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"INVESTIGATION AND DEVELOPMENT OF  
ALTERNATIVE METHODS FOR SHALE  
OIL PROCESSING AND ANALYSIS"

OCTOBER, 1979 - APRIL, 1983  
DEPARTMENT OF CHEMISTRY  
ALABAMA A.& M. UNIVERSITY

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(2) This project began in 1978 with retorting equipment on long term loan from Lawrence Livermore National Laboratory (LLNL), Livermore, California. This equipment has been modified and is currently still in use. The author expresses thanks to the oil shale group in the Chemistry and Materials Science division, particularly Drs. John H. Campbell and Alan Burnham for continuing it. Thanks is also extended to Messrs. Jim Taylor and George Koskinas for invaluable technical assistance.

In addition to the Chemistry and Materials Science Division, the author is indebted to Dr. Daniel Stuermer of the Environmental Science Division at LLNL for the Ames assay results on the raw oils as well as initial exposure to HPLC.

Finally, the author expresses his thanks to the O.E.O. Office at Livermore for support during several summers without which involvement in energy research would not have been made immediately possible. Particular thanks to Manual Perry, Marvin Smith, Jim Evans, Shirley McDavid and Omega Ware for making possible continued summer support at LLNL.

Essentially all of the retorting of oil shale and oil shale mixtures was done by Brian Thomas, a senior chemistry major, scheduled to graduate in May of 1983. Without his dedication and insight, this work could not have proceeded as rapidly.

INVESTIGATION AND DEVELOPMENT OF  
ALTERNATIVE METHODS FOR SHALE  
OIL PROCESSING AND ANALYSIS

Narrative

I. Effects of selected wastes on Oil Shale Retorting.

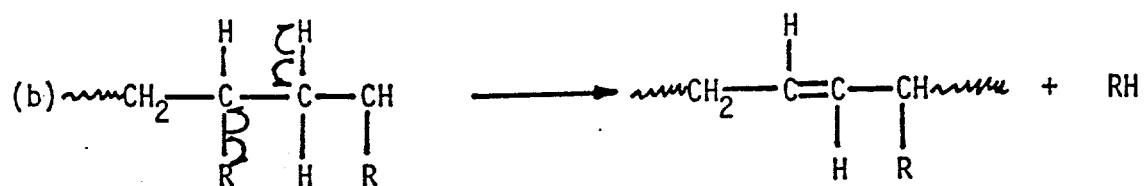
A. Background and Rationale.

Oil shale, a carbonaceous rock which occurs abundantly in the earth's crust, has been investigated for many years as an alternate source of fuel oil. The insoluble organic matter contained in such shales is termed "Kerogen" from the Greek meaning oil or oil forming. The kerogen in oil shale breaks down into oil-like products when subjected to conditions simulating destructive distillation. These products have been the subject of extensive investigations by several researchers and many of the constituents of shale oil have been identified. (1) Forsman (2) estimates that the kerogen content of the earth is roughly  $3 \times 10^{15}$  tons as compared to total coal reserves of about  $5 \times 10^{12}$ . Although the current cost per barrel estimate for commercial production of shale oil is higher than that of fossil oil, as our oil reserves continue to dwindle, shale oil technology will become more and more important.

When oil shale is heated, kerogen is said to undergo chemical transformation to usable oil in two steps (3): Kerogen (in oil shale)  $\xrightarrow{300-500^{\circ}\text{C}}$  bitumen  $\xrightarrow{\hspace{1cm}}$  Crude shale oil and other products. The crude shale oil so obtained differs from fossil oil in that: (1) kerogen is thought to have been produced from the aging of plant matter over many years; (2) shale oil has a higher nitrogen content than fossil oil; (3) non-hydrocarbons are present to a much greater extent in shale oil; (4) the hydrocarbons in shale oil are much more unsaturated than those in fossil oil (petroleum).

Investigators at Lawrence Livermore Laboratory have studied the kinetics of decomposition of Colorado oil shale (3). The activation energies so obtained (48-54 Kcal/mole) are significantly lower than those expected for thermal scission of C-C, C-H or C-O bonds (80-100 Kcal/mole), inferring that free radical processes must accompany kerogen decomposition into crude oil.

It is well known that the thermal decomposition of such disposable synthetics as polystyrene occurs to give small fragments through radical processes. The radical nature of the thermal decomposition of synthetic polymeric materials has been the subject of a number of papers and books for many years. The greatest amount of quantitative work on thermal degradation has been published on vinyl and related polymers in the 300-500°C temperature range (5). In general, degradation is by either (a) chain scission or (b) nonchain scission reactions:

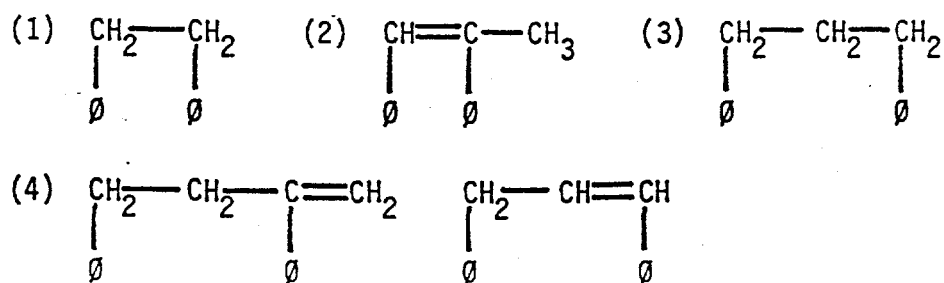


Thermal degradation via chain scission is a typical free-radical chain reaction involving separate initiation, propagation and termination steps. Initiation generally involves homolytic scission of C-C bonds to form two free radicals. An important consideration in the degradation process is to

determine whether or not the polymer degrades very rapidly to volatile monomer ("unzips") or if there are intermediate stages in which non-volatile fragments of intermediate molecular weight are produced which then undergo further fragmentation to form products. Studies have shown that certain polymers do unzip such as poly (methyl methacrylate) ( $E_a$  48 Kcal/mole) and poly ( $\alpha$ -methylstyrene) ( $E_a$  65 Kcal/mole), and give 92-100 weight percent of monomer upon being thermally decomposed. Most polymers, however, do not cleanly depolymerize. Polystyrene ( $E_a$  88 Kcal/mole) yields only 42% monomer and, in addition the products shown below:

Chart 1

Products from thermal decomposition of polystyrene at 350°C (4)



Linear and branched polyethylene yield only 0.03 percent monomer while poly (isobutene) and poly (methyacrylate) yield 0.7 and (18-32) percent.

The implication from these observations for the present study is that such synthetic polymers which decompose to give an array of products should interact with fragments generated in oil shale decomposition to give oils which have incorporated parts of the polymer into the product oil structure. Such interactions may provide a way of supplementing or enhancing the yields of oil produced on the laboratory scale and possibly commercial production of shale oil. The measured activation energies indicate that



oil shale and polymer decomposition should occur almost simultaneously as retorting proceeds. Such polymers as poly (methylmethacrylate) may not be as effective at intermingling with shale oil fragments and may give only a solution of monomer in shale oil or may volatilize and escape completely. Interaction of oil shale fragments with this type polymer would be determined by the rate of depolymerization versus the rate of chain transfer.

#### MATERIALS AND METHODS

The apparatus used for retorting oil shale is illustrated schematically below. This apparatus is patterned after the modified Fisher Assay apparatus as designed and used at Lawrence Livermore Laboratory, Livermore, California (6).

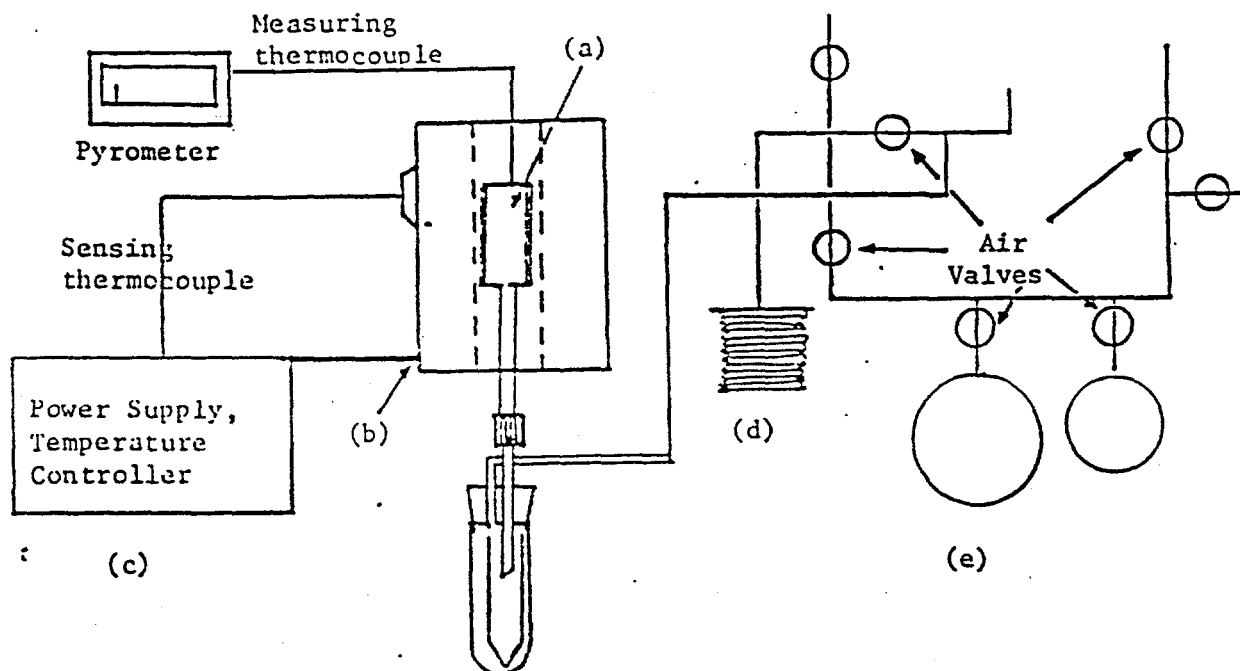


Figure 1, Fisher Assay Apparatus.

