Report to
Scientific Officer
Bureau of Naval Weapons
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TEST PROGRAM ON DURABILITY
OF
AIRCRAFT COATINGS (U)

Final Report

McCrone Associates Project No. 458

Prepared under Contract NOw-66-0391-c
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ABSTRACT

A laboratory investigation was conducted to develop methods for rapidly determining paint coating resistance to oxidation and photochemical degradation. The ultimate objective is development of a rapid test for expected life of paint coatings.

Accelerated aging of paint films has been achieved using ultraviolet radiation and oxidizing atmospheres. The resulting gases given off by the films have been analyzed by gas phase chromatography. The chemical nature of the paint film can be identified by this means, and the actual compounds evolved by a series of films have thus been identified. Interestingly enough, most belong to a homologous series of methyl ketones (acetone, methyl ethyl, methyl propyl, methyl butyl and methyl pentyl), although methanol, water, carbon monoxide and carbon dioxide were also found. A study of polyurethane films of known history showed a good relationship between service life for each film and the chemical composition of the gases evolved.

Further work is in progress to confirm these promising results and to extend the study to other paint compositions.

PURPOSE

The purpose of this program is to develop rapid tests useful in predicting the long-term service durability of exterior finishes on aircraft-tests based on observed accelerated chemical and physical steps in the coating degradation.

INTRODUCTION

Service life, or durability, of paints subjected to outdoor exposure and to the variable effects of flight conditions on aircraft surfaces depends upon resistance to the stresses of oxidation, ultraviolet radiation, heat, oil and abrasion.

Many methods and instruments have been developed for evaluating physical and chemical changes in the coating in an attempt to predict its performance. The most common instrument is the accelerated weathering machine. Ideally this device should simulate outdoor exposure conditions and thus rate coatings according to their expected serviceability. Even though these instruments are widely used, the results are often in question, and the search for further methods continues.

Another approach to the study of coatings is to determine changes in the physical properties of a film after it has been exposed to selected environmental conditions. Most often changes in flexibility, elasticity or tensile strength have been measured after exposure to oxidation, heat, cold, ultraviolet radiation or some other condition.

Still another approach is to follow chemical changes in the coating subjected to environmental factors. This is generally more difficult and time-consuming than physical tests but gives more fundamental information. Observing the chemical changes, however, has been hampered by the fact that many are very complex, and suitable or sufficiently sensitive methods or instrumentation simply did not exist.

In some cases, changes have been too small to be identified or measured, but with modern instruments, the chemist can now detect chemical changes in paint films which were undetectable just a few years ago.

In this experimental program, evaluation of chemical changes has been emphasized. We have used ultraviolet radiation and oxidating atmospheres, examined the resulting off-gases by infrared spectroscopy and gas chromatography and then studied the relation of these results to service life.

COATING SYSTEMS

The coatings of primary interest to the Navy are of the following four types:

(1) nitrocellulose acrylic MIL-L-19537
(2) plasticized acrylic AML-95K
(3) polyurethane AML-P43
(4) epoxy-polyamide MIL-C22750A

The contractor is not expected to expose paint panels; NAEC is to provide aged painted panels as they become available.

LABORATORY INVESTIGATION

Gas-chromatographic precolumn reactor

An ultraviolet precolumn reactor for the gas chromatograph, designed and built during the first year of this program, showed promising results. It was, however, greatly improved during the past year by reducing the internal volume of the reaction chamber. This allows the gaseous reaction products to be more readily swept into the gas chromatographic column, producing better-defined elution peaks. The present reactor will take a 2 x 3-in. paint film in a cylindrical cell. The inner wall is a Vycor tube, 7/8 in. in diameter, sealed with Teflon rings on each end. The reactor is connected to the bypass or sampling loop of a gas valve and can be filled with carrier gas or, optionally, with any other gas or reactive atmosphere. After the flash tube is fired, the gas valve is opened and the vaporized decomposition or reaction products are swept into the analytical column by the carrier gas. The new reactor design also allows easier sample changing. In addition there is less chance of heat effect since, 1) the paint or film is further from the flash tube, 2) it is shielded by the Vycor tube and 3) the cell volume is smaller, giving better gas chromatograms.

A difficulty still present in this program is the lack of Navy samples with completely known performance characteristics. In the absence of knowns from the Navy some clear polyurethane varnishes were available which had been tested in Florida to their ultimate failure. Cast films of each of the five were dried at least two weeks, then tested in the photoflash reactor to see if field testing could be correlated with laboratory testing. The gas chromatograph was equipped with a hydrogen flame detector and the electrometer was set to high sensitivity. Each film was flashed once, then the products were flushed into the column and separated. Under these conditions, all of the films showed a major chromatographic peak with retention time of fourteen minutes (Figure 1). If the varnishes are listed in the order of increasing height of this peak, they fall in the order of length of service in Florida. That is, the most stable film had the smallest peak; the least stable, the largest.

Three oil-based paints, differing only in the type of titanium dioxide pigment were tested similarly. One was pigmented with a non-chalking rutile pigment, a second with a less chalk-resistant rutile and a third with anatase. In normal exposures it would be expected that the nonchalking pigment would be most UV-resistant and the anatase pigment least chalk-resistant. When these paints were tested in the photoflash reactor, however, there was no clear-cut correlation between the chromatographic results and the expected exterior performance. This is seen in Figure 2, which shows chromatograms of the off-gases after UV flashing. Note that the oil-base paints (Figure 2) show a very different GC spectrum than the polyurethane-based varnishes (Figure 1).

Low-energy ultraviolet study

Decomposition products form when the paint films are subjected to light from the ultraviolet photoflash tube. This high-energy flash is accompanied by some heat, however, and we wished to check whether lowering the energy of the ultraviolet radiation would produce the same products.

Low-energy ultraviolet light was used during the initial stages of last year's work. Infrared spectra of the gaseous products and attenuated total reflectance (ATR) infrared spectra of the paint films were determined. These studies were stopped at that time because the spectra of the gaseous products were too weak and the ATR spectra were unsatisfactory. The films were subjected to very little heat, however, so that no thermal decomposition products would be expected to form.

During this year samples of a variety of films were studied after exposure to low-energy UV for up to twelve days. Two pigmented oil-base films were first exposed for periods ranging from 29 to 40 hr. Gas samples were then removed with a gas syringe and injected into the same gas chromatographic column that was used for the high-energy UV flash studies. Product concentrations (Figure 3) were much less with the low-energy radiation, due in part to cell size, but the retention times were similar to those produced in the photoflash reactor. (Compare with Figure 2.) A higher concentration of reaction products for identification could have been obtained by sweeping gas from the cell into a liquid nitrogen trap.

The effect of low-energy ultraviolet radiation on different films of MIL 19537, MIL 22750 and AML 95K was studied by infrared spectroscopy of the off-gases (Figure 4). Only the region from 2.5 to 8μ is shown since the rest of the spectrum, from 8 to 15μ, showed no significant absorption. The major effect of low-energy UV radiation is the increase of carbon dioxide in the off-gas shown by the band at 4.3μ. Occasionally other peaks show up, e.g. the 5.7μ band in the spectrum of the AML 95K sample. Absorption at 5.7μ decreases, however, with longer exposure. To determine the effect of still longer exposure, two polyurethane films were exposed to low-energy ultraviolet; one film had no stabilizer and a second, a phenolic modified polyurethane, contained an ultraviolet inhibitor. Infrared spectra are shown for the carbon dioxide absorption at 4.3μ and carbon monoxide at 4.6 and 4.75μ in Figure 5. These data seem to show little difference between the two films. However, ultraviolet radiation intensity was three to four times greater for the phenolic modified polyurethane. The greater stability of this film is indicated by the CO_2 being about the same and not greater than in the less stable film.

Oxidation precolumn reactor

In the first year's work, an oxidative reactor was developed into which 5-10 cc of oxygen could be injected. As in the UV experiments, the off-gas products could be separated and analyzed by gas chromatography. This reactor appeared promising, but the temperatures required to produce oxidation reactions which would give enough off-gas with some of the coatings were too high. It seemed desirable to develop a method which would yield oxidation products at lower temperatures, to which the paints would be exposed under more normal operating conditions.

