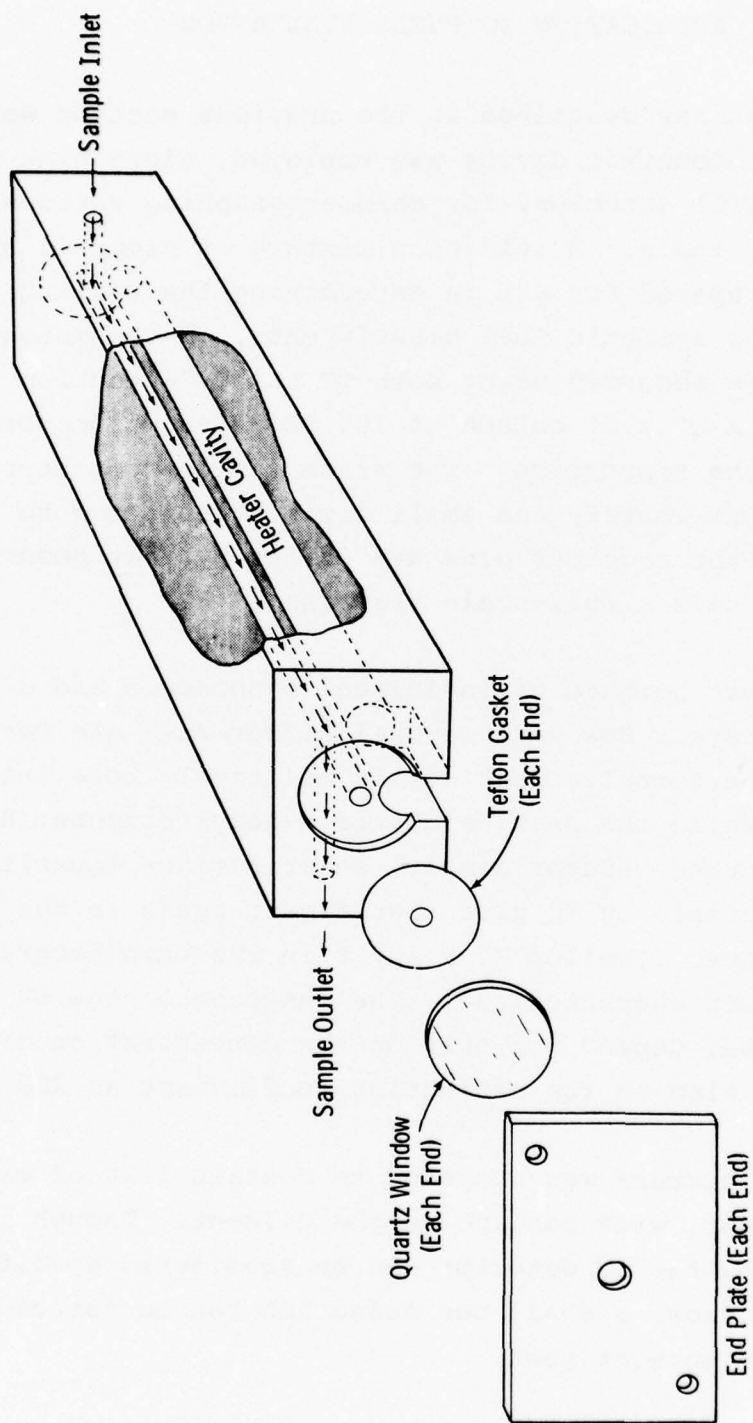


Figure 4. Redesigned Absorption Cell.

SECTION III

APPLICATION TO FUELS EVALUATION

After modifications described in the previous section were completed, the photometric device was employed, along with a thermal conductivity (TC) detector, for chromatographing various aircraft turbine engine fuels. A reference mixture of aromatic hydrocarbons was prepared for use in determining the boiling point distribution of aromatic fuel constituents. A chromatogram of the mixture was recorded using both TC and UV detection as shown in Figure 5. A $\frac{1}{4}$ " x 5' column of 10% SE-30 on chromosorb W-HP was used for the separation. The trace for one detector is shifted by approximately one small division relative to the other because the recorder pens are offset by that amount to allow each to have a full-scale traverse.

In general, peak heights of individual components are similar for the two detectors. However, several differences are worthy of notice. The tetralin peak (G) is relatively more intense in the UV trace while the peak height of n-hexyl benzene (H) is less in that trace. Surprisingly, several minor impurities which are just detectable by TC give significant peaks in the UV trace. These impurities, labelled X, Y and Z in the chromatogram, have not been further characterized. The response of the UV detector will, of course, depend not only on the concentration of the component but also on its extinction coefficient at 255 nm.

The reference mixture was prepared to contain 1-2% of each listed component, with pentane as the diluent. Though in practical terms the UV detector can be considered specific for aromatic compounds, a small pen deflection can be noticed for the massive alkane solvent peak.

