

Maintenance and Operation of the U.S. DOE Alternative Fuel Center

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1617 Cole Boulevard
Golden, Colorado 80401-3393
A national laboratory of the U.S. Department of Energy
Managed by the Midwest Research Institute
for the U.S. Department of Energy
Under Contract No. DE-AC36-83CH10093

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Executive Summary

Program Title: Maintenance and Operation of the U.S. DOE Alternative Fuel Center

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J. Erwin, "Vapor Pressure Interactions of Ethanol with Butane and Pentane in Gasoline", Symposium on Oxygenates as Fuel Additives, American Chemical Society (ACS) National Meeting, San Diego, Calif., March 1994.

Technical Objectives

Five tasks were defined for work in the Alternative Fuels Utilization Program to enhance the quality of alternative fuels and improve the utility and value of U.S. energy sources:

- Task 1 – Facility maintenance for the Alternative Fuel Center (AFC) of the Office of Energy Efficiency and Renewable Energy at Southwest Research Institute
- Task 2 – Production of two test fuels, including a) preparation of low-sulfur, low-olefin catalytically-cracked gasoline blendstock, and b) low-emission gasoline
- Task 3 – Other Government Research
- Task 4 – Industry Research (on a noninterference basis)
- Task 5 – Safety and Health Compliance.

Approach

A timeline was established to coordinate the uses of the hydrogenation pilot plant of the AFC among Task 2 project work, other government work, and work for industry. Consistent with assisting the AFUP in accomplishing its general goals, the work was done with all fuel producers, regulators, and users in mind. AFC capabilities and results were disseminated whenever possible.

Accomplishments

Hydrotreater maintenance was achieved through selected repairs on project and diligent upkeep on outside projects. The equipment was used for preparation of low-sulfur, low-olefin cracked gasoline blendstock, which was further investigated through economic analysis *via* linear programming. This material proved to be cost effective for meeting potentially lower limits on sulfur content of gasoline.

Linear programming was also used to devise a "minimum emissions" gasoline from hydrocarbon sources which could be produced from alternative or conventional blendstocks. This formulation showed half the reactivity of the AQIRP Test Fuel A.

Work for other government programs and industry is shown below. In addition to the new alternative fuels knowledge produced this year by the AFC project, use of the AFC by other government agencies and industries for outside projects has contributed to better fuels and alternative fuel sources. This testifies to the widespread interest and value of both the AFUP and the AFC.

Table E-1. Utilization of the AFC by Industries and Government Agencies

User or Fuel Recipient	AFC Activity*	Type of Fuel**	General Objectives
US EPA	B	D	Develop an emissions-reducing component
US EPA	B	G	RVP study
NREL	D,B	G	RVP study
DOE Fossil Energy Division	D	D	Ignition quality, Fischer-Tropsch fuels
Oil Company	B	D	Fuel producing reduced emissions
Industrial Association	B	D	Fuel partially derived from biomass
Oil Company	H,D,B	D	Fuel producing reduced emissions
DOE Fuels & Chemicals Research Division	H,D	D	Fundamental data on emissions
Oil Company	H,B	D	Fuel producing reduced emissions
Oil Company	B	G	Additive testing

* Type of activity: H hydrotreating, D distillation, B blending.

** Type of fuel: D diesel, G gasoline.

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Introduction

In 1982, the Department of Energy (DOE) and Southwest Research Institute (SwRI) jointly established what is now called the Alternative Fuel Center (AFC) to provide drum quantities of finished transportation fuels from a variety of sources. Since 1978 the Alternative Fuels Utilization Program (AFUP) of the Office of Energy Efficiency and Renewable Energy has investigated the possibilities and limitations of expanded and replacement transportation fuels from alternative sources to complement conventional petroleum fuels. DOE funded the design, construction, and installation of a hydrogenation pilot plant capable of performing a range of hydrotreating, reforming, and hydrocracking operations. DOE also provided storage for finished fuels and blend stocks in a series of tanks ranging in size from 500 to 10,000 gallons (1893 to 37,854 liters), and a piping system to facilitate delivery, transfer, blending, and shipping. SwRI provided the building, utilities, laboratory, and safety systems needed for the pilot plant. Later, the U.S. Navy provided a pilot scale continuous distillation unit, and SwRI provided batch distillation equipment, which are conveniently housed in the same building as the hydrotreater pilot plant, but are not formally part of the AFC.

Since then, overall objectives of the program have broadened somewhat. The present goal of the AFC is to enhance the quality of fuels and improve the utility and value of our energy alternatives. The emphasis is on gasoline and diesel transportation fuels, but in the past research extended to military specification fuels and emergency fuels and hydrocarbon products. SwRI has operated the AFC and associated facilities to provide custom-processed and specially blended fuels for both government and industry. The specially processed and blended fuels became a major resource in a wide variety of research efforts to improve fuels. From the beginning, the AFC has helped industry and other government agencies meet their research needs on the basis of non-interference with its primary DOE mission. Typical quantities of hydrotreated or distilled product ranges from 5 to 500 gallons (19 to 1900 liters). Engine test requirements frequently dictate the actual quantities produced. Custom blends made in the facility range from partial drum quantities to 9000 gallon (34200 liters) quantities.

This report covers the first year of the three-year contract. The principal objective was to assist the AFUP in accomplishing its general goals with two new fuel initiatives selected for tasks in the project year:

- 1) production of low-sulfur, low-olefin catalytically-cracked gasoline blendstock, and
- 2) production of low-reactivity/low-emission gasoline.

Supporting goals included maintaining equipment in good working order, performing reformulated gasoline tests, and meeting the needs of other government agencies and industries for fuel research involving custom processing, blending, or analysis of experimental fuels. This year's work is summarized by topic and provides a chronology. Monthly progress reports – indexed for reference in Exhibit 1 – provide that chronology.

Task 1 Facility Maintenance

The AFC is comprised of samples, structures, equipment, and storage infrastructure on a specially diked work area spread over about an acre at Southwest Research Institute. Drawing 1 shows its principal components. The laboratory houses the hydrogenation pilot plant, a fractionation pilot plant originally supplied by the U.S. Navy, a batch still, quality control laboratory, and such temporary setups as are needed, including filters, coalescers, packed columns, centrifuge equipment, and batch reactors. Descriptions of the facilities are given in the exhibits at the end of this report.

Located outside the laboratory building are the tanks and connected piping for storage and blending. The tankage includes:

- Two 10,000-gallon insulated and heated tanks
- Two 5,000-gallon covered tanks
- Three 1,000-gallon tanks
- Two 500-gallon tanks.

The piping includes various transfer pumps for moving and blending components. Tanks are instrumented for continuous temperature and level measurement, and can also be nitrogen blanketed when required. There is a bulk loading/unloading terminal for tanker truck shipments. Other tanks are used as needed and are close enough to be interconnected to the transfer manifold via flexible hose and fittings.

Additional facilities at the AFC include:

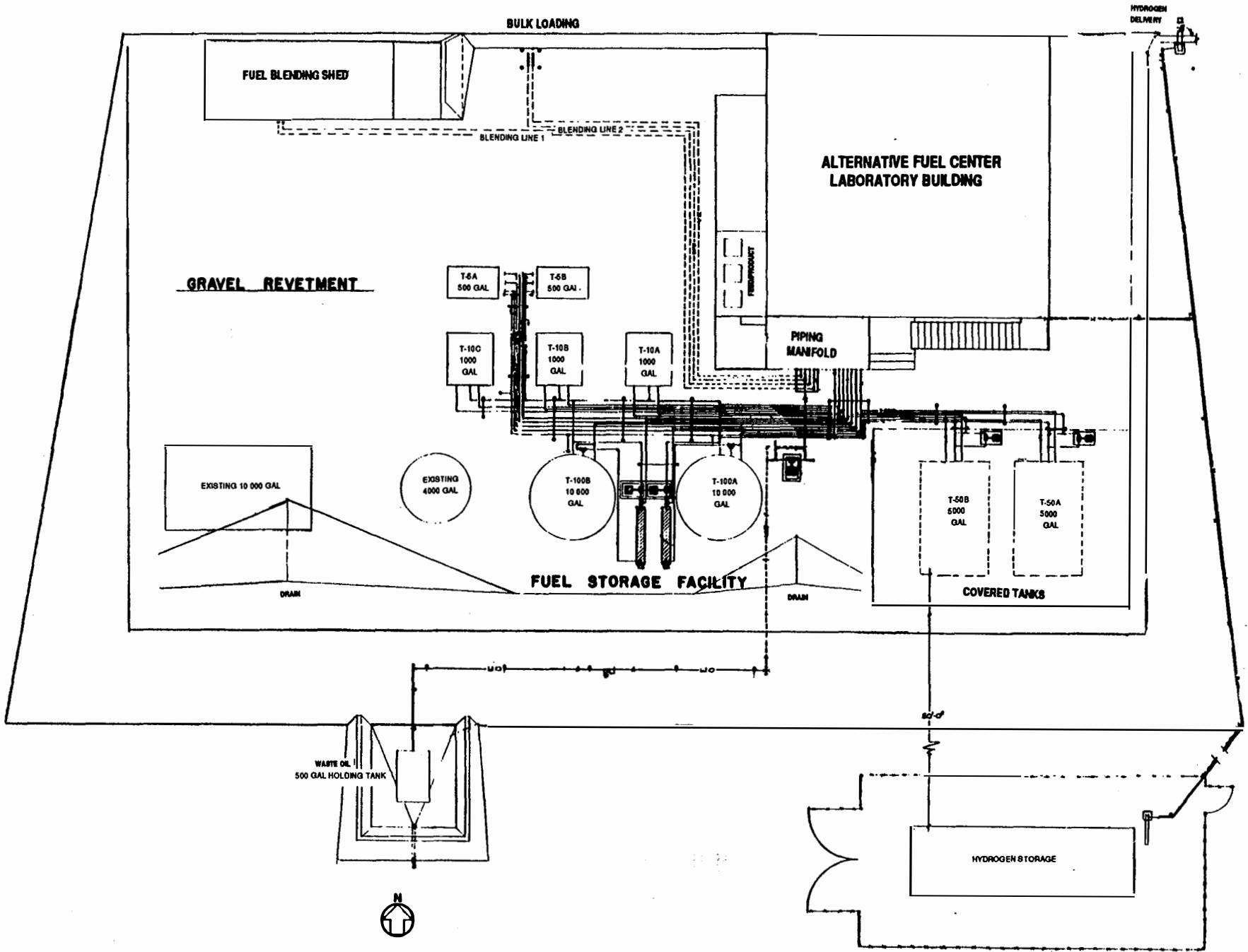
- Small sample storage
- Drum storage
- An outdoor blending shed
- Cold storage
- Waste sample disposal.

More intensive maintenance was required for the hydrotreater than for the building systems and other facilities. SwRI supplied sensors and alarms for the laboratory building; this safety equipment is an integral and necessary factor in hydrotreater operation. During routine checks of government equipment the project staff also examined building systems for proper function. Table 1 shows a summary of the findings during monthly inspections and exercises. The principal finding was a progressive failure of building safety alarm systems. All the sensors were recently sent to the manufacturer for evaluation, and SwRI plans a system renovation or replacement, depending on the results of the manufacturer's evaluation.