One way to obtain more oxidation products at lower temperatures is to increase the time that the oxygen is in contact with the coating. This was done by attaching the reactor to a gas valve in place of a conventional gas sample loop. With this setup, the vehicle under study is coated on an inert support as in previous tests and, after curing, is placed in the reactor. The precolumn reactor is then filled with ozone-enriched oxygen and allowed to stand for the desired time at the desired temperature. The oxidation products are then analyzed by gas chromatography. When the revised reactor was tested with oxygen in contact with the material for 10 to 20 min, we obtained sufficient oxidation products at 150°C. Previous tests with the same vehicle had produced insufficient products at 200°C. In the previous system, any thermal decomposition products were swept continuously from the reactor into the analytical column, resulting in a constant background. A slight disadvantage in the new system is that, because the reactor is not in the gas stream when it is filled with oxygen, any thermal decomposition products also accumulate along with oxidation products.

Use of ozone-enriched oxygen made it possible to reduce still further the amount of thermal decomposition. Ozone oxidation at 100°C yielded sufficient off-gas for the chromatographic analysis and at this temperature there is little thermal decomposition. The ozone generator which was built is described in the appendix.

The precolumn reactor was coated with 20% of the vehicle, as in the other oxidation tests. Ozone-enriched oxygen was injected into the reactor and allowed to react. The gas products were then swept into the analytical column of the gas chromatograph for analysis. A wide variety of experimental conditions were tested to find those which would yield sufficient oxidation products at a low enough temperature that interference from thermal degradation could be minimized. It was found that five cubic centimeters of two to three percent ozone, injected into the reactor at 100°C and shortly afterward into the analytical column, yielded sufficient oxidation products with essentially no thermal degradation products.

In experiments with ozone-oxygen at 30-35°C, there were no detectable off-gases with any of the tested vehicles, regardless of amount of oxygen or reaction times up to five minutes. With reactor temperatures of 70-75°C, five cubic centimeters or more of ozone-oxygen and reaction times less than one minute, occasional runs were obtained with sufficient off-gases from a coating of low stability, but not with a phenolic modified polyurethane coating containing a UV stabilizer. At 100°C, with 5 cc of oxygen, in more than 15 seconds, enough gas products were obtained from all of the coatings for gas chromatography using flame detection.

Even though we cannot eliminate all thermal degradation products, it is felt that their quantity is so small that they cannot change or negate results obtained to this point. In most cases such products have retention times similar to those of the oxidation peaks.

The same films that had been previously tested in the ultraviolet photoflash reactor were tested with the ozone-enriched oxygen atmosphere. Correlation of results with the oxidation precolumn reactor was not as clear-cut as with the UV flash reactor. Although the same general trend was followed, a few results deviated from the field test results. Chromatograms from the study are shown in Figure 6. The same major peak is shown by four of the polyurethanes, though its amplitude varies slightly.

One observation that can be made from the chromatograms of both the oxidation and the ultraviolet reactions is that the major peak of the series of films appears to be the same. Therefore we felt that, if we could identify the products from the oxidation reaction, it would help us to understand the film degradation mechanism as well as the reason why this major gas chromatographic peak seems to indicate a more stable film.

Identification of gas chromatograph peaks

Initial attempts were made to collect the gas peaks as they eluted from the gas chromatograph. Several successive samples were run and the products, condensed in a stainless steel coil, were kept in liquid nitrogen. However, when the material was heated and injected into the evacuated gas cell, there was not enough material to produce an identifiable infrared spectrum.

We therefore decided to liquefy the volatile products immediately after they left the reactor, rather than from the gas chromatograph. Several reaction runs were made with ozone-enriched oxygen and the products were collected in the stainless steel coil immersed in liquid nitrogen. The coil was then warmed to 35-40°C and the gases were injected into an evacuated infrared gas cell. Figure 7 shows the infrared spectra. In the upper curve the most prominent peak observed is the carbonyl peak, at 5.7μ; also present are C-H stretching at 3.4μ, the CO_2 peak at 4.3μ and the methanol peak at 9.7μ. A few other, minor bands are too broad or too vague to be interpreted. A second sample was injected from the same condenser heated to 100°C. Its infrared spectrum, the lower curve of Figure 7, shows only water and a little carbon dioxide.

Rather than continue to collect products from successive reactions till we had enough to identify some of the compounds by infrared spectroscopy, we decided to try a two-column gas chromatographic method for characterizing the off-gases.

Figure 8 shows the basis of this method of identification. The relative retention times of known compounds on two different types of liquid phases are plotted. It will be observed that the slopes are straight lines for each homologous series, and are characteristic of the functional groups. By chromatographing our reaction products under the same conditions as were used for the standards on these two columns, we can identify the types and number of homologous series.

The first column used was a 12-ft silicone grease column; the second, a 13-ft diisodecyl phthalate column. With the two-column method, we were able to identify most of the off-gases boiling above room temperature as a series of ketones from acetone through heptanone. From the infrared studies, it is also obvious that the major components are water, carbon monoxide and carbon dioxide. Some low-boiling hydrocarbons, such as methane and ethane, probably are present as well. The series of individual peaks from the gas chromatograms, however, are due to compounds boiling above room temperature.

Using the ozone-oxidation method and the precolumn reactor, we identified carbon monoxide, carbon dioxide, water, methanol, acetone, methy ethyl ketone, 2-butanone, 2-hexanone and 2-heptanone from the polyurethane films. We are more certain that the carbonyl is in the 2 position in 2-butanone, 2-pentanone and 2-heptanone than we are in the case of hexanone. The most prominent peaks observed in the chromatograms were assigned to 2-pentanone and 2-hexanone.

The gas chromatograms from ultraviolet flash degradation and also from the precolumn oxidation tests are broad and diffuse because the vehicle film acts as a liquid phase and retains the off-gases. This was confirmed by another experiment. The normal column or precolumn reactor used is about 18 inches long, but when this was replaced with a six-inch column, the resulting peaks were still broad and diffuse. This indicates that there is strong attachment or solubility with the coating, which has made identification more difficult.

Final identification was made more precise by condensing the off-gases from a series of runs into a stainless steel condenser and injecting this directly into the column. The much sharper resulting chromatograms can be observed in Figures 9 and 10. The oxidation products were chromatographed over silicone and over diisodecyl phthalate.

Besides the gaseous products, a relatively nonvolatile oil emerges from the precolumn reactor. This is most likely unchanged volatile material from the vehicle. This oil was washed out of the condensation tubing with a solvent and submitted to infrared examination. In Figure 11, the upper curve is that of the oil which was washed out of the condenser; the lower spectrum is for the dried vehicle film. Taking into account that there has been a certain amount of oxidation, the upper curve shows a general similarity to that of the vehicle. The volatile oil seems to have the general appearance of a phthalic plasticizer. However, it is possible that isocyanate groups and the aromatic UV stabilizer could give similar infrared spectra.

Electron microscopy

At the beginning of the program, panels were received of the two AML and MIL coatings that had been exposed in Florida for eight months. This was four months longer than the exposure of all those submitted during the first year except the polyurethane panel, which had been weathered for one year.

Electron micrographs of the four aged panels are shown in the appendix. This magnification is 33,000X, somewhat greater than that of the previous micrographs. The polyurethane coating AML-P-43 and the plasticized acrylic coating AML-P-95K appear to have continued to weather very well. The epoxy-polyamide has also weathered well but shows the presence of a little loose pigment. The nitrocellulose acrylic panel MIL-L-19537 has shown the greatest surface damage by weathering here as well as for the panel weathered four months.

DISCUSSION OF LABORATORY RESULTS

In an overly brief summary of the investigation we could state that the most interesting results have come from ultraviolet photoflash degradation experiments, identification of ketones in the oxidation off-gases and electron microscopy.

Good correlation with expected service life was obtained with a series of polyurethane films degraded by ultraviolet flash. The most prominent gas chromatograph peak obtained was largest with the least stable film and quite small with the most stable film. The data obtained from duplicate films degraded by the ozone-oxidation method were in general agreement; the best and poorest films gave the same results as with the ultraviolet method, but films of intermediate stability gave less conclusive results.

The series of gas chromatographic peaks in the oxidation method was identified as a homologous series of ketones, with the major peak being 2-hexanone. We believe that 2-hexanone is also the major peak obtained by ultraviolet degradation.

It seems somewhat surprising that most of the detectable off-gases boiling above room temperature are ketones. This may be truer for the polyurethanes than for the other three vehicles of interest to the Bureau of Weapons. More work could be done to identify the off-gases of the other coatings from both ultraviolet and oxidation methods, but since the objective of the program is to develop practical tests rather than to merely study the theory of paint degradation mechanisms, it is felt that most of the effort should be directed to method development. In the case of the polyurethanes, it seems that the ketones probably come from the alkyl groups of the ester portion of the polyols used to react with the isocyanates.