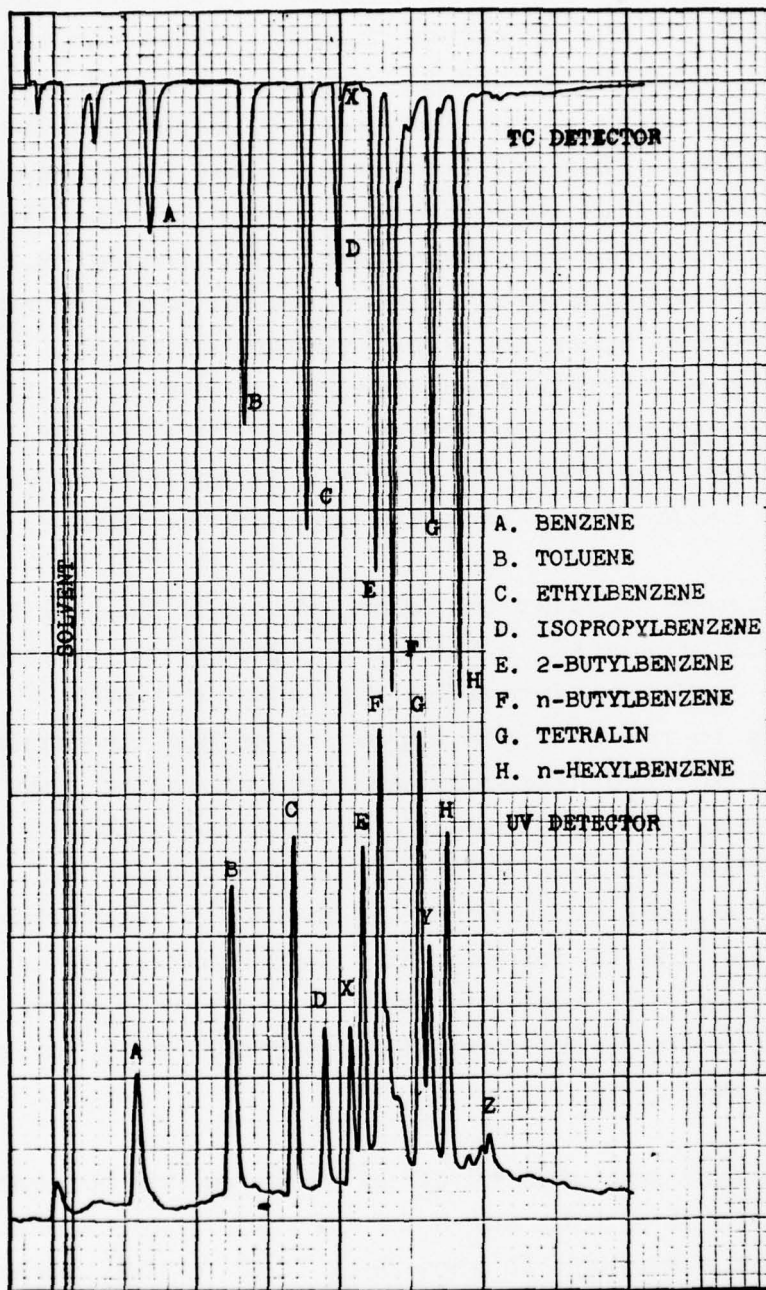


Figure 5. Aromatic Reference Mixture Using Dual TC/UV Detectors.

Figure 6 shows a plot of retention time versus boiling point for the components of the reference mixture. Based on this retention time/boiling point relationship, impurity X would have a boiling point of 165°C which agrees with that of methylethylbenzene (B.P. 165.15°C). Similarly, impurity Y has a calculated boiling point of 213°C. This component could be a C₆-substituted benzene (B.P. 211-213°C) or a C₂ alkyl-substituted indane (B.P. 213-214°C). Naturally as molecular weight increases so does the number of possible compound types and substitution positions. Identification using boiling point/retention time relationships is uncertain beyond the range of low molecular weight benzene derivatives. A plot such as that shown in Figure 6 is useful for characterizing the boiling range of the aromatic part of the sample, however.

A chromatogram of regular gasoline, obtained by dual TC/UV detection, is presented in Figure 7. Though the column and chromatographic conditions provide only moderate resolution, a number of the peaks in the UV trace appear to be due to single components. Responses of the two detectors were attenuated so that all peaks remained on scale. Accordingly, attenuation factors were 64 for the TC detector and 50 x 8 for the UV detector. Under the conditions employed, the UV detector is obviously substantially more sensitive than the TC detector for individual aromatic components. This is particularly apparent late in the chromatogram where the TC detector peaks are quite small though the UV detector peaks are at their greatest.