The largest AFC maintenance item, the hydrogen trailer (represented by Western Sales in Amarillo, Texas), was inspected and recertified in June 1993. Table 2 shows a summary of the hydrotreater maintenance log. Most repairs involved operational units, sensors, or instruments. The computer has been a continuing source of difficulties and processing shutdowns and needs to be upgraded. The hydrogen flowmeters are another source of continuing problems; they require either better protection from condensates and particulates, or replacement with less sensitive units.

Many in-house modifications continue to function and serve the hydrotreater well. An example is the added heat exchanger that follows the reactor.

3



Drawing 1. Alternative fuel center facilities

**Table 1. Summary of Synthetic Fuel Center Monthly Log
Items Checked and Comments**

Date	Fire and Smoke Alarms	Gas Alarms	Air and Water Systems ^a	Hydrogen and Lower Revetment ^b	Tanks ^c	Misc. ^d
9/92	✓	✓	✓	✓	✓	✓
10/92	✓	✓	✓	✓	✓	✓
11/92	✓	✓	✓	✓	✓	✓
12/92	1	2	✓	✓	✓	✓
1/93	1	2,3	✓	✓	✓	✓
2/93	1	2,3	✓	✓	✓	✓
3/93	1	2,3	✓	✓	✓	✓
4/93	1	2,3	✓	✓	✓	✓
5/93	1	2,3	✓	✓	✓	✓
6/93	1	2,3	✓	✓	✓	✓
7/93	1	2,3	✓	✓	✓	✓
8/93	1	2,3	✓	✓	✓	✓

- a. Includes all filters.
- b. Includes drum sample storage.
- c. Includes piping and blending facility.
- d. Includes emergency lights, oil traps and other upper revetment equipment.

Comments:

1. Main bay smoke detector not working.
2. A combustible gas detector failed.
3. Combustible gas detector not repairable. A new system is being considered as other detectors have a limited life.

Table 2. Hydrotreater Maintenance

Date	Type of Unit	Problem	Action
24 SEPT 92	Weigh scale	Inaccurate Reading	Calibration
29 SEPT 92	Manual valves	Leaking	Replaced stems and packings
16 OCT 92	Computer	Would not boot	Reseated IC's, cleaned board contacts, exercised jumpers
19 OCT 92	High pressure separator	Leaking	Changed seal ring
22 OCT 92	Column bottoms pump	Not working	Replaced pump, ordered spare
26 OCT 92	Pressure regulator	Leaking through	Cleaned lines, filter, and regulator
26 OCT 92	Mass flow meter	Not reading	Blew out obstruction with air
27 OCT 92	Pressure transducer	Unstable readings	Cleaned and tightened electrical connections
06 NOV 92	Smoke detectors	False alarms	Cleaned, changed capacitor
10 NOV 92	Mass flowmeter	Not reading	Blew out liquid with air
11 NOV 92	Transfer pump	Leaking	Tightened seals
07 JAN 93	Column bottoms pump	Not pumping, broken idler gear	Installed new pump
07 JAN 93	Column bottoms pump	Motor overheating	Installed a borrowed motor
07 JAN 93	Computer monitor	Blank screen	Sent out for repair, tube replaced
12 JAN 93	Transfer pump	Leaking	Replaced seals
14 JAN 93	Bottoms pump		Installed new motor, returned borrowed motor
15 JAN 93	Uninterruptible power supply	Would not turn on	Replaced batteries
18 JAN 93	Uninterruptible power supply	Burning fuses	Replaced bad fuse holder
15 APR 93	H2S detectors	Failed	Sent to supplier (Delphian) for evaluation
19 APR 93	Pressure regulator	Leaking	Rebuilt and cleaned
28 MAY 93	Control valve	Leaking through	Cleaned and adjusted
31 MAY 93	Hydrogen trailer	Inspection expired	Serviced lights and brakes, vented hydrogen, and prepared for transportation
18 JUN 93	Control valve	Leaking through	Installed new valve trim and seat
22 JUN 93	Electric to air pressure transducer	Failed	Replaced, first with borrowed unit, later with a new unit
28 JUN 93	Pressure regulator	Leaking	Rebuilt

We have recently inventoried AFC test fuels and components in storage. AFC project materials (with brief descriptions) are listed in Table 3 below, including those AFC materials used in earlier government projects.

Table 3. AFC Fuels and Fuel Components in Storage

SwRI ID No.	Product	No. of 55 Gal. Drums	Description
FL-1309	Middle Distillate		High nitrogen shale oil hydrotreated in run 10
FL-1330	Oil		Paraho shale oil blend
FL-1393	Naphtha		Wilsonville coal liquid hydrotreated in run 12
FL-1418	Naphtha	1	Wilsonville coal liquid hydrotreated in run 13
FL-1440	Oil, DF range	5	Coker gas oil from Texaco
FL-1442	Oil, DF range	0.5	Low sulfur, light coker gas oil hydrotreated in run 14
FL-1443	Oil, DF range	1	Low aromatics, light coker gas oil hydrotreated in run 14
FL-1538	Oil, DF range	1	Light cycle oil
FL-1615	Oil, DF range	1	Low sulfur, light cycle oil, hydrotreated
FL-1627	Diesel fuel	13	Straight run, petroleum derived
FL-1840	Diesel fuel		Fischer-Tropsch Diesel
FL-1873	Diesel fuel		Low aromatics, hydrotreated, straight run diesel
FL-1932	Oil	23	Paraho shale oil
FL-2028	Naphtha	2	FCC product, hydrotreated in run 26
FL-2032	FCC naphtha	6	FCC product
FL-2062	Naphtha	2	FCC product, hydrotreated in run 30
FL-2065	Oil		Coal liquid, direct liquefaction, paraffinic
FL-2066	Solvent	1	Blend, paraffinic solvent and methanol

Task 2a. Production of Low-Sulfur, Low-Olefin, Catalytically-Cracked Gasoline

This task experimented with reducing the sulfur concentration of reformulated gasoline. Sulfur must be (and always is) removed from reformer feed to protect the catalyst. With respect to catalytic converters, sulfur in gasoline is a reversible poison which reduces the capacity of the catalyst to operate. As shown in California, reducing sulfur from 300 ppm to 50 ppm can have a significant impact on lowering tailpipe emissions from of HC, CO, and NOx. Sulfur reduction was accomplished by hydrotreating the straight-run stocks blended into finished gasoline. Increasingly stringent emission-control regulations require an assessment of the means for removing the remaining sulfur.

One source of sulfur is catalytically-cracked (FCC) stocks. These are stocks produced by fluidized bed catalytic cracking. Hydrotreating under mild conditions effectively removes sulfur, nitrogen, and olefins from FCC products. This allows the refiner to take advantage of the lower costs associated with operating at comparatively low temperature, low hydrogen pressure, and high volumetric throughput. Mild hydrotreating to remove sulfur, nitrogen, and olefins, with a minimal effect on aromatics, has a minimal impact on octane quality.

Task 2 required acquisition and hydrotreating of two different catalytically-cracked gasoline blendstocks. The feedstocks represented a broad range naphtha from a California source and a narrow range naphtha from an East Coast source. Each feedstock was hydrotreated in a trial run and also in a longer production run to produce sufficient material for blending specification gasoline and engine testing (sometimes desired in other projects).

Engine testing was not performed in the current AFC project, but data on the effects of various blending schemes on emissions were presented in a collaborative paper prepared by the Bechtel Corporation, Southwest Research Institute, (SwRI) and National Renewable Energy Laboratory (NREL). Meeting advanced reduced sulfur emissions regulations and economically producing reformulated gasoline were the principal goals of this joint effort. The modeling used the properties of the two hydrotreated products as inputs to quantify the impact on a typical refinery. The Bechtel Corporation provided their linear programming-based Process Industry Modeling System (PIMS). The PIMS model optimizes gasoline blending through oxygenate purchase and/or adjustments to reforming severity to satisfy specifications. The refinery configuration for modeling was typical of Petroleum Administration for Defense District (PADD) 2, (the Midwest). The results showed that mild hydrotreating of the FCC stock had significant economic advantages over other processing schemes. A paper by Poddar, et al., (1990) provides the complete information about the modeling and economic analysis.

Each trial run consisted of brief hydrotreater experiments to provide guidance for selecting operating conditions for the two longer runs. The trial run results shown in Tables 4 and 5 indicate the effects of process severity on selected properties. No single parameter uniquely defines process severity. In general, the five parameters in the tables all affect it to some extent. However, the pressure was held constant (600 psi), and both of the hydrogen flow rates were in a range where variations make only minor changes in the extent of hydrogenation. As a result, the temperature and space velocity factors dominated, allowing process severity to be represented two dimensionally. A process severity map, Figure 1, at a single pressure provides a convenient illustration of those effects for the East Coast feedstock. The map shows operating conditions (indicated by X's) on a grid of temperature versus liquid hourly space velocity. The lower left parts of the grid (severity map) represent the mildest combination of operating conditions, while the upper right represents the most severe. Figures 1 and 2 display the effect of process severity on research octane number (RON) and fluorescent indicator analysis (FIA) aromatics respectively for the narrow range, East Coast feed.

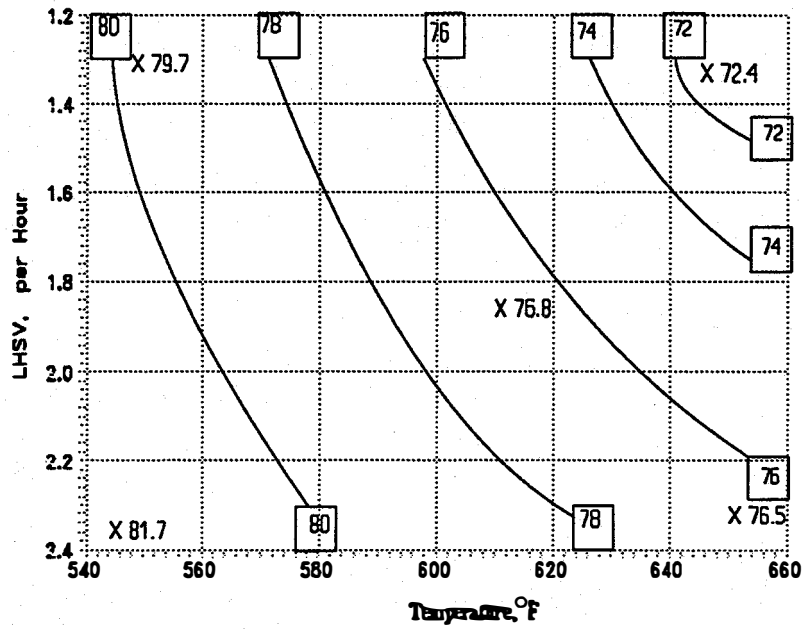


Figure 1. The effect of processing severity on RON, shown as isopleths on an operating temperature/LHSV grid