Further study of ultraviolet and oxidative degradation data obtained on the two AML, the two MIL and the subsequent polyurethane coatings suggests that either method yields "fingerprints". That is, the gas chromatograms of the off-gases are characteristic of the basic paint vehicle. Therefore it now seems unlikely that the methods can be used, without modification or simplification, as indicators of service life. The methods are useful for evaluating the most stable coating of various ones as long as they are of the same chemical family. This was illustrated above with the polyurethanes.

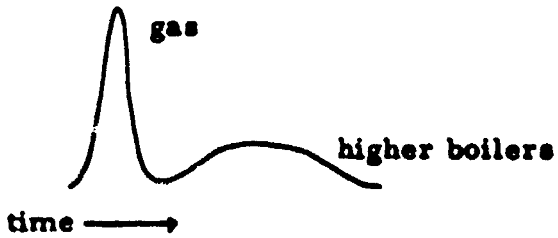
What, then, can we do with the knowledge gained thus far? One thought is that it should be possible to simplify the chromatogram system so that all of the higher-boiling products are eluted from the chromatograph as a single peak following the earlier gas peaks. One way of doing this is to catalytically oxidize these peaks to carbon dioxide and record them as a single measurement or peak. If the earlier peaks (with the exception of water) were also oxidized, we would have two carbon dioxide peaks and water. The two peaks would be expected to result in more discrimination than a single peak, although some experiments should also be conducted to indicate whether a single carbon dioxide measurement is suitable. At present, we do not think a single measurement will suffice because the low-energy ultraviolet studies show that carbon dioxide is a major off-gas and the higher boilers are trace components. The higher boilers would therefore tend to become lost in a single peak measurement.

Of the two degradation methods, the ultraviolet flash method appears to be more suitable here because it more closely duplicates environmental conditions. Some variables which have not yet been tested, due to time and cost limitations, are adding some ozone and/or moisture and slightly elevating the temperature to about 50°C. With slight modifications, the ultraviolet cell could also be made to operate under vacuum should it be found desirable to simulate upper-atmosphere pressure.

When we were making many consecutive oxidation runs at 150°C and condensing the volatile products, we noted that an appreciable quantity of oil was bleeding off due to either thermal or oxidative degradation. In either case there is the suggestion that, in high-velocity flight, the aircraft's surface temperature may be raised enough that some of the paint vehicle could be lost. This is an additional factor in favor of some form of gas chromatograph procedure.

RECOMMENDATIONS

The ultraviolet photoflash degradation method should be extended by adding moisture and ozone during the flashing. A slight elevation of temperature to 40 or 50°C may also be desirable. Then the gas chromatographic separation should be simplified to obtain fewer peaks — possibly only two or three — by shortening the separation column and/or reducing separation efficiency. A preliminary gas peak of the light gases could be followed by a second peak representing substances which boil above room temperature. This may be represented as follows:



Alternatively, we may find it more desirable to oxidize both peaks to carbon dioxide and to try to correlate the resulting data with coating life.

Degraded clear and pigmented coatings of known history should be submitted to cross-section examination by light and electron microscopy and by x-ray microprobe analysis to investigate migration, separation or the development of voids in the layers.