Regular gasoline may consist of up to 45 volume percent aromatics, in contrast to JP-4 fuel which is not more than 25 percent aromatic. Figures 8 through 10 show dual TC-UV chromatograms of several JP-4 specimens. The TC-chromatograms show that while the overall composition of these fuels are quite different, the aromatic portions are shown by the UV detector to be quite similar. Many aromatic constituents appear to be common to all three fuels.

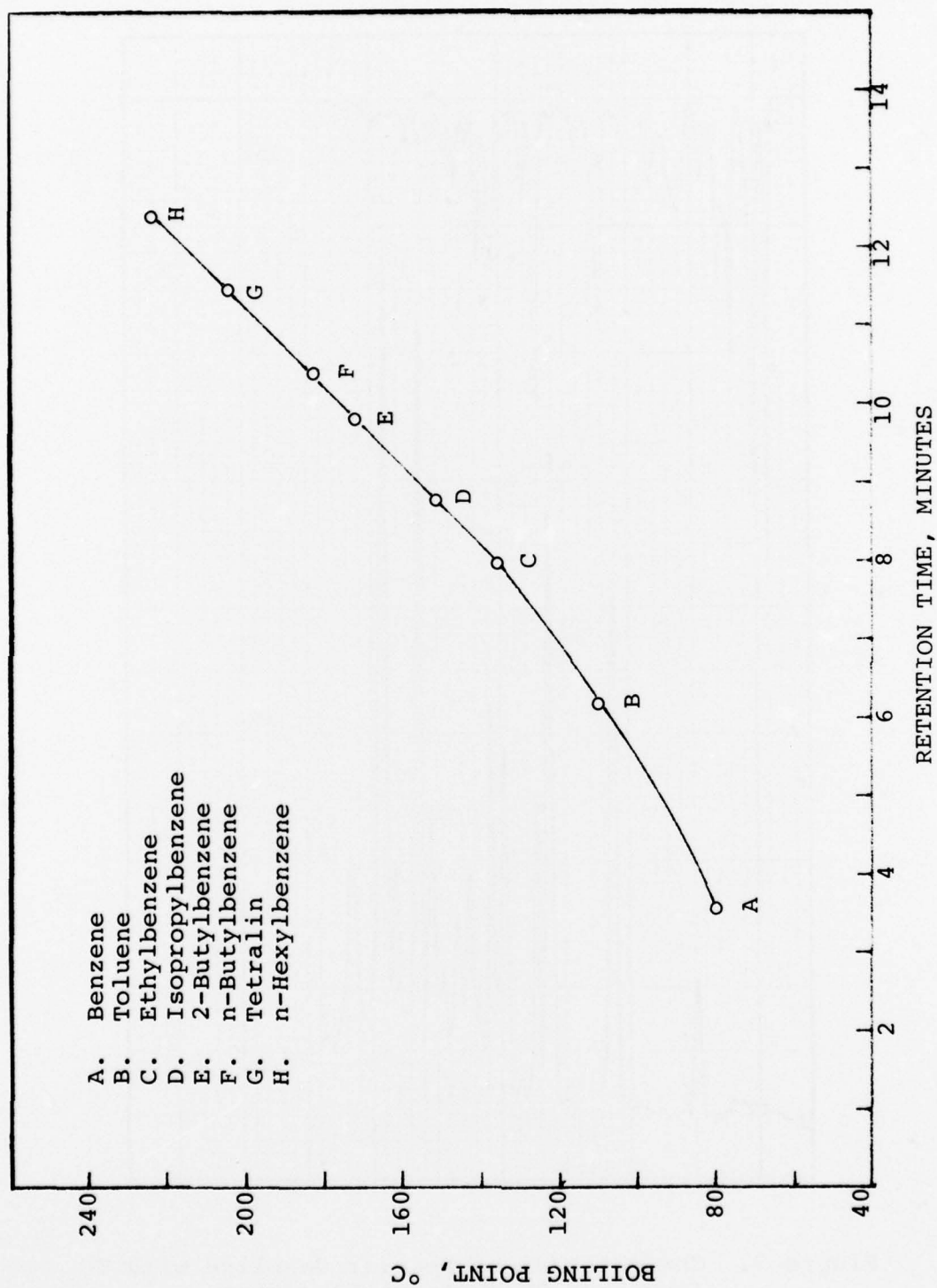


Figure 6. Retention Time Boiling Point Plot for Aromatic Reference Mixture.

