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# Effect of Deoxygenation and Prestressing on Hydrocarbon Fuel Thermal Stability

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# EFFECT OF DEOXYGENATION AND PRESTRESSING

## ON HYDROCARBON FUEL THERMAL STABILITY

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## INTRODUCTION

This report describes part of a continuing study of hydrocarbon fuel thermal stability. This stability is measured by a fuel's tendency to form harmful surface deposits and sediment when it is subjected to thermal stress. It is well known (refs. 1 to 4) that the relative amount of different types of hydrocarbon and organic heteroatom species in a practical fuel is one of the important factors which determines its stability. However, the detailed chemistry of the degradation process is difficult to understand from experiments with complex fuel mixtures. Therefore, a program was started to study thermal stability effects in several pure hydrocarbon types over a wide range of temperature.

In the first part of this work (ref. 5) four hydrocarbon types were studied. The types selected were a normal alkane, an alkene, a naphthene and an aromatic. Although alkenes are not a major component of practical fuels, they are produced when an alkane fuel is pyrolyzed at temperatures above 350° C. Their presence, even in small quantities, may have an effect on formation of solid materials during thermal stressing because of their very reactive characteristics. In the first work deposit formation was measured when the four fuels were thermally stressed, after being fully saturated with air, at temperatures ranging from 150° to 450° C. This temperature range covers two regimes in the oxidative decomposition of n-alkane hydrocarbons (ref. 6). In the low temperature regime (below 300° C) decomposition is by autoxidation, the direct reaction of the fuel with dissolved oxygen. In the intermediate temperature regime (300° to 450° C) decomposition is mainly by further reactions of the first autoxidation products and also by direct pyrolysis of the fuel molecule. Each of the four pure components was found to exhibit its own unique deposit and sediment formation characteristics.

In the present work the same four hydrocarbons were stressed after being thoroughly purged of dissolved oxygen. Previous work on practical hydrocarbon fuels showed (refs. 7 to 9) that removal of oxygen could improve thermal stability in several cases. However, the degree of improvement depended quite strongly on the composition of the fuel and also on trace impurity concentrations. Moreover, in some situations, deoxygenation actually caused a fuel to form more, rather than less, deposit. This occurred in presence of trace amounts of certain organic sulfides and disulfides (refs. 7 and 8). Experiments with deoxygenated pure hydrocarbon blends (ref. 10) showed that deposit formation was very dependent on the presence of small concentrations of olefinic and naphthenic hydrocarbons. Therefore, it was felt that information on deoxygenation effects for pure hydrocarbons would be useful in understanding these previous results. In a second series of experiments, the effect of

deoxygenated prestressing on deposit formation was studied for all the hydrocarbon fuels. This was done by reaerating the deoxygenated stressed fuel and stressing it a second time. F. R. Mayo has suggested (ref. 11) that deposit formation on metal tubes may depend strongly on the previous heating history of the fuel.

In recent work (ref. 12) S. R. Daniel has studied the storage and low-temperature thermal stability of a mixture of ten percent tetralin in n-dodecane. This binary mixture was chosen as a simpler system which could model the characteristics of a practical Jet A fuel. We have also used this "model fuel" in the present higher temperature study and compared its deposit formation with that of Jet A fuel. The same three experimental conditions were used, namely (1) fully aerated, (2) deoxygenated and (3) reaerated after deoxygenated stressing. The temperature range of the present work was from 250° to 400° C and all experiments were performed at a pressure of 500 psig. All fuels were thermally stressed using a commercially available jet fuel thermal oxidation tester (JFTOT) and a modification of the standard ASTM JFTOT procedure (ref. 13).

## EXPERIMENTAL DETAILS

### Fuels

The following pure hydrocarbons were used without further purification:

(1) n-Decane - pure grade (99 mole percent, minimum), from Phillips Petroleum Co.

(2) Cyclohexane - Fisher certified ACS (99 mole percent, minimum) from Fisher Scientific Co.

(3) 1-Hexene - pure grade (99 mole percent minimum) from Phillips Petroleum Co.

(4) Benzene, Fisher certified ACS (99 mole percent minimum) from Fisher Scientific Co.

(5) Tetralin (Tetrahydro naphthalene) - purified grade from Fisher Scientific Co.

(6) n-Dodecane - pure grade (99 mole percent minimum) from Phillips Petroleum Co.

Commercial Jet A was obtained from the NASA Lewis Research Center fuel supply and was filtered through Attapulgus clay to remove polar compounds.

### Apparatus

A commercially available JFTOT from Alcor, Inc. was used to thermally stress each fuel. A schematic diagram of the apparatus is shown in figure 1. Either standard length or long Alcor 316-stainless steel heater tubes were used. The fuel flows over the outer surface of the electrically heated tube at the right in the figure. Any sediment formed in the fuel is trapped by the

test filter indicated, causing a pressure drop which is measured by the U-tube manometer. Deposit formation on the cylindrical tube surface is rated by use of an Alcor Mark 9 tube deposit rater (TDR). With this instrument measurements of light reflectance from the rotating tube are converted to arbitrary readings which give an approximate measure of the deposit formation at any position along the tube.

## Procedure

The present work employed the same modification of the ASTM fuel testing procedure described in reference 5. Each fuel was stressed for 40 minutes at a pressure of 3447 kPa (500 psig) and a flow rate of 3 $cm^3$/min. The temperature for each run was indicated by a thermocouple inside the cylindrical tube, placed to measure the maximum wall temperature along the test section. A typical test section temperature profile is shown in reference 5. Temperatures of 250°, 300°, 350° and 400° C were used in this work.

A measure of the deposit amount formed on each tube was obtained by plotting TDR reading vs. tube position. The assumption was made that any reading is proportional to the amount of deposit per unit length on the tube at the given position. Then, the total area under this graph should be an approximate relative measure of the amount of deposit on the heater tube. Our previous work (ref. 5) showed that this assumption should be valid for TDR readings of about 40 or less, but tends to break down above this value.