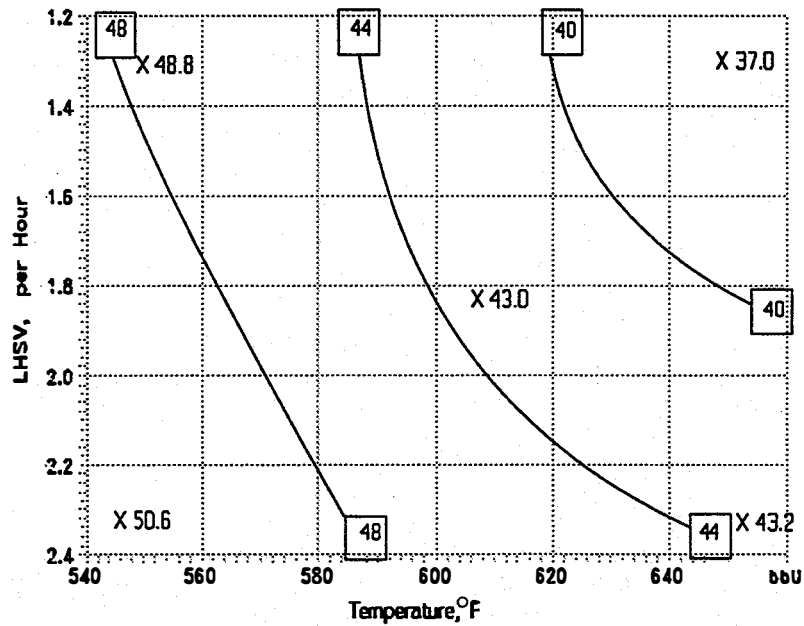


Figure 2. The effect of processing severity on aromatic concentration, shown as isopleths on an operating temperature/LHSV grid

The trial runs indicated that both feedstocks could be adequately hydrotreated at mild conditions. Table 6 shows the conditions used in the longer runs with each feedstock, and Table 7 shows the properties of each material before and after processing. Hydrotreating removed essentially all the sulfur, nitrogen, and olefins from both feeds. The RON decreased 7.5 numbers for the broad-range feed, and 6 numbers for the narrow-range feed, which received the mildest hydrotreatment. The MON decreased 4.9 numbers for the broad-range feed, and 4.1 numbers for the narrow-range feed.

Tables 4 and 5, show approximate octane decrease resulting from olefin loss. The amount of olefin removed (at constant aromatics content) is best shown in Column H in both tables. The octane decrease was research 5.3 and motor 3.8 for the narrow-range feed, and research 3.4, and motor 1.8 for the broad-range feed. Decreasing fractions of the total range from 44% to 70%, and average 62%. The products have potential value in reformulated gasoline, and relatively low production costs associated with the mild hydrotreating.

Table 4. Trial Run Conditions and Results, Broad-Range Naphtha, California Source

Parameter	Feed	A	B	C	D	E	F	G	H	I	J
LHSV ^a , Hr ⁻¹	n.a. ^b	1.24	1.21	1.33	1.57	1.97	1.99	1.96	2.26	2.30	2.29
Pressure, psig	n.a.	800	1700	800	1400	1700	800	800	600	600	600
Temperature, °F	n.a.	557	567	650	619	567	558	661	406	451	500
H ₂ Makeup, scf/bbl	n.a.	450	450	670	770	450	450	830	720	700	700
H ₂ Recycle, scf/bbl	n.a.	1950	2010	1830	1540	1230	1220	1240	1440	1400	1400
PRODUCT PROPERTIES											
FIA (Vol%) ASTM D 1319											
Aromatics	51.9	42.9	37.6	37.1	33.9	42.7	44.0	40.0	53.3	47.9	44.8
Olefins	9.9	1.0	0.7	0.8	0.6	1.0	0.8	0.5	1.6	0.8	0.2
Saturates	38.2	56.2	61.7	62.1	675.5	56.7	55.3	59.5	45.1	51.2	55.0
Sulfur											
WPPM ^c	4400	40	10	30	10	40	60	30	n.a.	n.a.	n.a.
WPPM ^d	-	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	213	65.4	17.1
Nitrogen (WPPM ^e)	-	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	108	81.4	16.4
RON ^f ASTM D 2699	91.1	81.0	79.7	77.6	76.4	80.8	81.3	79.8	87.7	86.0	85.0
MON ^g ASTM D 2700	80.6	74.8	73.2	72.4	70.4	74.2	78.4	73.5	78.8	77.5	76.4

a Liquid hourly space velocity; to obtain the actual flow rate in gallons per hour, multiply by the catalyst volume, 1.56 gallons.

b Not analyzed, or not applicable

c Weight per million, by ASTM D 2622

d Weight parts per million, by pyrofluorescence

e Weight parts per million, by D 4629

f Research Octane Number

g Motor Octane Number

h FIA (Fluorescent Indicator Analysis)

i Standard cubic feet.

**Table 5. Trial Run Conditions and Results
Narrow Range Naphtha, East-Coast Source**

Process Conditions:										
Parameter	Feed	A	B	C	D	E	F	G	H	I
LHSV ^a , Hr ⁻¹	n.a. ^b	2.31	2.31	1.82	1.28	2.32	2.31	1.33	2.28	2.28
Pressure, psig	n.a.	600	600	600	600	600	600	600	600	600
Temp. °F	n.a.	450	610	610	550	547	654	648	481	407
H ₂ Make-up scf/bbl	n.a.	700	700	562	1260	696	700	781	710	710
H ₂ recycle scf/bbl	n.a.	1400	1400	1775	2520	1392	1400	2435	1420	1420
Product Properties:										
FIA (Vol. %): ASTM D1319										
Aromatics	29.3	39.0	46.7	56.3	51.5	48.8	56.4	62.4	44.5	34.7
Olefins	16.7	1.5	0.5	0.7	0.7	0.6	0.5	0.6	0.7	2.6
Saturates	53.6	59.5	52.8	43.0	47.8	50.6	43.2	37.0	54.8	62.7
Sulfur, WPPM, ^c 3530 ^d Pyro.	1314	23.3	<10	<10	1.9	<10	<10	<10	218	2410
Nitrogen, WPPM, ^e 174 ^g Pyro.	<10	<10	<10	<10	2.0	<10	<10	<10	20	44
RON ^f	90.4	n.a.	n.a.	76.8	79.7	81.7	76.5	72.4	85.1	88.8
MON ^g	79.9	n.a.	n.a.	71.1	72.8	74.5	70.8	68.4	76.1	79.8

- a Liquid hourly space velocity; to obtain the actual flow rate in gallons per hour, multiply the catalyst volume, 1.56 gallons.
b Not analyzed, or not applicable.
c Weight parts per million.
d By ASTM D 2622.
e By ASTM D 4629
f Research Octane Number.
g Motor Octane Number.

Table 6. Production Run Processing Conditions

Parameter	FCC Products Used	
	FL-1860	FL-2032
Liquid Hourly Space Velocity, Hr ⁻¹	1.46	2.28
Pressure, psig	600	600
Temperature, °F	553	502
H ₂ MakeUp, scf/bbl	555	710
H ₂ Recycle, scf/bbl	1665	1420

Table 7. Properties of FCC Gasoline Before and After Hydrotreating

Property	Test Condition	Method	Broad Range, California Origin		Narrow Range, East Coast Origin		
			Feed FL-1860	Product FL-2028	Feed FL-2032	Product FL-2062	
Specific Gravity	60°F	D 1298	0.8208	0.8151	0.8260	0.8189	
API Gravity			40.9	42.1	39.8	41.3	
Distillation, °F	Vol% Evaporated	D 86	IBP	135	156	269	248
			5/10	185/213	204/228	283/289	272/282
			15/20	233/247	248/258	294/298	288/292
			30/40	271/295	282/302	306/313	301/306
			50/60	317/337	324/344	320/328	313/326
			70/80	359/380	362/383	337/349	334/344
			90/95	408/425	411/428	363/377	356/368
			EP	447	444	398	396
			RVP, psi	100°F	D 323	2.1	1.6
RON		D 2699	91.1	83.6	90.4	84.4	
MON		D 2700	80.6	75.7	79.9	75.8	
Composition							
Carbon, wt%		D 5291	88.25	87.78	88.24	86.08	
Hydrogen, wt%		D 5291	11.34	12.46	11.74	11.82	
Sulfur, WPPM		D 2622	4400	20	3500		
Sulfur, WPPM		Fluorescence		12		164	
Nitrogen, WPPM		D 4629	174	<10	84	<10	
Heat of Combustion, Btu/lb	Gross	D 240	19240	19266	18837	11.82	
	Net		18200	18129	17766		
FIA, Vol%		D 1319					
Saturates			38.2	52.1	29.3	47.3	
Olefins			9.9	1.0	16.7	0.7	
Aromatics			51.9	46.9	53.6	52.0	
Aromatic							
Carbon by	Total		54.3	25.5	24.8	21	
UV analysis,	Mono		28.5	24.4	8.3	21	
wt%	Di		9.2	1.1	3.3	0	
	Tri		16.5	0	13.2	0	
Unwashed Gum mg/100mL		D 381	3.1		17.3		

Task 2b. Production of Low-Emission Gasoline

Under this task, researchers were to produce five drums of low-emission/low-reactivity gasoline. Their task subgoals included selecting criteria for making low-emissions/low-reactivity gasoline, finding components and calculating a recipe, and producing the test fuel. Although no restrictions were placed on the recipe to conform to American Society for Testing and Materials (ASTM) D4814 or any other gasoline specification, an effort was made to make the test fuel as realistic as possible, while targeting the lowest emissions/reactivity possible. For this reason, distillation range, Reid vapor pressure (RVP) volatility, and octane number were used to guide the test fuel composition. These properties, together with fuel component densities and the model and correlation described below, were used to minimize calculated emissions/reactivity of the blend.

Researchers selected nine components which they believed had the key properties needed in gasoline, and which also had compositions believed to contribute low reactivity to the engine-out emissions. These components included the two hydrotreated FCC stocks produced in Task 2A. The remaining components, and their properties — except butane, which has well-known properties, and methyl tertiary butyl ether (MTBE), an oxygenate excluded for reasons stated below — are listed in Table 8.

The reactivity of engine exhaust emissions depends on their composition. The quantity and composition of exhaust emissions is known to be dominated by engine factors. Assuming these factors to be equal, however, gasoline composition will affect the unburned hydrocarbons and the products of chemical equilibrium which form in the plasma of the combustion chamber. Therefore, the effects due to composition include the composition of the hydrocarbons surviving combustion, and the quality of the combustion process itself. The quality of combustion, in turn, controls the total amount of hydrocarbons emitted and the amounts of certain hydrocarbons formed during combustion.

Adequate information to establish the relationships between exhaust emissions and gasoline composition has been published. Hochhauser et al, (1992) examined the effects of changing the aromatics, olefins, and MTBE concentrations, as well as the effects of 90% distillation temperature on combustion emissions and their reactivities. Although their results varied among the various reactivity measures available and between vehicle fleets, both olefins concentration and the 90% distillation temperature strongly correlated with more reactive emissions. Aromatics concentration was less important, and the MTBE concentration generally contributed to more reactive emissions. Leppard et al., (1992) has published a correlation between the fuel components (by species type) and the exhaust components, showing that the exhaust concentrations of most components are proportional to their fuel concentrations, while other compounds are entirely or partly formed during combustion. Exhibit 3 lists additional sources of information on this topic.

