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# THERMAL STABILITY OF SOME AIRCRAFT TURBINE FUELS DERIVED FROM OIL SHALE AND COAL

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# THERMAL STABILITY OF SOME AIRCRAFT TURBINE FUELS DERIVED FROM OIL SHALE AND COAL

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## SUMMARY

Thermal stability breakpoint temperatures are shown for 32 jet fuels prepared from oil-shale and coal-syncrudes by various degrees of hydrogenation. Low severity hydrotreated shale oils, with nitrogen contents of 0.1 to 0.24 weight percent, had breakpoint temperatures in the 477 to 505 K ($400^{\circ}$ to $450^{\circ}$ F) range. Higher severity treatment, lowering nitrogen levels to 0.008 to 0.017 weight percent, resulted in breakpoint temperatures in the 505 to 533 K ($450^{\circ}$ to $500^{\circ}$ F) range.

Coal-derived fuels showed generally increasing breakpoint temperatures with increasing weight percent hydrogen, fuels below 13 weight percent hydrogen having breakpoints below 533 K ($500^{\circ}$ F).

Comparisons are shown with similar literature data.

## INTRODUCTION

This report presents thermal stability breakpoint temperatures data on a series of aircraft turbine type fuels prepared from oil shale and coal syncrudes.

Little information exists in the literature on the general properties and characteristics of fuels derived from synthetic crude oils. The Lewis Research Center of NASA is engaged in a program to study the possible impacts of obtaining and using aircraft turbine type fuels derived from oil shale and coal syncrudes. As part of this program a series of such fuels was prepared from TOSCO, H-Coal and COED syncrudes by the Atlantic-Richfield Company (ARCO) under a contract with NASA (ref. 1). The purpose of this ARCO preparation contract was to determine the processing steps and conditions necessary to meet certain yield and specification requirements for the final product fuels. The ARCO contract was to determine the processing conditions and the product quality at two yields (about 20 and 40 percent) and at two levels of hydrogenation severity for each yield, of TOSCO shale oil and for two levels of hydrogenation severity for the H-Coal and COED coal syncrudes. The yields from the TOSCO syncrude were varied by using hydrocracking to attain the higher yield. The two levels of hydrogenation severity for all three syncrudes were obtained by varying the pressures ($10.3\times10^6$ to $17.2\times10^6$ $N/m^2$, or 1500 to 2500 psi), temperatures (607 to 675 K or $634^\circ$ to $755^\circ$ F), and weight hourly space velocities (0.36 to 1.5). Each process stream was further split by distillation to give four distillation ranges. The product specifications that were required to be met were the levels of hydrogen, nitrogen, and sulfur content. All the physical and chemical tests required for aircraft turbine fuels were reported by ARCO for the 32 fuels.

Thermal stability data for such fuels are especially scarce. The evaluation of the thermal stability of a fuel should reveal any tendency of that fuel toward instabilities which could affect its performance in an aircraft fuel system. For example, there could be the tendency toward gum or deposit formation on heated surfaces or the tendency to form particulates which might plug small passageways in the fuel system.

The purpose of the work presented herein was to determine the thermal stability breakpoint temperatures on the fuels prepared by ARCO and to see if any correlations of breakpoint temperatures with fuel properties or processing would be evident. The breakpoint temperature data determined at NASA are compared with the single temperature ($260^\circ$ C) determinations made by ARCO on these same fuels.

The data cover 32 fuels. Sixteen of these fuels were from a TOSCO shale oil syncrude, 8 from an H-Coal syncrude, and 8 from a COED (coal-derived) syncrude. The breakpoint temperature range investigated was 477 to 589 K ($400^\circ$ to $600^\circ$ F).

## EXPERIMENTAL PROCEDURE

### Apparatus

The thermal stability data were obtained using the Alcor jet fuel thermal oxidation tester (JFTOT) apparatus and procedure which are described in detail in ASTM D 3241 (ref. 2). A cross-sectional sketch of the test section is shown in figure 1.

Filtered, aerated fuel flows upward through an annulus formed between an outer housing and an inner heated tube and then out through a test filter. The aluminum heater tube is heated electrically. Figure 2 shows a typical longitudinal temperature profile which was obtained by a traversing thermocouple located inside the heater tube. The maximum temperature, at a position index of 39 (39 mm from the fuel inlet position), is the temperature recorded as the JFTOT temperature. Some measurements and calculated values of flow velocity and residence time relating to the test section are also noted in figure 1. At the design flow rate of 3 cubic centimeters per minute the flow velocity through the annulus is about 0.5 centimeter per second and the residence time in the annulus is approximately 12 seconds.

The fuel is pressurized with nitrogen ($N_2$) to $3.4\times10^6$ newtons per square meter (500 psig) to prevent fuel vaporization at the test temperature (ref. 3). The test lasts for $2\frac{1}{2}$ hours and requires at least 450 cubic centimeters of fuel for a test run.

Two types of fuel instabilities that may affect performance of a jet fuel in an aircraft fuel system are expected to be in evidence in this type of test. First, the tendency to form gum or deposits on heat exchanger tubes or other heated surfaces would show up as deposits on the test heater surface; second, the tendency of the fuel to form particulates which might clog fuel orifices or filters would show up as an increasing pressure drop with time across the test filter.

The test filter pressure drop is recorded during the test procedure. The heater tube deposit is checked at the end of the run. The tube deposit can be rated visually (by comparison with a color standard) and given a numerical rating of 0 (clean tube) to 4 (heavy deposit) or it can be rated with an Alcor Mark 8A tube deposit rater (TDR). All the tube deposits cited in this report have been made with the TDR.

The TDR is a light reflectance measurement device in which the heater tube can be spun on its axis to give an average circumferential reading. While the tube is being spun, it can be scanned axially. The TDR scale is so calibrated that a zero reading indicates a clean tube and a 50 reading (the maximum) indicates a very heavy deposit.

For the results reported herein, it has arbitrarily been assumed that a maximum TDR spun rating of 13 or below is a pass condition for the test. Some rationale for using this value can be noted from figure 3 (ref. 4), which indicates that a TDR spun rating of 13 would have received a visual pass rating of 2 or less on all the tests used for this particular comparison. Since the value of 13 is also in agreement with the pass value used by Exxon Research and Engineering (ref. 5), the results of both sets of experiments are more readily comparable.

The standard procedure in ASTM D 3241 calls for a test at 533 K (500° F). If the fuel does not pass the stability criteria at this temperature, a second test at 519 K (475° F) is made, and the results at both temperatures are reported. In the tests herein it was attempted to select test temperatures that would bracket the spun TDR value of 13 and to label the temperature at which a maximum value of 13 was indicated as the "break point temperature". Where the break point temperature was indicated to be above the highest temperature used, it was simply labelled "above T". No runs were made above 589 K (600° F).

## Fuels

The fuels used in this study were prepared under a contract study by Atlantic Richfield Company (ARCO). The details of preparation and properties of the finished samples are reported in reference 1. The preparations are now described briefly.

The flow system schematics of figures 4(a) to (c) show the principal details of the processing that was carried out on the three syncrudes. It can be noted in figure 4(a) that the low yield TOSCO samples were obtained by hydrotreating only the 361 to 616 K ($190^o$ to $650^o$ F) cut from the crude. The high yield samples, however, also contain material from the 616 to 783 K ($650^o$ to $950^o$ F) cut of the crude which has been hydrocracked. The H-Coal samples (fig. 4(b)) received only a single stage hydrotreatment but at more severe conditions than the comparable range for TOSCO processing. The COED samples (fig. 4(c)) contain hydrotreated IBP to 561 K (IBP to $550^o$ F) crude material and hydrocracked 561 to 700 K ($550^o$ to $800^o$ F) crude products, similar to the high-yield TOSCO samples.

Each of the streams labelled "Final sample blends" in figure 4 actually consisted of two separate hydrotreatment severity runs. And, each of these separate run streams was fractionated into the group of four different boiling range final products.

The properties of the final sample blends as determined by ARCO (ref. 6) are shown in table I. It should be emphasized that the objective in processing these fuels was not to produce finished fuels that would necessarily meet all aircraft turbine fuel specifications. Rather, the objective was to meet (1) the yield, (2) the processing severity to meet the H, N, and S levels, and (3) the boiling point range conditions. The full range of aircraft turbine fuel specification tests was then carried out on these blends.

A recently completed similar study by Exxon Research and Engineering (ref. 5) produced a series of aircraft turbine fuels of the JP-4 and Jet A type from five syncrudes: Paraho, TOSCO, and Garrett shale syncrudes and H-Coal and Synthoil coal syncrudes. In this study, also, the effect of varying the severity of processing on the final product properties was investigated. The flow system schematics of figures 5(a) to (3) show the principal details of the processing that was carried out on these five syncrudes. The thermal stability data (JFTOT) obtained by Exxon on these fuels and included in reference 5 will be used in some of the later comparisons of results.

## RESULTS AND DISCUSSION

The spun TDR values measured on the 32 ARCO samples are shown in figure 6. The ARCO fuel sample designations are used on the figures for identification. Scans were made from tube position index values of 20 through 54 (see fig. 2).

The TOSCO shale sample TDR values show a general symmetry around the maximum axial temperature location (position index, 39) as do the low-severity H-Coal spun TDR values. The high-severity H-Coal TDR values are comparatively more random; however, they are also all fairly low (max. value shown is ≤7.0). Most of the COED sample TDR values show no axial symmetry either. No significance is attached to this observation at the present time, it is simply noted.

The maximum values of spun TDR are plotted against test temperature in figure 7. In most cases no pressure drop buildup across the test filter was observed during the $2\frac{1}{2}$-hour runs. In those few cases where filter $\Delta P$ buildup did occur, the data are shown in figure 8. In only one case, with fuel number 33430, did the fuel fail to pass the $\Delta P$ test while still not showing much tube deposit.

The breakpoint temperatures, defined as the temperatures at which a maximum spun TDR value of 13 is expected, were determined from the plots of figure 7 (where maximum spun TDR was the criterion), or they were estimated from figure 8 where $\Delta P$ was the criterion (i.e., where $\Delta P$ exceeds 25 mm Hg before the end of the test).

These breakpoint temperature data are summarized in table II along with some of the fuel properties for which comparisons are subsequently made. Also shown in this table are the visual tube ratings taken by ARCO of JFTOT tests on these same materials at 533 K ($500^{\circ}$ F). A similar table made from the Exxon data (ref. 5) is presented herein as table III for comparison purposes.

A comparison of the breakpoint temperature data taken at Lewis with the visual ratings obtained on the same sample materials at ARCO is shown in figure 9. In the visual rating method, a value of less than 3 at a test temperature of 533 K ($500^{\circ}$ F) is required for a pass condition. It can be seen that for all but four fuels the pass or fail criterion was in agreement by either rating procedure. Three of the four not in agreement were very close, probably within the range of repeatability of the tests. Only one fuel sample seemed to be in marked disagreement, sample 33318. This is a high nitrogen content fuel, and the visual rating reported seems to be out of line with the other samples in the TOSCO low yield - low severity treatment group.

Figure 10 shows the breakpoint temperatures for the shale fuels plotted against weight percent nitrogen. The low-severity treated shale fuels, with nitrogen levels of 0.1 to 0.24 weight percent, had thermal breakpoint temperatures in the 477 to 505 K ($400^{\circ}$ to $450^{\circ}$ F) range. The higher severity treated fuels, with nitrogen levels of 0.008 to 0.17 weight percent, had breakpoint temperatures in the 505 to 533 K ($450^{\circ}$ to $500^{\circ}$ F) range. The fuels with nitrogen levels below 0.008 weight percent generally had breakpoint temperatures in excess of 533 K ($500^{\circ}$ F). There was little variation in the weight percent hydrogen in the shale fuels, and the sulfur levels were all below 0.0044 weight percent.

The coal-derived fuel samples all have very low nitrogen levels. The ARCO samples were less than or equal to 6 ppm, the Exxon samples were less than or equal to 67 ppm. Figure 11 shows the variation of thermal breakpoint temperature with hydrogen content for the low-nitrogen content fuels. In figure 11(a), which shows the coal fuels only, the samples show an increasing level for the breakpoint temperature with increasing weight percent hydrogen. Except for the synthoil, fuels with the hydrogen content below 13.0 weight percent had breakpoint temperatures below 533 K ($500^\circ$ F); only two of the coal-derived fuels with $H \geq 13.5$ weight percent had breakpoint temperatures below 533 K ($500^\circ$ F). One of these two was the sample that had the breakpoint temperature determined by the $\Delta P$ across the test filter rather than by tube deposit rating. The synthoil-derived fuels have a significantly higher level of breakpoint temperature for the same hydrogen content. Synthoil fuel samples with hydrogen levels of 12 to 12.3 weight percent had breakpoint temperatures equal to or greater than 533 K ($500^\circ$ F).