The only indication of sediment formation is an increase in the pressure drop Δp across the test filter from its initial value of zero. The variation of Δp with time has been found to be only a qualitative indication of sediment formation. The pressure changes are too small and too irreproducible to be used even as an approximate measure of the amount of sediment formed. Therefore, in the following section we can only discuss sediment formation qualitatively.

## RESULTS AND DISCUSSION

### Relative Importance of Deposit and Sediment Formation

Formation of surface deposit was much more important than that of sediment in the present study. Only two fuels, benzene and the tetralin-n-dodecane mixture, showed any measureable sediment formation. In all cases, the measured Δp values were small (less than 10 mm Hg) and, for benzene, the results were quite irreproducible. For example, three consecutive experiments at 300° C on the same day with deoxygenated benzene from a single loading of the JFTOT reservoir gave widely different results. Final Δp values were 6, 0, and 4 mm Hg for the three experiments. Two different experiments at 300° C using different batches of prestressed, reaerated benzene gave final Δp values of 0 and 9.5 mm Hg. For tetralin and n-dodecane the largest Δp observed was 4.5 mm Hg for the aerated fuel at 250° C, the lowest temperature used. For both fuels, the highest Δp values were obtained at the lowest temperature used. This result agrees with results reported in our earlier work (ref. 5) for a no. 2 heating oil, an unstable practical fuel. We concluded from our pressure measurements that sediment formation is much less

important than deposit formation. Also we do not have a really reliable meas-
ure of the amount of sediment formation. Qualitatively, there is an indica-
tion that, for some situations, sediment formation may be more important at
lower temperatures (250° - 300° C) than at our highest temperature used
(400° C), but this trend will have to be checked by additional work. In the
following sections only deposit formation will be used to quantitatively com-
pare the thermal stability of the fuel studied.

Effect Of Deoxygenaticn And Deoxygenated

Prestressing For Hydrocarbons

Standard length heater tubes were used for all experiments with the pure
hydrocarbons. These tubes have a test-section length of 60 mm, whereas the
long tubes have a 120 mm test-section length. This is the only difference
between the two types of tube. Typical examples of heater tube deposit forma-
tion for pure hydrocarbon types are shown in figure 2. This black and white
photograph does not give a completely accurate depiction of the actual
deposit. It does show some effects of deoxygenation and of deoxygenated pre-
stressing of a fuel. For example, deoxygenated cyclohexane gives much less
discoloration (the middle tube) than either the untreated or the prestressed
aerated fuels. For benzene, however, the deoxygenated fuel gives more discol-
oration on the tube than the untreated aerated fuel. As pointed out in refer-
ence 5, the photograph also shows the differences in the deposit pattern among
the different fuel types under the same experimental conditions.

For a better comparison of deposit formation, figure 3 shows plots of TDR
reading vs. tube position for the four pure hydrocarbons studied at three dif-
ferent nominal temperatures. Results for n-decane are not given at 300° C
(the lowest temperature) because no measurable deposit was formed. The nomi-
nal temperature for each experiment is the maximum value measured along the
test section temperature profile, as described in the "Procedure" section.
For each fuel and temperature three TDR reading profiles are shown. These are
for (1) the untreated, fully aerated fuel, (2) the thoroughly deoxygenated
fuel, and (3) the stressed, deoxygenated fuel, which has been reaerated. To
determine the effect of deoxygenation on deposit formation, profile 2 is com-
pared to profile 1. To study the effect of deoxygenated prestressing on fuel
stability, profile 3 is compared to profile 1. As stated previously, TDR
readings of 40 or less should be proportional to the deposit formation per
unit length at any tube position. The graphs of figure 3 show that only a few
of the TDR readings are greater than 40, except at the highest temperature of
400° C. Even at 400° C, most of the readings are 40 or below. Therefore we
have made the assumption that for all our TDR reading vs position profiles,
the area under the curve gives a valid relative measure of deposit formation.
Even a qualitative inspection of figure 3, however, shows different effects of
deoxygenation for different fuels. For cyclohexane, profile 2 generally shows
much reduced readings compared to profile 1. This indicates that deoxygena-
tion has reduced deposit formation. The opposite is true for benzene. Com-
parison of the two profiles shows either no reduction or an increase in
deposit formation for the deoxygenated fuel. The qualitative effect of deoxy-
genated prestressing on the stability of aerated n-decane can also be seen
from figure 3. At both 350° and 400° C profile 3 is always below profile 1,
indicating that the prestressing has reduced deposit formation for n-decane.

4

For more quantitative comparisons the area under each profile of figure 3 was measured and the results plotted as bar graphs. In each of figures 4, 5, and 6 relative deposit amounts (areas) are shown for all four hydrocarbons at one constant temperature (300°, 350°, and 400° C, respectively). In each of figures 7, 8, 9, and 10 all deposit data for one pure hydrocarbon are shown as a function of temperature. We can now readily observe the effect of deoxygenation and of nonoxidative prestressing as a function of hydrocarbon type and temperature.

Effect of deoxygenation. - Figures 4 to 6 show different effects of deoxygenation for different hydrocarbon types. Only for the naphthene, cyclohexane, is there a large reduction of deposit formation. Figure 10 shows that this occurs at all temperatures used. This effect would be expected if direct reaction with oxygen is the rate controlling step in the formation of deposit precursors. For 1-hexene and n-decane deoxygenation gives only a small to moderate deposit reduction. This is true over the entire temperature range (figs. 7 and 8). For benzene, however, deoxygenation causes either a small or moderate increase in deposit formation at all temperatures used (fig. 9). This result is interesting in view of one of the main findings of our first work (ref. 5) with aerated fuels. Benzene always formed much less deposit than any of the other pure hydrocarbons, even at nominal temperatures of 400° and 450° C. This was explained by the known resistance of the benzene ring to attack by molecular oxygen. It is possible that the products of any direct benzene-oxygen reaction (e.g., phenol) are not deposit precursors. Therefore, if most of these actual deposit precursors are formed by the direct pyrolysis of benzene, then the removal of the competing oxygen reactions would increase the amount of deposit precursor formation.