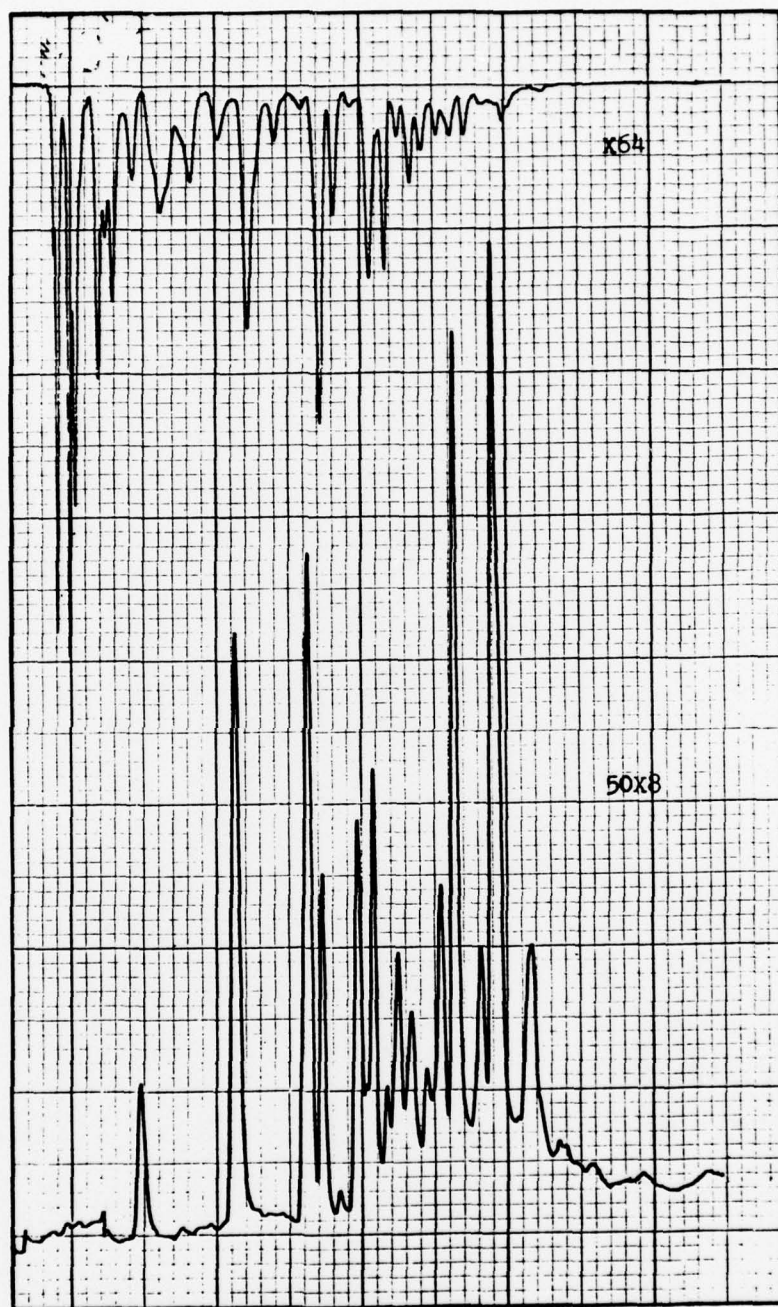


Figure 7. Chromatogram of Regular Gasoline with TC (top) and UV (bottom) Detectors.