Table 8. Minimum Emissions Gasoline Components

Property	Test Condition	Test Method	Alkylate FL-2103	Reformate FL-2101	Alkylate FL-2099	Isomerase FL-2105	Reformate FL-0863
API Gravity	60°F	D 1298	70.1	41.0	70.5	-	45.6
Density	60°F		0.7017	0.8199	0.7003	-	
Distillation	Vol% Evaporated	D 86					
	IBP		88	114	108	79	117
	5		133	156	158	91	168
	10		165	178	190	91	192
	20		200	211	206	92	221
	30		214	234	214	93	240
	40		217	253	218	94	254
	50		223	270	220	95	266
	60		227	283	222	97	278
	70		229	298	228	99	293
	80		234	315	234	105	309
	90		244	338	256	111	334
	95		315	360	349	113	358
	EP		370	407	382	128	404
RVP, psi	100°F	D 323	6.55	4.64	5.50	16.73	1.8
RON		D 2699	97.3	100-101 Est	92.5	-	95
MON		D 2700	93.8	90	91.8	-	84.07
Sulfur, %		D 2622	0.003	0.001	0.001	-	<0.1
FIA		D 1319					
Aromatics			-	70.5	-	-	53.8
Olefins			-	1.0	-	-	0
Saturates			-	28.5	-	-	46.2

Procedure

General Description

The procedure required models to predict the quantity and quality (composition) of the emissions. It also required a method for formulating the optimal blend composition for minimizing the emissions predicted by the model. The best and most readily available optimization procedure, linear programming (LP), requires that the predicted emissions relate linearly with the properties and concentrations of each blend component. Linear programming thus provides the single best-blend formulation for meeting the gasoline specifications. A single solution, however, does not reveal how the formulation would change with variations in any particular property: exploring such a variation requires imposing a progression of limits and obtaining multiple solutions, which satisfy the various values imposed for the limit.

The Interim version of the U.S. Environmental Protection Agency (EPA) Complex Model was viewed as an acceptable model for predicting emissions quantities. This model, however, had three problems. First, an examination of the model indicated many second-order terms used to calculate the emissions from component properties, so its linearity was a concern. Of course, if the emissions really were significantly non-linear with respect to blend component properties, and concentrations, a different optimization technique would be required. However, because of the ease and efficiency of linear programming researchers considered it well worthwhile their while to attempt to vary the inputs to the model and to see if the outputs varied linearly. They did. Second, the Interim model was designed for fuels which met, or were close to the specification limits. However, individual blend components often differ widely from the limits; the sum of the components compensates for the blend itself meeting the limits. Experimenting with the model showed that components yielding unrealistic values were used only in small quantities, so the net result was reasonable. Third, the output provided only a global, or overall, emissions quantity, not quantities of the individual species needed to characterize the reactivity.

The emissions quantities provided by the EPA Complex Model for each prospective blend component were used to calculate the blend formulations. Linear programming was used to solve the blend composition meeting gasoline specifications and having minimum predicted emissions. There were some uncertainties in the results caused by nonlinear blending of the 90% distillation temperatures. Therefore, variations in composition with octane number were explored by imposing, then varying, an upper octane number limit. Researchers selected a final composition based on a minimum in the 50% distillation temperature (which does blend linearly), which was coincident with a minimum in the 90% distillation temperature, a major factor in both emissions quantity and reactivity.

Researchers needed to know the quantities of individual species in order to determine a predicted reactivity. This information came from Leppard's correlations, which related species concentrations in the exhaust to their concentrations in the fuels, as well as to combustion factors. The concentrations obtained using Leppard's correlations were multiplied by the overall emissions quantities from the EPA Complex Model to obtain the needed quantities of individual species. Researchers then used a straightforward application of the Carter reactivity factors to provide the emissions reactivity of the low-emissions blend.

Detailed Approach

An interim version of the (EPA) Complex Model for emissions was used in this project to predict emissions quantities (termed "model emissions") based on commonly measured fuel properties. This model did not predict emissions reactivity. However, some of the same properties used by the model which contribute to higher values for pollutants in the EPA Complex Model are associated with more reactive emissions in Hochhauser's work (particularly the olefins and aromatics concentration, and the 90%

distillation temperature. Although Hochhauser did not examine the 50% distillation temperature in relation to reactivity, it too should correlate positively with higher reactivity because it correlates positively with the 90% distillation temperature in most fuels.

Among the other properties, sulfur should not affect reactivity, and RVP should generally make a small negative contribution. High RVP in fuels comes from four- and five-carbon hydrocarbons, which are usually paraffins, rather than olefins. Paraffins contribute less to reactivity than do olefins. A simple replacement of light olefins with light paraffins, however, would not make the linear reduction expected due to the differences in their reactivities alone. This is because combustion produces light olefins. The combustion temperature allows the composition to approach thermodynamic equilibrium, so the olefin production is inherent, and cannot easily be avoided.

Oxygen concentration in the EPA Complex Model contributes to reduced emissions quantity; however, as noted in Hochhauser's work, it also increased reactivity. While methanol blends are well known to decrease both emission and emission reactivity, they are not yet regarded as preferred fuels. Rather, industry has shown a strong preference for MTBE and ethanol. The concentrations of oxygenates are often set by law or vehicle tolerance limits, rather than being allowed to vary in blend optimization calculations. As a result, we felt that the hydrocarbons should provide the main focus of low-emission fuel testing. Therefore, because of the effect of oxygen concentration on the reactivity and emissions quality of the target test gasoline formulation, MTBE and other oxygenates were excluded from consideration at the outset. The only remaining factor considered in the EPA Complex Model, benzene, contributes to the toxic emissions (which were not considered in this study), but not to reactivity. The benzene molecule is stable compared to other emissions (e.g. olefins) and so is less reactive. We concluded that non-oxygenated fuels with low EPA Complex Model emissions could be expected to have low emissions reactivity.

Our approach included a preliminary screening of the EPA Complex Model emissions (excluding toxics) for each of the prospective blending components. The next step used linear programming to determine a minimum emissions blend meeting RVP, boiling range, and octane requirements for gasoline. The LP calculation used the EPA model results as inputs, and solved for the minimum emissions formulation. Changing other restrictions on the properties requirements and re-optimizing provided several blend formulations, and from these a particular formulation was chosen in accord with literature-based factors that indicated low reactivity. Because the 90% distillation temperatures were used directly in the Interim version of the EPA Complex Model, as well as in the Hochhauser study, they were used directly in the LP model. This was done even though linear combination of the temperatures, rather than the volumes distilled at temperature, provides only a crude estimate of the blend distillation temperatures. The alternative conversion to a volume-based limit would have required tenuous assumptions and introduced other errors. The correlations in Leppard's paper and EPA Complex Model results were used to calculate a predicted emissions reactivity.

Detailed Methods

The first step was to determine whether the EPA Complex Model provided results suitable for linear combination to represent blending. In addition to the distillation temperature problem noted above, the Interim version of the model available to us contained a large number of second-order terms, which generally do not combine in a linear manner. To determine the relative importance of the second-order terms to the overall model results, model emissions were calculated using only the linear terms, and repeated using the complete model. The fuel properties used for the calculations included the mean properties from the 1990 fuel data set, which will be the base fuel for compliance with the EPA regulation requiring 15% improvement in emissions. Solutions were also obtained for properties differing by one

standard deviation in the direction of both higher and lower emissions. The results are shown in Figures 3 through 8. Figure 3 shows the calculated volatile organic compound (VOC) from fuel with the low, mean or (average), and high emissions properties for the EPA "normal emitter" vehicle fleet. Figure 4 shows VOC for the "high emitter" fleet, and Figure 5 for the combined fleet. Figures 6 through 8 show the same information for oxides of nitrogen (NO_x). In all cases, the calculations using only linear terms were good approximations of the calculations using the complete model with linear and nonlinear terms, and the variation over one standard deviation of fuel properties appeared linear. Interestingly, most of the nonlinear terms present in the Interim version were dropped in the final version of the model by EPA.

The properties of real blending components generally fall well outside the range of normal fuels to which the EPA Complex Model applies. However, gasolines require only small proportions of blendstocks, such as butane, which have the most extreme properties. Also, the exclusion of non-linear terms in computing their model emissions, researchers obtained more realistic results than if they had used second-order terms, which can go extremely high or low for fuel properties beyond normal ranges. Table 9 shows the model emissions for the nine components, the unweighted total of VOC, plus the NO_x as the objective function in the LP calculation to solve for the minimum emissions blend.

Because the inputs to the calculations included the 90% distillation temperature, an important parameter known to blend nonlinearly, the results had to be regarded as somewhat approximate. To resolve the problem, variations in blend formulation were obtained by imposing, then varying, an additional limit. Maximum octane number limit was chosen because our particular set of blending components provided blends with unusually high octane numbers, and excess octane numbers do not, per se, benefit emissions or performance; they may in fact discourage production because they are too expensive. Raising the maximum octane number changed the blend formulations, and the calculated emissions decreased from octane number 88 through 90, then remained almost flat with further octane increases, as shown in Figure 9. RVP contributes to the model emissions, and it began moving off its upper limit (9 psi) at about octane number 90 (see Figure 10), so its subsequent decrease contributed lower model emissions to the results in Figure 9.

Figure 11 shows the effects of changing the upper octane limit on the distillation properties. The LP model 50% and 90% distillation temperatures each have a minimum near octane number 90. Since low 90% distillation temperature correlates strongly with low emissions reactivity, and only slight improvement could be obtained in model emissions by using a higher octane number blend, the distillation minimum became the final basis for choosing the blend formulation. Table 10 shows the blend formulation and its calculated and measured properties. Figure 12 shows the variations in blend composition as a function of octane number.

Calculating the predicted emissions reactivity relied heavily on the correlations by Leppard. He obtained correlations for two fuels, designated A and H in the Auto/Oil Industry study. Fuel H contained MTBE, which caused significant differences in emissions compared to non-oxygenated fuels. Our low emissions fuel more closely resembled Fuel A, so we used the Fuel A correlations in our calculations.

Leppard's correlations contain a set of factors for components which survive the combustion process unaltered, and another set of factors for components produced during combustion. Our calculation used the average of the three factors Leppard obtained from each three vehicles. The survival factor for paraffin, aromatics, or olefin, times the component concentration in the fuel, yields exhaust concentration as a fraction of total exhaust hydrocarbons. The quantities obtained provide the portion of the emissions which came directly from the fuel having survived the combustion process chemically intact.