Effect of deoxygenated prestressing. - Comparison of bars (1) and (3) for each fuel in figures 4 to 6 and each temperature in figures 7 to 10 gives the effect of nonoxidative prestressing on deposit formation for an aerated hydrocarbon. For n-decane at 350° and 400° C, the pretreatment reduces deposit formation the same amount that direct deoxygenation does. For benzene, the pretreatment increases deposit formation about as much as deoxygenation does at all temperatures. For cyclohexane prestressing had no effect except at 300° C, where deposit formation increases moderately. Figure 8 shows that prestressing 1-hexene increased its deposit formation slightly at 300° C, but this trend steadily reversed as the temperature was raised to 400° C, where a moderate decrease was observed. So deoxygenated prestressing can be either beneficial or detrimental to fuel stability depending on both the type of hydrocarbon and on temperature. Information must be obtained on the chemical mechanisms involved in order to understand these effects. Since pyrolysis becomes more important compared to other reactions as the temperature increases, one observation can be made. The pyrolysis products of benzene seem to form more deposit precursors than the pyrolysis products of the other hydrocarbons studied. The latter pyrolysis products apparently inhibit deposit formation.


Effect Of Deoxygenation and Deoxygenated

Prestressing For Two Fuel Mixtures

Figure 11(a) shows experimental results for the thermal stressing of the model fuel, 10 percent tetralin in n-dodecane. Results for commercial Jet A

5

fuel, which the binary mixture models quite well in low-temperature work (ref. 13) are shown in figure 11(b). Standard length tubes were used for the Jet A experiments and long tubes for the model-fuel runs. Since we are only comparing qualitative trends between the two fuels, the difference in test-section length should have no effect on the conclusions. One difference in behavior was noted between these two fuels in the temperature range 250° - 350° C. The model fuel formed significant deposit at 250° C, whereas Jet A had to be heated to 300° C to obtain about the same amount of deposit formation. This can be seen qualitatively in the plots of TDR reading vs. tube position of figure 11. Relative deposit formation for these two fuels using our three different experimental conditions is shown in the bar graphs of figures 12 and 13.

Effect of deoxygenation. - Comparison of bars (1) and (2) for each temperature shows that deoxygenation significantly reduces deposit formation for both fuels at 300° C and below. At 350° C, however, deoxygenation has essentially no effect in reducing deposit for either fuel. Thus, deoxygenation effects are quite similar for the model fuel and for Jet A. These results for Jet A can be compared with those of Taylor using a similar JP-5 type fuel. The work is reported in a survey report by Szetela (ref. 14). Taylor's work (in a thermal reactor quite different from the JFTOT) showed a greater reduction in deposit formation at 300° C than we observed, due to deoxygenation. He also found a significant stabilizing effect of deoxygenation at 350° C, which we did not. However, the deposit rates obtained by Taylor for aerated and deoxygenated fuel do approach each other and become the same at a temperature of about 500° C. Therefore, our work agrees qualitatively but not quantitatively with that of Taylor. This is not unexpected in view of the differences between our reactors and also, undoubtedly, differences in the composition of the two fuels.

Effect of deoxygenated prestressing. - Comparing bars (1) and (3) in figures 12 and 13 shows that nonoxidative pretreatment consistently has a stabilizing effect on the aerated model fuel at all temperatures from 250° to 350° C. Deposit formation is reduced by about 50 percent. For Jet A, this pretreatment causes a small increase in deposit formation at 300° and 350° C. Thus we observed different behavior for the model and the practical fuel when they were nonoxidatively prestressed. Taylor, in work described in an internal government report, performed some deoxygenated prestressing experiments with JP-5 fuel and found lower deposit formation when the fuel was stressed a second time. However, this second stressing was done deoxygenated rather than aerated, as in our experiments. Therefore, these two sets of results are not directly comparable.

The reduction in deposit formation caused by prestressing the tetralin-n-dodecane mixture can be explained by the known reactions of tetralin with tetralin hydroperoxide (Daniel, ref. 12). The deoxygenated mixture will still contain a small amount of tetralin hydroperoxide which reacts with tetralin to form tetralol as a major product, especially at elevated temperatures. Daniel has found that tetralol inhibits deposit formation in his experiments at 130° C.

In summary, our deposit formation studies have shown both similarities and differences in behavior between Jet A fuel and the binary tetralin-n-dodecane mixture that is often used to model it.

## CONCLUDING REMARKS

This study has shown the variety of effects that deoxygenation has on the stability of pure hydrocarbons. Only cyclohexane showed significantly improved thermal stability in the absence of dissolved oxygen over a wide temperature range (300° - 400° C). Benzene showed a decrease in stability under the same conditions for the same temperature range. It should be pointed out that benzene's behavior may be different from that of straight-chain substituted benzenes and condensed aromatics. These latter types of compounds probably constitute most of the aromatic fraction of a practical fuel such as Jet A. Deoxygenated prestressing does not in general have a large effect on the stability of the reaerated pure hydrocarbons. Only benzene showed a consistent trend toward lower thermal stability under this treatment. Again, its behavior may be different from that of other substituted or condensed aromatics. The single unsubstituted ring, although resistant to oxidation, may undergo pyrolysis forming species which can then be oxidized to form deposit precursors during the reaerated thermal stressing.

The binary model of Jet A, 10 percent tetralin in n-dodecane, appears to be less stable than Jet A to thermal stressing. Although this result should be verified using a highly purified binary mixture, these first results are interesting. They emphasize the need for further kinetic studies to determine the mechanism of deposit formation in this mixture. This knowledge may help determine what species and reactions in Jet A inhibit these deposit forming reactions. Although the effect of deoxygenation on the model and practical fuels is the same, we found differences in the effect of nonoxidative prestressing. This treatment significantly stabilizes the reaerated tetralin-n-dodecane mixture, but has little effect on Jet A. Further kinetic studies on the oxidative and nonoxidative thermal decomposition of the model fuel are needed to help explain this difference in behavior.