Figure 11(b) shows the breakpoint temperature data for the few shale-derived fuels which had nitrogen contents less than or equal to 67 ppm superimposed on the coal fuels plot. Of the five shale fuels that met this low nitrogen criterion, only one (a low-severity treated fuel) had a breakpoint temperature significantly below the general level of the coal fuel data.

## CONCLUDING REMARKS

This report has presented thermal stability breakpoint temperature data, obtained on the ALCOR JFTOT apparatus, for 32 aircraft turbine type fuels prepared from shale and coal syncrudes. These fuels were the result of specifying the yield and severity of hydroprocessing. The final fuel samples represented four different distillation ranges for each processing sequence, nominally 366 to 561 K ($200^\circ$ to $550^\circ$ F), 366 to 616 K ($200^\circ$ to $650^\circ$ F), 422 to 561 K ($300^\circ$ to $550^\circ$ F), and 422 to 616 K ($300^\circ$ to $650^\circ$ F).

The shale-derived fuels showed a variation in breakpoint temperature with nitrogen content. The higher nitrogen level fuels, 0.1 to 0.24 weight percent nitrogen, had breakpoint temperatures in the 477 to 505 K ($400^\circ$ to $450^\circ$ F) range. The lower nitrogen level fuels, 0.008 to 0.017 weight percent, had breakpoint temperatures in the 505 to 533 K ($450^\circ$ to $500^\circ$ F) range. With the shale-derived fuels of nitrogen content less than about 0.008 weight percent nitrogen, there appeared to be no general trend of breakpoint temperature with nitrogen content.

The improved thermal stability with reduced nitrogen content does not prove that nitrogen containing compounds are the sole, or even major, contributors to thermal instability. The increased hydrogenation severity that is required to reduce the nitrogen content should also reduce the concentrations of other unstable species such as oxygen containing organics or olefinic hydrocarbons.

The nitrogen levels of the coal-derived fuels were all fairly low, less than 70 ppm. The breakpoint temperatures of the coal-derived fuels showed generally increasing breakpoint temperature with increasing weight percent hydrogen, although the correlation is not a very strong one. None of the ARCO fuels below 13.0 weight percent hydrogen had breakpoints equal to or greater than 533 K ($500^\circ$ F), and only two of the coal fuels with hydrogen content greater than or equal to 13.5 weight percent hydrogen had breakpoint temperatures below 533 K ($500^\circ$ F). There appears to be a significantly higher level of breakpoint temperature for the Exxon Synthoil derived fuels than for the other coal-derived fuels for the same hydrogen content. The Synthoil fuels with hydrogen levels of 12 to 12.3 weight percent had breakpoint temperatures equal to or greater than 533 K ($500^\circ$ F).

Again, the improved thermal stability with increased hydrogen content is not necessarily the result of hydrogen concentration alone, but more probably it results from the saturation or removal of trace amounts of unstable species by more drastic hydrogenation.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 17, 1977,
505-04.

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TABLE I. - FINAL PRODUCT INSPECTIONS

Data from ref. 1.

(a) Low yield shale products

| Property | Low severity | | | | High severity | | | |
|---|---|---|---|---|---|---|---|---|
| | Boiling range, K ($^{\circ}$F) | | | | | | | |
| | IBP-616 (IBP-560) | 394 to 616 (250 to 650) | 394 to 561 (250 to 550) | IBP-561 (IBP-550) | IBP-616 (IBP-650) | 394 to 615 (250 to 650) | 394 to 561 (250 to 550) | IBP-561 (IBP-550) |
| | Fuel number | | | | | | | |
| | 33315 | 33316 | 33317 | 33318 | 33340 | 33341 | 33342 | 33343 |
| Specific gravity | 0.8040 | 0.8170 | 0.8068 | 0.7945 | 0.7977 | 0.8081 | 0.8022 | 0.7914 |
| Reid vapor pressure, kN/m$^2$ (psi) | 1.1 (0.15) | ---------- | ---------- | 0.34 (0.05) | 2.8 (0.40) | ---------- | ---------- | 1.1 (0.15) |
| Flash point, K ($^{\circ}$F) | --------- | 315 (108) | 311 (100) | ---------- | --------- | 312 (102) | 312 (102) | --------- |
| Freezing point, K ($^{\circ}$F) | 255 (0) | 258 (5) | 229 (-47) | 225 (-54) | 255 (1) | 258 (5) | 231 (-44) | 229 (-47) |
| Viscosity at 239 K (-30$^{\circ}$ F), m$^2$/sec | (a) | (a) | $6.781\times10^{-6}$ | $4.736\times10^{-6}$ | (a) | (a) | $6.990\times10^{-6}$ | $5.059\times10^{-6}$ |
| Net heat of combustion, J/g | 43 836 | 43 652 | 44 120 | 43 631 | 44 120 | 43 857 | 43 190 | 43 928 |
| Existing gum, mg | 16.2 | 51.4 | 40.2 | 32.2 | 0.8 | 19.2 | 8.6 | 9.0 |
| Smoke point | 21 | 20 | 21 | 22 | 26 | 26 | 24 | 26 |
| Aromatics, percent | 21.9 | 25.9 | 22.2 | 19.0 | 13.7 | 17.4 | 17.1 | 13.5 |
| Olefins, percent | 1.1 | 0.8 | 1.1 | 1.0 | 0.8 | 1.0 | 1.2 | 0.9 |
| Naphthalenes, percent | 1.0 | 1.2 | 0.5 | 0.5 | 0.4 | 0.4 | 0.2 | 0.2 |
| *Hydrogen, percent* | *13.64* | *13.66* | *13.68* | 13.73 | 13.82 | 13.86 | 13.95 | 13.76 |
| Nitrogen, percent | 0.1954 | 0.2233 | 0.2011 | 0.1750 | 0.0161 | 0.0168 | 0.0152 | 0.0132 |
| Total sulfur, percent | 0.0010 | 0.0044 | 0.0006 | 0.0006 | 0.0009 | 0.0003 | 0.0001 | 0.0002 |
| Mercaptans, percent | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0003 | 0.0002 | ---------- | --------- |
| Oxygen, percent | 0.03 | 0.05 | 0.06 | 0.04 | 0.03 | 0.05 | 0.09 | 0.09 |

[a] Solid.

TABLE I. - Continued.

(b) High yield shale products

| Property | Low severity | | | | High severity | | | |
|---|---|---|---|---|---|---|---|---|
| | Boiling range, K ($^{o}$F) | | | | | | | |
| | IBP-616 (IBP-650) | 394 to 616 (250 to 650) | 394 to 561 (250 to 550) | IBP-561 (IBP-550) | IBP-616 (IBP-650) | 394 to 616 (250 to 650) | 394 to 561 (250 to 550) | IBP-561 (IBP-550) |
| | Fuel number | | | | | | | |
| | 33365 | 33366 | 33367 | 33368 | 33408 | 33409 | 33410 | 33411 |
| Specific gravity | 0.7972 | 0.8146 | 0.8054 | 0.7874 | 0.7936 | 0.8100 | 0.8035 | 0.7874 |
| Reid vapor pressure, kN/m$^2$ (psi) | 7.6 (1.10) | ---------- | ---------- | 6.9 (1.00) | 8.6 (1.25) | ---------- | ---------- | 7.6 (1.10) |
| Flash point, K ($^{o}$F) | --------- | 314 (106) | 312 (102) | ---------- | --------- | 312 (102) | 309 (96) | --------- |
| Freezing point, K ($^{o}$F) | 251 (-8) | 255 (-1) | 227 (-50) | 222 (-59) | 250 (-9) | 254 (-3) | 226 (-52) | 223 (-58) |
| Viscosity at 239 K (-30$^{o}$ F), m$^2$/sec | (a) | (a) | $6.918\times10^{-6}$ | $4.093\times10^{-6}$ | --------- | ---------- | $7.060\times10^{-6}$ | $4.326\times10^{-6}$ |
| Net heat of combustion, J/g | 44 204 | 44 062 | ---------- | 44 442 | 44 329 | 43 882 | 44 066 | 44 371 |
| Existing gum, mg | 26.8 | 61.8 | 23.4 | 17.0 | 9.2 | 32.8 | 16.0 | 16.6 |
| Smoke point | 23 | 20 | 22 | 25 | 26 | 25 | 25 | 27 |
| Aromatics, percent | 15.7 | 20.3 | 17.9 | 13.7 | 12.1 | 15.4 | 13.2 | 11.4 |
| Olefins, percent | 0.8 | 0.9 | 1.3 | 0.8 | 0.6 | 1.0 | 1.0 | 0.8 |
| Naphthalenes, percent | 0.75 | 0.93 | 0.42 | 0.33 | 0.3 | 0.35 | 0.21 | 0.17 |
| Hydrogen, percent | 13.82 | 13.37 | 13.80 | 13.70 | 13.98 | 13.95 | 13.95 | 13.98 |
| Nitrogen, percent | 0.1305 | 0.1581 | 0.1397 | 0.1138 | 0.0101 | 0.0144 | 0.0076 | 0.0088 |
| Total sulfur, percent | 0.0014 | 0.0012 | 0.0006 | 0.0005 | 0.0011 | 0.0002 | 0.0002 | 0.0002 |
| Mercaptans, percent | 0.0001 | ---------- | ---------- | ---------- | <0.0001 | ---------- | ---------- | --------- |
| Oxygen, percent | 0.08 | 0.14 | 0.14 | 0.13 | 0.11 | 0.10 | 0.07 | 0.10 |

[a] Solid.

TABLE I. - Continued.

(c) Low yield H-Coal products

| Property | Low severity | | | | High severity | | | |
|---|---|---|---|---|---|---|---|---|
| | Boiling range, K ($^oF$) | | | | | | | |
| | IBP-616 (IBP-650) | 394 to 616 (250 to 653) | 394 to 561 (250 to 550) | IBP-561 (IBP-550) | IBP-616 (IBP-650) | 394 to 616 (250 to 650) | 394 to 561 (250 to 550) | IBP-561 (IBP-550) |
| | Fuel number | | | | | | | |
| | 33416 | 33417 | 33418 | 33419 | 33430 | 33431 | 33432 | 33433 |
| Specific gravity | 0.8493 | 0.8654 | 0.8565 | 0.8413 | 0.8338 | 0.8488 | 0.8468 | 0.8314 |
| Reid vapor pressure, kN/$m^2$ (psi) | 1.4 (0.20) | ---------- | ---------- | 1.1 (0.15) | 1.7 (0.25) | ---------- | ---------- | 3.1 (0.45) |
| Flash point, K ($^oF$) | ---------- | 312 (102) | 309 (96) | ---------- | ---------- | 312 (102) | 314 (106) | ---------- |
| Freezing point, K ($^oF$) | 251 (-8) | 237 (-32) | 217 (-68) | 211 (-79) | 255 (0) | 246 (-17) | 225 (-54) | 207 (-86) |
| Viscosity at 239 K (-30$^o$ F), $m^2$/sec | ---------- | $16.99 \times 10^{-6}$ | $6.785 \times 10^{-6}$ | $5.162 \times 10^{-6}$ | $9.757 \times 10^{-6}$ | $15.91 \times 10^{-6}$ | $9.102 \times 10^{-6}$ | $6.264 \times 10^{-6}$ |
| Net heat of combustion, J/g | 43 359 | 42 878 | 43 108 | 43 263 | 43 723 | 43 273 | 43 273 | 43 601 |
| Existing gum, mg | 6.0 | 74.0 | 92.0 | 10.2 | 4.8 | 110.8 | 11.2 | 9.8 |
| Smoke point | 14 | 15 | 15 | 16 | 24 | 21 | 24 | 25 |
| Aromatics, percent | 29.7 | 33.8 | 30.9 | 26.3 | 5.9 | 6.7 | 5.8 | 5.5 |
| Olefins, percent | 1.2 | 1.8 | 1.4 | 1.2 | 1.3 | 1.4 | 1.0 | 0.9 |
| Naphthalenes, percent | 0.54 | 0.66 | 0.31 | 0.27 | 0.064 | 0.077 | 0.065 | 0.055 |
| Hydrogen, percent | 12.73 | 12.47 | 12.64 | 12.79 | 13.56 | 13.26 | 13.31 | 13.73 |
| Nitrogen, percent | 0.0005 | 0.0006 | 0.0006 | 0.0001 | <0.0001 | <0.0001 | <0.0001 | 0.0001 |
| Total sulfur, percent | 0.0004 | 0.0004 | 0.0001 | 0.0001 | 0.0005 | 0.0005 | <0.0001 | 0.0001 |
| Mercaptans, percent | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- | ---------- |
| Oxygen, percent | 0.11 | 0.09 | 0.06 | 0.10 | 0.04 | 0.06 | 0.03 | 0.04 |

TABLE I. - Concluded.