The sample to be retorted is weighed into the retort vessel (a) and the top heliarc welded into place. The vessel is placed into the oven (b) and the entire system is flushed with an inert gas (argon; helium etc.). The gas

pressure within the system is then adjusted to approximately 2/3 atmosphere and the power supply (c) activated to heat at a rate of 12°C per minute. Oil generation begins around 250-275°C and continues until about 450°C. The gases generated are initially collected in the stainless steel bellows (d). When the bellows is fully extended the gases are transferred to the evacuated collection vessel (e) by activating a series of air valves. The temperature is then held at 500°C for thirty minutes after which the apparatus is allowed to cool. The collected gas and oil are weighed and compared with the weight of the retort vessel before and after heating to give material balance. The oil density is determined using a pycnometer and oil yield in liters per tonne and gallons per ton is determined.

The apparatus and procedure outlined above have been used to generate data on about fifty samples to date. This data, selectively summarized in Table 2, has been collected for mixtures of oil shale with (1) polystyrene, (2) polyethylene, (3) sawdust, (4) SBR rubber, (5) corrugated cardboard, (6) polyvinylchloride (PVC) and (7) polyethylene terephthalate. Both Alabama and Colorado shale have been used for oil generation. In all cases except for corrugated cardboard, small to substantial increases in oil yield (above Fischer Assay) have been noted. The oil integrity as determined by infrared and gas chromatographic analyses seems to have been changed least for sawdust, corrugated cardboard, and SBR rubber runs.

It is well known that shale oil has somewhat more mutagenic effects than fossil oil. Since the processing of waste materials with oil shale produces altered shale oils we were very interested in having the mutagenic properties of our oils determined and compared with the assay oils. During the summer of 1982 several oils from our laboratory were subjected to Ames

assay at Lawrence Livermore Laboratory. The results of these analyses are summarized in Table 1. We were very pleased to find that the altered oils were in no cases more mutagenic than the assay oils. We concluded from these tests that the waste substances used to date do not impart any harmful effects to the oils being generated in this manner.

Table 1 Ames Assay Results On Shale Oils Generated Using Selected Wastes.

| Oil Shale          | % Additive      | Counts, Minimum | Counts, Maximum | # of Revertants Per mg. |
|--------------------|-----------------|-----------------|-----------------|-------------------------|
| Colorado (R-1)     | 0 (Assay)       | 161             | 325             | 0.21                    |
| Colorado (R-10)    | 15% SBR Rubber  | 144             | 342             | 0.23                    |
| Colorado (R-12-13) | 4% Cardboard    | 85              | 231             | 0.22                    |
| Colorado (R-19)    | 5% polystyrene  | 97              | 156             | 0.037                   |
| Colorado (R-21)    | 5% polyethylene | 184             | 346             | 0.076                   |
| Colorado (R-27)    | 15% sawdust     | 75              | 135             | 0.066                   |
| Alabama (R-8-14)   | 0 (Assay)       | 45              | 80              | 0.13                    |
| Alabama (R-16)     | 15% SBR Rubber  | 28              | 54              |                         |
| Alabama (R-9-23)   | 5% polystyrene  | 63              | 111             | 0.027                   |

#### B. Summary of Research to Date

Table 2 is a summary of all retort runs involving both Colorado and Alabama oil shale mixed with various natural and synthetic waste products. Retort runs involving polystyrene, polyethylene and SBR rubber all resulted in substantial increases in oil yield. Selected retort runs showing percent changes for all additives used are given in table 3. Infrared spectra (appendix I) of the oils produced show that polystyrene shale oils have very enhanced aromatic character whereas most other oils do not exhibit significant differences from

Table 2 Oil Shale Retort Runs

| Shale Type  | Run # | Wt. of Shale | Wt. of Additive | Total Oil (gms.) | Total Water | Total Gas | Oil Density g/ml. | Oil Yield (Gal./ton) | Based on Total Weight | Material Balance |
|-------------|-------|--------------|-----------------|------------------|-------------|-----------|-------------------|----------------------|-----------------------|------------------|
| Col.        | R-1   | 100.0388     | 0.0             | 11.0537          | 0.8881      | 2.20      | 0.8856            | 29.63                | 29.63                 | 96.4             |
| Col.        | R-18  | 92.5268      | 0.0             | 9.9423           | 0.6307      | 2.16      | 0.8914            | 28.89                | 28.89                 | 97.7             |
| Col.        | R-2   | 78.2448      | 0.0             | 9.0192           | 0.3342      | 2.10      | 0.8927            | 27.75                | 27.75                 | 99.9             |
| Col.        | R-5   | 59.3463      | 8.8063 (a)      | 15.2162          | 0.5151      | 2.00      | 0.8371            | 73.40                | 63.92                 | 99.3             |
| Col.        | R-21  | 59.9926      | 2.9988 (a)      | 9.4406           | 0.4388      | 1.44      | 0.8678            | 43.46                | 41.39                 | 98.2             |
| Col.        | R-6   | 53.2714      | 1.3488 (b)      | 7.2375           | 0.5555      | 1.60      | 0.8968            | 36.31                | 35.41                 | 102.8            |
| Col.        | R-19  | 50.0035      | 2.5012 (b)      | 6.4766           | 0.3395      | 0.98      | 0.8805            | 35.25                | 33.57                 | 84.2*            |
| Col.        | R-10  | 59.2788      | 10.4065 (c)     | 11.2351          | 0.5032      | 1.80      | 0.8901            | 51.03                | 43.38                 | 95.5             |
| Col.        | R-20  | 59.9972      | 2.9976 (c)      | 8.1951           | 0.5788      | 1.85      | 0.8885            | 36.84                | 35.09                 | 102.8            |
| Rubber Tire | R-34  | 0.0          | 27.6987         | 11.8985          | 1.1111      | 2.90      | 0.8989            | 29.52                | 114.53                | 101.9            |
| Col.        | R-12  | 82.9396      | 3.3401 (d)      | 9.0153           | 1.4794      | 2.70      | 0.8823            | 28.38                | 28.38                 | 99.4             |
| Col.        | R-15  | 66.5000      | 3.5000 (e)      | 7.7575           | 0.8794      | 2.60      | 0.8986            | 31.11                | 29.55                 | 106.1            |
| Col.        | R-25  | 59.9915      | 3.0001 (e)      | 6.9125           | 1.1456      | 2.70      | 0.8989            | 30.72                | 29.26                 | 103.7            |
| Col.        | R-27  | 42.4876      | 7.4938 (e)      | 5.8041           | 2.5473      | 3.30      | 0.8932            | 36.65                | 31.16                 | 101.1            |
| Col.        | R-28  | 21.0014      | 8.9984 (e)      | 4.0616           | 2.5994      | 2.40      | 0.9122            | 50.81                | 35.57                 | 101.3            |
| Col.        | R-32  | 56.9897      | 2.9984 (f)      | 9.5334           | 0.1404      | 1.90      | 0.8588            | 46.68                | 44.35                 | 101.3            |
| Col.        | R-33  | 56.9951      | 3.0034 (g)      | 6.3517           | 0.7442      | 2.70      | 0.8903            | 29.99                | 28.50                 | 100.8            |
| Ala.        | R-7   | 79.8119      | 0.0             | 3.1967           | 2.7415      | 2.40      | 0.9081            | 10.57                | 10.57                 | 92.4             |
| Ala.        | R-13  | 90.7982      | 0.0             | 3.7119           | 2.5651      | 2.00      | 0.9003            | 10.85                | 10.85                 | 94.5             |
| Ala.        | R-14  | 87.5517      | 0.0             | 3.1618           | 2.6891      | 2.40      | 0.9307            | 9.13                 | 9.13                  | 99.3             |
| Ala.        | R-11  | 70.0000      | 3.5000 (a)      | 5.8712           | 1.8527      | 2.60      | 0.8270            | 25.58                | 24.31                 | 95.2             |
| Ala.        | R-9   | 49.8384      | 2.6000 (b)      | 3.5829           | 2.0308      | 1.40      | 0.8975            | 19.20                | 18.24                 | 94.6             |
| Ala.        | R-16  | 59.2780      | 10.4605 (c)     | 8.3783           | 0.8354      | 2.50      | 0.9240            | 36.66                | 31.16                 | 96.0             |
| Ala.        | R-29  | 56.9969      | 2.9990 (e)      | 2.6667           | 3.3498      | 3.40      | 0.9279            | 12.08                | 11.48                 | 104.5            |
| Ala.        | R-30  | 35.0009      | 15.0003 (e)     | 5.5319           | 5.0400      | 3.10      | 0.9360            | 40.47                | 28.33                 | 93.1             |
| Col.        | R-13  | 8.9985       | 2.8782 (b)      | 6.3714           | 2.1683      | 1.50      | 0.9159            | 185.27               | 56.28                 | 95.2             |
| Ala.        | R-17  | 50.0000      | 2.5000 (a)      | 6.0583           | 1.1601      | 1.60      | 0.8877            | 36.35                | 29.74                 | 102.3            |

(a) Polyethylene (b) Polystyrene (c) SBR (rubber tire) (d) Corrugated cardboard (e) Sawdust  
 (f) Polyvinylchloride (PVC) (g) Polyethylene terephthalate (PET)