Respectfully submitted,

W. C. McCRONE ASSOCIATES, INC.

Richard E. Putscher

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Senior Research Chemist

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APPENDIX

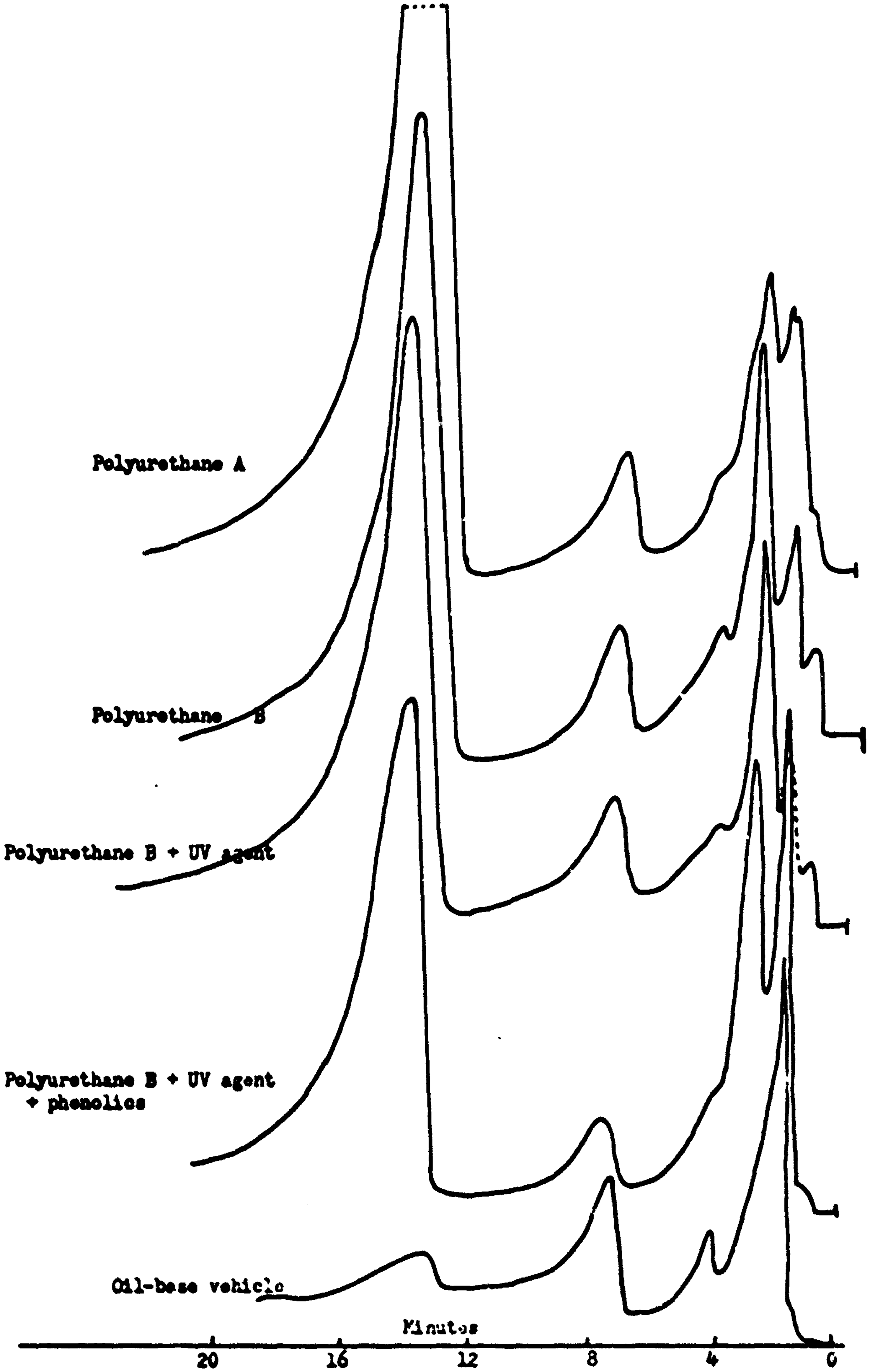


Figure 1. Volatile products from ultraviolet photoflash of clear vehicle films

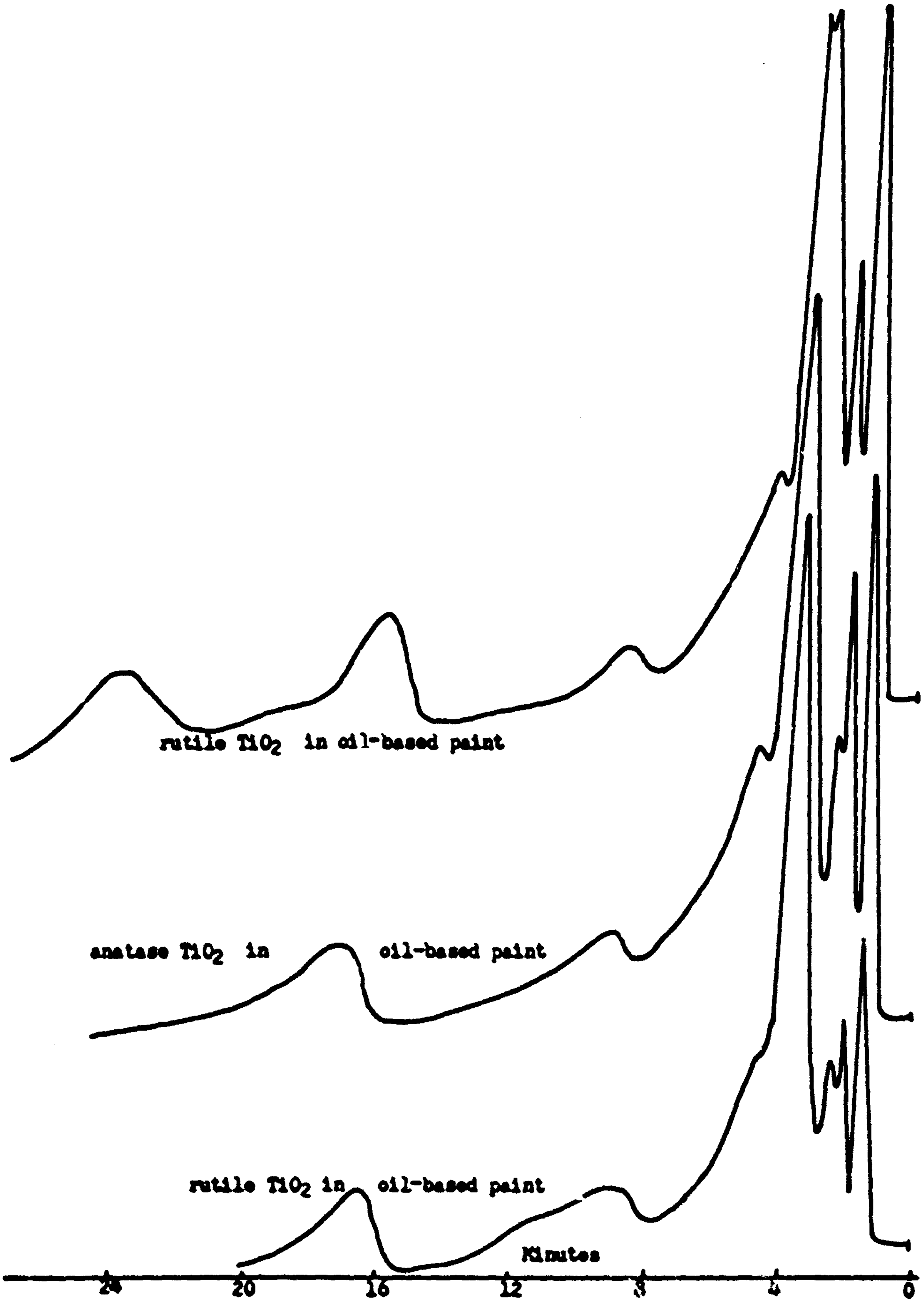


Figure 2. Off-gases from ultraviolet flash of pigmented films

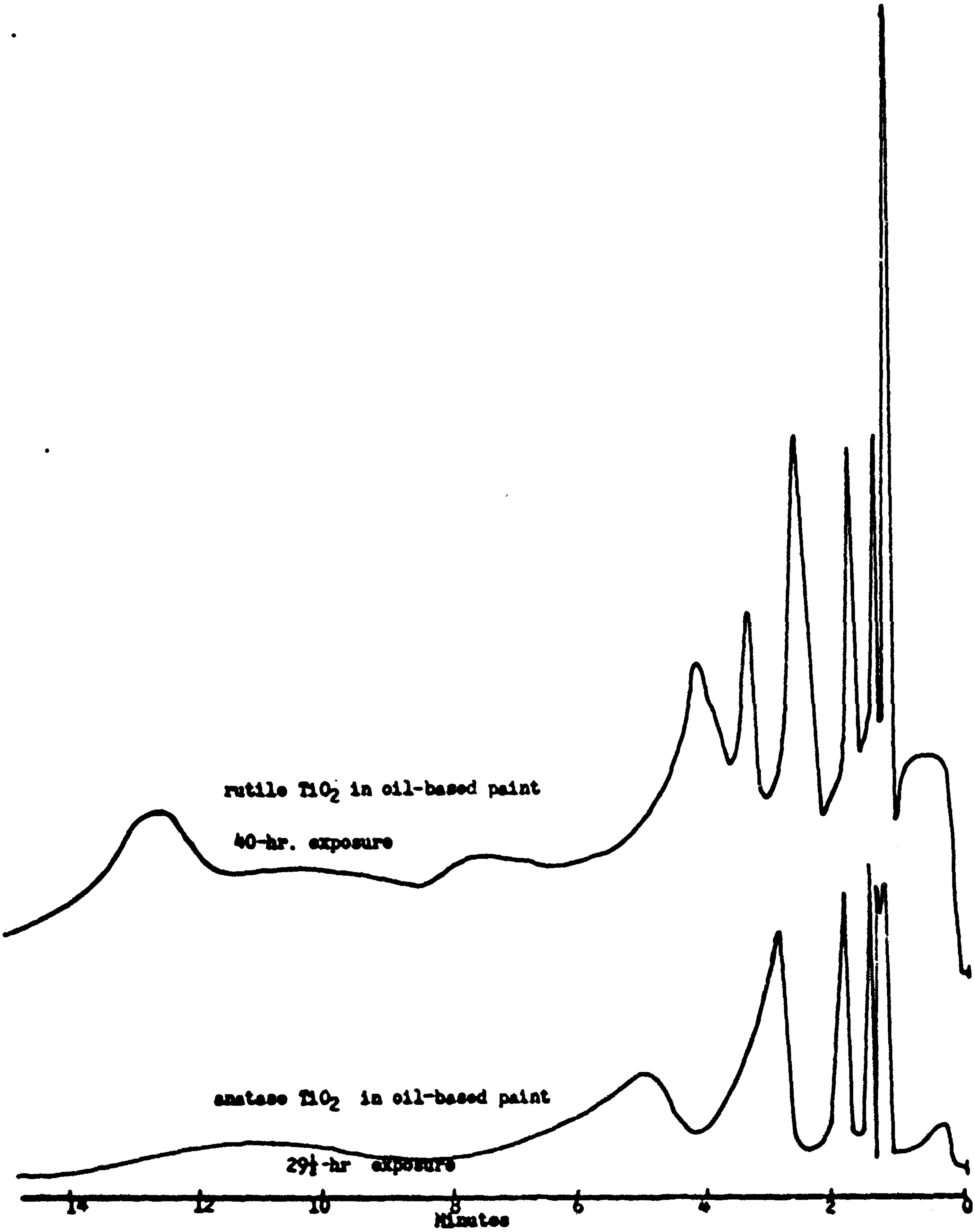


Figure 3. Off-gases from ultraviolet radiation of pigmented films

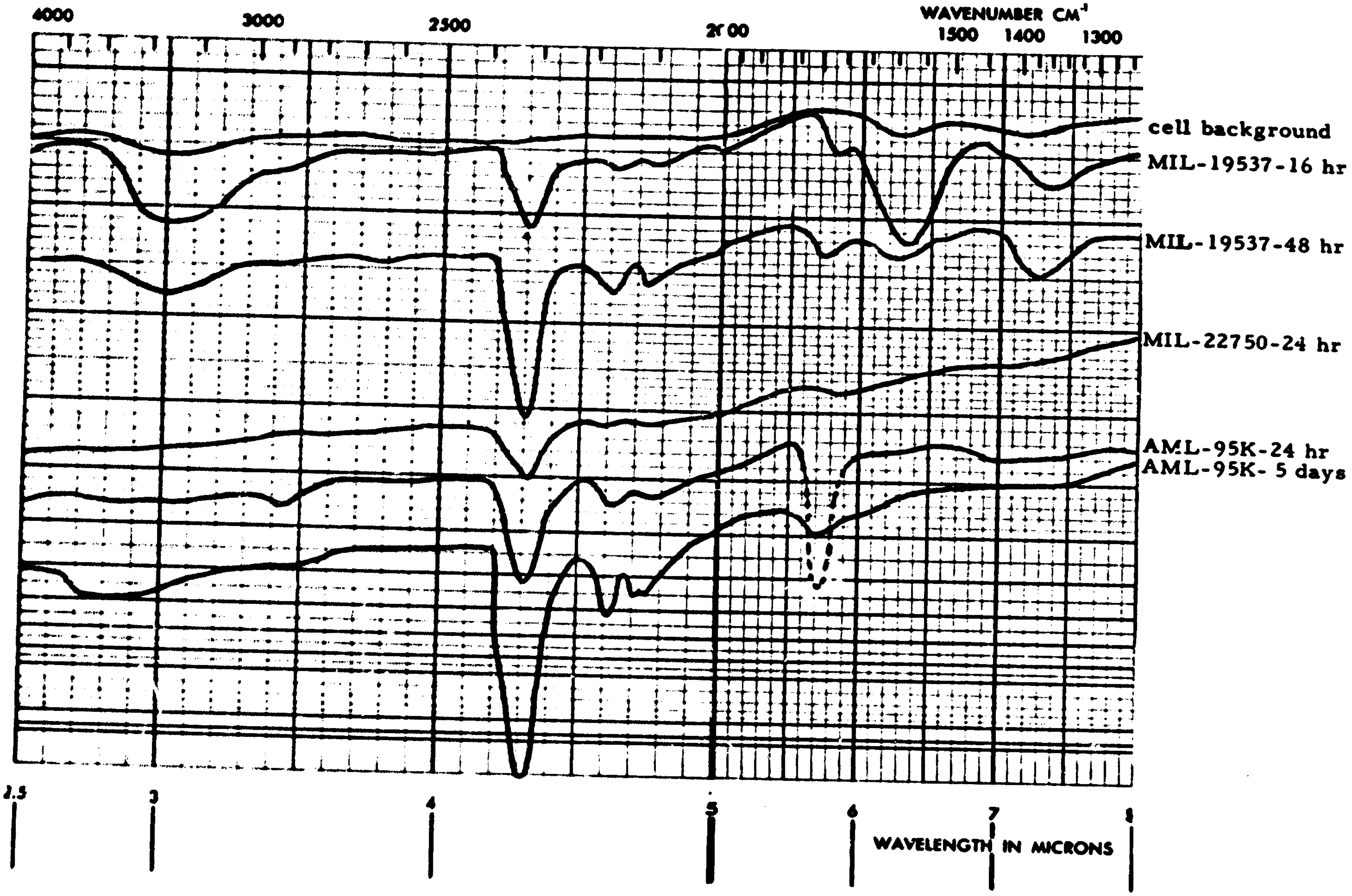


Figure 4. Effect of low-energy ultraviolet on three NAEC films

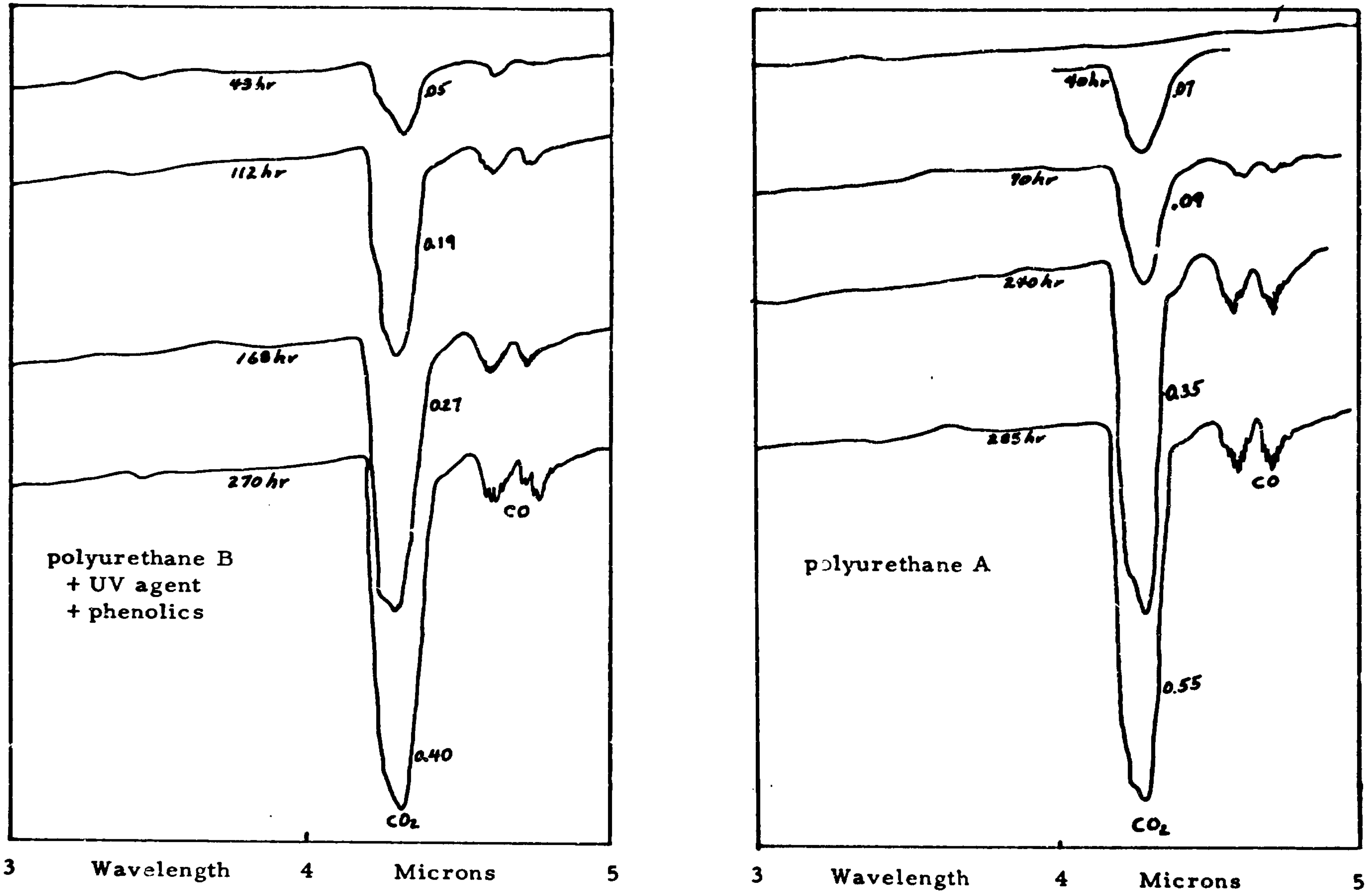


Figure 5. Effect of low-energy ultraviolet radiation on two polyurethane films

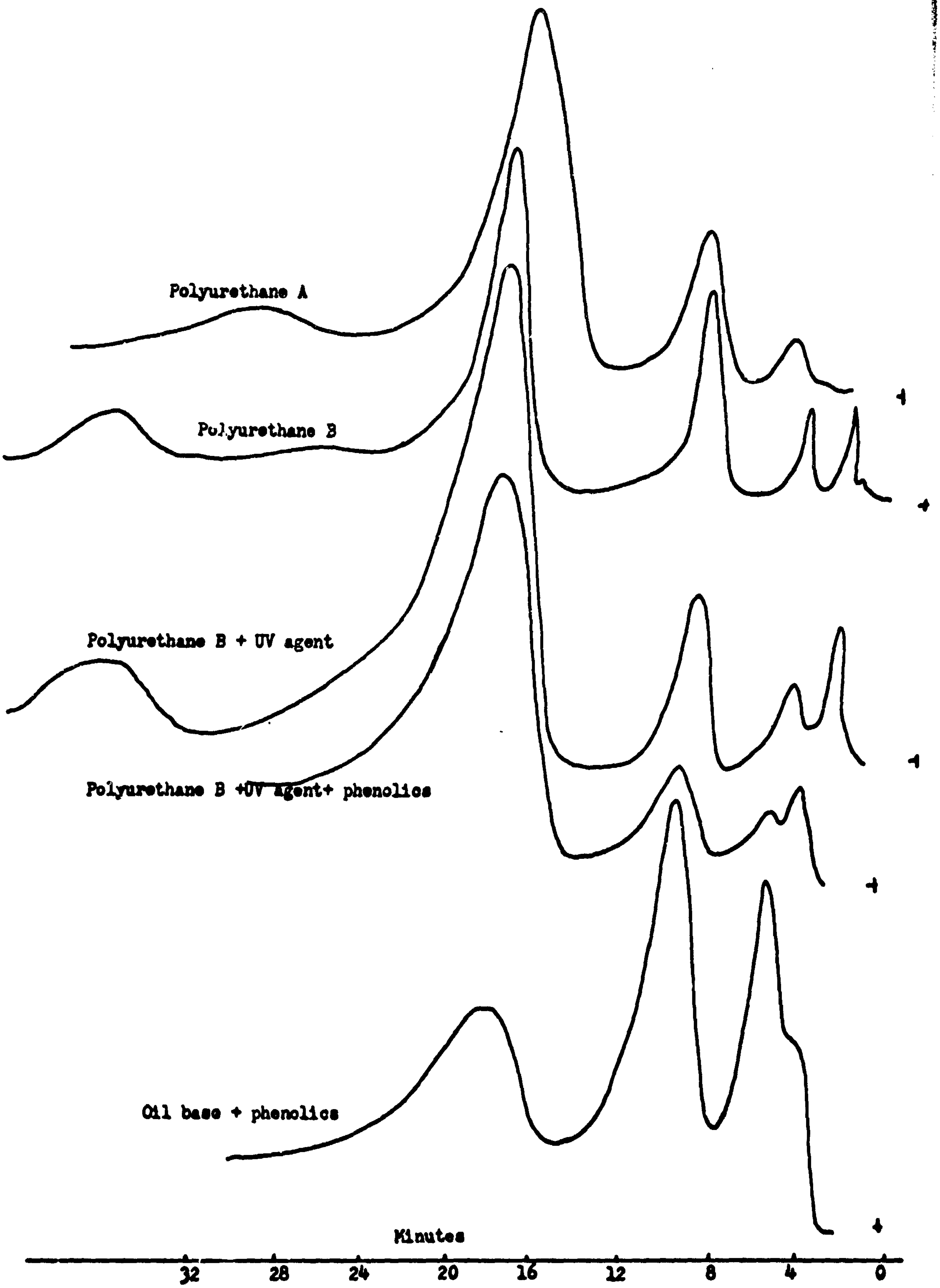