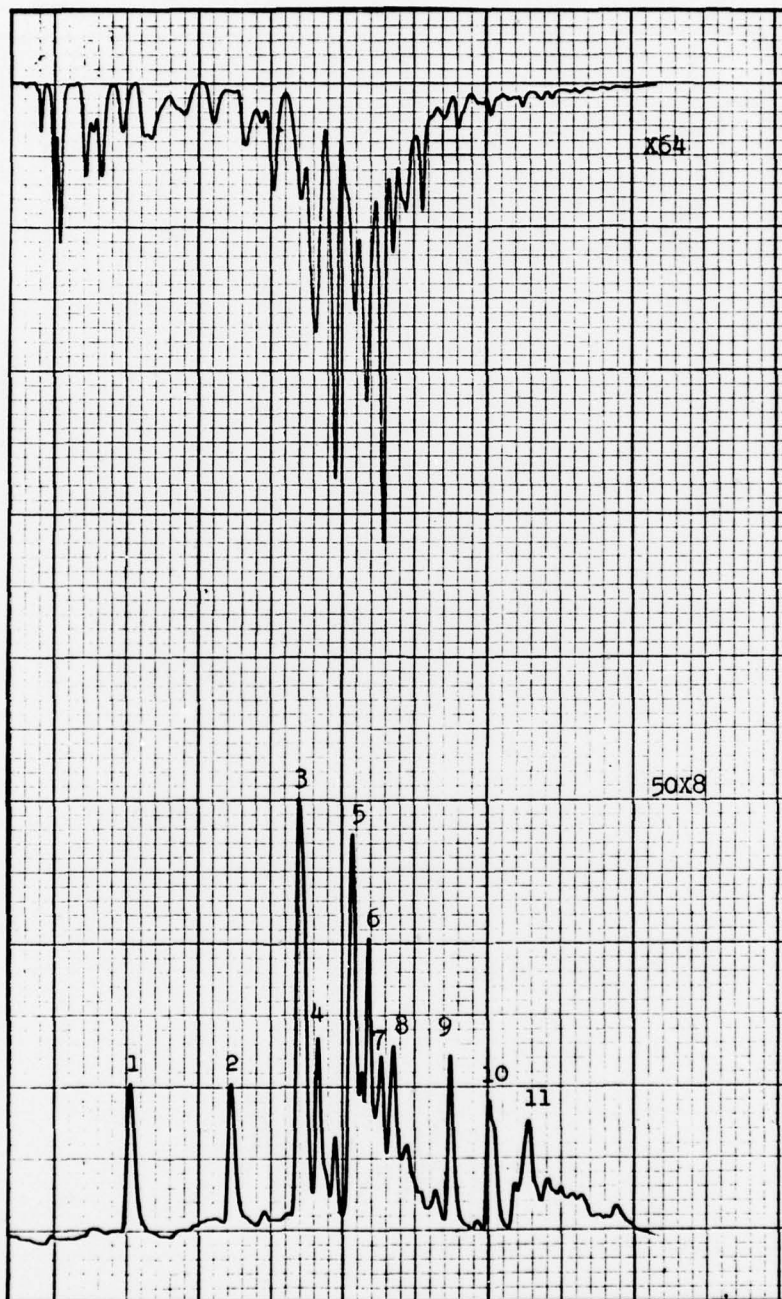


Figure 8. Chromatogram of JP-4 (Code 1019) with TC (top) and UV (bottom) Detectors.

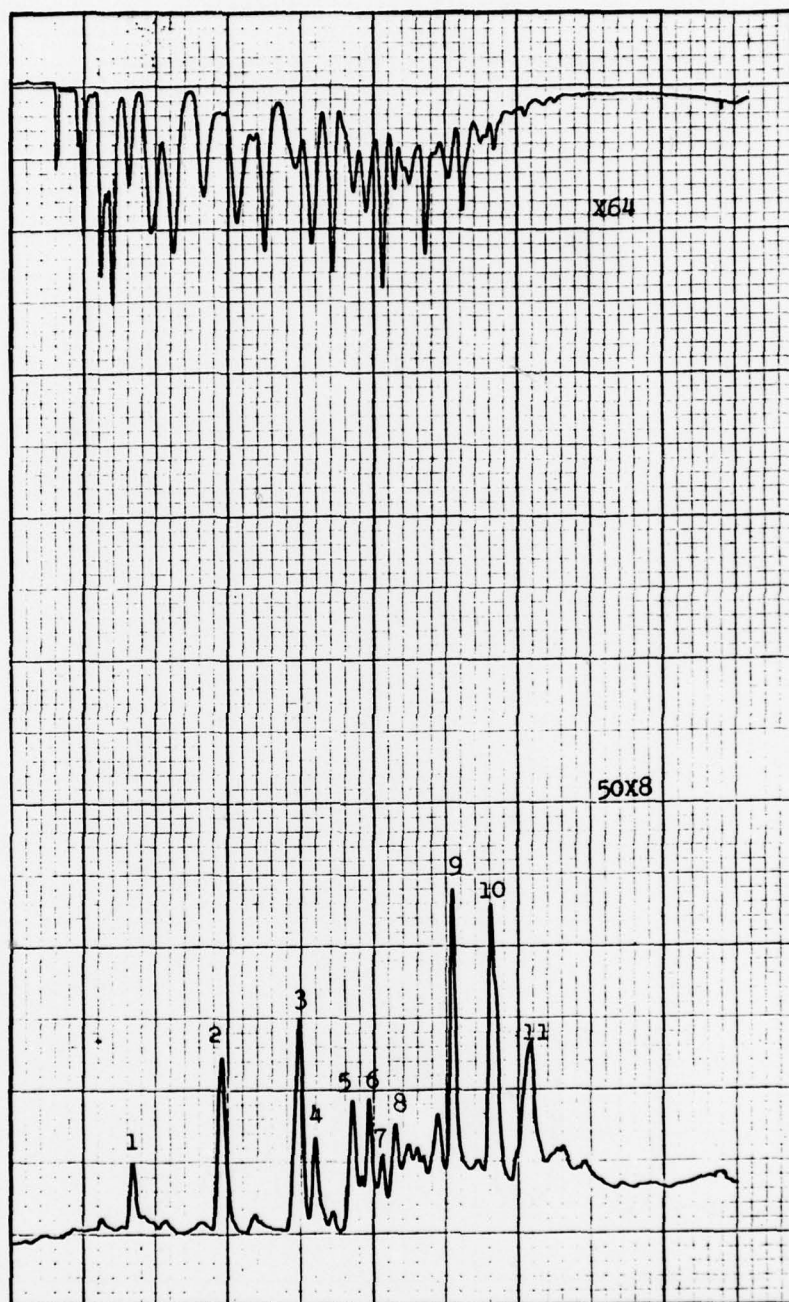


Figure 9. Chromatogram of JP-4 (Code 1044) with TC (top) and UV (bottom) Detectors.