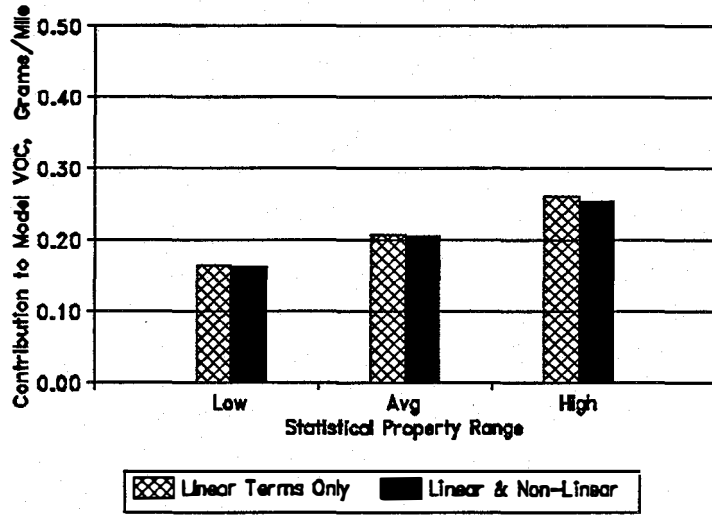


Figure 3. VOC, linear and total terms, high emitters only

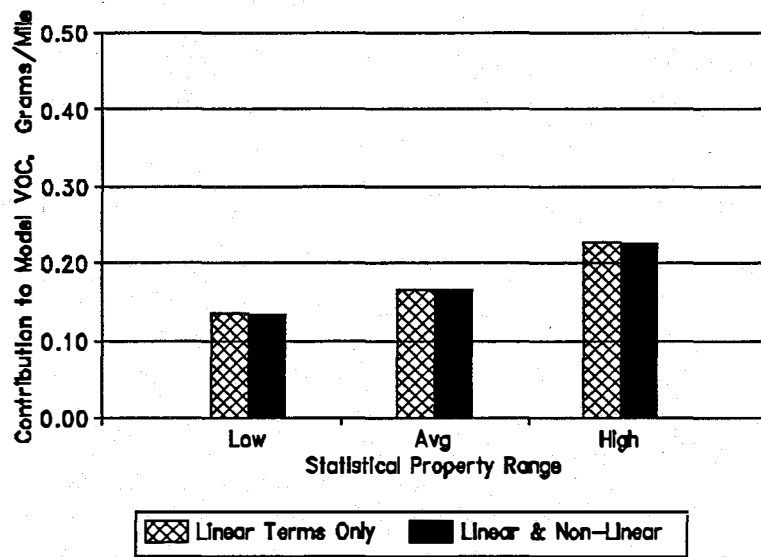


Figure 4. VOC, linear and total terms, normal emitters only

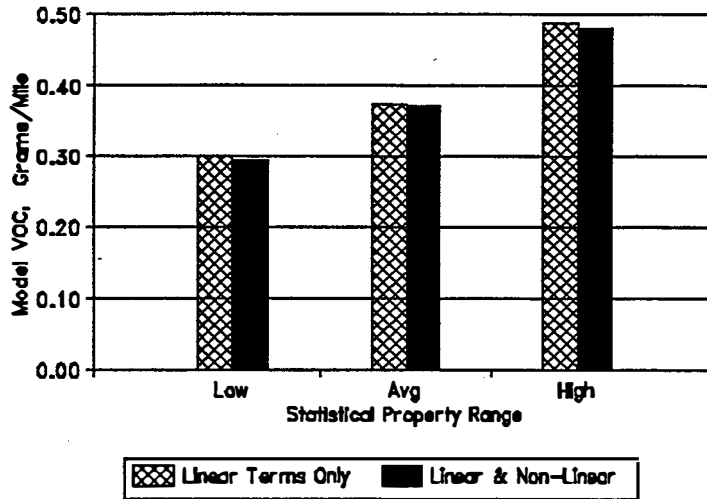


Figure 5. VOC, linear and total terms, all emitters

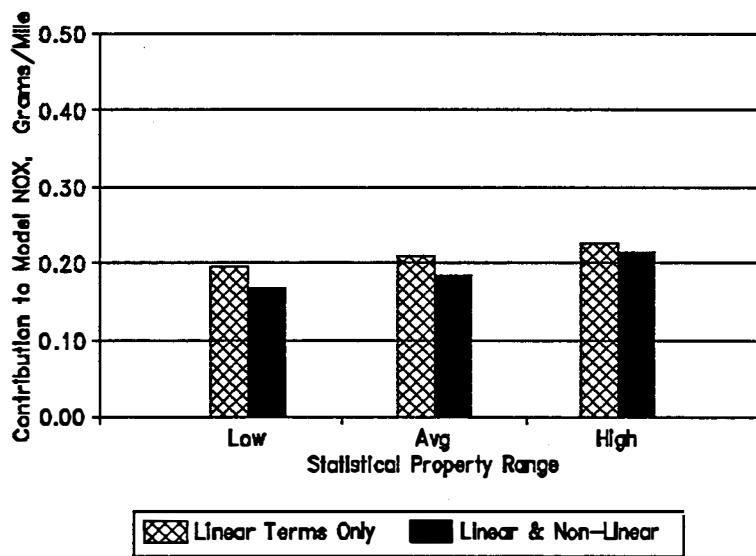


Figure 6. NOx, linear and total terms, normal emitters only

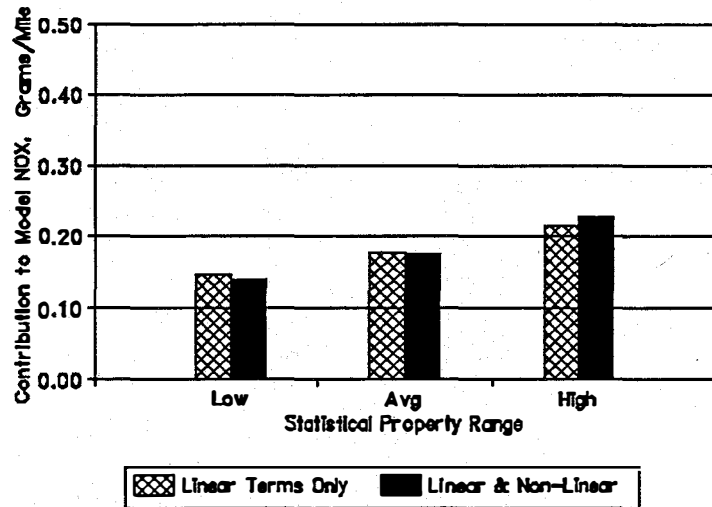


Figure 7. NOx, linear and total terms, high emitters only

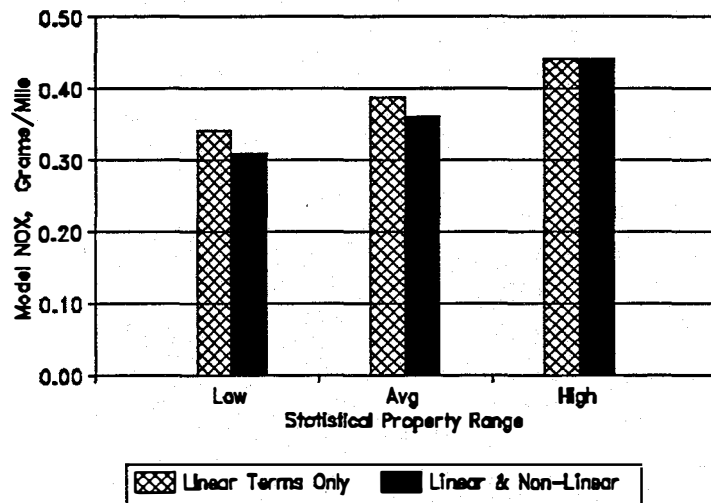


Figure 8. NOx, linear and total terms, all emitters

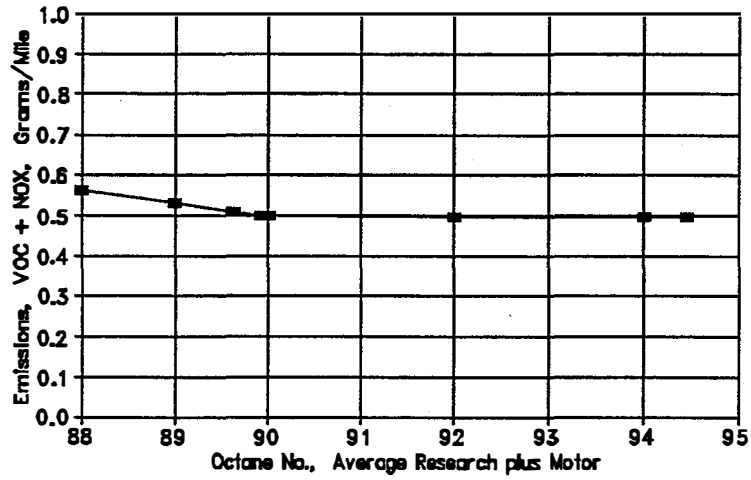


Figure 9. Calculated emissions versus octane, minimum emissions (VOC + NOx) blends

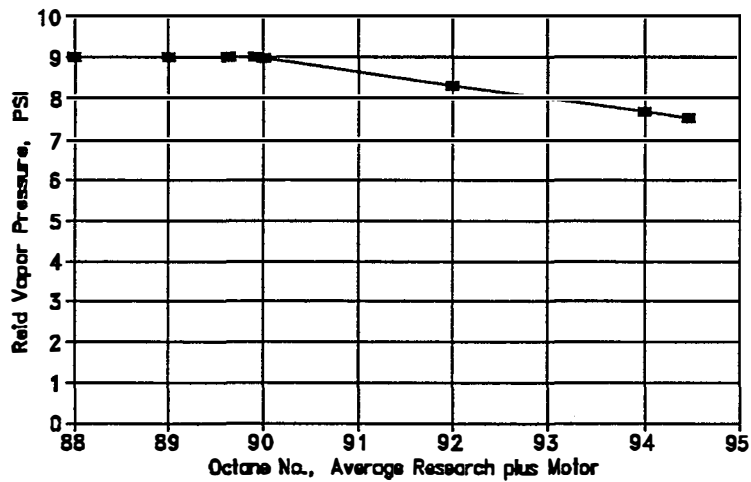


Figure 10. Reid vapor pressure versus octane, minimum emissions (VOC + NOx) blends

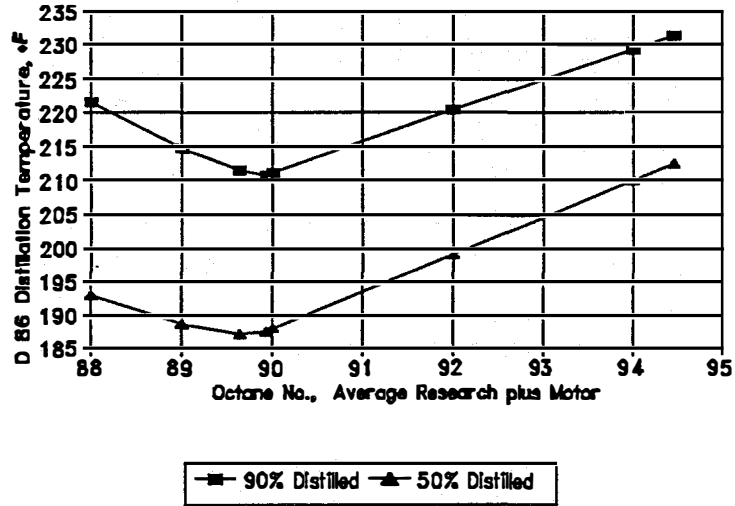


Figure 11. Distillation properties versus octane, minimum emissions (VOC + NOx) blends