Figure 6. Oxidative products from precolumn reactor using ozone-enriched oxygen

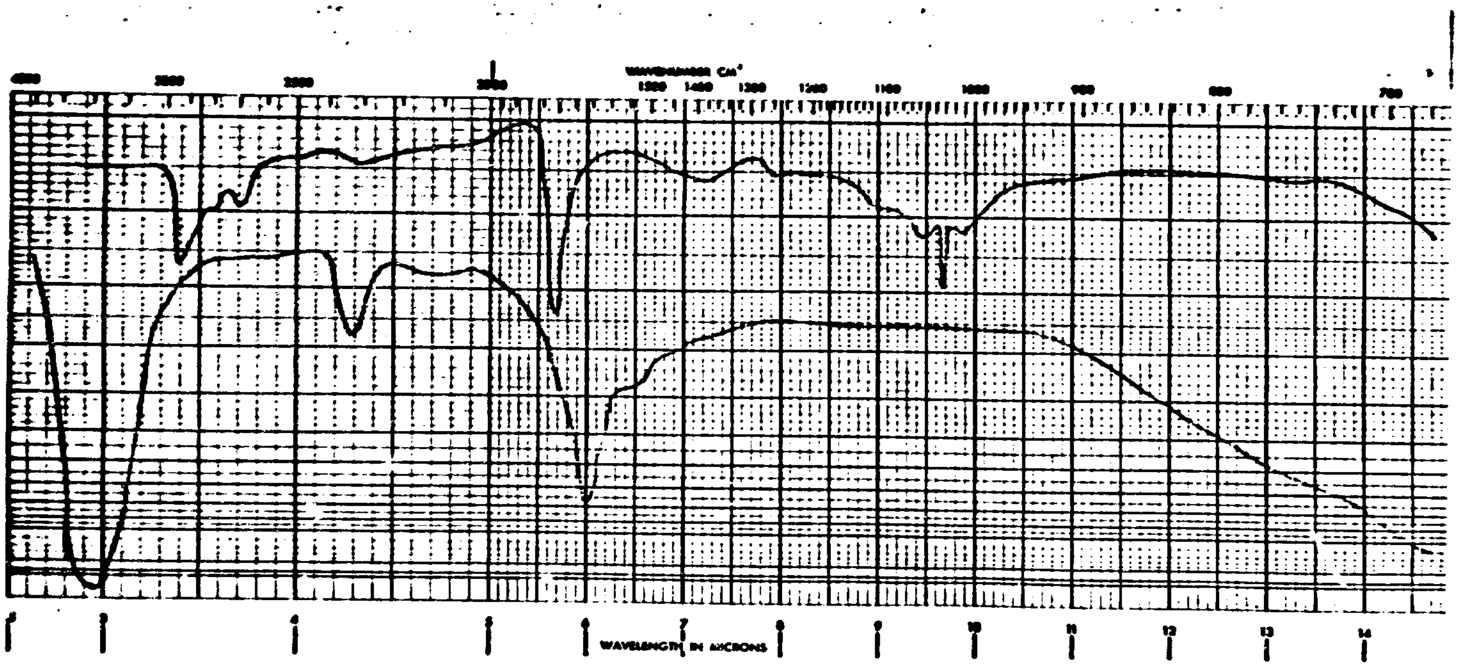


Figure 7. Infrared spectra of volatile products from precolumn oxidation reactor

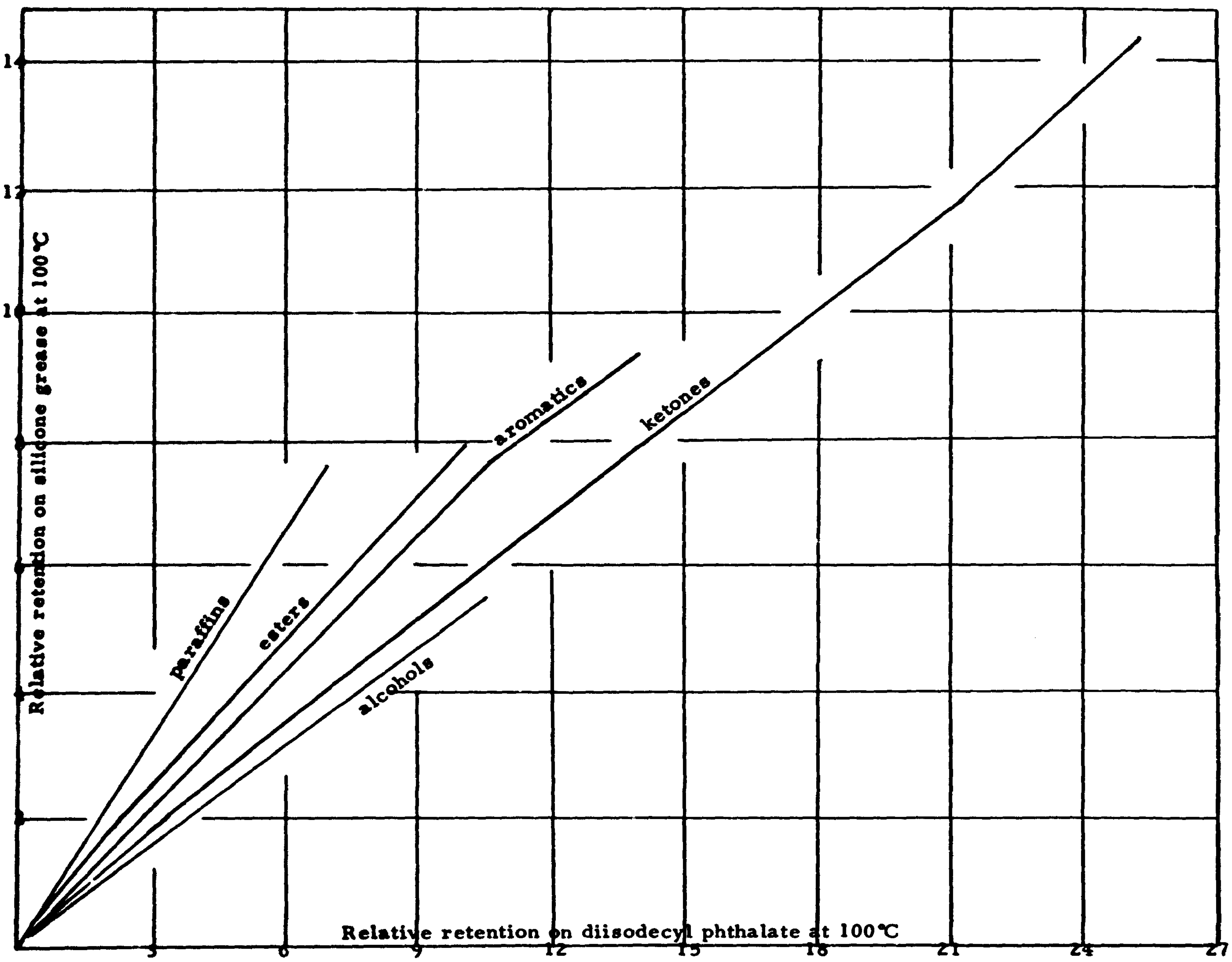


Figure 8. Relative retention chart for identification of gas chromatograph peaks

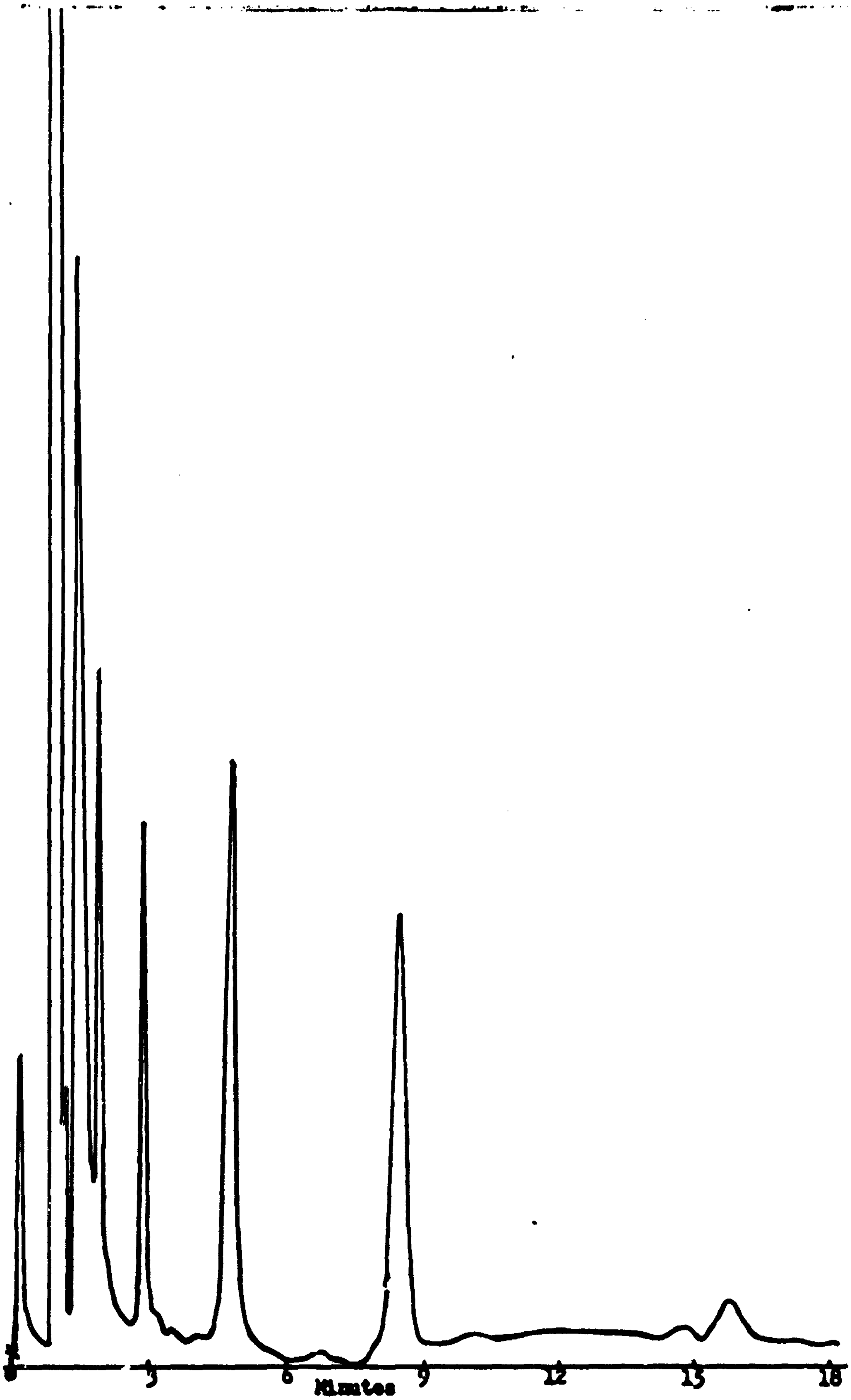


Figure 9. Oxidation products gas chromatographed over silicone grease

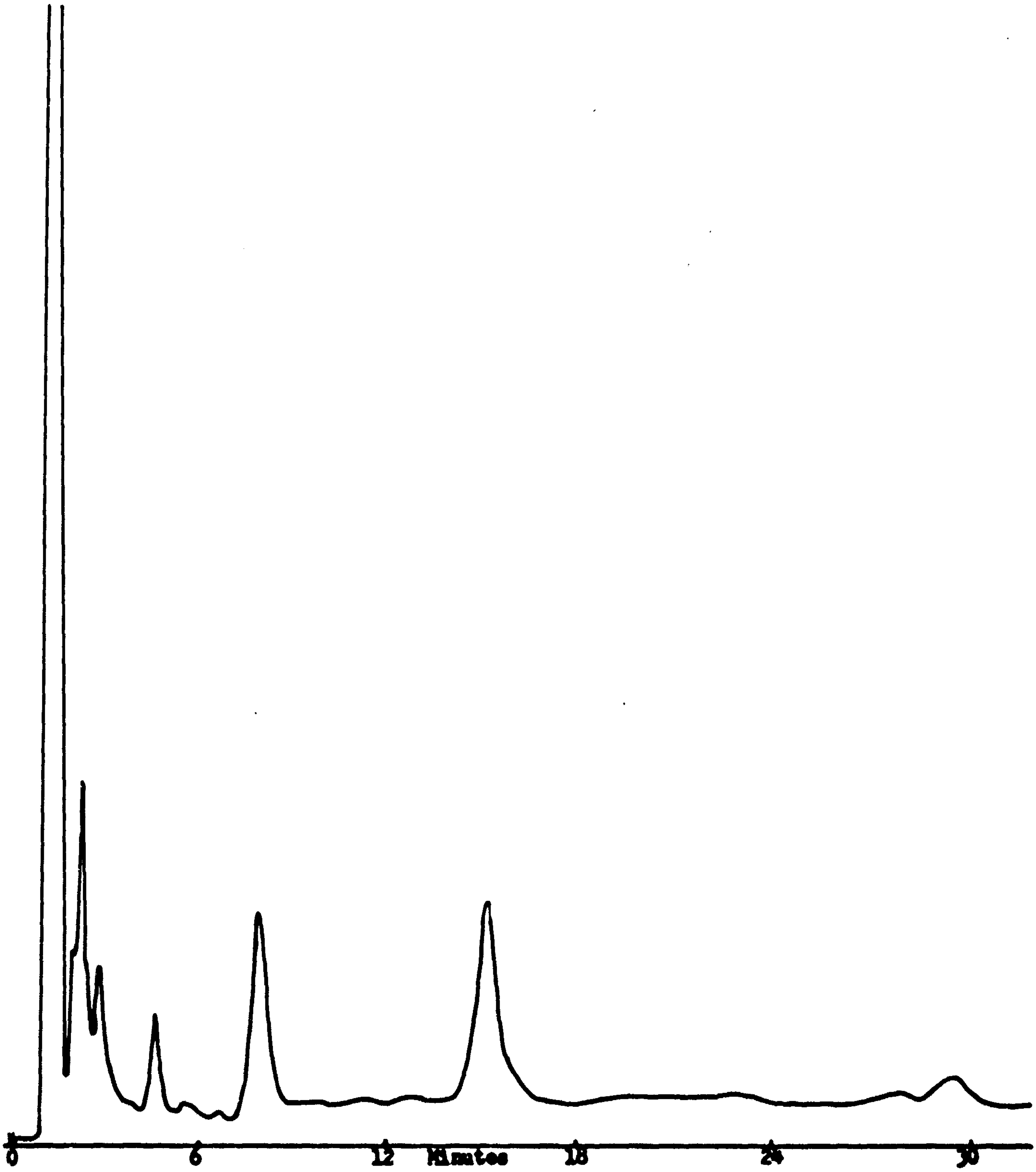


Figure 10. Oxidation products gas chromatographed over diisodecyl phthalate

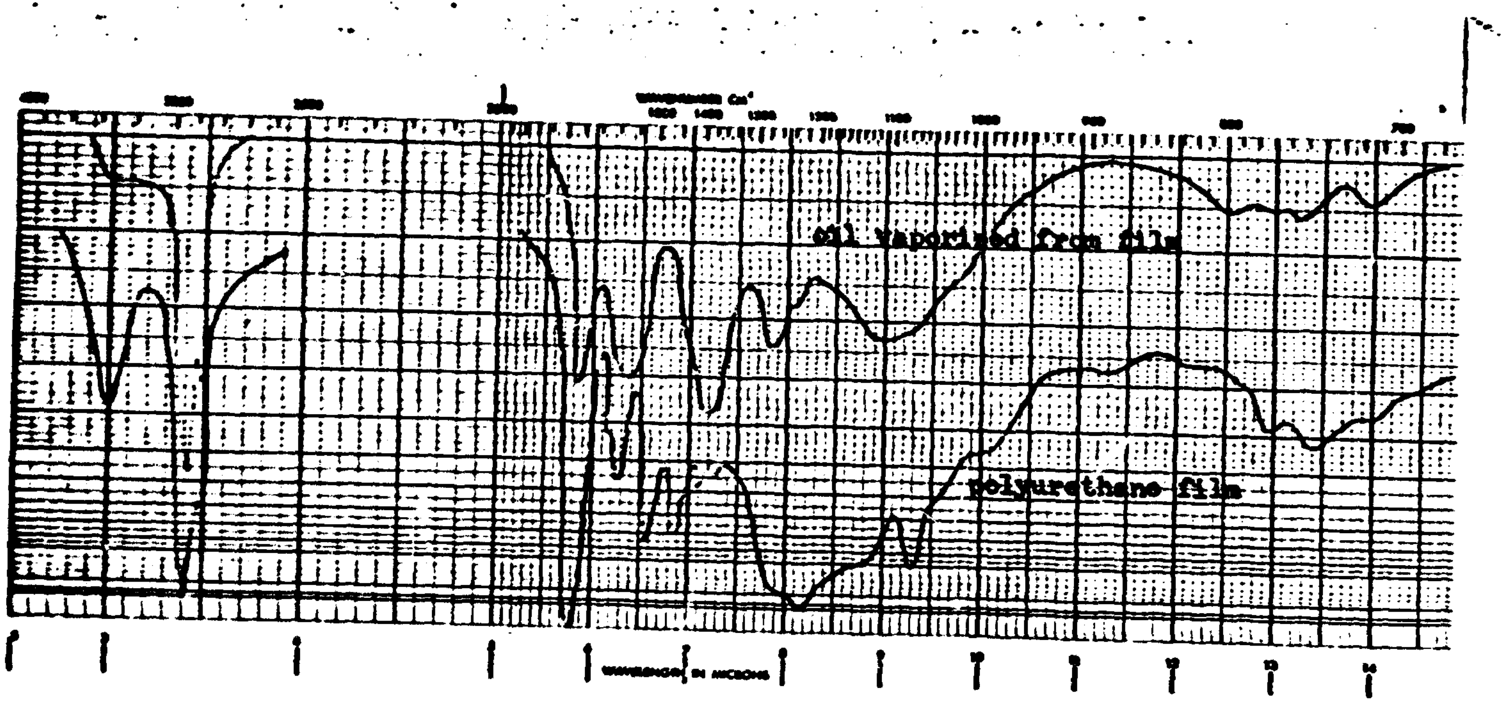
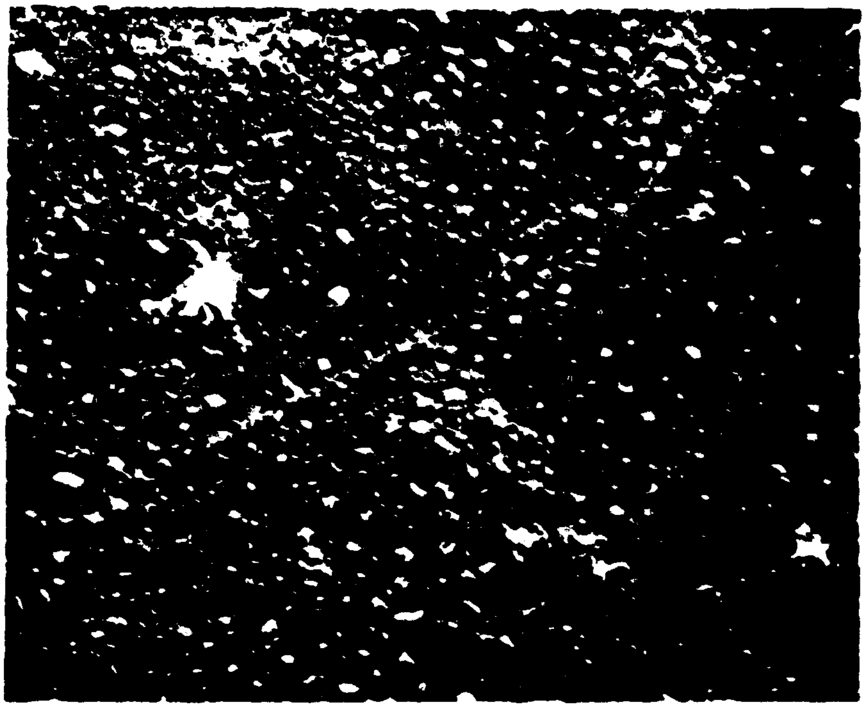
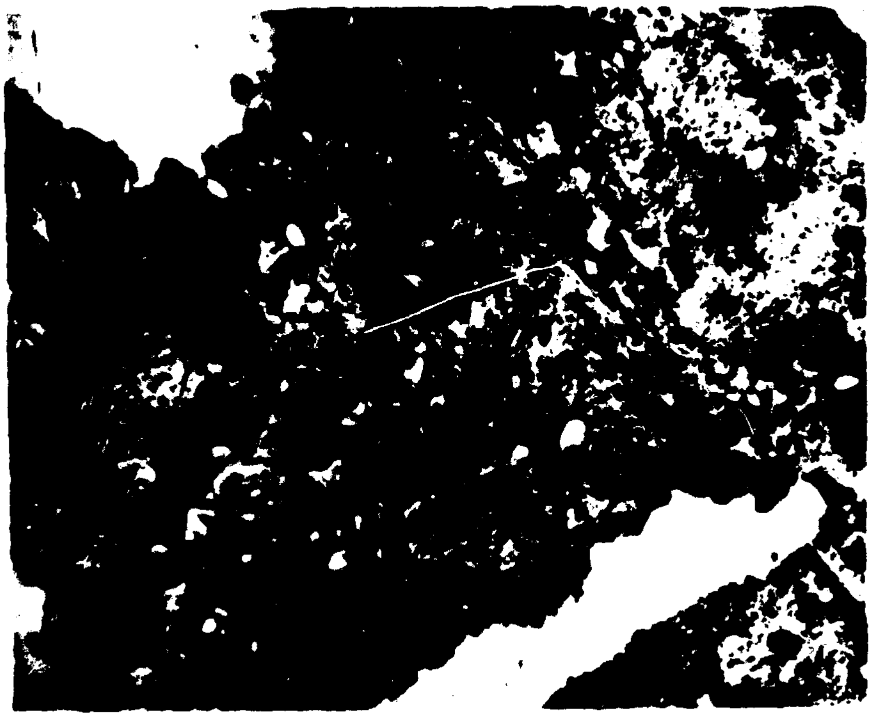


Figure 11. Infrared spectra of oil and polyurethane films



Electron micrograph of AML-P-43 panel, after eight months of Florida exposure.