## SUMMARY OF RESULTS

The effect of deoxygenation and of deoxygenated prestressing on the thermal stability of four pure hydrocarbons and two fuel mixtures was studied by stressing the fuels in a Jet Fuel Thermal Oxidation Tester (JFTOT). Deposit and sediment formation was evaluated over the temperature range 250° to 400° C using 316 stainless steel tubes. A series of three conditions was employed for each fuel. The fuel was first stressed fully air saturated. Second, another sample was stressed after being deoxygenated by nitrogen purging. Finally the deoxygenated and stressed fuel was reaerated by air saturation and stressed a second time. The pure fuels used were n-decane (a normal alkane), 1-hexene (an alkene), cyclohexane (a naphthene), and benzene (an aromatic). The fuel mixtures used were commercial Jet A and also a mixture of 10 percent tetralin in n-dodecane, which has been used as a simpler model of Jet A by other investigators. The important results are as follows:

(1) Deoxygenation of pure hydrocarbon fuels does not always reduce deposit formation. For cyclohexane, a significant reduction is observed, but for benzene an increase is obtained. The other two fuels show moderate decreases in deposit amount.

(2) Deoxygenated prestressing may have either a stabilizing or destabilizing effect on the reaerated hydrocarbon. The result depends on both the

7

type of hydrocarbon and the temperature. For cyclohexane, a small increase in deposit formation is observed at 300° C, but no change is observed at the higher temperatures. For benzene the prestressing increases deposit formation at all temperatures. For hexene at 300° C a slight increase in deposit formation is found. This effect reverses, however, as the temperature is increased to 400° C, where the prestressing causes less deposit formation. For n-decane prestressing causes less deposit at both 350° and 400° C, compared to the aerated straight hydrocarbon.

(3) Deoxygenation of the tetralin-dodecane mixture greatly reduces deposit formation at 250° C. The extent of the reduction decreases as temperature is increased, however. At 400° C, the reduction in deposit formation is negligible. Deoxygenated prestressing also stabilizes this binary mixture significantly over the 250° - 350° C temperature range, compared to the aerated, untreated mixture.

(4) Deoxygenation of Jet A at 300° C causes a significant reduction in deposit formation. At 350° C, however, there is no significant effect of deoxygenation on deposit formation. This trend is similar to that for the model fuel. However, the effect of deoxygenated prestressing on Jet A is different from that on the model fuel. Deposit formation for the reaerated Jet A is very slightly higher than for aerated untreated Jet A.

(5) The model binary fuel mixture shows significant deposit formation at a lower temperature than Jet A does. Only further kinetic experiments on this binary mixture will help elucidate the mechanism of deposit formation and explain the differences in behavior between the model and real fuels.

(6) No significant sediment formation was observed for any of the pure hydrocarbons or fuel mixtures used in this study.

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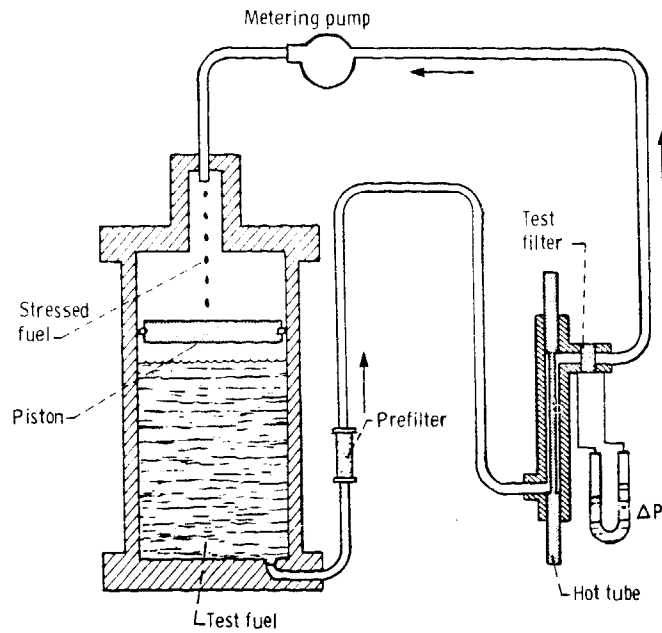
Figure 1. - Schematic of jet fuel thermal oxidation tester (JFTOT).