(d) High yield COED products

| Property | High severity | | | | Low severity | | | |
|---|---|---|---|---|---|---|---|---|
| | Boiling range, K (°F) | | | | | | | |
| | IBP-616 (IBP-650) | 394 to 616 (250 to 650) | 394 to 561 (250 to 550) | IBP-561 (IBP-550) | IBP-616 (IBP-650) | 394 to 616 (250 to 650) | 394 to 561 (250 to 550) | IBP-561 (IBP-550) |
| | Fuel numbers | | | | | | | |
| | 33502 | 33503 | 33504 | 33505 | 33516 | 33517 | 33518 | 33519 |
| Specific gravity | 0.8255 | 0.8458 | 0.8368 | 0.8165 | 0.8358 | 0.8586 | 0.8493 | 0.8270 |
| Reid vapor pressure, kN/m$^2$ (psi) | 5.8 (0.85) | ---------- | ---------- | 5.8 (0.85) | 5. (0.85) | ---------- | ---------- | 7.2 (1.05) |
| Flash point, K (°F) | --------- | 313 (104) | 314 (106) | ---------- | --------- | 319 (114) | 313 (104) | --------- |
| Freezing point, K (°F) | 242 (-23) | 239 (-30) | 220 (-64) | 215 (-72) | 256 (-23) | 256 (2) | 221 (-62) | 217 (-70) |
| Viscosity at 239 K (-30° F), m$^2$/sec | (a) | $19.25\times10^{-6}$ | $9.676\times10^{-6}$ | $5.565\times10^{-6}$ | (a) | (a) | $9.851\times10^{-6}$ | $5.586\times10^{-6}$ |
| Net heat of combustion, J/g | 43 865 | 44 074 | 43 865 | 44 129 | 43 212 | 43 627 | 43 518 | 43 873 |
| Existing gum, mg | 0.8 | 23.0 | 6.6 | 7.6 | 1.6 | 21.2 | 2.4 | 3.2 |
| Smoke point | 20 | 20 | 24 | 27 | 16 | 14 | 16 | 19 |
| Aromatics, percent | 9.3 | 11.6 | 7.2 | 5.4 | 22.4 | 28.5 | 25.2 | 20.1 |
| Olefins, percent | 0.7 | 1.1 | 0.9 | 0.5 | 0.6 | 1.2 | 0.8 | 0.5 |
| Naphthalenes, percent | 0.49 | 0.62 | 0.13 | 0.11 | 0.68 | 0.86 | 0.38 | 0.31 |
| Hydrogen, percent | 13.6 | 13.44 | 13.63 | 13.69 | 13.07 | 12.88 | 12.96 | 13.24 |
| Nitrogen, percent | 0.0002 | 0.0002 | 0.0002 | 0.0002 | 0.0003 | 0.0002 | 0.0002 | 0.0002 |
| Total sulfur, percent | 0.0003 | 0.0003 | 0.0001 | 0.0001 | 0.0003 | 0.0001 | 0.0001 | 0.0001 |
| Mercaptans, percent | --------- | ---------- | ---------- | ---------- | --------- | ---------- | ---------- | --------- |
| Oxygen, percent | 0.05 | 0.05 | 0.05 | 0.03 | 0.03 | 0.03 | 0.03 | 0.03 |

[a] Solid.

## TABLE II. - ARCO FUEL SAMPLES

| ARCO number | Crude | Yield level | Severity level | ASTM boiling range | | Weight percent | | | Jet fuel thermal oxidation tester breakpoint temperature | | Jet fuel thermal oxidation tester visual rating at 533 K (500° F) | Process conditions | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | K | °F | H | N | S | K | °F | | Pressure | | Temperature | | Space velocity, WHSV, hr$^{-1}$ |
| | | | | | | | | | | | | N/m$^2$ | psi | K | °F | |
| 33315 | TOSCO | Low | Low | 372 to 594 | 211 to 610 | 13.64 | 0.195 | 0.001 | 491 | 424 | 4 | 10.3×10$^6$ | 1500 | 625 | 665 | 1.5 |
| 33316 | ↓ | ↓ | ↓ | 425 to 596 | 305 to 613 | 13.66 | .2233 | .0044 | 495 | 431 | 4 | | | | | |
| 33317 | | | | 425 to 543 | 305 to 518 | 13.68 | .2011 | .0006 | 501 | 442 | 4 | | | | | |
| 33318 | | | | 374 to 545 | 213 to 521 | 13.73 | .175 | .0006 | 495 | 431 | 1 | | | | | |
| 33340 | TOSCO | Low | High | 377 to 594 | 219 to 610 | 13.82 | 0.016 | 0.0009 | 515 | 467 | na$^a$ | 10.3×10$^6$ | 1500 | 652 | 715 | 1.5 |
| 33341 | ↓ | ↓ | ↓ | 424 to 596 | 303 to 613 | 13.86 | .0168 | .0003 | 514 | 466 | 4 | | | | | |
| 33342 | | | | 424 to 550 | 304 to 531 | 13.95 | .0152 | .0001 | 535 | 504 | 3 | | | | | |
| 33343 | | | | 380 to 554 | 224 to 537 | 13.76 | .013 | .0002 | 516 | 469 | 4 | | | | | |
| 33365 | TOSCO | High | Low | 349 to 591 | 168 to 605 | 13.82 | 0.131 | 0.0014 | 485 | 414 | 4 | 13.8×10$^6$ | 2000 | $^b$(666 to 675<br>622) | $^b$(740 to 755<br>660) | $^b$(0.36 to 0.43<br>0.75) |
| 33366 | ↓ | ↓ | ↓ | 422 to 593 | 301 to 608 | 13.37 | .1581 | .0012 | 480 | 405 | 4 | | | | | |
| 33367 | | | | 423 to 550 | 302 to 530 | 13.80 | .1397 | .0006 | 509 | 456 | 4 | | | | | |
| 33368 | | | | 350 to 549 | 170 to 528 | 13.70 | .114 | .0005 | 502 | 444 | 4 | | | | | |
| 33408 | TOSCO | High | High | 352 to 592 | 175 to 607 | 13.98 | 0.010 | 0.0011 | 524 | 483 | 4 | 13.8×10$^6$ | 2000 | $^b$(666 to 675<br>622) | $^b$(740 to 755<br>660) | $^b$(0.36 to 0.43<br>0.75) |
| 33409 | ↓ | ↓ | ↓ | 424 to 594 | 304 to 609 | 13.95 | .0144 | .0002 | 521 | 478 | 4 | | | | | |
| 33410 | | | | 425 to 552 | 306 to 535 | 13.95 | .0076 | .0002 | 506 | 452 | 4 | | | | | |
| 33411 | | | | 369 to 552 | 204 to 534 | 13.98 | .0088 | .0002 | 514 | 465 | 4 | | | | | |
| 33416 | H-Coal | Low | Low | 376 to 624 | 218 to 663 | 12.73 | 0.0005 | 0.0004 | 510 | 458 | 4 | 13.8×10$^6$ | 2000 | 611 | 640 | 0.5 |
| 33417 | ↓ | ↓ | ↓ | na | na | 12.47 | .0006 | .0004 | 509 | 456 | 4 | | | | | |
| 33418 | | | | 416 to 550 | 290 to 531 | 12.64 | .0006 | .0001 | 508 | 455 | 4 | | | | | |
| 33419 | | | | 380 to 557 | 225 to 543 | 12.79 | .0001 | .0001 | 516 | 470 | 3 | | | | | |
| 33430 | H-Coal | Low | High | 383 to 615 | 230 to 648 | 13.56 | <0.0001 | 0.0001 | 525 | 485 | 0 | 17.2×10$^6$ | 2500 | 630 to 636 | 675 to 685 | 0.5 to 0.45 |
| 33431 | ↓ | ↓ | ↓ | 424 to na | 304 to na | 13.26 | <.0001 | .0005 | >561 | 550 | 1 | | | | | |
| 33432 | | | | 424 to 545 | 304 to 522 | 13.31 | <.0001 | <.0001 | >561 | 550 | 0 | | | | | |
| 33433 | | | | 380 to 558 | 224 to 545 | 13.73 | .0005 | .0001 | >583 | 590 | 1 | | | | | |
| 33502 | COED | High | High | 360 to 592 | 188 to 606 | 13.6 | 0.0002 | 0.0003 | 541 | 515 | 0 | 13.8×10$^6$ | 2000 | $^c$(641 to 647)<br>$^d$(658 to 675)<br>$^e$(609 to 631) | $^c$(695 to 705)<br>$^d$(725 to 755)<br>$^e$(636 to 676) | $^c$0.5<br>$^d$0.7<br>$^e$1.0 |
| 33503 | ↓ | ↓ | ↓ | 425 to 593 | 305 to 608 | 13.44 | .0002 | .0003 | 517 | 471 | 3 | | | | | |
| 33504 | | | | 427 to 551 | 309 to 533 | 13.63 | .0002 | .0001 | 553 | 536 | 2 | | | | | |
| 33505 | | | | 360 to 557 | 189 to 544 | 13.69 | .0002 | .0001 | 589 | 600 | 1 | | | | | |
| 33516 | COED | High | Low | 362 to 591 | 192 to 605 | 13.07 | 0.0003 | 0.0003 | 536 | 506 | 0 | 13.8×10$^6$ | 2000 | $^c$(616 to 622)<br>$^d$(658 to 664)<br>$^e$(607 to 631) | $^c$(650 to 660)<br>$^d$(725 to 735)<br>$^e$(634 to 676) | $^c$(0.65 to 0.8)<br>$^d$0.7<br>$^e$1.0 |
| 33517 | ↓ | ↓ | ↓ | 425 to na | 305 to na | 12.88 | .0002 | .0001 | 512 | 506 | 3 | | | | | |
| 33518 | | | | 424 to 551 | 304 to 532 | 12.96 | .0002 | .0001 | 541 | 514 | 4 | | | | | |
| 33519 | | | | 360 to 553 | 189 to 536 | 13.24 | .0002 | .0001 | 548 | 527 | 0 | | | | | |

[a] Not available, na.

[b] Hydrotreatment and hydrocracking conditions. Final sample blends also contain low-yield material.

[c] Hydrotreatment of IBP to 561 K (IBP to 550° F) fraction.

[d] Hydrotreatment of 561 to 700 K (550° to 800° F) fraction.

[e] Hydrotreatment of 561 to 700 K (550° to 800° F) fraction

(c, d, e) Final sample blends contain products from treatment of both fractions.

## TABLE III. - EXXON FUEL SAMPLES

[Data from ref. 5.]

| EXXON number | Crude | Fuel type | Severity level | ASTM boiling range | | Weight percent | | | Jet fuel thermal oxidation tester breakpoint temperature | | Process conditions | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | K | °F | H | N | S | K | °F | Pressure N/m$^2$ | Pressure psi | Temperature K | Temperature °F | Space velocity, LHSV, hr$^{-1}$ |
| 113 | TOSCO | JP-4 | Low | 384 to 517 | 231 to 471 | 13.05 | 0.0093 | 0.0005 | 552 | 534 | 5.5×10$^6$ | 800 | 644 | 700 | 1.2 |
| 113 | | Jet A | Low | 443 to 539 | 338 to 511 | 13.41 | .017 | .0012 | 523 | 482 | 5.5 | 800 | | | 1.2 |
| 17-B | | JP-4 | Medium | 384 to 521 | 231 to 478 | 14.3 | .0062 | .0003 | 577 | 580 | 10.3 | 1500 | | | .93 |
| 17-B | | Jet A | Medium | 445 to 540 | 342 to 512 | 13.87 | .0063 | .0009 | 637 | 688 | 10.3 | 1500 | | | .93 |
| 410 | | JP-4 | High | 395 to 516 | 252 to 469 | ----- | .0019 | .0011 | >541 | >515 | 15.1 | 2200 | | | .56 |
| 410 | | Jet A | High | 438 to 534 | 329 to 501 | ----- | .0034 | .0008 | >575 | >575 | 15.1 | 2200 | | | .56 |
| 111 | Paraho | Jet A | Low | 437 to 536 | 328 to 505 | 13.02 | 0.24 | ------ | 477 | 400 | 5.5×10$^6$ | 800 | 644 | 700 | 1.08 |
| 11-B | Paraho | Jet A | Medium | 463 to 527 | 374 to 490 | 13.45 | .0036 | 0.0004 | 572 | 570 | 10.3 | 1500 | 644 | 700 | .93 |
| 414 | Paraho | Jet A | High | 451 to 523 | 352 to 482 | ----- | .0032 | <.0001 | >583 | >590 | 15.1 | 2200 | 644 | 700 | [a](0.35 to 0.50) |
| 115 | Garrett | Jet A | Low | 434 to 542 | 321 to 517 | 13.41 | 0.0052 | 0.0019 | 502 | 445 | 5.5×10$^6$ | 800 | 644 | 700 | 0.91 |
| 103 | | Jet A | Medium | 447 to 534 | 345 to 502 | 13.58 | .0030 | .0004 | 586 | 585 | 10.3 | 1500 | | | .99 |
| 404 | | JP-4 | High | 392 to 533 | 246 to 500 | ----- | .0027 | .0056 | [b]>533 | [b]>500 | 15.1 | 2200 | | | .48 |
| 404 | | Jet A | High | 454 to 530 | 358 to 495 | ----- | .0026 | .0036 | >597 | >615 | 15.1 | 2200 | | | .48 |
| 405 | | Jet A | High | 459 to 526 | 366 to 488 | ----- | .0015 | .0009 | >602 | >625 | 15.1 | 2200 | | | .42 |
| 203 | Synthoil | Jet A | Low | 444 to 546 | 340 to 524 | 11.08 | 0.0067 | 0.0005 | 487 | 418 | 5.5×10$^6$ | 800 | 644 | 700 | 0.88 |
| 105 | | | Medium | 439 to 538 | 331 to 509 | 12.17 | .0030 | .0022 | 555 | 540 | 10.3 | 1500 | | | .81 |
| 107 | | | Medium | 434 to 536 | 322 to 505 | 11.96 | .0057 | .0005 | 544 | 520 | 10.3 | 1500 | | | .95 |
| 202 | | | Medium | 442 to 541 | 337 to 514 | 12.33 | .0016 | .0029 | 533 | 500 | 10.3 | 1500 | | | .69 |
| 416 | | | High | 396 to 527 | 254 to 489 | ----- | .0029 | .0004 | >566 | >560 | 15.1 | 2200 | | | [a].54 |
| 304 | H-Coal | JP-4 | Low | 369 to 490 | 205 to 422 | 12.61 | 0.0026 | 0.0003 | 519 | 475 | 5.5×10$^6$ | 800 | 644 | 700 | 0.99 |
| 304 | | Jet A | Low | 448 to 522 | 347 to 480 | 11.7 | .0027 | .0006 | 467 | 382 | 5.5 | 800 | | | .99 |
| 209 | | JP-4 | Medium | 361 to 492 | 190 to 427 | 14.29 | .0024 | .0001 | 552 | 535 | 10.3 | 1500 | | | .95 |
| 209 | | Jet A | Medium | 444 to 520 | 340 to 476 | 12.66 | .0047 | .0016 | 536 | 505 | 10.3 | 1500 | | | .95 |
| 419 | | Jet A | High | 452 to 519 | 354 to 474 | ----- | .0026 | <.0001 | 541 | >515 | 15.1 | 2200 | | | .49[a] |