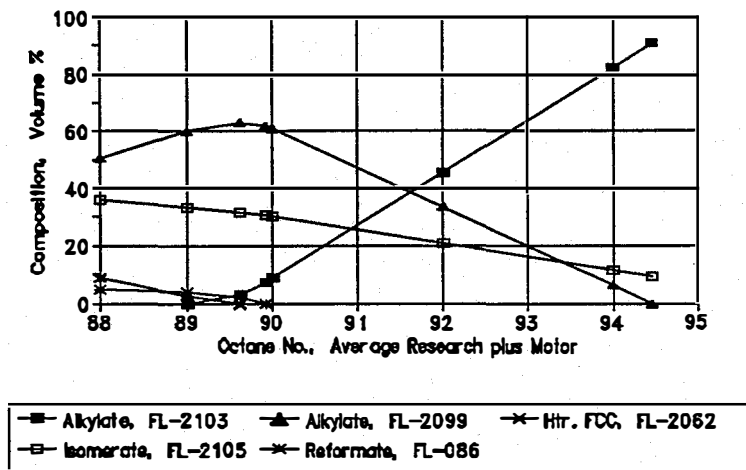


Figure 12. Gasoline composition versus octane, minimum emissions (VOC + NOx) blends

Table 9. Interim EPA Complex Model Emissions of Blend Components, Calculated Using Linear Terms, and Total of All Terms (grams/mile)

Blend Component	VOC Linear	VOC Total	NOX Linear	NOX Total	Both Linear	Both Total
FL-2103, Alkylate	0.247	0.472	0.246	0.314	0.493	0.786
FL-2101, Reformate	0.565	0.916	0.577	0.351	1.142	1.267
FL-2102, MTBE	0.228	5.151	0.230	0.497	0.458	5.648
FL-2099, Alkylate	0.248	0.415	0.245	0.311	0.493	0.726
FL-2028, FCC CA	0.461	1.756	0.518	0.409	0.979	2.165
Normal Butane	1526	1.6E+40	0.990	1.2E+11	1527	1.6E+40
FL-2062, FCC EC	0.424	1.234	0.472	0.286	0.896	1.520
FL-2105, Isomerate	0.276	12.911	0.237	1.412	0.513	14.323
FL-0863, Reformate	0.453	0.609	0.454	0.280	0.907	0.889

Table 10. Composition and Properties of Low Emission Gasoline - FL-2114

Composition:		Type	Identification	Volume Fraction
		Alkylate	FL-2103	0.0741
		Alkylate	FL-2099	0.6214
		Isomerate	FL-2105	0.3045
Properties:	Test Condition	Method	Measured	LP Model
Gravity, Spec.	60°F	D 1298	0.6823	*
Gravity, °API	60°F	D 1298	75.9	*
Distillation, °F	Vol% Evaporated	D 86		
	IBP		89	*
	5/10		105/120	*/*
	15/20		128/137	*/*
	30/40		157/178	*/*
	50/60		199/214	188/*
	70/80		222/230	*/*
	90/95		250/330	211/*
	EP		392	*
RVP	100°F	D 5191	9.60	9.0
RON		D 2699	91.1	*
MON		D 2700	89.5	*
Avg (R+M)/2			90.3	89.9

* Indicates the property was not calculated.

The second set of factors, called "combustion factors", provided the hydrocarbons produced during combustion. Leppard's paper did not explicitly state the amounts of all the hydrocarbons formed during combustion, so they were estimated from his graphs (which gave the totals). For those components present in the fuel, the amount in the exhaust predicted by the survival correlation was subtracted from the total to give the combustion-produced fraction. This is the same way Leppard treated the data; that is, he did not use the combustion-produced portions in calculating the survival correlations. The product of Leppard's survival factor times the fuel concentration added to the combustion factor yields the total concentration in the exhaust hydrocarbons in units of weight fraction. The combustion factors and Leppard's survival correlation factors are given in Table 11.

Table 11. Factors for Estimating Exhaust Concentrations Using Fuel Concentrations, Non-oxygenated Fuel, After Leppard, et al., (1992)

Hydrocarbon	Leppard's Survival Factor, Vehicle Average	Combustion Factor, Vehicle Average
Methane	0.50 ^a	0.0334
Ethane	0.50 ^a	0.0131
Propane	0.50 ^a	0.0043 ^b
n-Butane	0.50	0.0017
All other paraffins	0.50	0.0
Benzene	0.82	0.0297
Toluene	0.82	0.0213
Styrene	0.82	0.0073
All other aromatics	0.82	0.0
2-Methylpropene	0.64	0.0090
<i>trans</i> -2-butene	0.64	0.0074
<i>cis</i> -2-butene	0.64	0.0031
2-Methyl-1,3-butadiene	0.64	0.0038
Cyclopentadiene	0.64	0.0034
All other olefins	0.64	0.0

a. Not present in Leppard's fuels, and not used in deriving correlation.

b. Not reported by Leppard; interpolated from methane, ethane and n-butane values.

Results

These estimated combustion factors provided an estimated concentration of each hydrocarbon species as a fraction of total exhaust hydrocarbons. However, total exhaust hydrocarbons should differ from Leppard's totals for Fuel A by a factor related to the fuel's compositional differences, assuming no significant vehicle factors. The EPA Complex Model was used to calculate the relative quantities based on composition. The VOC model output represents all the hydrocarbons except methane and ethane. The best estimate of these would be to multiply the model outputs by a common factor; however, because we planned to only apply the ratio of the model outputs, the factor was not needed. The ratio of FL-2114 total model VOC to the Fuel A total model VOC was obtained using the FL-2114 properties, and the Fuel A properties which, though not included in Leppard's paper, were reported by Gerry et al., (1992).

The ratio, 0.582, times the 1.96 g/mile total hydrocarbons in Leppard's work (Fuel A, bag composite, and average for the three vehicles) provided an estimated 1.14 g/mile total exhaust hydrocarbons for FL-2114. This allowed us to calculate the estimated exhaust species concentration, and by applying the Carter maximum incremental reactivity (MIR) and maximum ozone reactivity (MOR) factors, it provided predicted exhaust reactivities. The results are shown in Table 12. Fuel FL-2114 has an estimated reactivity that is 50% of AQIRP Fuel A.

Table 12. Engine-Out Exhaust Reactivities

Type of Reactivity	Fuels Used in Engine	
	SwRI Fuel FL-2114	AQIRP Fuel A ^a
Measured Specific MIR, gO ₃ /gNMOG	N.M. ^b	3.10 ^c
Predicted Specific MIR, gO ₃ /gNMOG	2.68	3.96
Predicted MIR, gO ₃ /Mile	3.06	7.76
Predicted Specific MOR, gO ₃ /gNMOG	1.20	1.48
Predicted MOR, gO ₃ /Mile	1.37	2.89

a. Based on properties and composition reported by Gerry et al.⁴
b. Not Measured. Measurements are planned under a separate DOE contract with Mantech Environmental Technology, Inc.
c. After Leppard, et al,¹ average of three vehicles.

The above data provide only one comparison to date between a predicted and measured value. The predicted specific MIR for Fuel A exceeds the measured value by 28% which can be viewed as good agreement because of the uncertainties in the data and the complex nature of the procedures. A second comparison will become available when the results of the specific reactivity measurements on the SwRI fuel FL-2114 are published.

Five drums of the test fuel were blended. The characterization was given in Table 10. This batch was sent to Mantech Environmental Technology, Inc. for emissions measurements in their test program.

Tasks 3 and 4 - Other Government and Research and Industry Research

AFC objectives are supported when other government and industrial clients use the AFC. The nation benefits when the AFC assists in developing higher quality fuels and improving our ability to utilize alternative fuel sources. In addition, using the equipment helps keep it in good working order, and the repair parts purchased on these projects help pay for routine maintenance. During the year covered by this report, the only fuels provided as part of the contract were those discussed in Task 2 above. Table 13 outlines the principal uses of the AFC for studies performed in addition to the basic DOE subcontract.

Table 13. Utilization of the AFC by Industries and Government Agencies Beyond the Operating Contract

User/Recipient	AFC Activity*	Type of Fuel**	General Objectives
EPA	B	D	Develop an emissions-reducing component
EPA	B	G	RVP study
NREL	D,B	G	RVP study
DOE Fossil Energy Div.	D	D	Ignition quality, Fischer-Tropsch fuels
Oil Company	B	D	Fuel producing reduced emissions
Industrial Association	B	D	Fuel partially derived from biomass
Oil Company	H,D,B	D	Fuel producing reduced emissions
DOE Fuels & Chem. Research Div.	H,D	D	Fundamental data on emissions
Oil Company	H,B	D	Fuel producing reduced emissions
Oil Company	B	G	Additive testing

* Type of activity: H hydrotreating, D distillation, B blending.
 ** Type of fuel: D diesel, G gasoline.

Conclusions

1. The AFC has been maintained in good working order. The hydrogen trailer was recertified, and plans are underway for a control system upgrade. The Institute plans to renovate or replace the safety sensors in the building.
2. Mild hydrotreating of FCC products effectively removes sulfur and olefins with minimal effects on aromatics and octane quality. This approach to making reformulated gasoline has significant economic advantages.
3. Calculated VOC and NOX emissions using only the linear terms in the interim EPA Complex Model differ only slightly from those obtained using the complete model.
4. With guidance obtainable from the EPA Complex Model, linear programming can be used to select gasoline formulations which should produce low engine-out emissions.
5. For non-oxygenated fuels, an interim EPA Complex Model prediction of low emissions is an indicator of low engine-out emissions reactivity.
6. An estimated or predicted, emissions reactivity can be calculated based on fuel speciation and published information.
7. In addition to the new alternative fuels knowledge produced this year by the AFC project, use of the AFC by other government agencies and industries for outside projects has contributed to better fuels and alternative fuel sources. This is clear evidence of the widespread interest in, and value of, the Alternative Fuels Utilization Program and the Alternative Fuel Center.

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Hochhauser, A.M. et al., (1992), Speciation and Calculated Reactivity of Automotive Exhaust Emissions and their Relation to Fuel Properties - Auto/Oil Air Quality Improvement Research Program, Society of Automotive Engineering Paper 920325, Presented at the International Congress and Exposition, Detroit, MI., 24-28 February, 1992.

Leppard, W.R. et al., (1992), Effects of Gasoline Composition on Vehicle Engine-Out and Tailpipe Hydrocarbon Emissions - The Auto/Oil Air Quality Improvement Research Program, Society Automotive Engineering Paper 920329, International Congress and Exposition, Detroit, MI., 24-28, February, 1992.

Poddar, S.K. et al., (1993), "Evaluation of Catalytically Hydrotreated Cracked Stocks for Reformulated Gasoline by LP Modeling," Presented at the American Institute for Chemical Engineers Annual Meeting, St. Louis, MO., 7-12 November, 1993.