n-DECANE          1-HEXENE          BENZENE          CYCLOHEXANE

CONDITIONS FOR EACH FUEL (a) AERATED, (b) DEOXYGENATED, (c) STRESSED DEOXYGENATED
AND REAERATED.
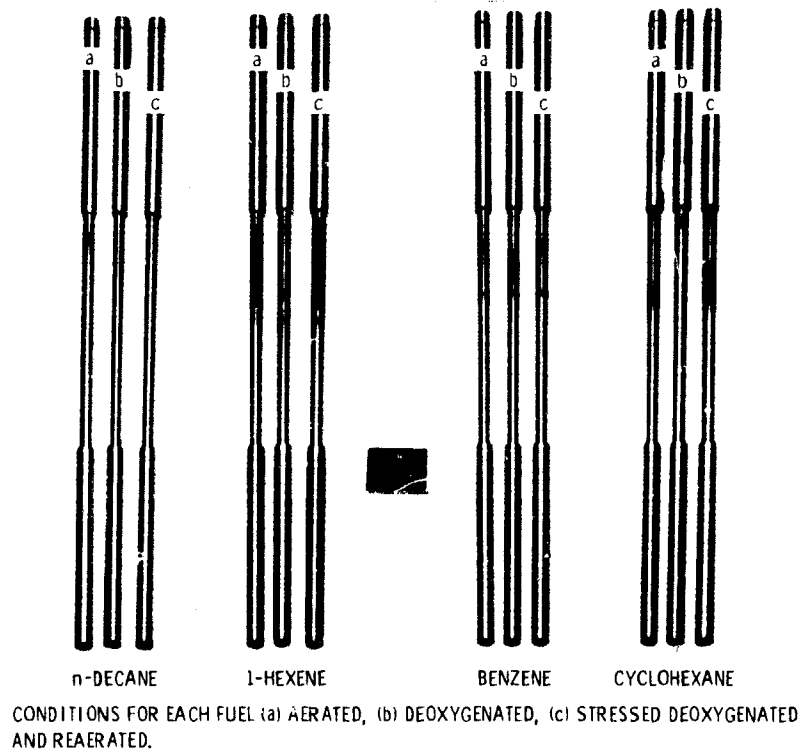
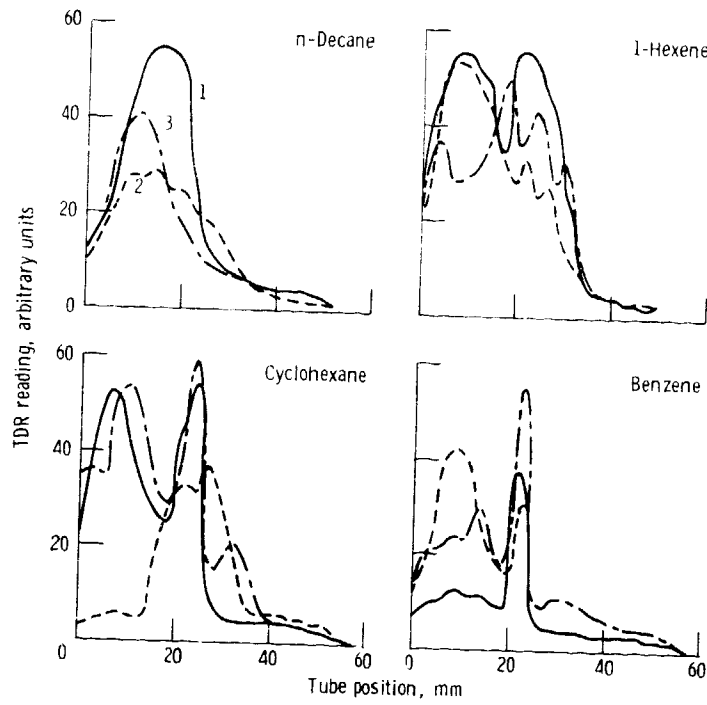Figure 2. - Deposit tubes for four pure hydrocarbons at $350^\circ$ C.     C-83-1394
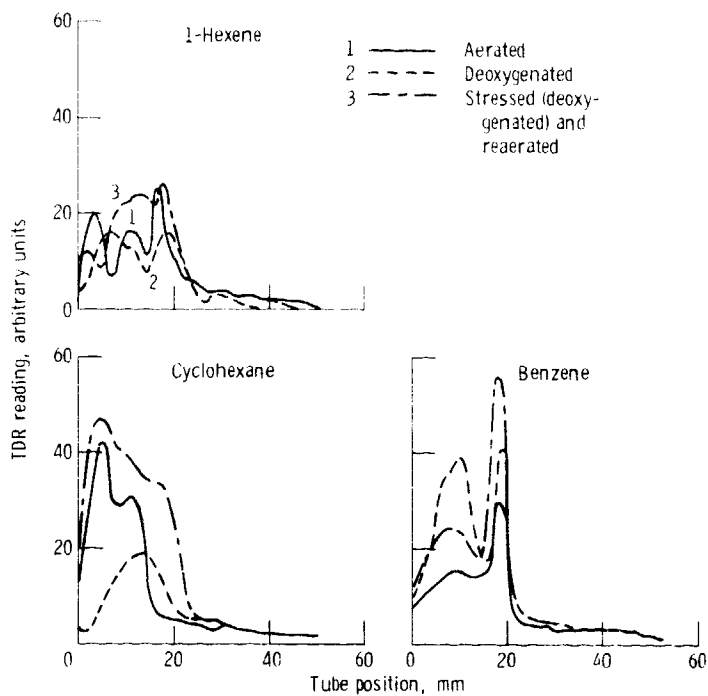
(a) Temperature 300° C.

Figure 3. - Deposit formation for pure hydrocarbons.
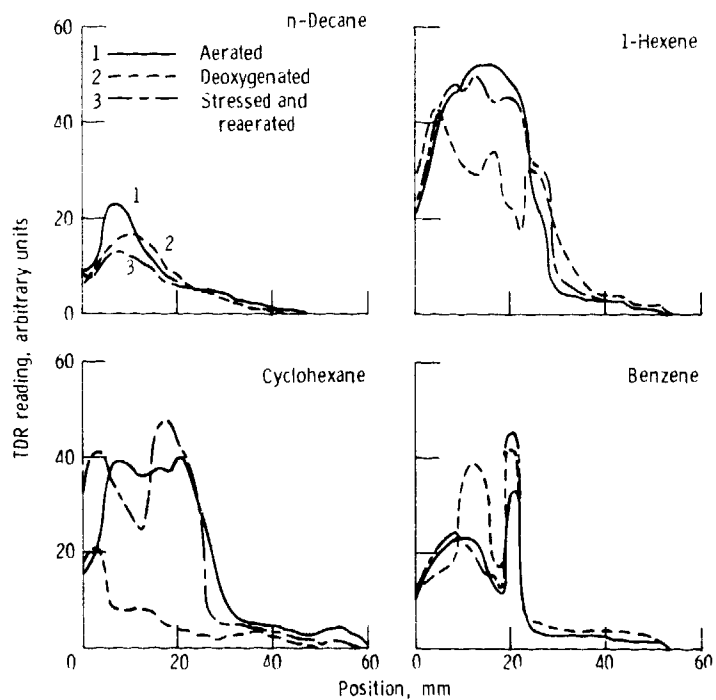


(b) Temperature 350° C.

Figure 3. - Continued.

(c) Temperature 400° C.