Table 3. Changes in Oil Yield Resulting from Retorting Oil Shale With Various Additives Based On Average Shale Assay (Colorado Shale, 28.76 gal/ton; Alabama Shale 10.18 gal./ton)

| Run # | Shale Type | Weight of Shale | Additive     | Weight of Additive | % Additive | Oil Yield (gal./ton) | % Change in Oil Yield |
|-------|------------|-----------------|--------------|--------------------|------------|----------------------|-----------------------|
| R-21  | Colorado   | 59.9926         | Polyethylene | 2.9988             | 5.0        | 43.46                | + 51.11               |
| R-5   | Colorado   | 59.3463         | Polyethylene | 8.8063             | 15.0       | 73.40                | + 155.11              |
| R-6   | Colorado   | 53.2714         | Polystyrene  | 1.3488             | 5.0        | 35.25                | + 26.25               |
| R-19  | Colorado   | 50.0035         | Polystyrene  | 2.5012             | 5.0        | 35.25                | + 22.56               |
| RE-4* | Colorado   | 60.5639*        | Polystyrene  | 6.0484*            | 9.0*       | 34.89*               | + 278.00*             |
| R-10  | Colorado   | 59.2788         | SBR          | 10.4605            | 15.0       | 51.03                | + 77.43               |
| R-20  | Colorado   | 59.9972         | SBR          | 2.9976             | 5.0        | 36.84                | + 28.10               |
| R-12  | Colorado   | 82.9396         | Cardboard    | 3.3401             | 4.0        | 29.52                | + 2.64                |
| R-15  | Colorado   | 66.5000         | Sawdust      | 3.5000             | 5.0        | 31.11                | + 8.10                |
| R-25  | Colorado   | 59.9915         | Sawdust      | 3.0001             | 5.0        | 30.72                | + 6.82                |
| R-27  | Colorado   | 42.4876         | Sawdust      | 7.4938             | 15.0       | 36.65                | + 27.43               |
| R-28  | Colorado   | 21.0014         | Sawdust      | 8.9984             | 30.0       | 50.81                | + 76.67               |
| R-32  | Colorado   | 56.9897         | PVC          | 2.9984             | 5.0        | 46.68                | + 62.31               |
| R-33  | Colorado   | 56.9951         | PET          | 3.0034             | 5.0        | 29.99                | + 4.28                |
| R-11  | Alabama    | 70.0000         | Polyethylene | 3.5000             | 5.0        | 25.58                | + 151.20              |
| R-9   | Alabama    | 49.8384         | Polystyrene  | 2.6000             | 5.0        | 19.20                | + 88.54               |
| R-16  | Alabama    | 59.2780         | SBR          | 10.4605            | 15.0       | 36.66                | + 260.00              |
| R-29  | Alabama    | 56.9969         | Sawdust      | 2.9990             | 5.0        | 12.08                | + 18.62               |
| R-30  | Alabama    | 35.0009         | Sawdust      | 15.0003            | 30.0       | 40.47                | + 297.41              |
| R-13  | Colorado   | 8.9985          | Polystyrene  | 2.8782             | 10.0       | 185.27               | + 544.20              |
|       |            |                 | SBR          | 8.8852             | 30.0       |                      |                       |
|       |            |                 | Sawdust      | 8.8587             | 30.0       |                      |                       |
| R-17  | Alabama    | 50.0000         | Polyethylene | 2.5000             | 4.5        | 36.35                | + 257.00              |
|       |            |                 | Polystyrene  | 2.5000             | 4.5        |                      |                       |

the assay oils. The pouring ability of the oils is drastically affected by some additives. Although the actual pour points of the oils were not determined, it is quite obvious that the polystyrene oils were quite fluid and had much lower pour points than other oils. The oil generated from shale and polyethylene is a tacky mass at room temperature whose density was difficult to determine at first. The oil produced from Colorado shale and PVC had characteristics very similar to those of polyethylene oils, in that it too was a very tacky semi-solid with a relatively high pour point.

Oils produced from SBR, sawdust, and PET seemed to be "normal" oils with physical and spectral characteristics very similar to those of the assay oils. The point should be emphasized that without further analysis of spent shale, we do not know if any additional oil is obtained from the shale itself. The oil yields are probably due mostly to the assay yield of oil being supplemented by the oil produced from the additive. It is interesting however that the additive usually seems to decompose completely to give supplementary oil as compared to the expected shale oil yield from assay results. Table 4 gives a comparison of expected oil yields versus actual oil yields (from table 2). Calculation of expected oil yields as based on the average oil assay from Colorado and Alabama shales.

Expected Oil Yield (E) = Expected Assay Yield + wt. of Additive  
(grams)

Expected Oil Yield (E) =  $\frac{(\text{Av. Yield}) (\text{Weight of Shale}) (\text{Density}) (4.1727) +}{(\text{grams}) \quad 1000}$

Wt. of Additive

Av. Yield — 28.76 gal./ton

Weight of Shale — 59.3463

Density — 0.8371

4.1727 — Conversion factor metric to English units.

A sample calculation is given below for R-5 (table 2)

$$\text{Expected Assay Oil Yield (E.A.Y.)} = \frac{(28.76) (59.3463) (0.8371) (4.1727)}{1000}$$

$$\text{E.A.Y.} = 5.9617 \text{ grams.}$$

Weight of additive plus EAY = Total Predicted Oil Yield, E

$$5.9617 + 8.8063 = 14.7680 \text{ g oil.} = E$$

$$\text{Actual Oil Produced} \text{ — } 15.2162 \text{ g.} = A$$

$$\% \text{ Difference} = \frac{A - E}{E} \times 100$$

$$= \frac{15.2162 - 14.7680}{14.7680} \times 100$$

$$= + 3.03 \%$$

Note that this treatment assumes that (1) the assay value remains constant with change in sample size and (2) all of the additive decomposes to give oil. Assumption (1) is probably closer to being accurate than is (2). In an earlier run involving only polystyrene it was found that at least 16% of the polymer decomposes to give gaseous product. Studies on the sealed tube thermal decomposition of polystyrene cite figures as high as 42% monomer being formed. Further study is needed to determine exact effects on oil yield and to determine if intermingling of waste and kerogen fragments occurs in the oil formation process.

Table 4. Comparison of Expected Oil Yields\* and Actual Oil Yields for Shales Plus Additives.

| Run# | Shale Type | Wt. of Shale | Additive     | Wt. of Additive | Expected (E) Oil Yield (gms.) | Actual (A) Oil Yield (gms.) | % difference (A-E)/E |
|------|------------|--------------|--------------|-----------------|-------------------------------|-----------------------------|----------------------|
| R-5' | Colorado   | 59.3463      | polyethylene | 8.8063          | 14.7680                       | 15.2162                     | + 3.03               |
| R-6  | Colorado   | 53.2714      | polystyrene  | 1.3488          | 7.0820                        | 7.2375                      | + 2.20               |
| R-20 | Colorado   | 59.9972      | SBR          | 2.9976          | 9.3949                        | 8.1951                      | - 12.77              |
| R-12 | Colorado   | 82.9396      | cardboard    | 3.3401          | 12.1219                       | 9.0153                      | - 25.62              |
| R-25 | Colorado   | 59.9915      | sawdust      | 3.0001          | 9.4716                        | 6.9125                      | - 27.02              |
| R-32 | Colorado   | 56.9897      | PVC          | 2.9984          | 8.8715                        | 9.5334                      | + 7.46               |
| R-33 | Colorado   | 56.9951      | PEI          | 3.0034          | 9.0925                        | 6.3517                      | - 30.14              |
| RE-2 | Colorado   | 77.4895      | polystyrene  | 2.8331          | 9.1636                        | 8.9757                      | - 2.09**             |
| RE-4 | Colorado   | 60.5639      | polystyrene  | 2.1093          | 8.1577                        | 7.9738                      | - 2.31***            |
| R-11 | Alabama    | 70.0000      | polyethylene | 3.500           | 5.951                         | 5.8712                      | - 1.47               |
| R-9  | Alabama    | 49.8384      | polystyrene  | 2.600           | 4.5000                        | 3.5829                      | - 20.38              |
| R-16 | Alabama    | 59.2780      | SBR          | 10.4605         | 12.7871                       | 8.3783                      | - 34.48              |
| R-29 | Alabama    | 56.9969      | sawdust      | 2.9990          | 5.2455                        | 2.6667                      | - 49.16              |

\*Calculated based on average yields of Colorado and Alabama Shale

\*\*Based on Colorado Shale assayed at 21.66 gal./ton.

\*\*\*Based on Colorado Shale assayed at 9.23 gal./ton.