Exhibit 1. Index of Monthly Progress Reports

XS-2-12130-1 Summary of Monthly Progress Reports (03-5151)														
TOPICS	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Sample Inventory	T	●			●								●	
Maintenance Log	T		●			●	3F	●	●			●	●	●
Equipment Custody	●			●										
Hydrotreat Cracked Gasoline	T1	T1	T1,2,3	T1	T1	T1	T1	T1, F1, F2	T1-5	T1				
Travel/Contacts	●			●	●									
Outside Processing		●	●	●	●				●	●	●	●	●	
Hydrogen Trailer				●	●	●	T	T	●	●	●			
Low Reactivity /Emissions Gasoline								F, T2	T6		F1-10 T1-3		●	●
Environmental Safety												●		
Control Upgrade														●
● Topic covered T Table (number) F Figure (number)														

Exhibit 2. Description of Facilities

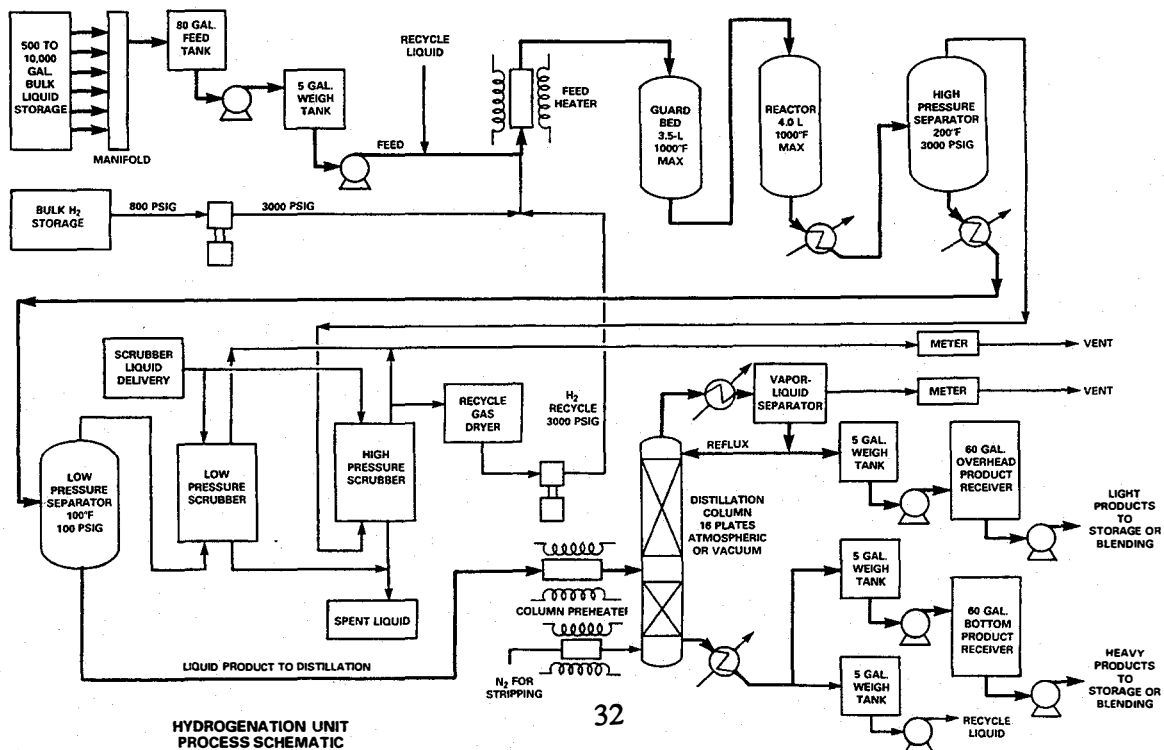
Hydrogenation Unit Capabilities

The pilot unit was designed with flexibility to handle a range of hydrogenation operations. Nominal feed rate is 1.0 to 2.2 gal/hr. The reactor section operates at pressures to 3000 psig and temperatures to 1000°F. Hydrogen circulation capacity of 250 scf per hour is equivalent to about 4.800 scf per barrel at maximum feed rate. Appropriate operating conditions and catalyst types can be selected for the following product objectives at various levels of severity:

SEVERITY	PRODUCT OBJECTIVE
Low	Hydrotreat to reduce sulfur and nitrogen content of reformer feed or distillate fuel.
Moderate	Hydrotreat to prepare feedstocks for hydrocracking or to increase hydrogen content of fuel.
Intermediate	Hydrogenate aromatics to produce low-emission diesel fuel.
High	Hydrocrack light cycle oil to make high energy density jet fuel.
High	Catalytic reforming of low octane naphtha.

Test fuels or blending components have been made in quantities of 50 to 500 gallons for many fuel evaluation projects. The unit is used to make fuels from shale oil and coal liquids for the Department of Energy (DOE) Alternatives Fuels Utilization Program.

The attached process schematic of the unit shows feed joined by hydrogen through a preheater to two fixed-bed reactors in series. Reactor effluent is cooled and liquid product is recovered in two stages of separation. Recycle hydrogen and vent gases are scrubbed to remove contaminants. The liquid product goes to a distillation column, which is used as a stripper to remove H₂S or adjust the flash point. Alternatively, the distillation column can take a light product overhead at atmospheric pressure or under vacuum. The column bottoms may be collected as product or recycled to the reactor section. The recycle pump can also be used to increase total feed rate to 3.5 gal/hr.

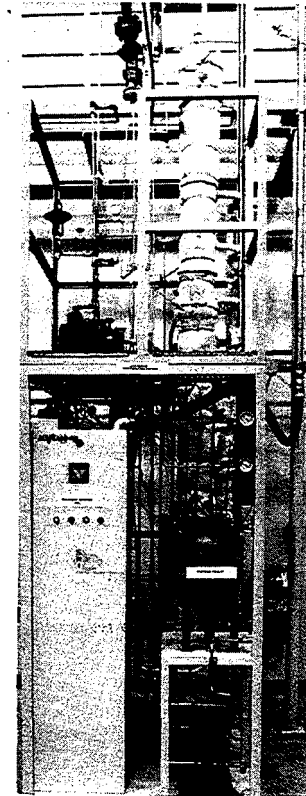


HYDROGENATION UNIT
PROCESS SCHEMATIC

Continuous Fractionation Unit

A pilot scale Continuous Distillation Apparatus is available for research projects with 1- to 5-day run times. The distillation equipment was funded by the U.S. Navy Air Propulsion Center in cooperation with the U.S. Army Belvoir Research and Development & Experimental Center. The facility is housed in the Synthetic Fuel Center on the grounds at SwRI and includes all tankage lines, pumps, heat exchangers, and automatic controls for independent operation. The column has the capacity to fractionate approximately 120 gal/day of distillable feed, producing overhead products in the range of 10% to 90% of the feed, with the remainder as bottoms product. The column is also equipped for vacuum distillation. Column specifications are:

Column Type:	Continuous w/removable packing
Pressure Range:	0.2 - 14.7 psi
Temp. Range:	150° to 600°F (900°F under vacuum)
Feed Rate:	Nominally 5 gal/hr
Overhead Product:	10% to 90% of feed
Reflux Ratio:	Variable
Theoretical Plates:	10-40 (depending on operating conditions, packing)



Continuous Distillation System

The distillation system is designed for unattended fractionation of feedstocks over the range of operating conditions listed above. Process control and data acquisition is through a dedicated microcomputer system linked directly to the process. A sophisticated safety system is part of the operating program and contains dissimilar alarm logic to provide, on one level, troubleshooting actions, and on a higher level, controlled system shutdown. Feed enters the column via a preheater through any of five ports. Light product is condensed overhead and directed back to the column as reflux or to the overhead product receiver. Bottoms product is drawn from the reboiler at the bottom of the column as the level in the reboiler rises.

Program Title: Storage, Processing, Inspection, and Analysis of Petroleum Products Including Unfinished Fuels, Blends, and Synfuels

Sponsor: U.S. Department of Energy,
Office of Vehicle and Engine Research and Development

Contract No.: DEAC01-84CE-50070

SwRI Project No.: 02-7117

Start/Complete Dates: 7 June 1982 - 1 September 1985

Reports or Publications: Sefer, N.R. and Erwin, J., "Reforming and Hydrotreating of Shale- and Coal-Derived Products for Making Test Fuels," presented at the Windsor Workshop on Alternative Fuels, Energy, Mines, and Resources, Canada, June 24-26, 1985, Windsor, Ontario.

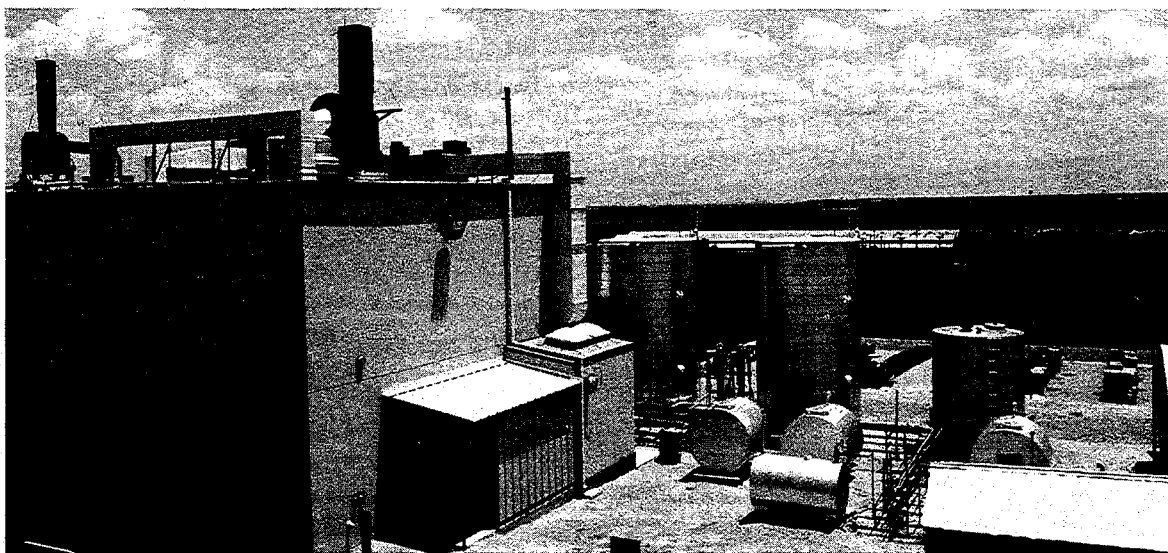
Sefer, N.R., Erwin, J., and Russell, J.A., "Synthetic Fuel Center Construction and Alternative Test Fuels Production," Final Report for Contract DE-AC01-84CE-50070, U.S. Department of Energy Report DOE/CS/50070-1, UC-96, Southwest Research Institute No. SwRI-7117/1, September 1985.

PROGRAM SYNOPSIS

Technical Objectives: The Synthetic Fuel Center was established by the Department of Energy as part of the Alternative Fuels Utilization Program. The main function was to provide test fuels in 5- to 500-gallon quantities for research projects on the utilization of alternative fuels.

Approach: Each test fuel required unique study and preparation. In all cases, the attempt was made to meet the test fuel experimental property and composition objectives while using stocks and techniques relevant to the current petroleum refining industry.

A hydrogenation pilot plant was installed in the new laboratory building shown below for handling synthetic feedstocks from oil shale and coal. Moderate-severity upgrading of shale oil was carried out, and the unit



02-MS-09

is capable of intermediate to high severity processing of shale oil and coal liquids. Catalytic reforming of shale-derived naphthas at low pressure raised the octane of these paraffinic materials from less than 50 to above 90 Research Octane Number. Other processing capabilities include distillation, adsorption, filtration, and centrifuging. Most test fuels required a blending step which was performed by rigorous technique.

Storage tanks from 500- to 10,000-gallon capacity were installed. These are connected through piping and a manifold to the processing unit and other tanks for storage or blending. Fuel blending to target properties or compositions was a major activity. Complete characterizations were made of all feedstocks and products.