[a] Final blends result from multistage treatments. Conditions cited here are for last stage only.

[b] Considered indeterminate due to excessive vaporization in test apparatus.

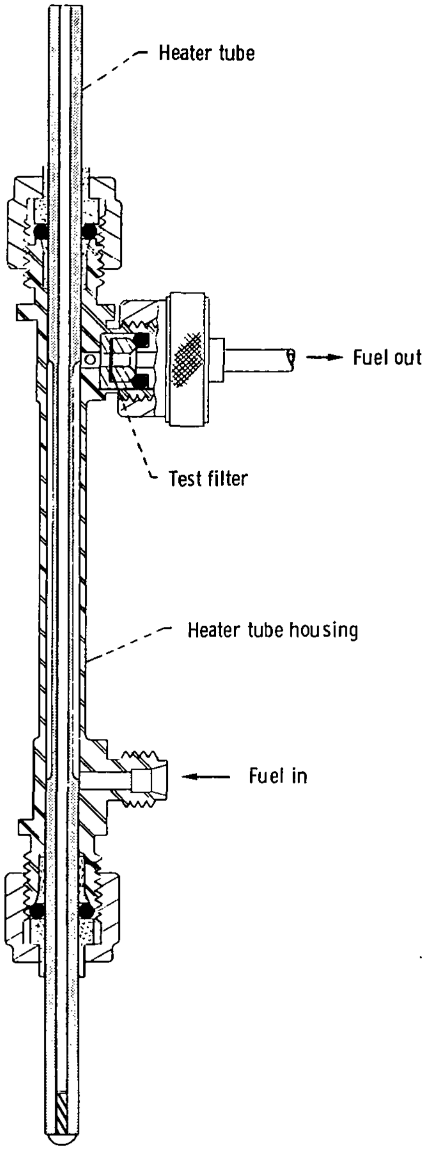


Figure 1. - Assembly drawing of heater tube section. Heated length, 6.0 centimeters; tube outside diameter, 0.325 centimeter; flow rate, 3.0 cubic centimeter per minute; residence time, 2.0 seconds per centimeter length; flow velocity, 0.5 centimeter per second.

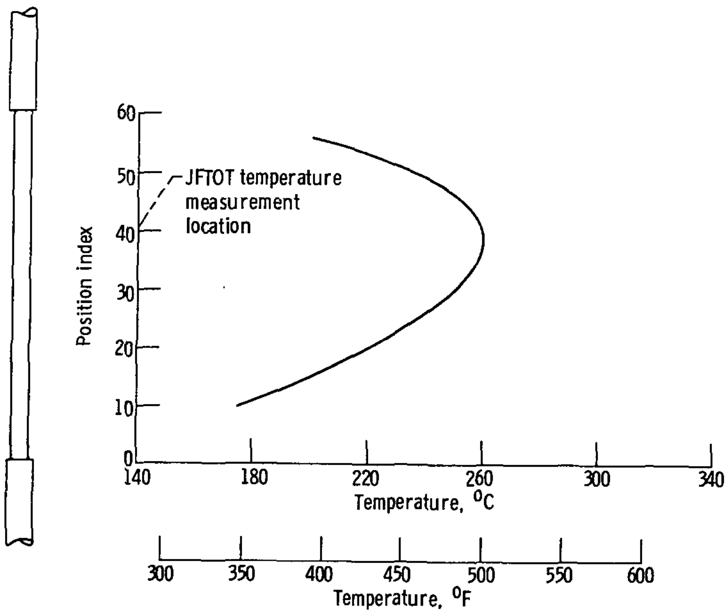


Figure 2. - Typical temperature profile in JFTOT tubes.

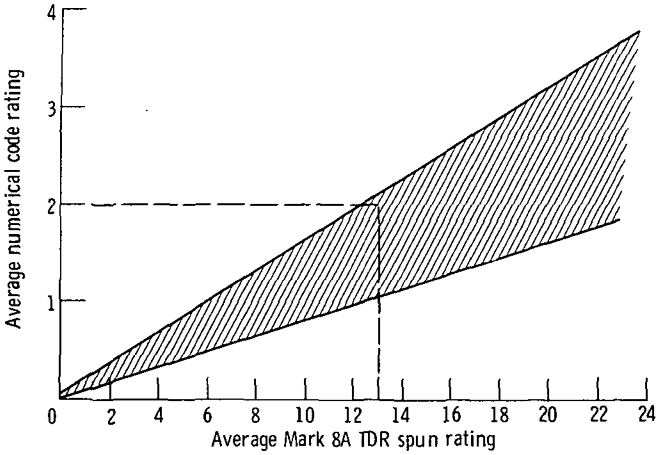
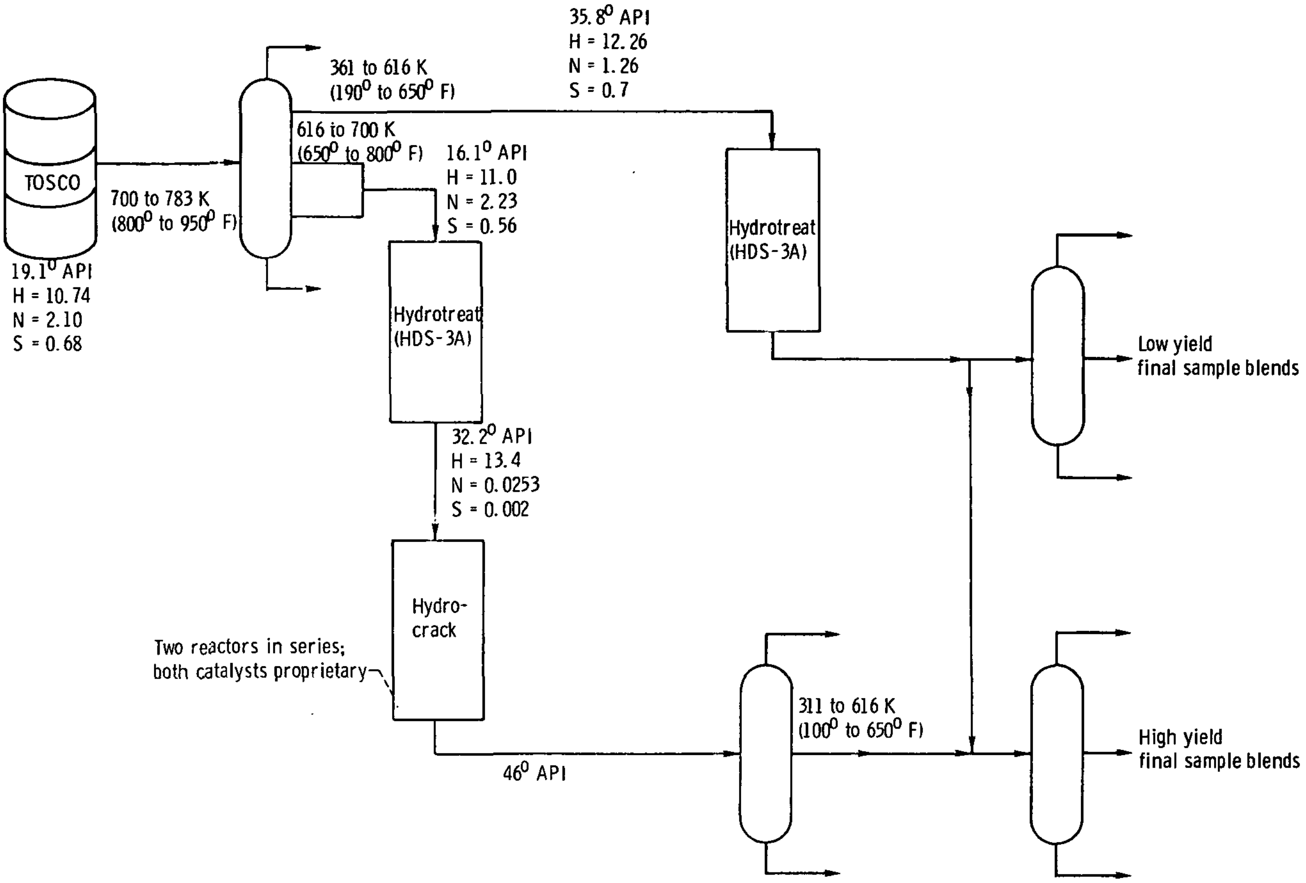
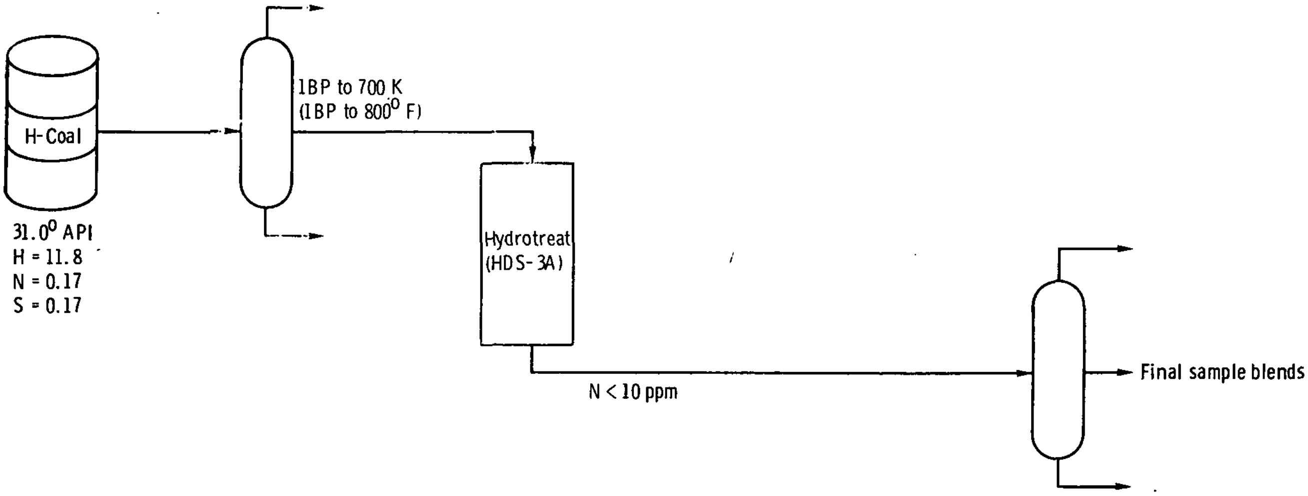


Figure 3. - Comparison of visual code ratings and Mark 8A spun ratings (from ref. 4).