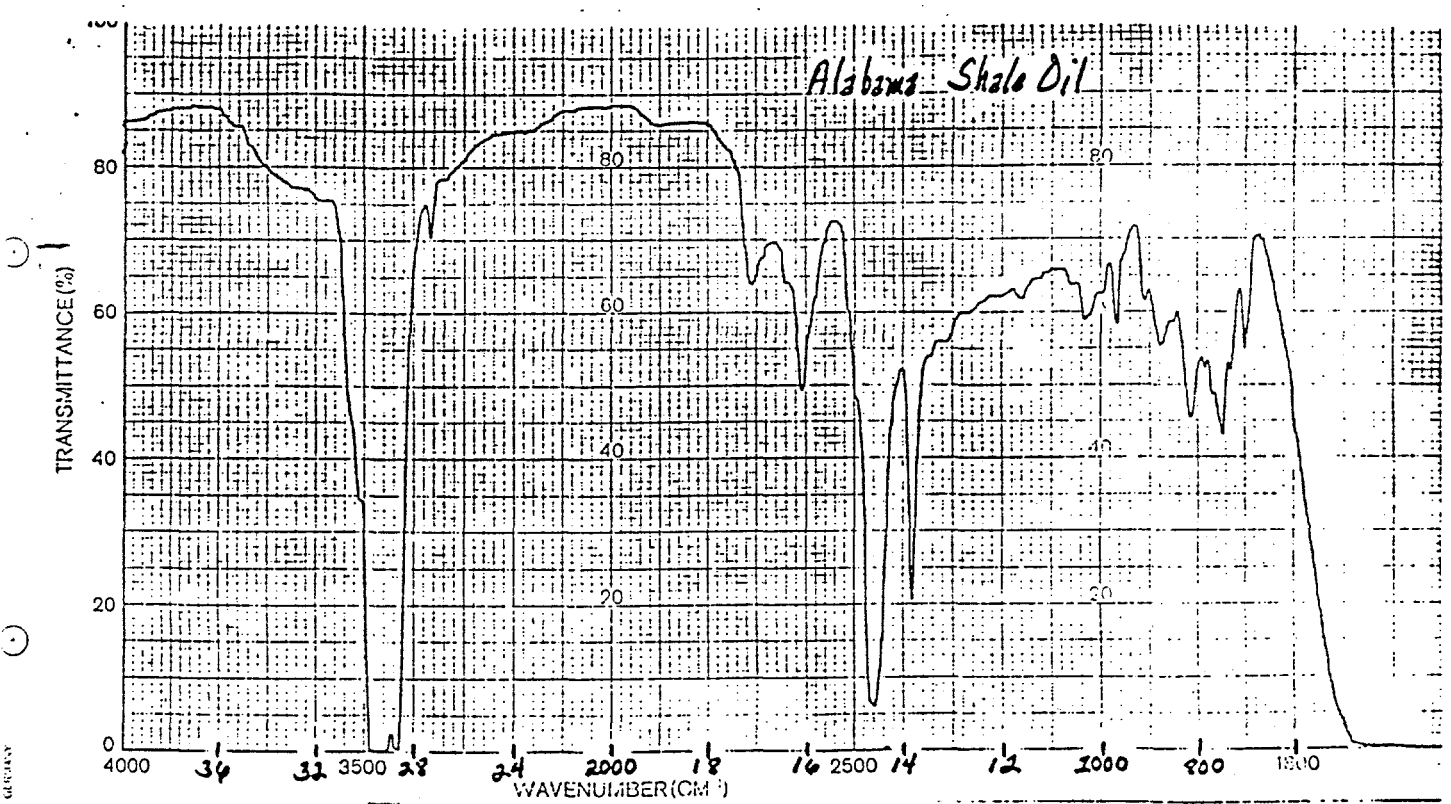
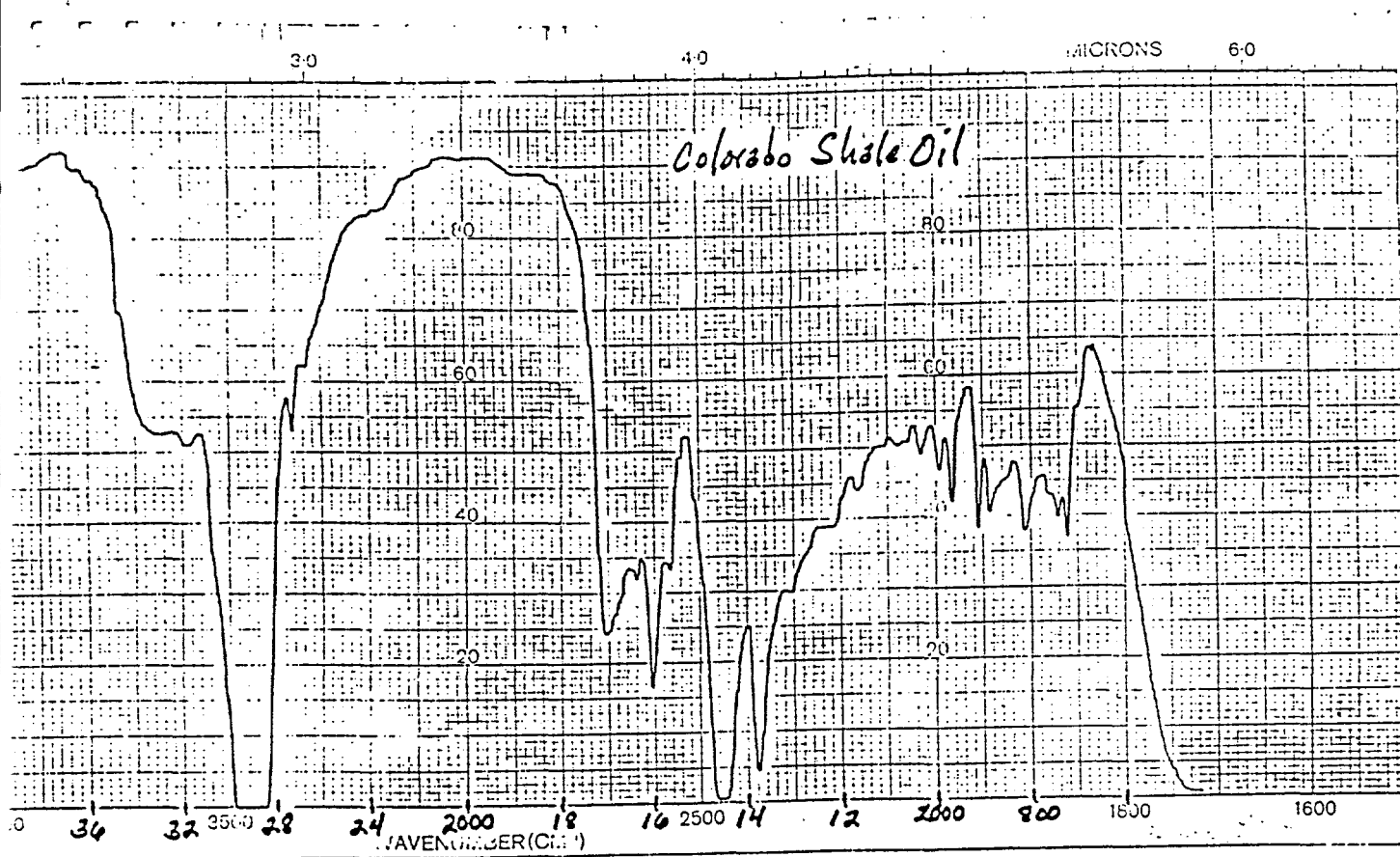


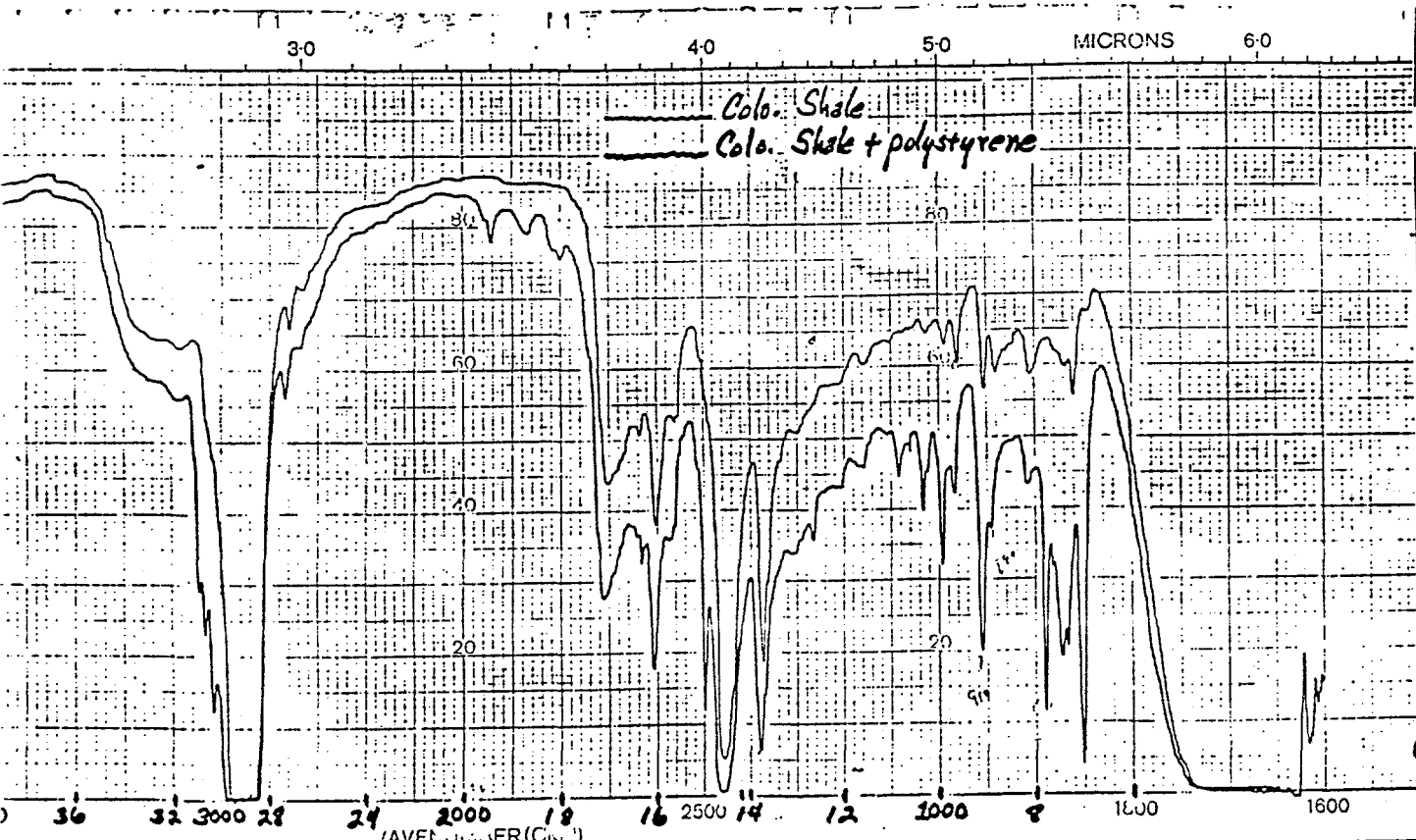
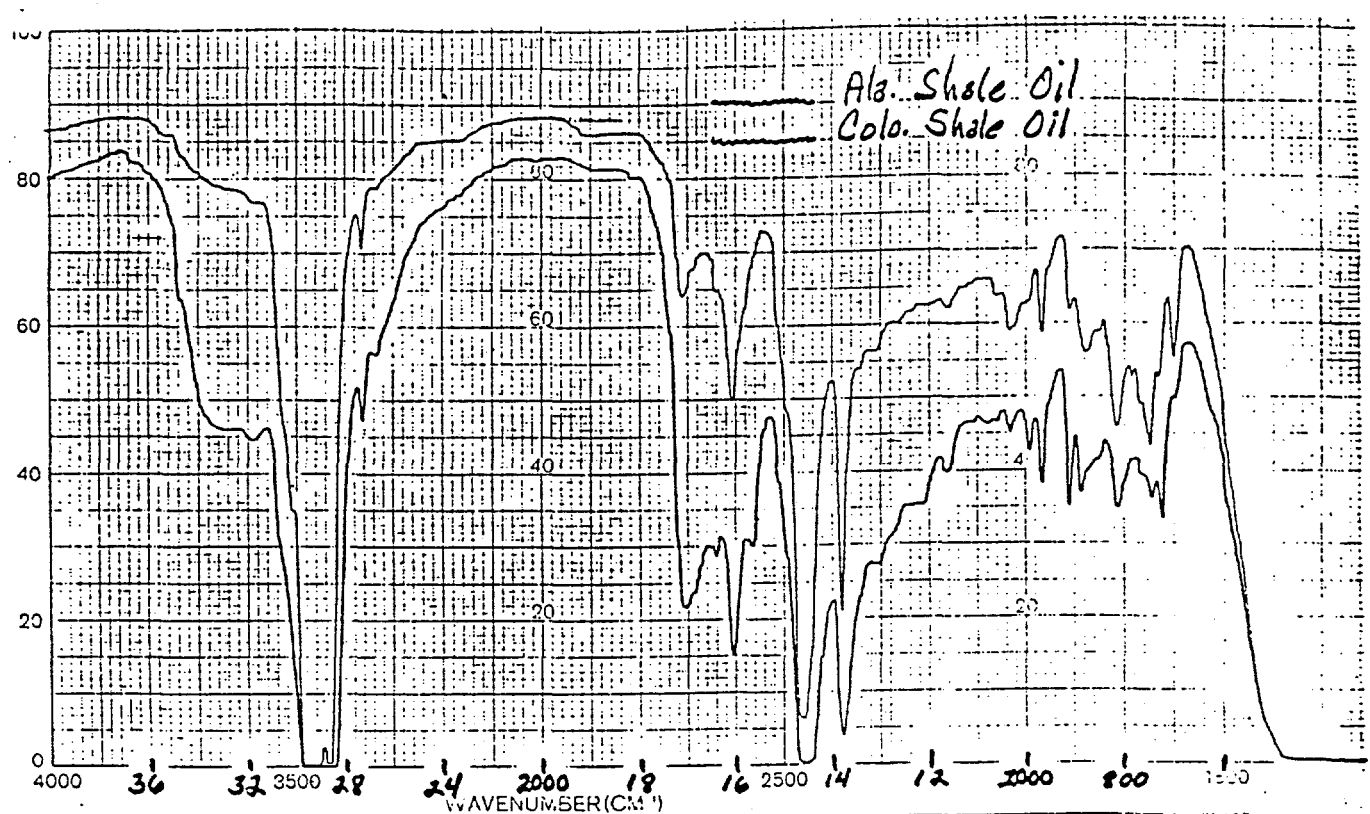
## INFRARED SPECTRA OF SELECTED OILS