Accomplishments: In the three-year report period, 26 fuels were prepared for 11 projects. Quantities ranged from 50 to 200-gallons of each fuel; the total production was 2,490 gallons. Starting materials for processing or blending included two shale oils, two shale-derived naphthas, and two coal-derived middle distillates. The table below lists the test fuels produced.

Amount, Gallons	Type	Project Destination	Characteristics	Processing Description
50 50 50 50	Diesel Fuel	Wisc. & Purdue	1-ring 42.6CN 31.2CN 2-ring 41.1CN 30.1CN	Blend of specification jet fuel and aromatic solvents selected by hydrocarbon type and blended to a target aromatic concentration.
110	Coal-Derived Diesel	Ricardo, Ltd.	Caustic extracted to reduce phenol in the SRC-II	Simulated coal-derived diesel fuel made from SRC-II middle distillate that was extracted with caustic to remove phenolic compounds.
110	Partially Upgraded	Ricardo, Ltd.	Suntech distilled residual from Air Force project	Diesel fuel made from partially upgraded (mild hydrotreating) shale oil distilled to diesel boiling range.
150 150 150	"Broadcut" D-2 SR Naphtha	MTI, Inc.	Blend to composition	Blended test fuel to give extended boiling range resembling a broad distillation cut from crude oil.
150 100	Gasoline No. 1 Shale 62V%	Univ. of Miami So. Illinois Univ.	Match unleaded Base Gasoline from Phillips	Distill shale-derived naphtha from Caribou. Cat reform 47 RON overhead cut to 91 RON. Blend to gasoline specs with alkylate plus butane.
150 100	Gasoline No. 2 Shale 52V%	Univ. of Miami So Illinois Univ.	Blend to match Gasoline No. 1 properties with controlled composition related to base gasoline	Similar to above with different shale naphtha from Caribou. Blend 90 RON reformat with different petroleum stocks.
200 200	Turbine fuel Turbine Fuel	Purdue Univ.	27.6 1-ring, 27.6 2-ring aromatic	Procure JP-7 base stock (2% aromatics) plus 1-ring and 2-ring aromatics concentrates to blend.
80 80 80 80	Diesel Fuel	Univ. of Wisc.	Volatility High High Low Low Cetane High Low Low High	Assign quantitative values to low and high volatility and cetane. Devise blending approach, find suitable stocks, purchase, test and blend.
1200	Diesel Boiling Range	Multiple	Caustic extraction of phenolic compounds from EDS	Subcontract to Merichem in Houston. Transfer product from tank car at Kelly AFB and transport to and from Houston.
150 50 50 50 50 50	Gas Turbine Fuel	NASA-Lewis	Phillips D-2 Base Fuel EDS Extracted EDS/D-2 Blend Canadian 1990 DF-2 Unleaded Gasoline Methanol	Order for direct shipment Ship from inventory Blend and ship Ship from inventory Buy and ship Buy and ship
50	Diesel Fuel	SwRI	High sulfur feed	Activate nickel-moly catalyst, practice hydrotreating at high pressure.
100	Diesel Fuel	AFLRL	Improved stability and engine deposits	Hydrotreat Oxy shale and to reduce nitrogen, sulfur and olefin contents.

Program Title: Synthetic Fuel Center Operation

Sponsor: Martin Marietta Energy Systems, Inc., Oak Ridge National Laboratory for U.S. Department of Energy

Contract No.: 86X-22027C

SwRI Project No.: 02-8929

Start/Complete Dates: November 1985/December 1987

Reports or Publications: Sefer, N.R. and Bowden, J.N., "Shale Light Oil as a Diesel Fuel," Western Research Institute, Confab 86, Silver Creek, CO, July 23, 1986.

Sefer, N.R. and Erwin, J., "Synthetic Gasolines and Diesel Fuels From Processing of Shale Oils and Coal Liquids," Society of Automotive Engineers, International Fuels and Lubricants Meeting, Transactions, SAE Paper No. 861542, Philadelphia, PA, October 1986.

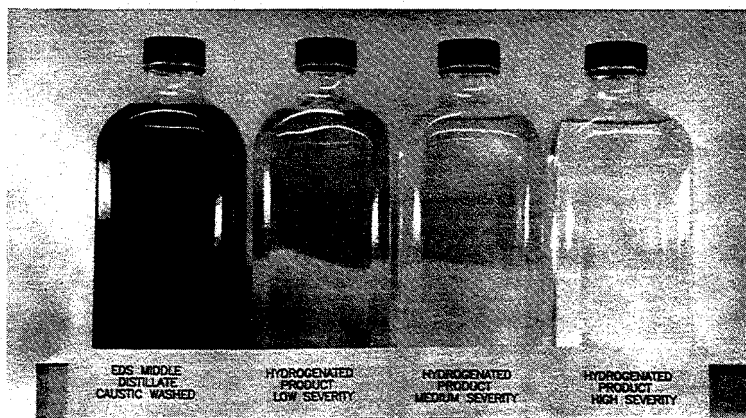
Erwin, J., Sefer, N.R., and Glavincevski, B., "Production and Analysis of EDS Coal-Derived Middle Distillate Test Fuels From Hydrogenation at Three Levels of Severity," Society of Automotive Engineers, 1987 International Fuels and Lubricants Meeting and Exposition, SAE Paper No. 872038, Toronto, Ontario, Canada, November 2, 1987.

PROGRAM SYNOPSIS

Technical Objectives: This work was the continuation of Contract DEAC01-84CE-50070 in which the Synthetic Fuel Center was established. The previous contract was responsible for 26 test fuels for 11 research projects and totaling 2490 gallons. The enumerated objectives of the statement of work, and special requests during the project, were directed toward supplying the research projects of the Alternative Fuels Utilization Project (AFUP) with test fuels having defined composition or properties. At other times, test fuels were made to conform to assessments of future fuels. In addition, full characterization of the test fuels and archiving of AFUP information was accomplished.

Approach: Test fuels were made from sources including shale, coal, and petroleum stocks. Specific fuel property problems were relieved or desired compositions obtained by a combination of blending and processing. The primary processing operation was catalytic hydrogenation, which was augmented by distillation, stripping, filtration, and other unit operations. At all times relevance to refinery practice and similarity to realistic fuel properties were observed.

Accomplishments: The test fuels made during the second contract segment of 2 years duration were more complex than in the first 3-year period and required more processing. Often multiple property adjustments were made, as for example, in the series of hydrogenated products made from EDS coal liquid shown in the photograph below. In all, 26 test fuels were prepared for 10 projects resulting in about 2010 gallons of fuel. The entire list of test fuels is given in the table on the reverse side. Many observations of product properties and processing conditions were made and reported during two contractor-coordination meetings and a fuels roundtable.



Hydrogenated products showing varying degrees of severity

02-MS-17

Amount, Gallons	Type	Project Destination	Characteristics	Source	Description	
50	Turbine fuel	NASA-Lewis	11.5 M% hydrogen	Caustic treated EDS middle distillate	Low severity hydrogenation to increase hydrogen content.	
52	Diesel fuel blend	Pennsylvania State Univ.	50 vol% EDS/50 vol% D-2	Untreated EDS middle distillate	Blended to composition for 35 cetane number	
			<u>Unleaded Premium</u>			
30	Low aromatics gasoline	Univ. of Tennessee	11.0 vol% aromatics	Petroleum stocks	Blended to range of aromatics with matched RVP and octanes	
30	Medium aromatics gasoline	Univ. of Tennessee	24.5 vol% aromatics			
30	High aromatics gasoline	Univ. of Tennessee	34.0 vol% aromatics			
100	Coal-derived gasoline #1	Southern Illinois Univ.	Unleaded regular with coal-derived reformat and petroleum stocks	SRC-II naphtha	Processed coal naphtha and blended to specifications	
140	Coal-derived gasoline #1	Univ. of Miami				
100	Coal-derived gasoline #2	Southern Illinois Univ.	Unleaded regular with coal-derived reformat and petroleum including cat cracked gasoline	SRC-II naphtha	Processed coal naphtha and blended to specifications	
150	Coal-derived gasoline #2	Univ. of Miami				
53	Diesel fuel blend	Pennsylvania State Univ.	44 vol% EDS/56 vol% D-2	Caustic treated EDS middle distillate	Blended to match 35 cetane number of 50/50 blend	
30	Coal-derived gasoline	Univ. of Tennessee	Unleaded premium with 24.3 vol% aromatics	SRC-II naphtha and petroleum stocks	Processed coal naphtha and blended to match medium aromatics	
28	EDS product #1 (hydrotreated)	Pennsylvania State Univ.	38.2 cetane number	Caustic treated EDS middle distillate	High severity hydrogenation to increase cetane number	
50	Shale diesel fuel	SwRI Division 03	Partially upgraded	Suntech/USAF	Diesel fraction distilled from mild hydrotreating of shale oil	
100 5	Canadian 1990 diesel	SwRI Division 03 and Michigan Tech. Univ.	28 vol% tar sand stocks	Tar sands & petroleum	Blended by Canadian National Research Council	
160 5	Diesel fuel blend	SwRI Division 03 and Michigan Tech. Univ.	57 vol% EDS/43 vol% D-2	Caustic treated EDS	Blended to 33 cetane number	
			<u>Hydrogen, M%</u>	<u>Aromatics, Vol%</u>		
50	Coal-derived diesel fuel	Pennsylvania State Univ. 18 gal. each severity	11.9	45.5	Caustic treated EDS	Series of fuels hydrogenated at three severity levels
50			12.7	21.6		
50			13.1	10.2		
156 20 156 20 156 20	Alternative diesel test fuels	Ford Motor Co. and Rutgers University	Base fuel	Petroleum stocks from Phillips Petroleum	Diesel control fuel	
			Alternative fuel #2		Light cycle oil	
			Alternative fuel #1		Equal parts D 2/LCO	
47 52 53	Shale-derived test fuel Series - diesel boiling range	Not assigned	250 ppm nitrogen 730 ppm 1890 ppm	Caribou distillate shale crude (12300 ppm nitrogen)	Reduce nitrogen content for stable products	
10 57	Shale Naphtha Shale Diesel Oil	Not assigned	140°-336°F Distillation 396°-599°F Distillation	Indirect-heated Paraho Shale Oil	Controlled-severity hydrogenation of shale oil plus distillation	

Exhibit 3. Bibliography for Low-Emission/Low-Reactivity Test Fuels

Bibliography for Low-Emission/Low-Reactivity Test Fuels

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13. ABSTRACT (<i>Maximum 200 words</i>) The Alternative Fuels Utilization Program (AFUP) of the Office of Energy Efficiency and Renewable Energy has investigated the possibilities and limitations of expanded scope of fuel alternatives and replacement means for transportation fuels from alternative sources. Under the AFUP, the Alternative Fuel Center (AFC) was created to solve problems in the DOE programs that were grappling with the utilization of shale oil and coal liquids for transportation fuels. This report covers the first year of the 3-year contract. The principal objective was to assist the AFUP in accomplishing its general goals with two new fuel initiatives selected for tasks in the project year: (1) Production of low-sulfur, low-olefin catalytically cracked gasoline blendstock (2) Production of low-reactivity/low-emission gasoline. Supporting goals included maintaining equipment in good working order, performing reformulated gasoline tests, and meeting the needs of other government agencies and industries for fuel research involving custom processing, blending, or analysis of experimental fuels.			
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