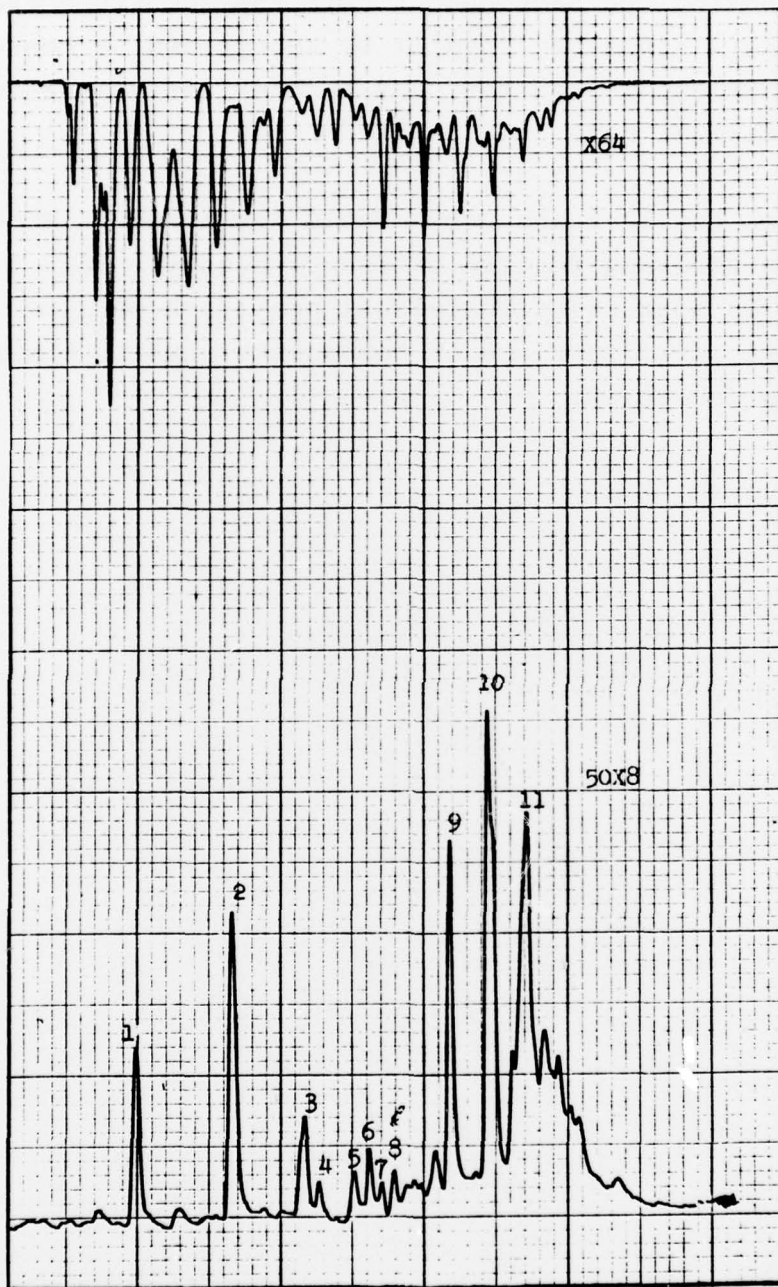


Figure 10. Chromatogram of JP-4 (Code 1061) with TC (top) and UV (bottom) Detectors.

To aid in comparing major components, each peak is numbered and components having the same number have the same retention time. Components 1, 2 and 3 can readily be recognized as benzene, toluene and xylenes. However, any further assignments would require additional data.

Chromatograms for JP-8 and Jet A fuels are shown in Figures 11 and 12. The striking feature of the UV detector data is its simplicity; in most cases individual peaks are obtained rather than the unresolved envelope of components usually observed by TC or FID detection.