All infrared spectra were run using a Perkin Elmer Model 599B Infrared Spectrophotometer and were run between plates.

### LEGEND

|            |                                                                                                                                            |
|------------|--------------------------------------------------------------------------------------------------------------------------------------------|
| Figure IR1 | Colorado Shale Oil between plates                                                                                                          |
| Figure IR2 | Alabama Shale Oil between plates                                                                                                           |
| Figure IR3 | Superimposed Spectra: top spectrum, Alabama Shale Oil; bottom spectrum, Colorado Shale Oil.                                                |
| Figure IR4 | Superimposed Spectra: top spectrum, Colorado shale oil; bottom spectrum, resultant oil from retorting Colorado oil shale with polystyrene. |
| Figure IR5 | Superimposed Spectra: top spectrum, resultant oil from retorting Alabama oil shale with polystyrene; bottom spectrum, Alabama shale oil.   |
| Figure IR6 | Resultant oil from retorting Alabama oil shale with polystyrene showing identification of bands due to aromatic and C=C absorptions.       |
| Figure IR7 | Resultant oil from retorting Alabama oil shale with polyethylene.                                                                          |
| Figure IR8 | Resultant oil from retorting Colorado oil shale with SBR rubber (rubber tire donated by Dunlop Tire Company, Huntsville, Alabama).         |



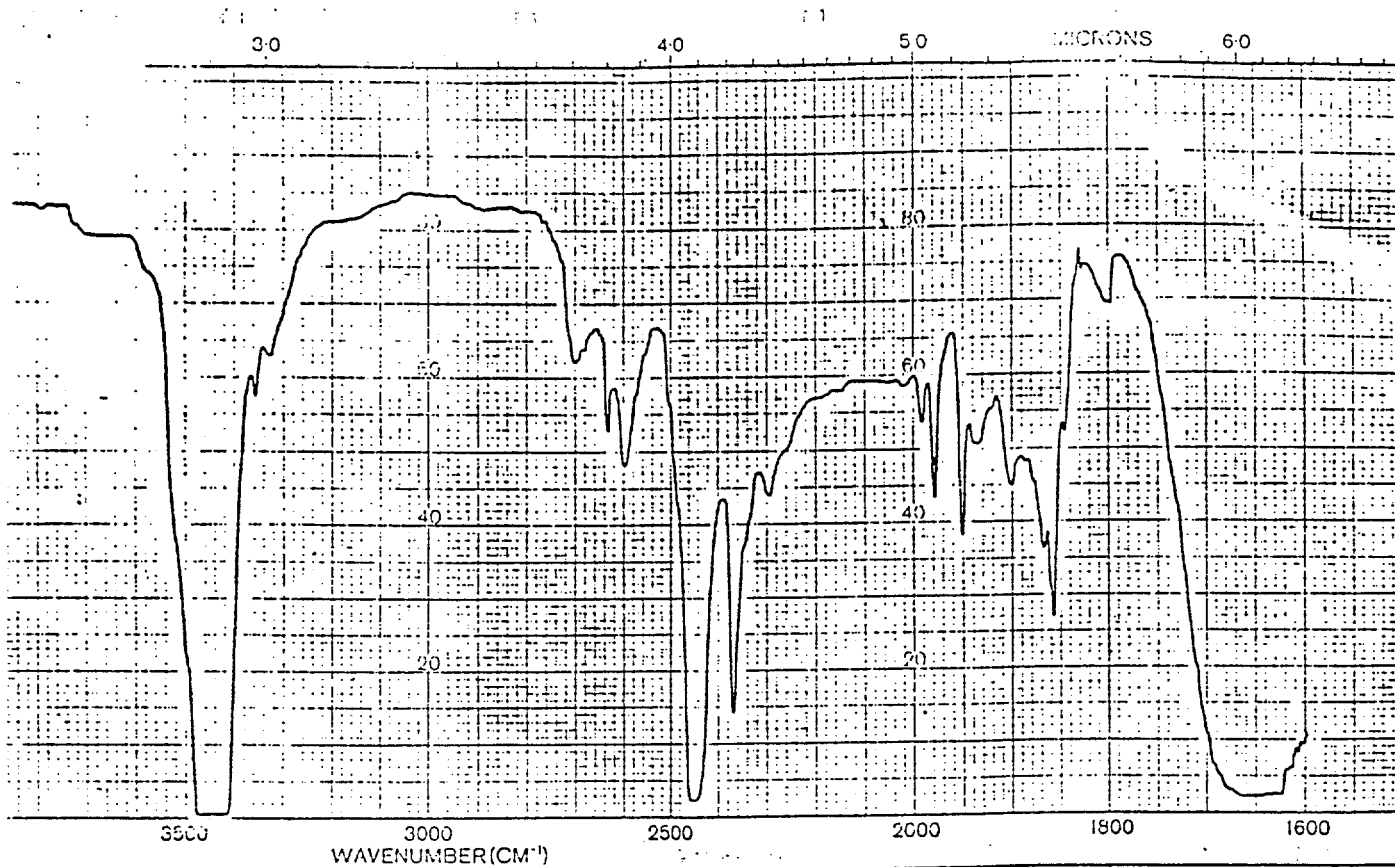


NOTE (1) Colorado Shale  
(2) Colo. Shale + 2.479 polystyrene

SOLVENT \_\_\_\_\_  
CONCENTRATION \_\_\_\_\_

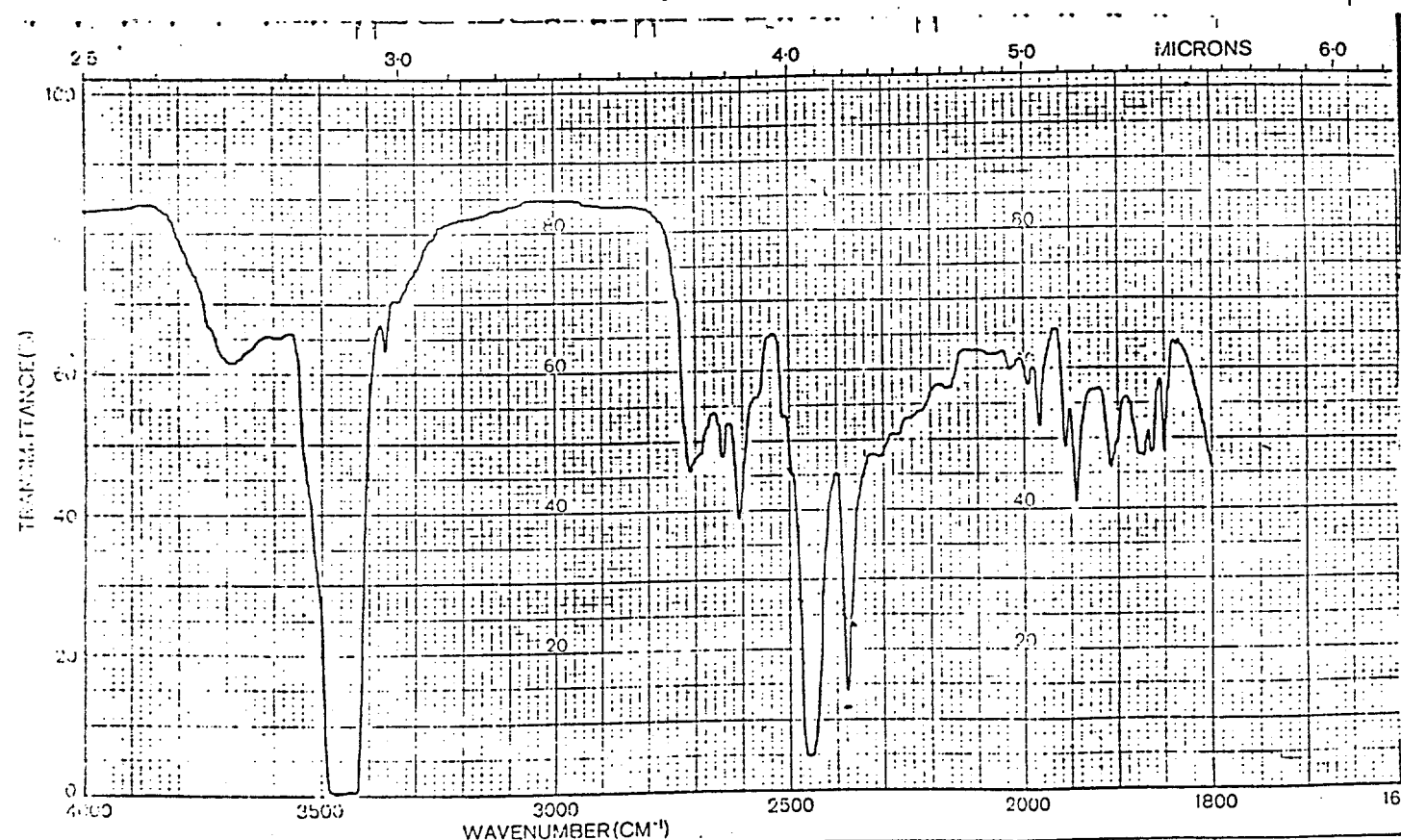
REMARKS \_\_\_\_\_





R11-Ah-82  
 4/30/81

|                                                                                                   |                                                                        |
|---------------------------------------------------------------------------------------------------|------------------------------------------------------------------------|
| SOLVENT... <u>none</u><br>CONCENTRATION... <u>neat</u><br>CELL PATH...<br>REFERENCE... <u>0.5</u> | REMARKS (min)<br><u>Alk. Shale + P. Ethylene</u><br><u>BT - 5/1/81</u> |
|---------------------------------------------------------------------------------------------------|------------------------------------------------------------------------|