Electron micrograph of AML-P-95K panel, after eight months of Florida exposure.



Electron micrograph of MIL-L-19537 panel, after eight months of Florida exposure.

Electron micrograph of MIL-C-22750 panel, after eight months of Florida exposure.

WALTER C. McCRONE ASSOCIATES

Ozone generator

So that ozone would be readily available, an ozone generator, similar in design to the one described by P. L. Hanst and others (Anal. Chem. 33, 1113 (1961)) was built. The all-glass generator has an outer tube, approximately eight inches long, closed off with inlet and outlet stopcocks. A center tube, extending the length of the outer tube, serves as an electrode well. The entire apparatus is immersed in a water jacket which also serves as the outer electrode. The power source for the generator is a transformer producing up to 12,000 v at 30 ma.

The generator is operated by flushing the tube with oxygen and then applying approximately 10,000 v.

Ozone concentration is determined by passing the gas through a known volume of potassium iodide solution and determining the amount of liberated iodine with a standardized sodium thiosulfate solution. Several tests were made under different flow conditions. The maximum ozone concentration produced under static conditions was three mole percent.

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13. ABSTRACT

A laboratory investigation was conducted to develop methods for rapidly determining paint coating resistance to oxidation and photochemical degradation. The ultimate objective is development of a rapid test for expected life of paint coatings. Accelerated aging of paint films has been achieved using ultraviolet radiation and oxidizing atmospheres. The resulting gases given off by the films have been analysed by gas phase chromatography. The chemical nature of the paint film can be identified by this means, and the actual compounds evolved by a series of films have thus been identified. Interestingly enough, most belong to a homologous series of methyl ketones (acetone, methyl ethyl, methyl propyl, methyl butyl and methyl pentyl), although methanol, water, carbon monoxide and carbon dioxide were also found. A study of polyurethane films of known history showed a good relationship between service life for each film and the chemical composition of the gases evolved. Further work is in progress to confirm these promising results and to extend the study to other paint compositions.

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