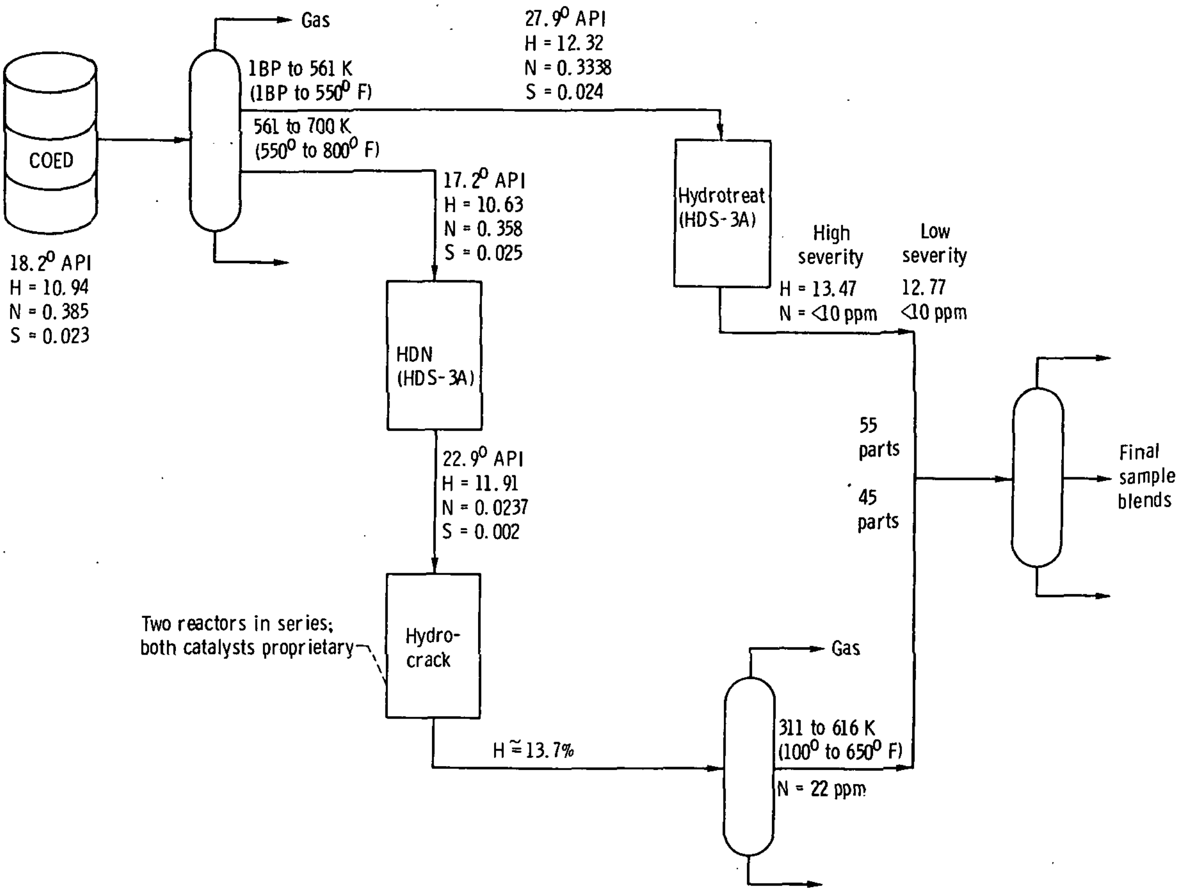


(a) TOSCO shale oil.



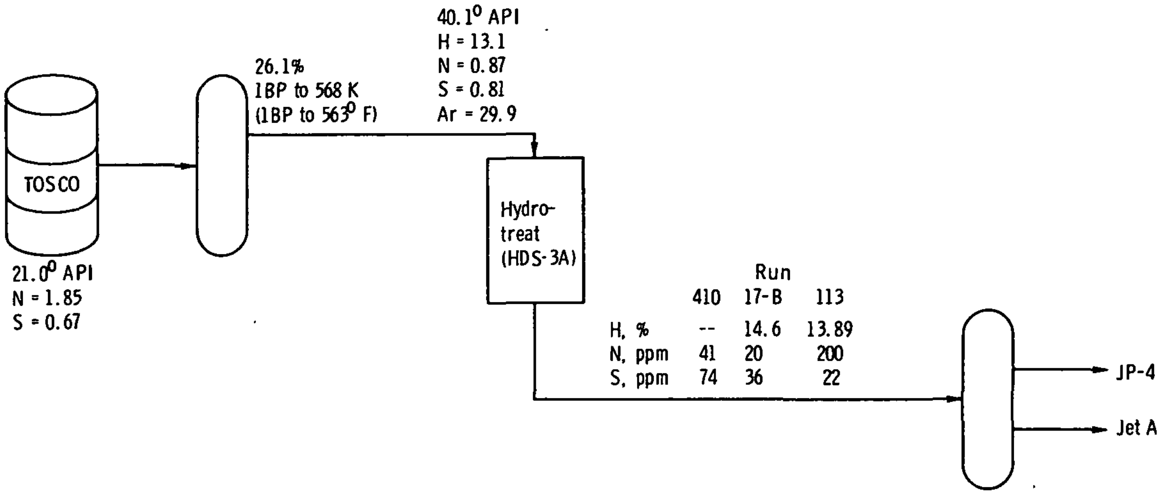
(b) H-Coal.

Figure 4. - Schematic diagram of ARCO product treatments.



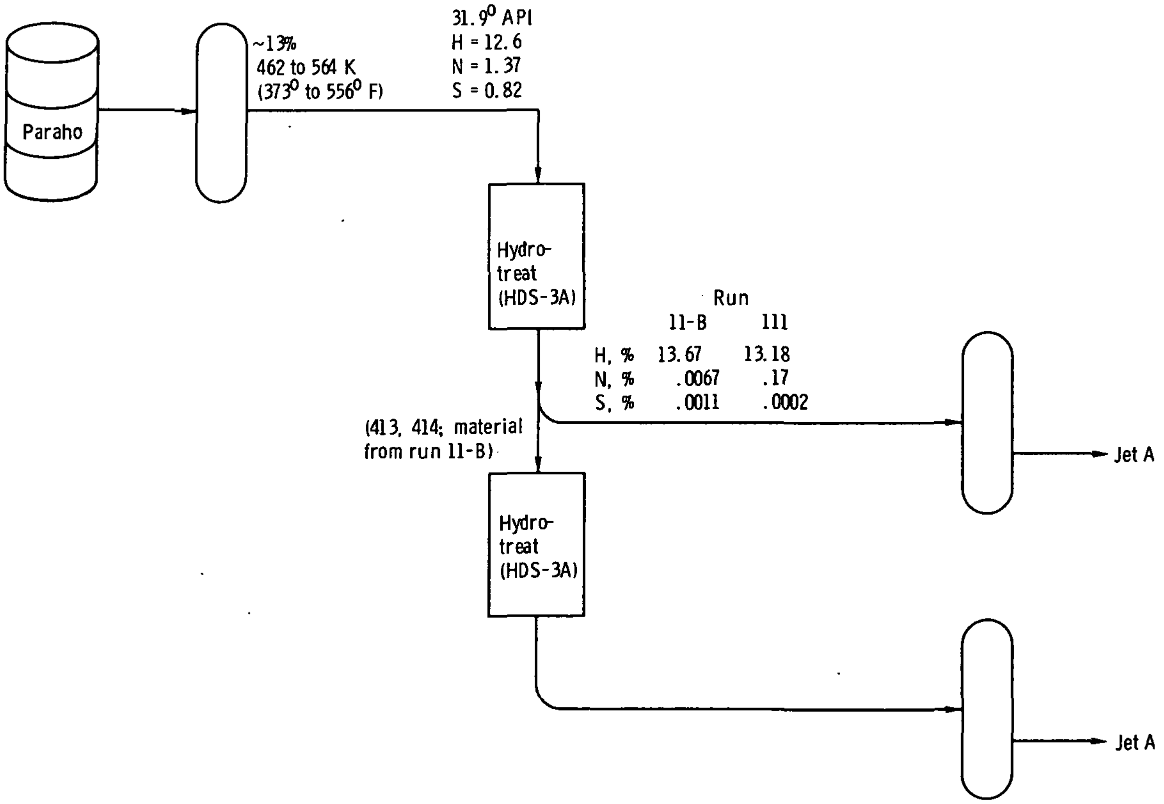
(c) COED.

Figure 4. - Concluded.



|  | Run 410 | Run 17-B | Run 113 |
|---|---|---|---|
| H, % | -- | 14.6 | 13.89 |
| N, ppm | 41 | 20 | 200 |
| S, ppm | 74 | 36 | 22 |

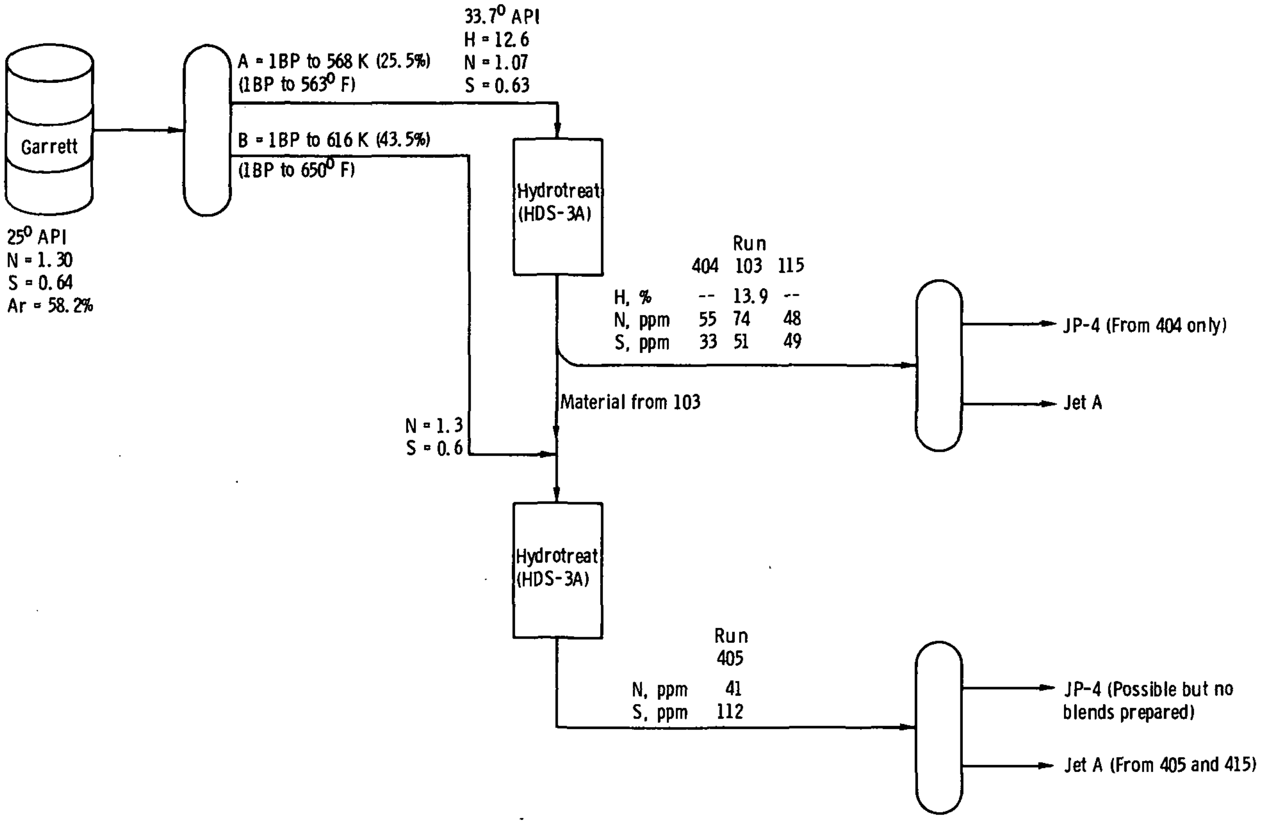
(a) TOSCO shale oil.



|  | Run 11-B | Run 111 |
|---|---|---|
| H, % | 13.67 | 13.18 |
| N, % | .0067 | .17 |
| S, % | .0011 | .0002 |