Colored Shale + Rubber Tire

|                                                |         |
|------------------------------------------------|---------|
| SOLVENT...<br>CONCENTRATION...<br>CELL PATH... | REMARKS |
|------------------------------------------------|---------|

# GAS CHROMATOGRAMS OF SELECTED OILS

All chromatograms were run on a Perkin Elmer Sigma 3 Gas Chromatograph at the conditions below.

|                    |                                                                                         |
|--------------------|-----------------------------------------------------------------------------------------|
| Column Temperature | Programmed between ambient and 250° Centigrade. Temperature held at 250 for 30 minutes. |
| Column             | 10% SP 2100 (Silicone Oil) on 100/120 Supelcoport, 10' x 1/8" stainless steel.          |
| Detector           | Dual flame F.I.D. at 250° C.                                                            |
| Flow rate          | Adjusted to 25 p.s.i. on console.                                                       |

Data was taken using a Perkin Elmer Sigma 10B Data Station.

|           | LEGEND                                             | Run #(Table 2) |
|-----------|----------------------------------------------------|----------------|
| Figure C1 | Colorado shale oil (Assay 29.63 gal./ton)          | R-1            |
| Figure C2 | Colorado oil shale + 5%polystyrene.                | R-6            |
| Figure C3 | Colorado oil shale + 5% corrugated cardboard       | R-12           |
| Figure C4 | Colorado oil shale + 5% polyethylene terephthalate | R-33           |
| Figure C5 | Colorado oil shale + 5% sawdust                    | R-28           |
| Figure C6 | Colorado oil shale + 5% rubber tire                | R-10           |
| Figure C7 | Alabama shale oil (Assay 10.57 gal./ton)           | R-7            |
| Figure C8 | Alabama oil shale + 5% rubber tire                 | R-16           |