Figure 3. - Concluded.
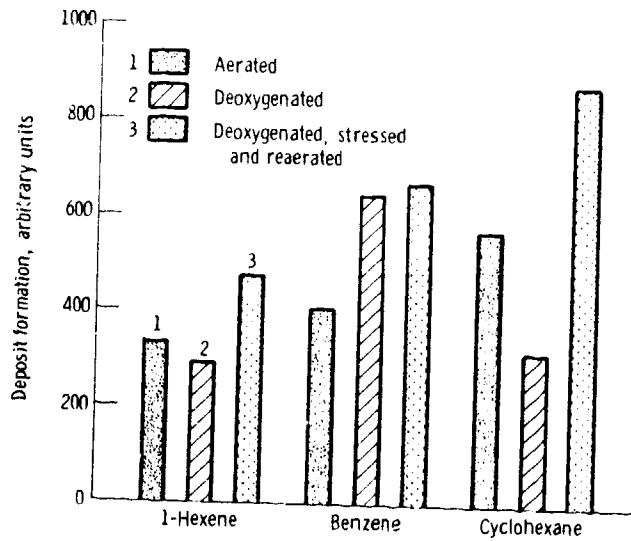


Figure 4. - Deposit formation for three pure hydrocarbons at
T = 300° C - effect of oxygen content and prestressing.
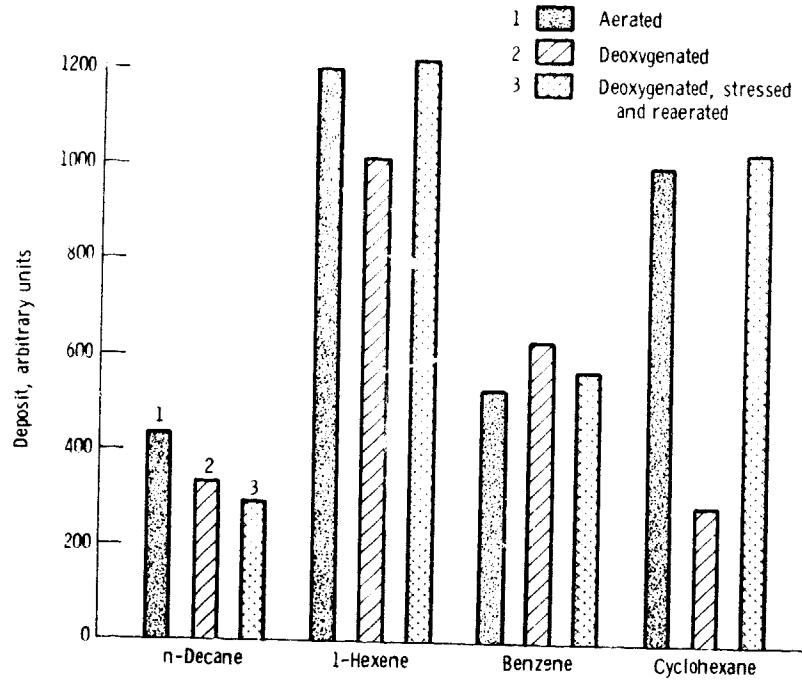
Figure 5. – Deposit formation for four pure hydrocarbons at T = 350° C – effect of deoxygenation and prestressing.
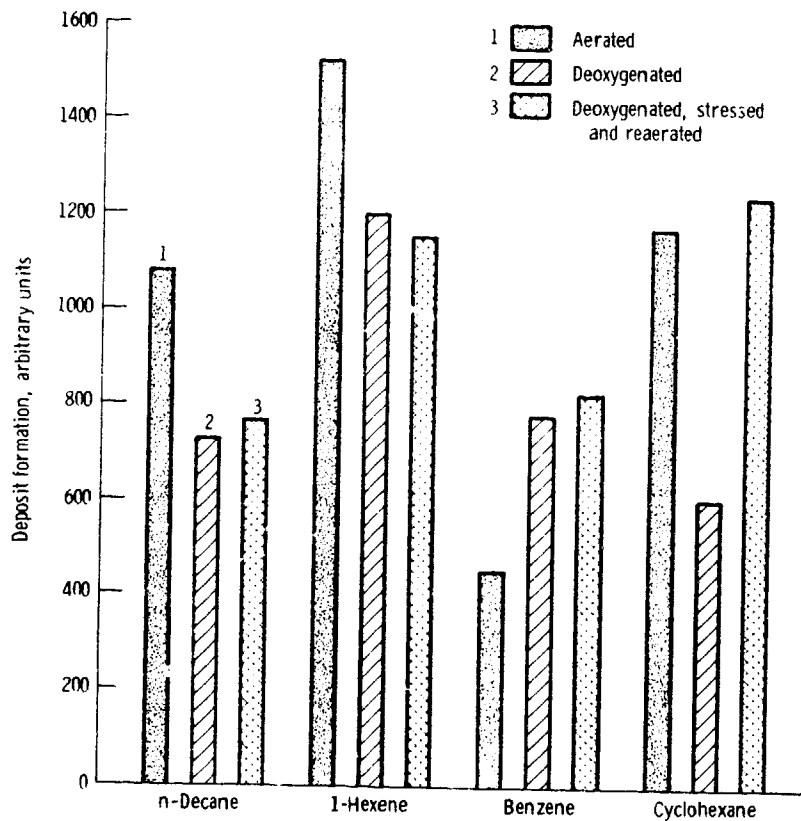