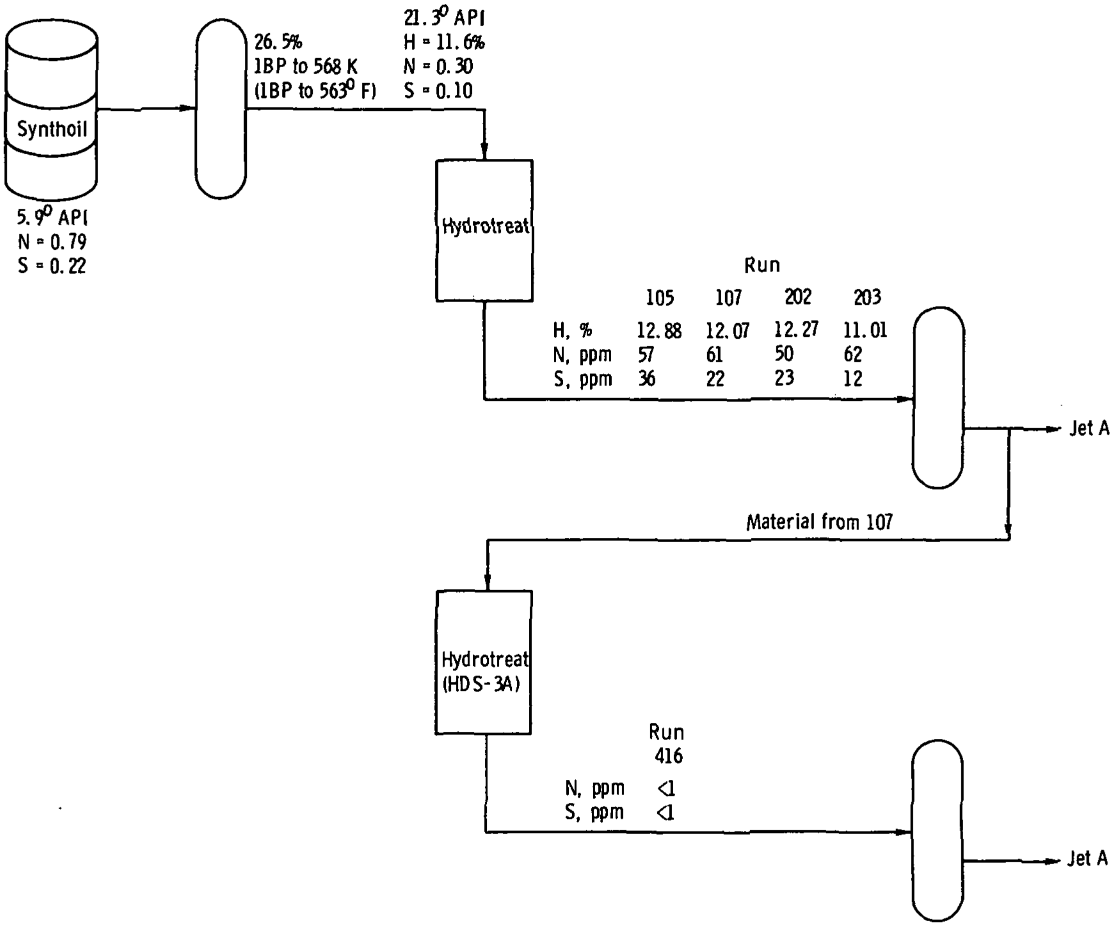
(b) Paraho shale oil.

Figure 5. - Schematic diagram of Exxon product treatments.



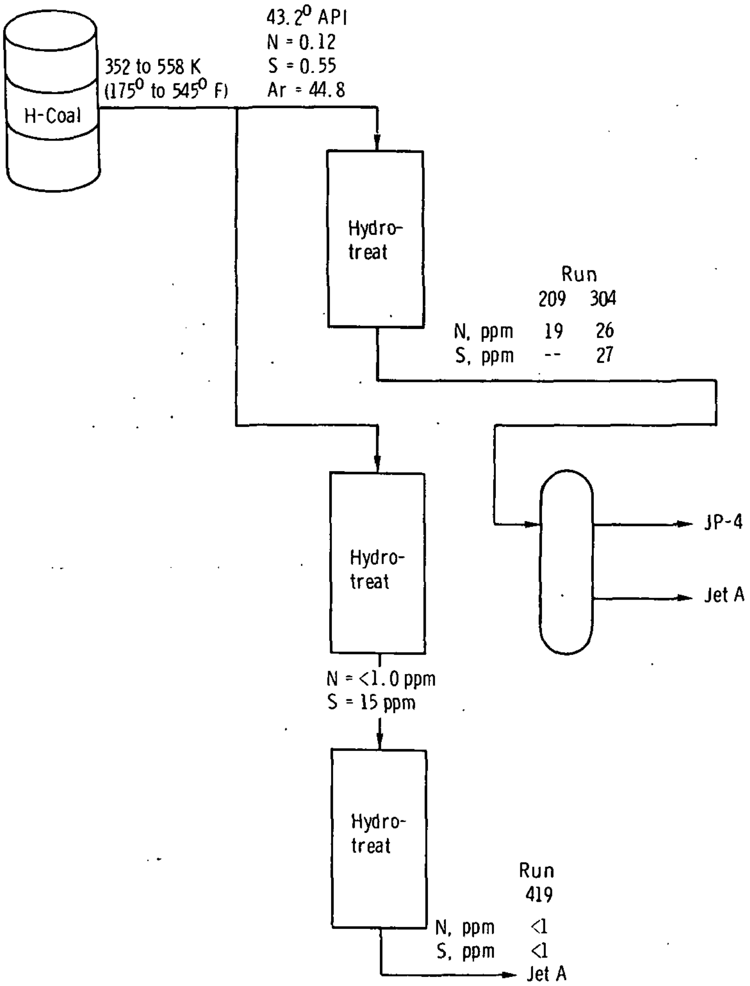
(c) Garrett shale oil.

Figure 5. - Continued.



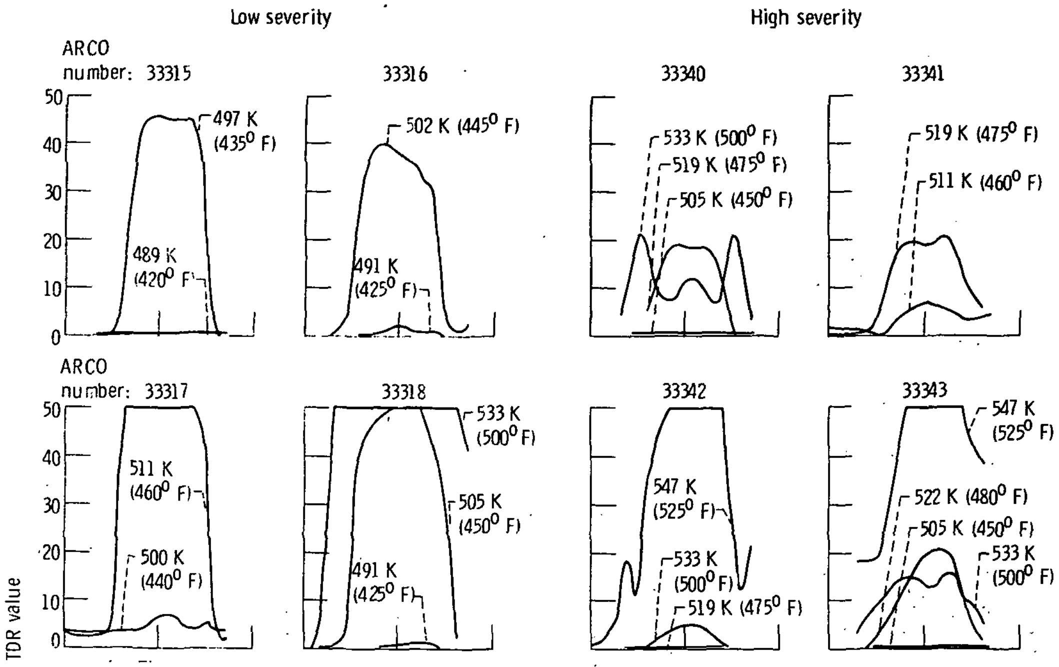
(d) Synthoil.

Figure 5. - Continued.

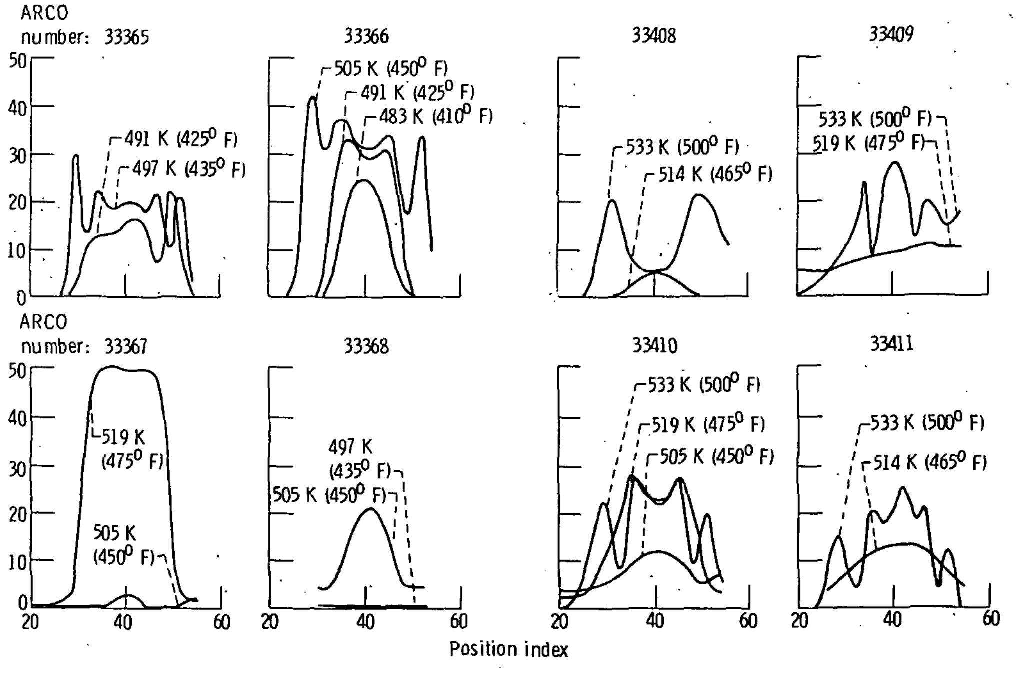


(e) H-Coal.

Figure 5. - Concluded.

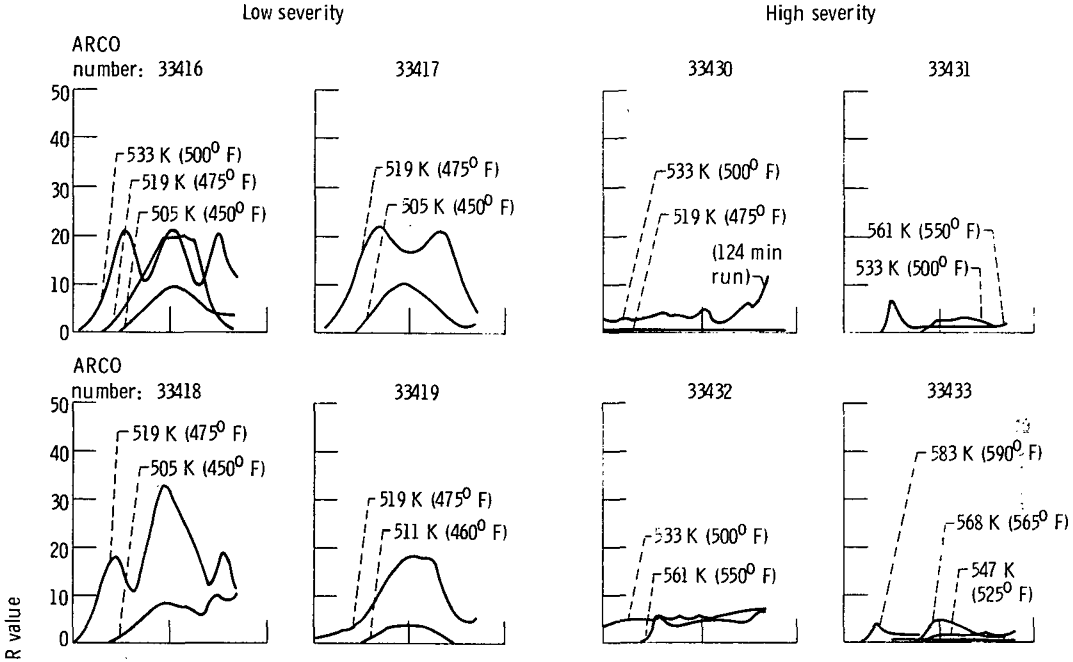


(a) TOSCO - low yield.