SENSITIVITIES 50 4

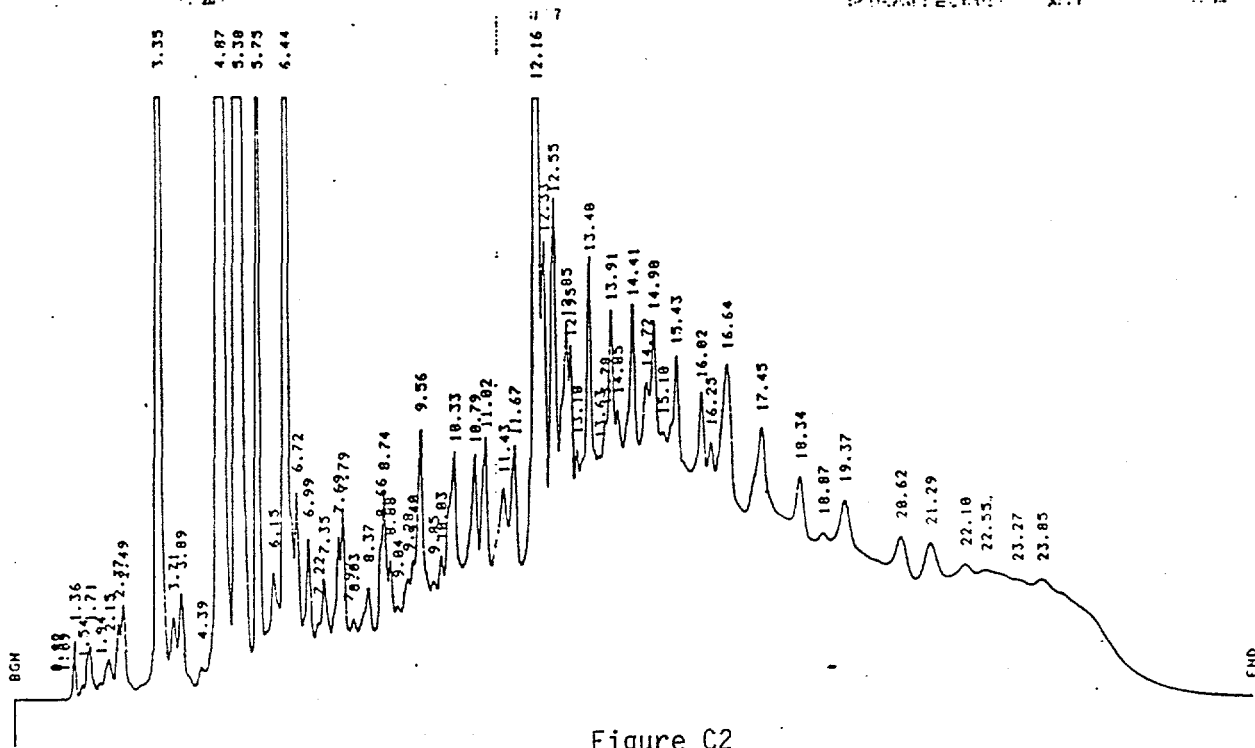


Figure C2

INST 1 METH 1 FILE 26

PJN 2 BULK SHALE OIL 9 : 2.2 0 / 17 / 0

SENSITIVITIES 50 4

TIME AREA BC RRT RF C NAME

BGH

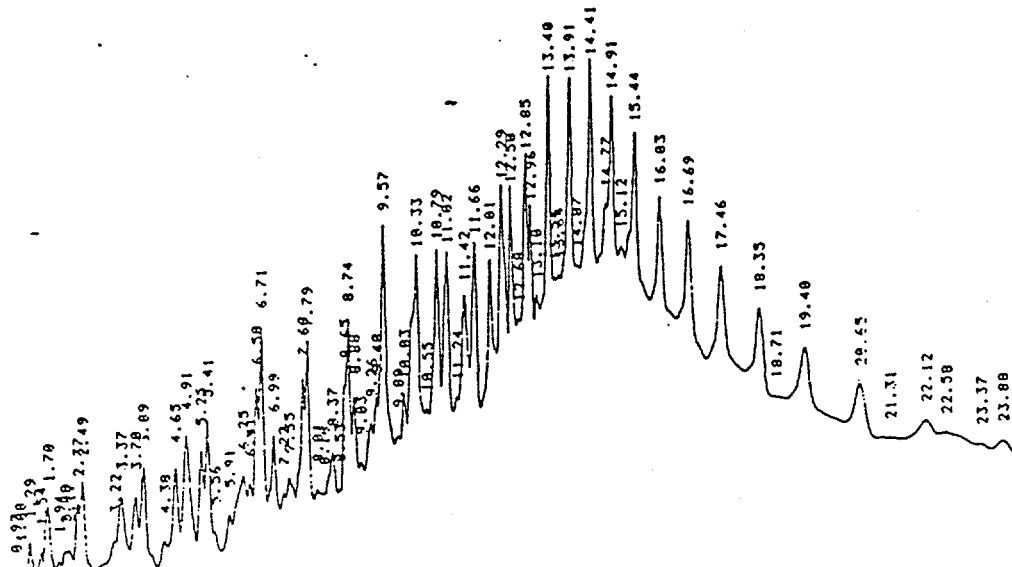


Figure C1

DUN 3 BULK SHALE OPIL + 1/16 Cnd band Cnle.  
 SENSITIVITIES 50 4 1.0  $\mu$ l

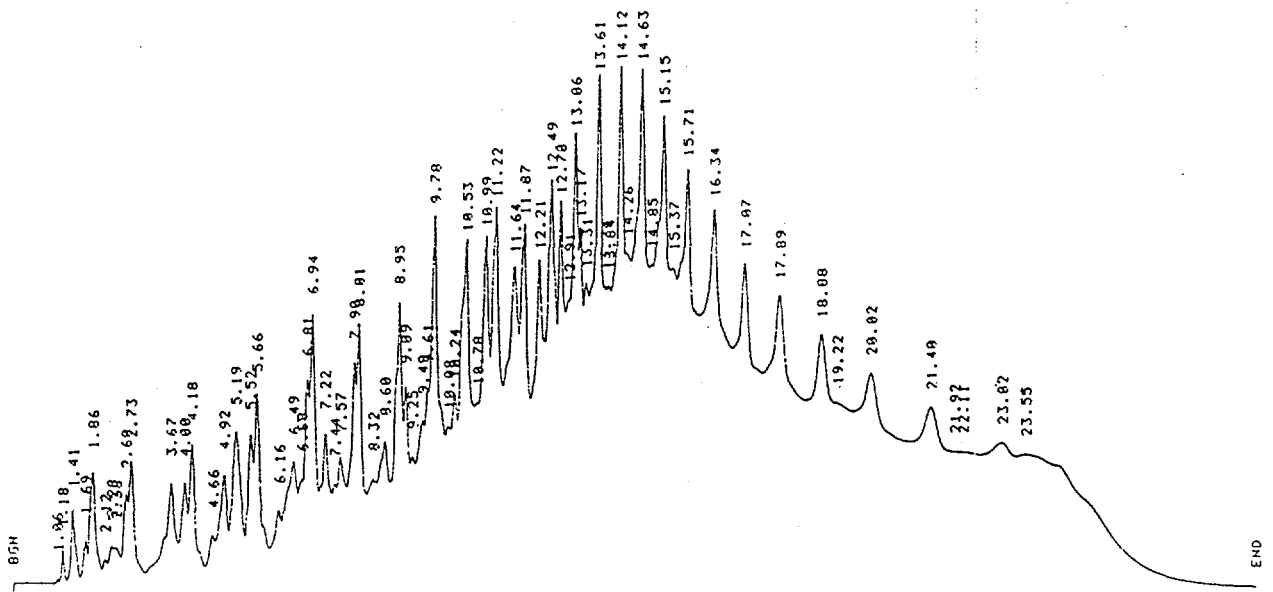


Figure C3

INST 1 METH 1 FILE 22

SENSITIVITIES 50 4 1.0  $\mu$ l

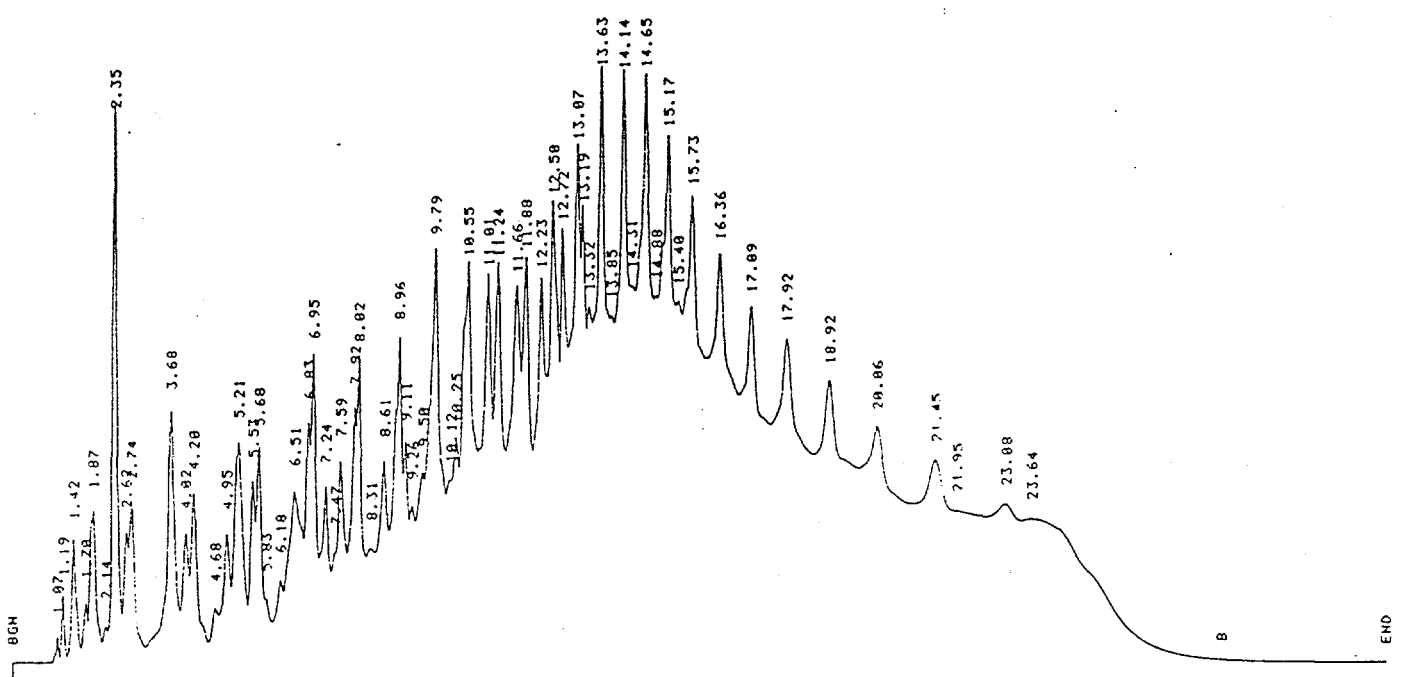


Figure C4

INST 1 METH 1 FILE 21



PIN 1 BULK SHALE OPTIL & Syndet-80 Chardo  
 SENSITIVITIES 50 4 1.046

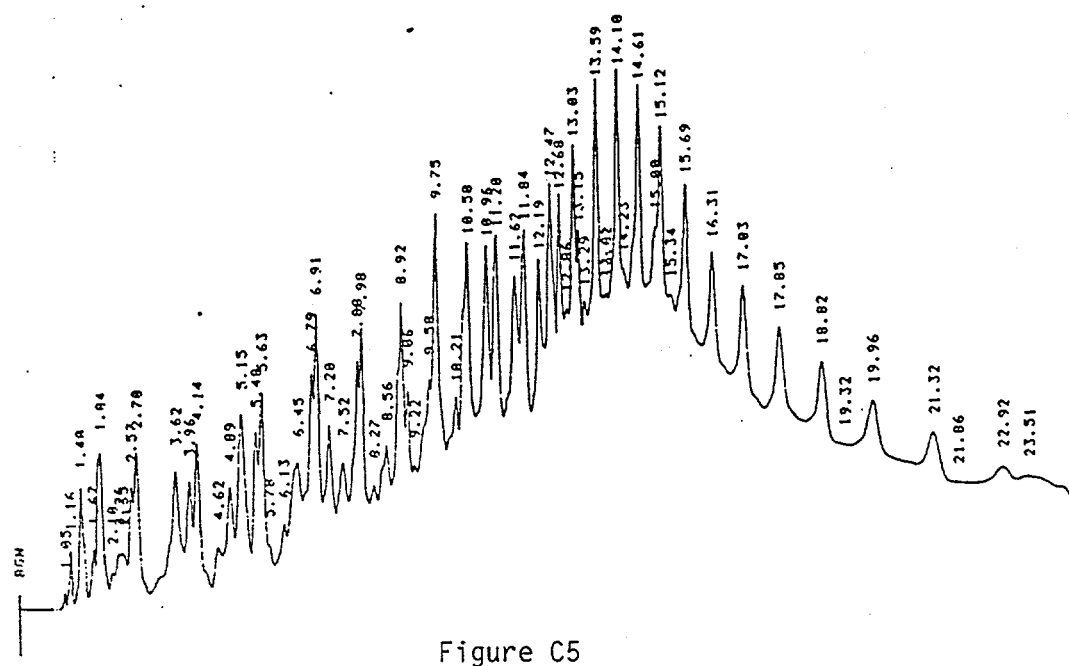


Figure C5

PIN 2 BULK SHALE OPTIL & Syndet-80 Chardo  
 SENSITIVITIES 50 4 1.046

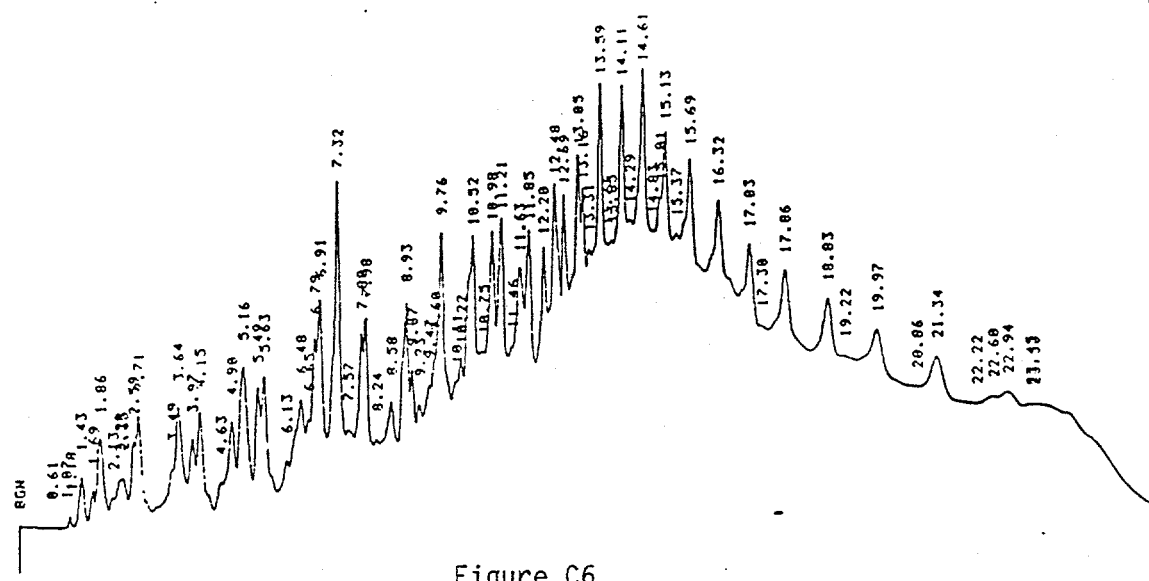
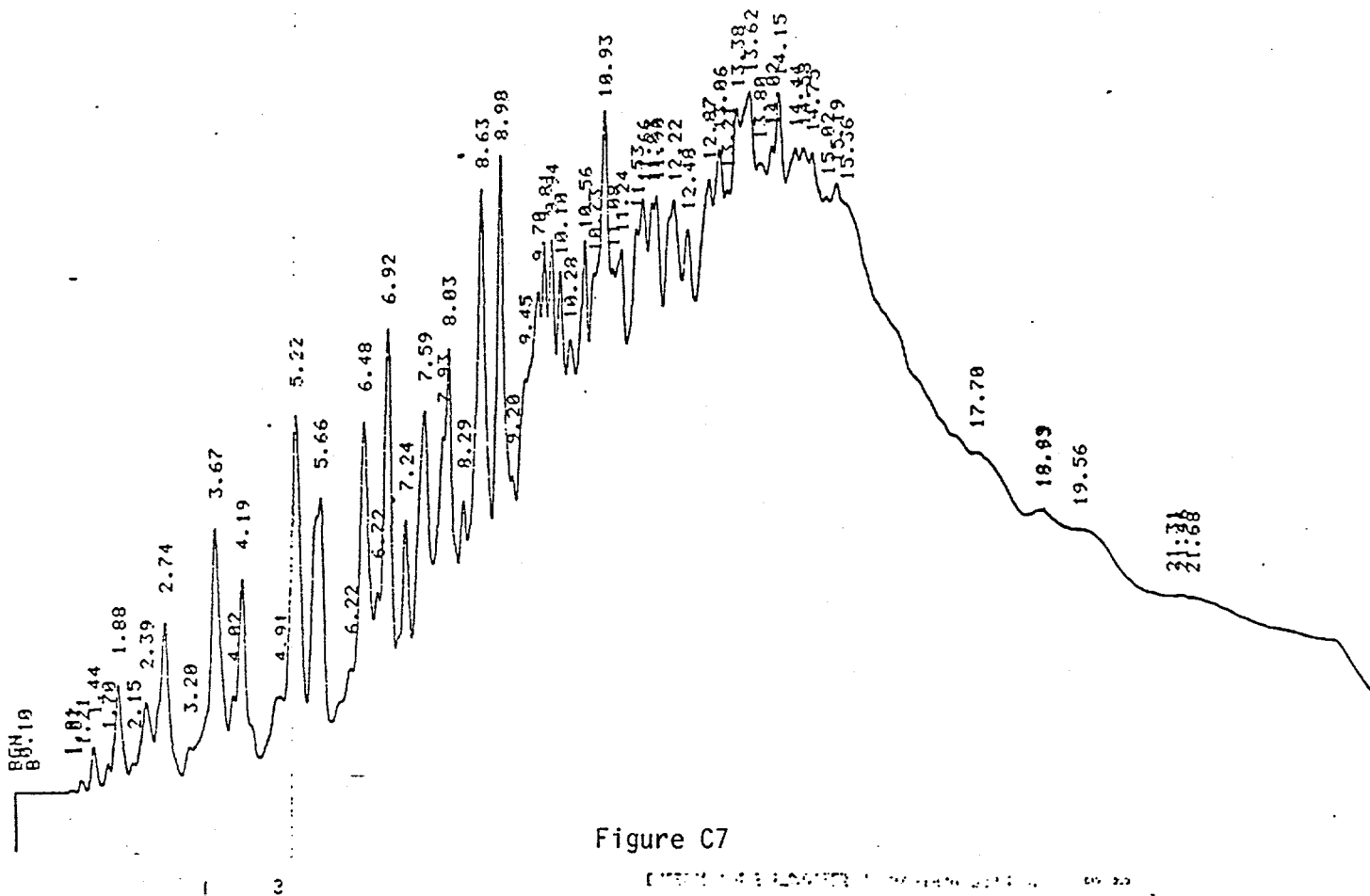