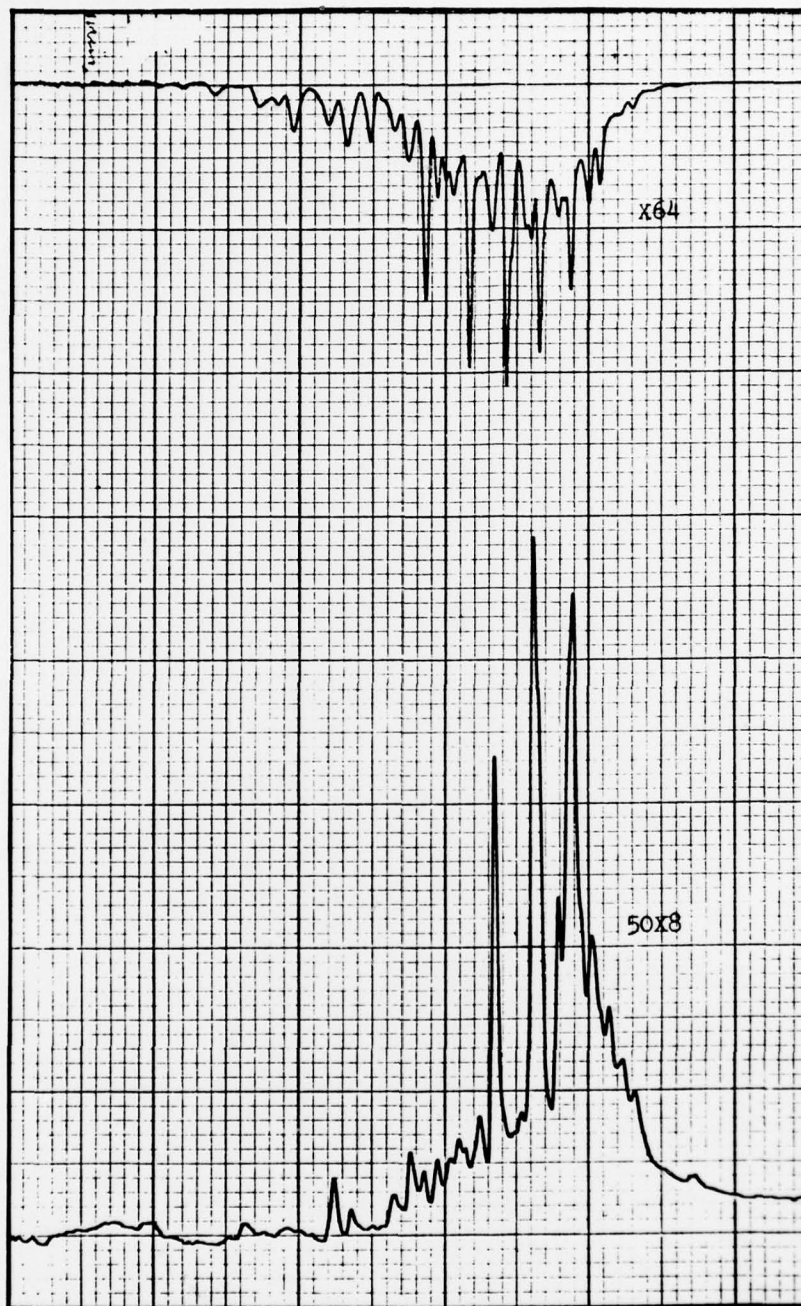


Figure 11. Chromatogram of JP-8 with TC (top) and UV (bottom) Detectors.