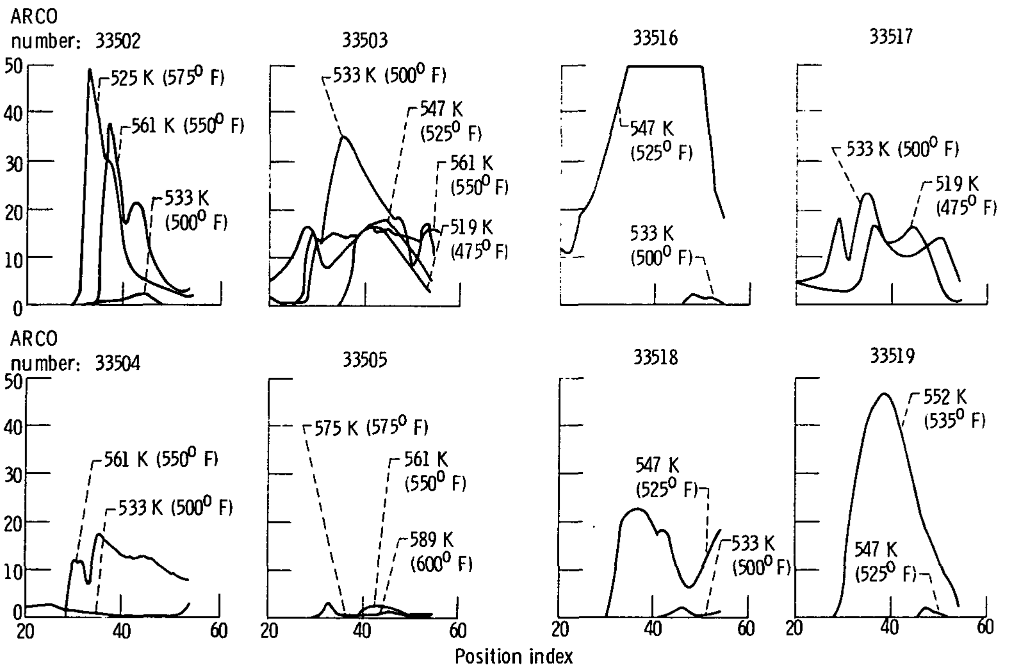


(b) TOSCO - high yield.

Figure 6. - Axial scan of Mark 8A spun tube deposit ratings.

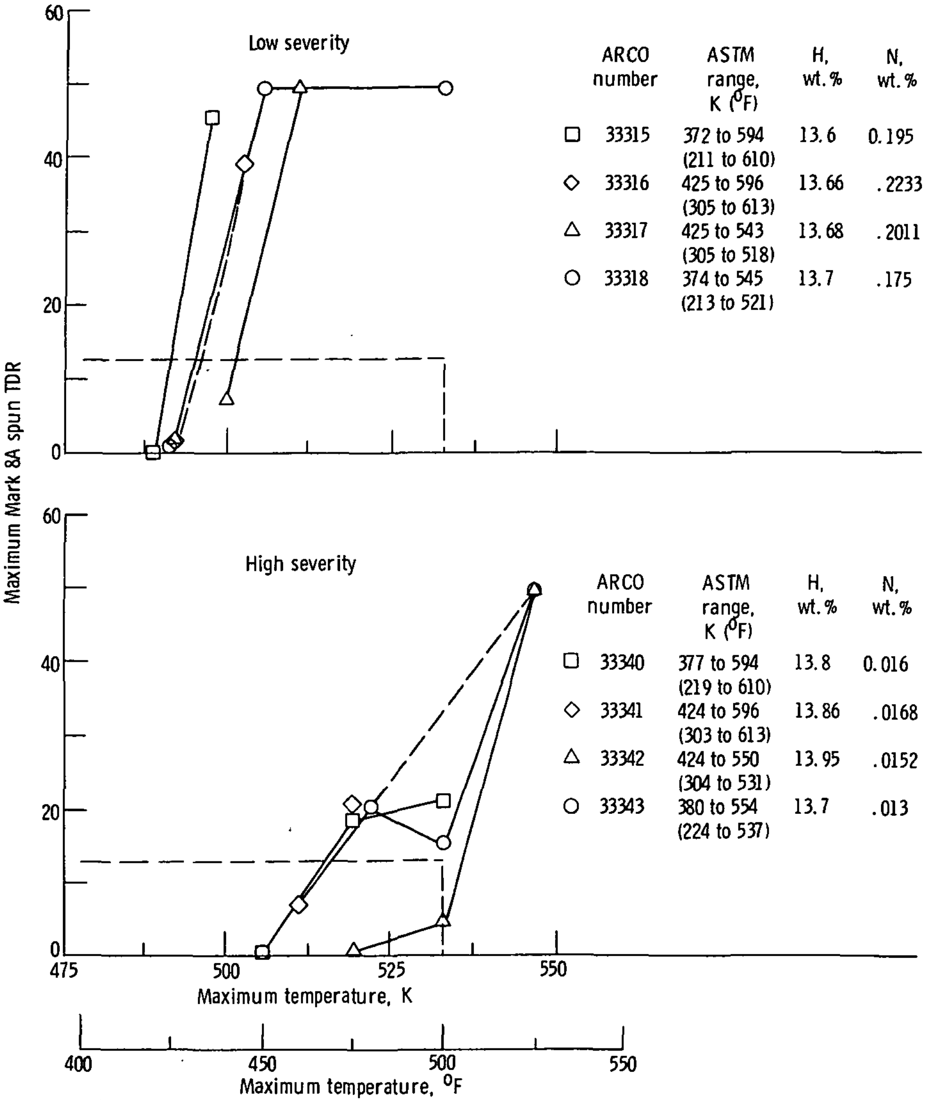


(c) H coal low-yield.



(d) COED high yield.

Figure 6. - Concluded.



Low severity

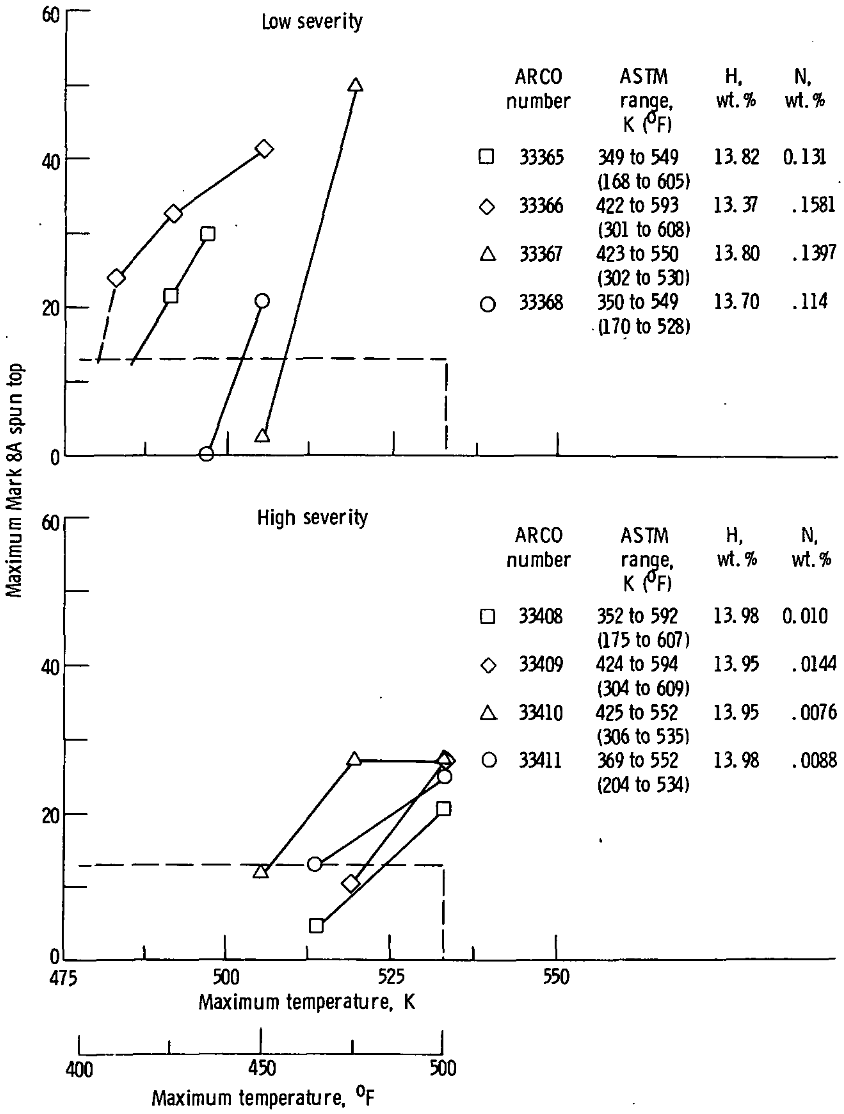
| | ARCO number | ASTM range, K (°F) | H, wt.% | N, wt.% |
|---|---|---|---|---|
| □ | 33315 | 372 to 594 (211 to 610) | 13.6 | 0.195 |
| ◇ | 33316 | 425 to 596 (305 to 613) | 13.66 | .2233 |
| △ | 33317 | 425 to 543 (305 to 518) | 13.68 | .2011 |
| ○ | 33318 | 374 to 545 (213 to 521) | 13.7 | .175 |

High severity

| | ARCO number | ASTM range, K (°F) | H, wt.% | N, wt.% |
|---|---|---|---|---|
| □ | 33340 | 377 to 594 (219 to 610) | 13.8 | 0.016 |
| ◇ | 33341 | 424 to 596 (303 to 613) | 13.86 | .0168 |
| △ | 33342 | 424 to 550 (304 to 531) | 13.95 | .0152 |
| ○ | 33343 | 380 to 554 (224 to 537) | 13.7 | .013 |

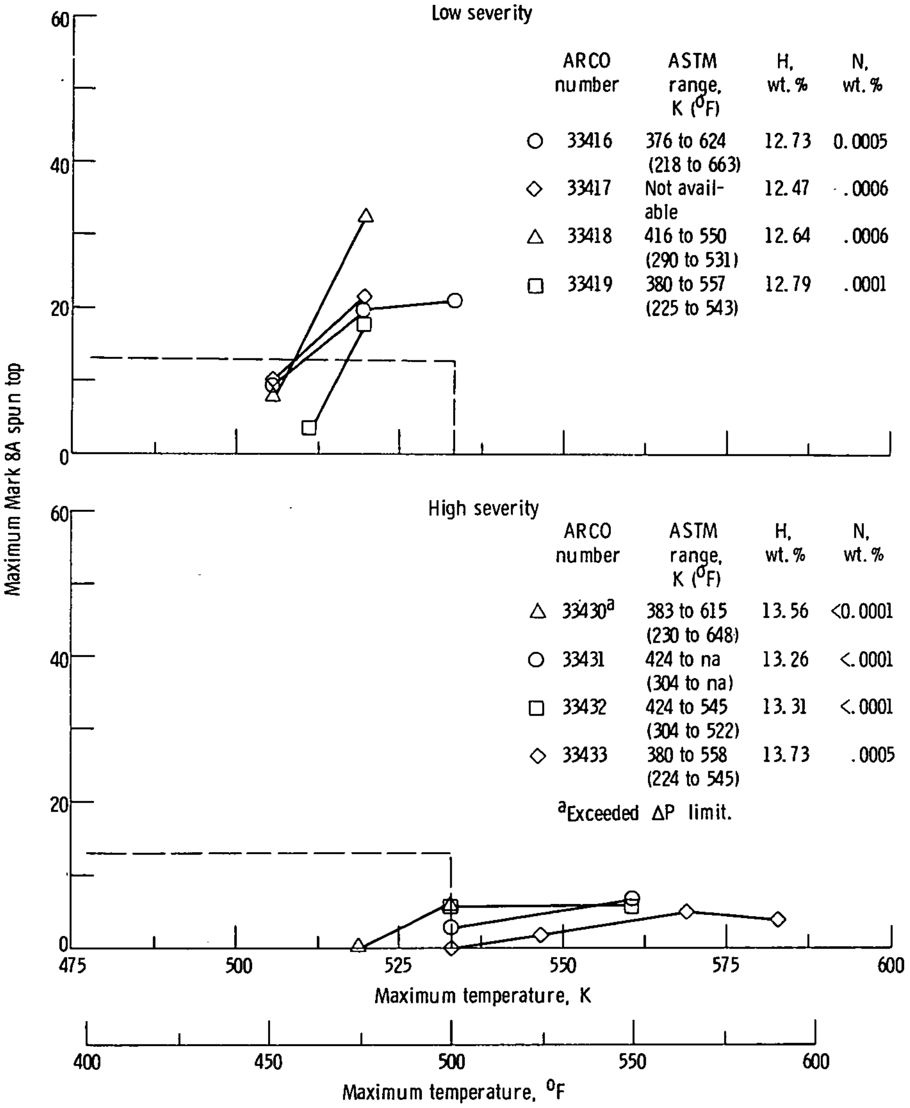
(a) TOSCO low yield.

Figure 7. - Variation of maximum Mark 8A spun TDR with temperature.



(b) TOSCO high yield.

Figure 7. - Continued.