Figure C6



News Articles, Shale Oil Project

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processing.

Publications Pertaining to Shale Oil Project

*Removed for separate  
processing.*

Vita, Principal Investigator

2/3/89  
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Presently professor and chairman of a chemistry department consisting of ten other full-time staff including one laboratory supervisor. Administrative duties include curriculum and budgetary responsibilities, scheduling of courses, grant administration, teaching and research.

#### ACADEMIC EXPERIENCE

Instructor of chemistry, Morgan State College, Baltimore, Maryland, 1962-1963. General and organic chemistry.

Instructor of chemistry, Alabama A.& M. College, Normal, Alabama, 1963-1965. General and analytical chemistry.

Assistant Professor of Chemistry, Alabama A.& M. College, 1965-1968. General, analytical and organic chemistry.

Associate Professor and Chairman, Department of Chemistry, Alabama A.& M. University, 1971-72. General and analytical chemistry.

Professor and Chairman, Department of Chemistry, Alabama A.& M. University, 1972-present. General, Analytical and Organic Chemistry.

#### INDUSTRIAL EXPERIENCE

Upjohn Pharmaceutical Company, Kalamazoo, Michigan, Summer, 1961. Duties: upgrading and testing gas-liquid chromatographic equipment.

All-Tile Chemical Co., Kalamazoo, Michigan. Duties: assisting with the development and implementation of a new process for insulation of cold storage warehouses using a polyurethane foam procedure.

Kaiser Aluminum and Chemical Co., Baton Rouge, La., Summer, 1963. Duties: quality control analyses of plant effluent from the bauxite caustic extraction process used in the production of alumina.

Lawrence Livermore National Laboratory, Livermore, Ca., Summer, 1977. Duties: Infrared analyses of shale oil.

Lawrence Livermore National Laboratory, Livermore, Ca., Summer, 1979. Duties: construction and operation of automated laboratory scale apparatus for oil shale retorting.

Lawrence Livermore National Laboratory, Livermore, Ca., Summer, 1981-1982. Duties: HPLC analysis of shale oil constituents; mutagenic testing of compound fractions from shale oil.

Lawrence Livermore National Laboratory, Livermore, Ca., Summer, 1983. Duties: Participant, nuclear chemistry class.

Lawrence Livermore National Laboratory, Livermore, Ca., Summers, 1984-1987. Duties: assisting with development of analytical procedures associated with the Radionuclide Migration Project, Nuclear Chemistry Division.

Lawrence Livermore National Laboratory, Livermore, Ca., Summer, 1988. Duties: Fourier Transform Infrared Spectroscopy (FTIR): devising a transfer line to transport samples efficiently between a gas chromatograph, FTIR, and a mass spectrometer (GC-FTIR-Mass Spec). Analytical Chemistry division of Chemistry and Materials Science.

#### EDUCATION

Tougaloo College, Tougaloo, Mississippi, B.S., Chemistry, 1959.

Western Michigan University, Kalamazoo, Michigan, M.A., Chemistry, 1963.

Louisiana State University, Baton Rouge, La., Ph.D., Physical Organic Chemistry, 1971.

#### PROFESSIONAL ACTIVITIES

##### DEPARTMENTAL

1. Academic Year Extension Grant (NSF), "Investigation of the Reactions of Medium Ring Compounds", 1966-1968.

2. Director, NSF COSIP-D Grant, "Novel Rearrangement Reactions of Medium Ring Compounds", 1972-1974.

3. Director, NSF Instructional Scientific Equipment Grant, 1973-1975.

4. Director, NSF MISIP Grant, an interdisciplinary science improvement program, 1974-1976.

5. Project Investigator, MSBS Natural Pesticide Project, NIH, 1976-1978.

6. Principal Investigator, Department of Energy Project, "An Investigation of Selected Disposables on Powdered Oil Shale in a Laboratory Retort", 1979-80; 1980-81.

##### INSTITUTIONAL

Chairman of committee which developed the initial Advanced Institutional Development Program (AIDP) proposal for Alabama A.& M. University, 1974. This program was funded for \$3,000,000.00 over a five-year period, one of the largest grants to any institution to date.

Coordinator, Alabama A.& M. University/Lawrence Livermore National Laboratory cooperative program, 1984-present. This program involves the departments of Computer Science, Chemistry, Physics and the Learning Resources Center.

##### COMMUNITY AND OTHER

Service on six Southern Association of Colleges and Schools visiting committees: Lubbock Christian College, 1973; Vorhees College, 1973; Texas A.& I. University, 1974; Northwest Louisiana State University, 1975; Fort Valley State College, 1979; George Mason University, 1981. Responsibilities have been evaluation of the sciences, faculty and research.

Consultant, Educational Opportunity Center Summer Science workshop for high school teachers. "Inexpensive Substitutions for the Laboratory".

#### PAPERS PRESENTED AND PUBLICATIONS

1. "The Acetolysis of cis and trans-2-methoxycyclooctyl tosylates, R.A. Evans and J.G. Traynham, presented before the 45th annual meeting of the Louisiana Academy of Sciences, April 23, 1971.
2. "Solvolyses of cis and trans-2-methoxy-1-cyclooctyl p-toluenesulfonates; Effect of Neighboring Methoxy on Cyclooctyl Cation Reactions". James G. Traynham and Richard A. Evans, presented before the 167th American Chemical Society National Meeting, Los Angeles, California, April 1, 1974.
3. "Oil Shale Retorting: Correlation of Selected Infrared Absorbance Bands with Oil Yield". Final technical report, Lawrence Livermore Laboratory, Chemistry and Materials Science Division, Livermore, Ca., August, 1977.
4. "Evans, R.A. And Campbell, J.H., "Oil Shale Retorting: A Correlation of Selected Infrared Absorbance Bands with Process Heating Rates and Oil Yield", In Situ, 3 (1), 33-51, 1979.
5. Evans, R.A., Thomas, Brian and Fowler, Josephine, "An Investigation of the Effects of Selected Disposables on Oils Obtained from Powered Oil Shale in a Laboratory Retort", Proc. ACHE Science Symposium, (5) 32-37, 1980.
6. Evans, R.A. and Thomas, Brian, "Oil Yield and Mutagenicity Studies on Colorado and Alabama Shale Oils", presented before the 7th ACHE Science Symposium, Talladega College, November 6, 1983.
7. Evans, R.A. Parker, Khristal and Dickerson, Harold, Jr., "Polymer Film Characterization Via Infrared Analysis: An Undergraduate Organic Laboratory Exercise", presented before the 7th ACHE Science Symposium, Talladega College, November 6, 1983.
8. Evans, R.A., and Fowler, Josephine R., "Sodium Induced Rearrangement of C8 Medium Ring Diols", presented before the 5th ACHE Science Symposium, Miles College, November 20, 1980.
9. Silva, R.J., Evans, R., Rego, J.H. and Buddemeier, R.W., "Methods and Results of Tc99 Analysis of Nevada Test Site Groundwaters", UCRL Preprint 96399, Lawrence Livermore National Laboratory, March 30, 1987.
10. Silva, R.J., Evans, R., Rego, J.H. and Buddemeier, R.W., "Methods



and Results of Tc99 Analysis of Nevada Test Site Groundwaters", Journal of Radioanalytical and Nuclear Chemistry, Vol 124, No. 2, (1988) 397 - 405.

#### CURRENT RESEARCH

1. Reaction of cis and trans-1,2-cyclooctane diols with sodium; a novel rearrangement of a medium ring diol.
2. Reaction of trans-2-methoxycyclooctanol with thionyl chloride; a product distribution study to determine extent of transannular reaction.
3. Investigation of the effect of various disposable or waste materials when mixed with powdered oil shale under retorting conditions. This work partially supported through an equipment loan from Lawrence Livermore National Laboratory and by the Department of Energy.
4. Evaluation of anion resin batch # and equivalency in the quantitative separation of Tc99 from solution as pertechnetate anion.