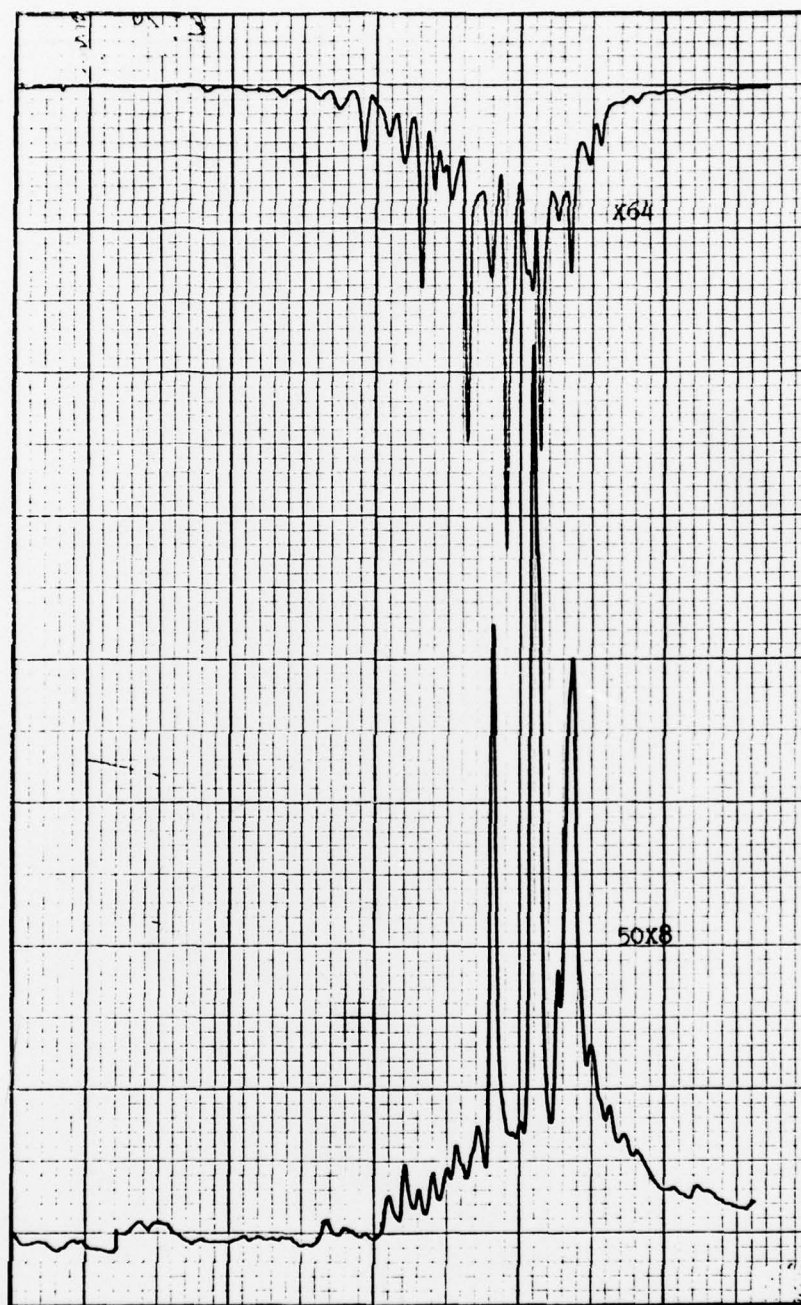


Figure 12. Chromatogram of Jet A Fuel With TC (top) and UV (bottom) Detectors.

SECTION IV

CONCLUSIONS

This investigation has shown that a UV photometric detector can be used in gas chromatography to enable a detailed examination of the aromatic portion of hydrocarbon fuels. While sophisticated analytical techniques such as gas chromatography/mass spectrometry (GC/MS) and nuclear magnetic resonance (NMR) can be used to characterize aromatic constituents, they lack the simplicity and attendant low cost of the UV detector. The purpose of this work has been to show the feasibility of a UV detector by construction of a workable unit. The device which was constructed during the course of this project has been used for chromatographing a variety of fuels and its potential for analyzing fuel aromatic constituents has been fully demonstrated.

SECTION V

POTENTIAL APPLICATIONS

Quantitative analytical methods have not been developed as a part of this work and these could logically be the subject of other investigations. However, even without further method development, the device can be extremely useful in qualitative applications or for simple comparisons of fuels. For example, it is frequently desirable to strip aromatics from a fuel for their further analysis. If the composition of the aromatic fraction is altered by its removal from the fuel, errors in subsequent analyses will result. The UV detector can be used to monitor the stripping process by comparing the chromatogram of the fuel before removal of aromatics, with the chromatogram of the aromatic fraction after its removal. The two chromatograms should be very similar for a good separation. If volatiles are lost, or other changes occur, it would be immediately apparent.

The usefulness of the UV detector extends beyond fuels analysis to a variety of other applications. Substituted aromatic hydrocarbons are known to participate in atmospheric photochemical reactions leading to smog formation. Consequently their deliberate release in significant amounts into the atmosphere must meet rigid Air Quality Standards which set the permissible emission levels for various aromatic constituents. Hydrocarbon emissions resulting from large-scale paint spraying and drying operations can readily be out of compliance if paints or solvents having high concentrations of certain aromatics are used. Olefins, while also photochemically reactive, are generally not important constituents in paint formulations. The UV detector could be used to establish procedures whereby the exact aromatic composition

of paints and solvents is quickly and inexpensively determined.

Benzene has been shown by clinical and epidemiological data to be a leukemia causing agent. The Occupational Health and Safety Administration has recently issued an emergency temporary standard for occupational exposure to benzene. Accordingly, mixtures containing one percent or more of benzene are to be treated as controlled substances. Many materials containing petroleum distillates, including paints, polishes, adhesives, cleaners, solvents, etc. may contain benzene at restricted levels. Separation of benzene from other paraffinic and naphthenic hydrocarbons by conventional gas chromatography requires high-resolution conditions, or a special column material such as one of the several currently being used to retard elution of aromatics relative to aliphatic constituents. Using the UV detector, practically any column which will pass benzene may be used for its measurement.

Extensions of the detector's capability could be made by changing the wavelength of absorption. The device was intentionally kept simple by elimination of costly and fragile optical components such as a monochromator or a lens system requiring critical alignment. The wavelength, however, can be readily altered, by changing to a different band-pass filter. Naphthalenes, for example, could be selectively detected in hydrocarbon distillates by their absorption at 275 nm wavelength. The amount of naphthalenes in aircraft fuels strongly influences combustion characteristics and the permissible level of these compounds is usually specified separately from the value for total aromatics.

Wavelength selection by use of appropriate band-pass filters can potentially allow the detector to enhance selectivity for virtually any UV-absorbing compound. This capability could greatly simplify the gas chromatographic analysis of complex mixtures such as those of biological, environmental or pharmaceutical origin.

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