Figure 6. – Deposit formation for four pure hydrocarbons at T = 400° C – effect of oxygen concentration and prestressing.
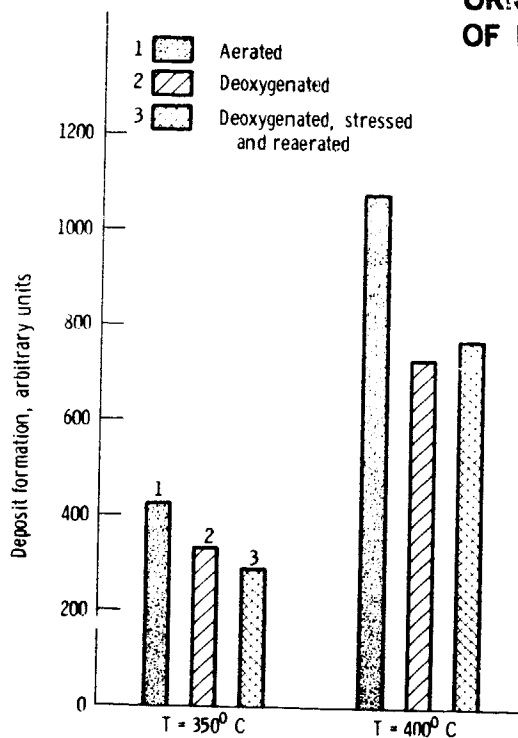
Figure 7. – Effect of oxygen content and prestressing on deposit formation for n-decane at several temperatures.
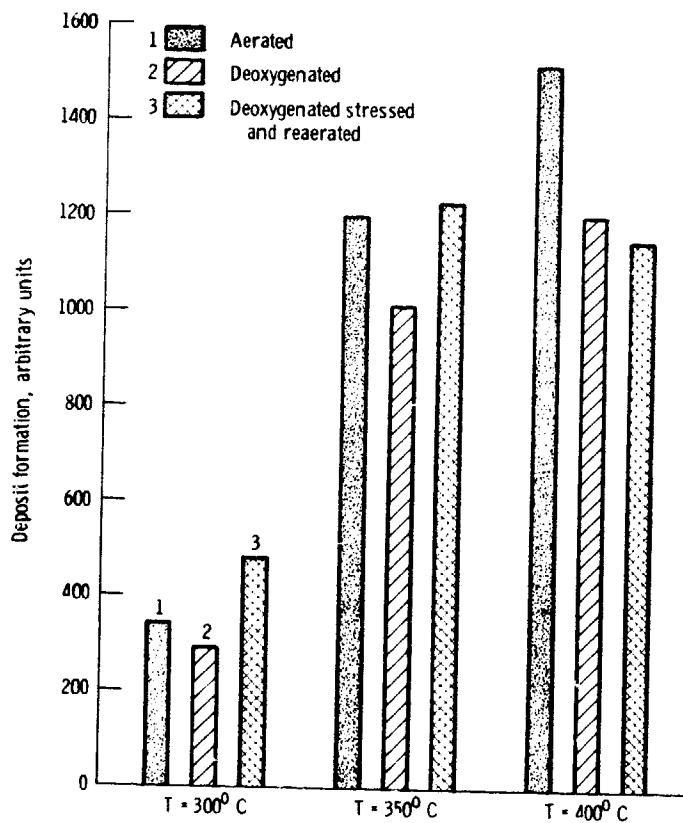


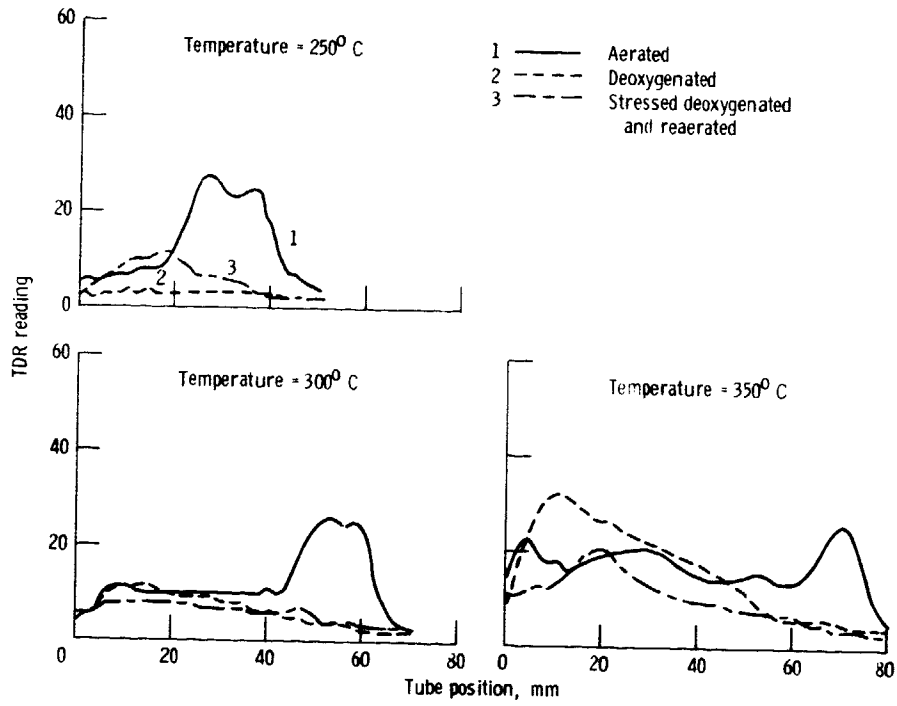Figure 8. – Effect of oxygen content and prestressing on deposit formation for 1-hexene at several temperatures.
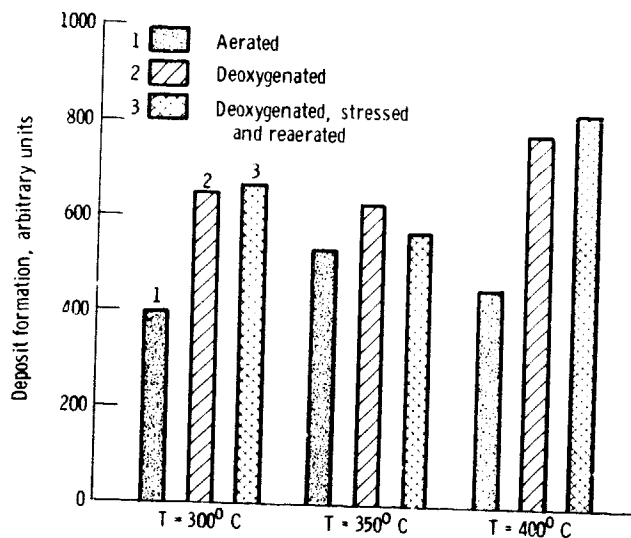
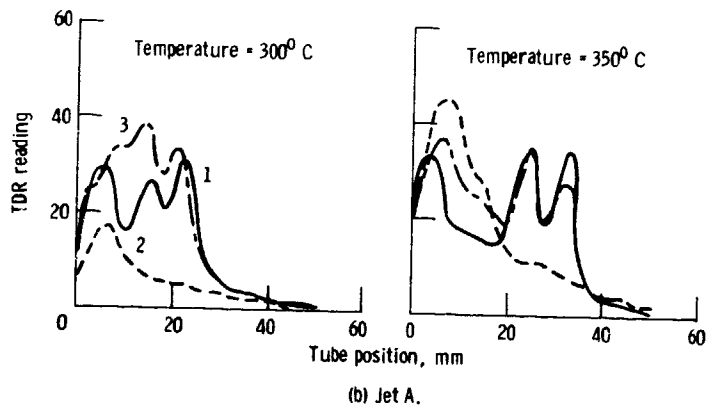Figure 9. - Effect of oxygen content and prestressing on deposit
formation for benzene at several temperatures.
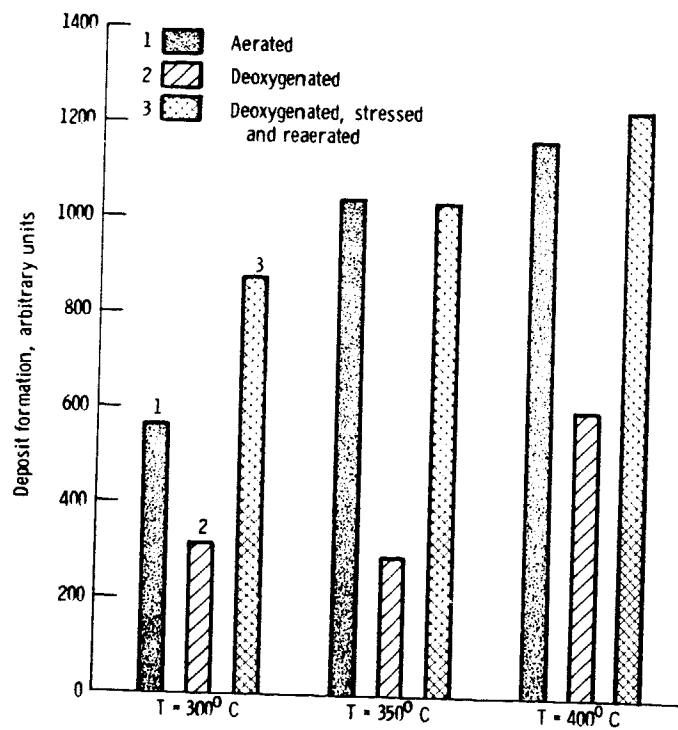
Figure 10. - Effect of oxygen content and prestressing on deposit
formation for cyclohexane at several temperatures.