Low severity

| | ARCO number | ASTM range, K (°F) | H, wt.% | N, wt.% |
|---|---|---|---|---|
| ○ | 33416 | 376 to 624<br>(218 to 663) | 12.73 | 0.0005 |
| ◇ | 33417 | Not available | 12.47 | .0006 |
| △ | 33418 | 416 to 550<br>(290 to 531) | 12.64 | .0006 |
| □ | 33419 | 380 to 557<br>(225 to 543) | 12.79 | .0001 |

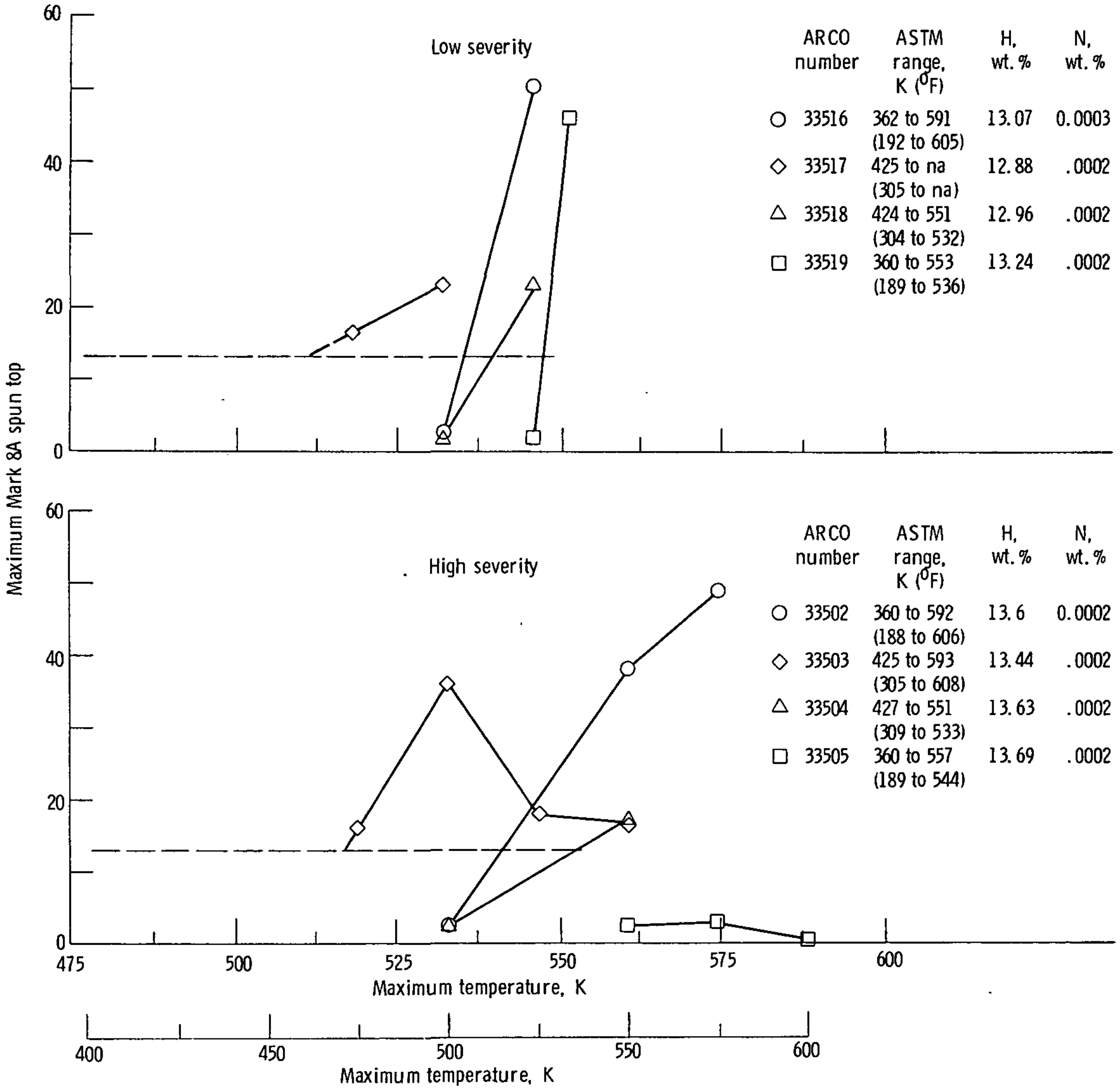
High severity

| | ARCO number | ASTM range, K (°F) | H, wt.% | N, wt.% |
|---|---|---|---|---|
| △ | 33430[a] | 383 to 615<br>(230 to 648) | 13.56 | <0.0001 |
| ○ | 33431 | 424 to na<br>(304 to na) | 13.26 | <.0001 |
| □ | 33432 | 424 to 545<br>(304 to 522) | 13.31 | <.0001 |
| ◇ | 33433 | 380 to 558<br>(224 to 545) | 13.73 | .0005 |

[a] Exceeded ΔP limit.

(c) H-Coal low yield.

Figure 7. - Continued.



(d) COED high yield.

Figure 7. - Concluded.

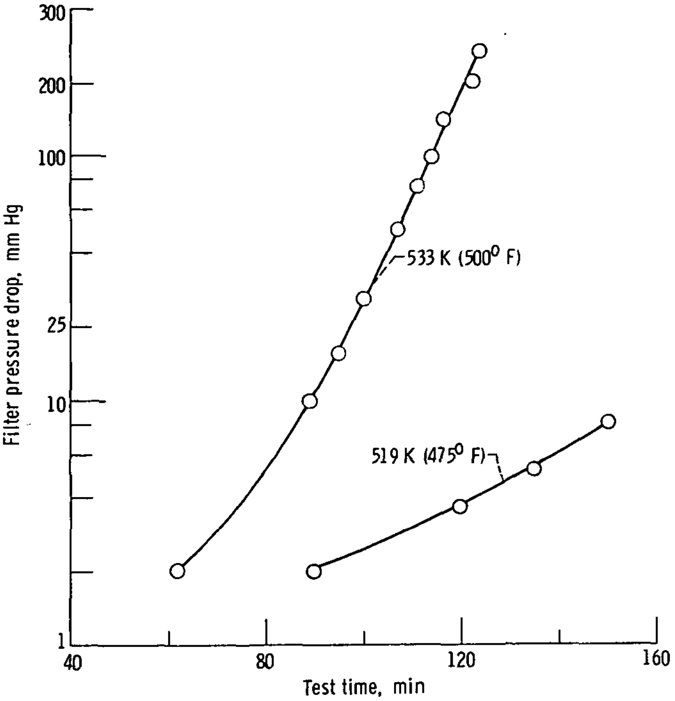


Figure 8. - Time variation of pressure drop through JFTOT test filter for ARCO sample 33430 (H-Coal).

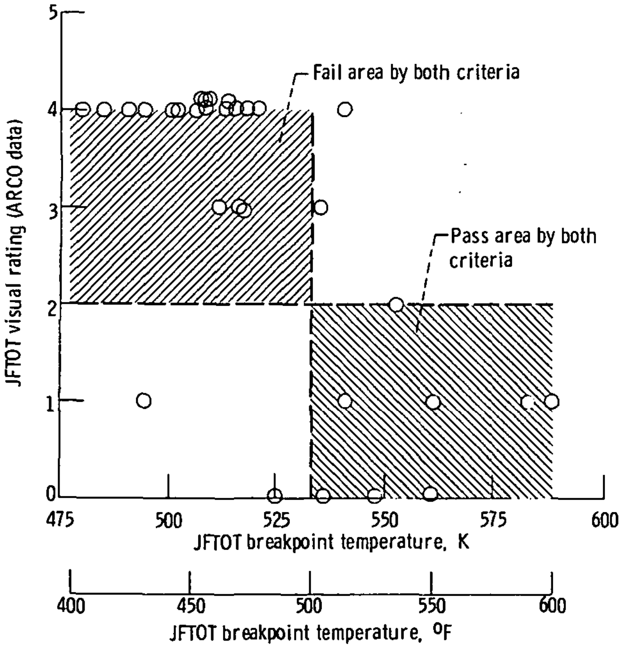


Figure 9. - Comparison of breakpoint temperature data (Lewis) with visual rating data (ARCO) on same sample.

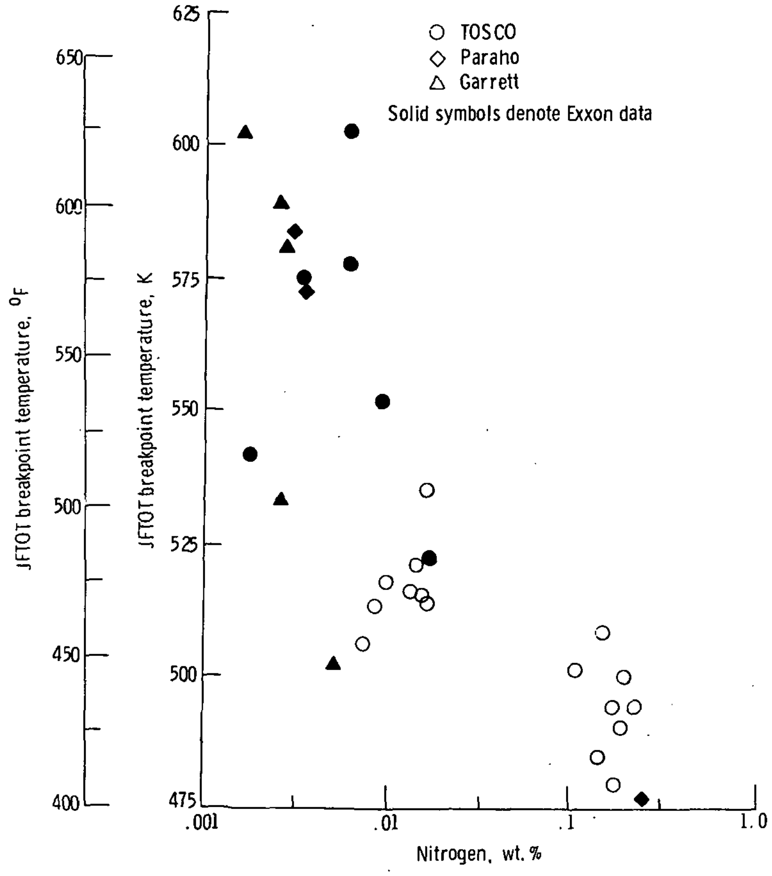
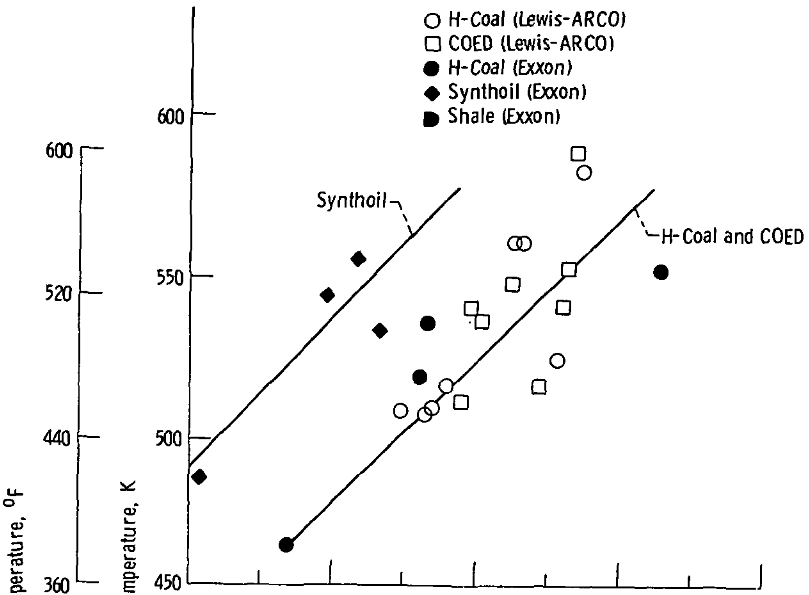
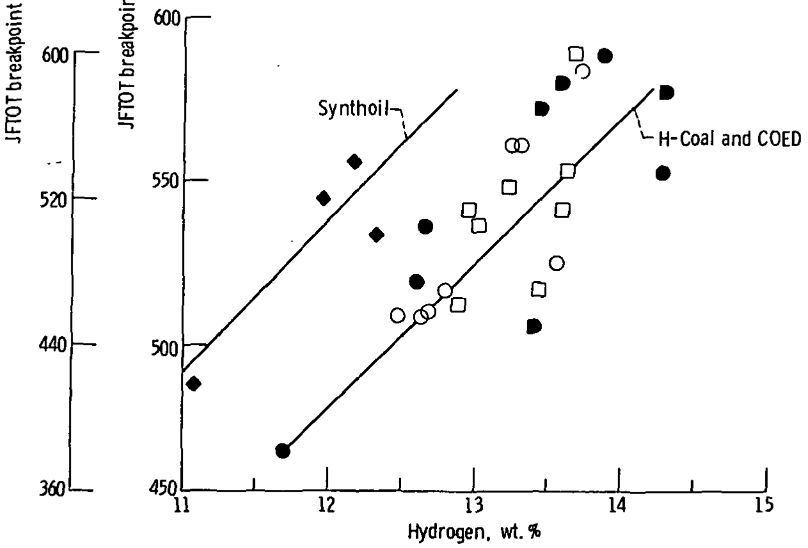


Figure 10. - Variation of JFTOT breakpoint temperature with nitrogen level after hydrotreatment.



(a) Coal fuels.



(b) Coal and shale fuels.

Figure 11. - Variation of breakpoint temperature with hydrogen content of low-nitrogen ($\leq 67$ ppm) fuels.

NASA-Langley, 1977 E-9070

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