(a) Ten percent tetralin in n-dodecane.

Figure 11. - Deposit formation for fuel mixtures.
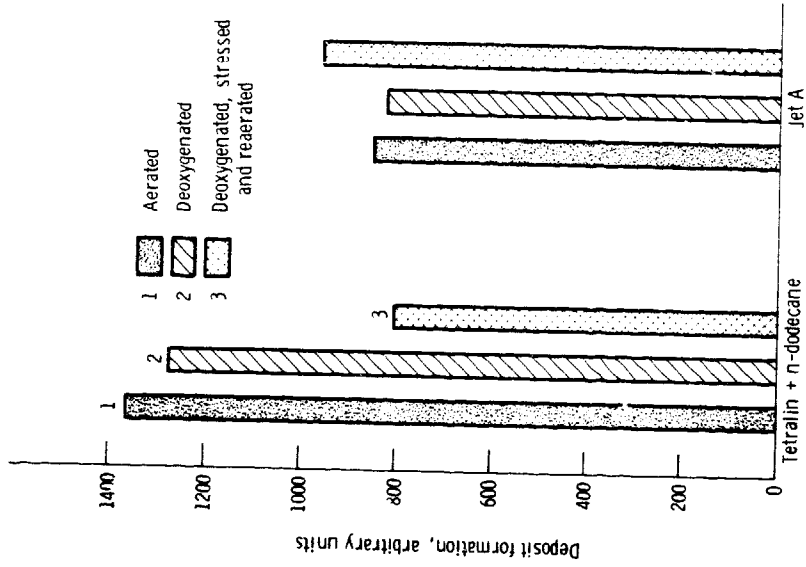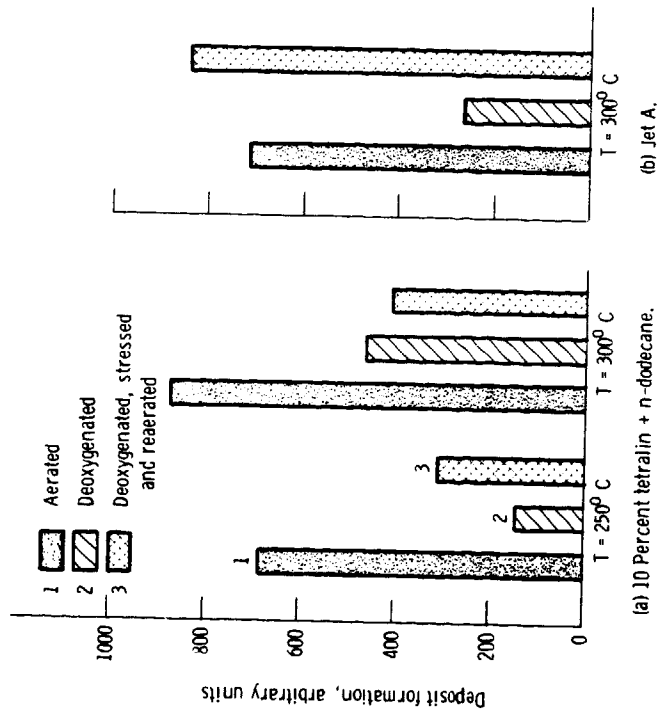


(b) Jet A.

Figure 11. - Concluded.

Figure 13. - Effect of oxygen content and prestressing on deposit formation for 10 percent tetralin in n-dodecane and for jet A at T ≈ 250° C.



(a) 10 Percent tetralin + n-dodecane.

(b) Jet A.

Figure 12. - Effect of oxygen content and prestressing on deposit formation for 10 percent tetralin in n-dodecane and for jet A at T = 250° and 300° C.

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