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FUEL COMPONENT AND HETEROATOM EFFECTS ON DEPOSITS AND WEAR

INTERIM REPORT
BFLRF No. 190

By

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Southwest Research Institute
San Antonio, Texas**

Under Contract to

**U.S. Army Belvoir Research
and Development Center
Materials, Fuels and Lubricants Laboratory
Fort Belvoir, Virginia**

Contract No. DAAK70-85-C-0007

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December 1985

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REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified		1b. RESTRICTIVE MARKINGS None	
2a. SECURITY CLASSIFICATION AUTHORITY N/A		3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited	
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE			
4. PERFORMING ORGANIZATION REPORT NUMBER(S) Interim Report BFLRF No. 190		5. MONITORING ORGANIZATION REPORT NUMBER(S)	
6a. NAME OF PERFORMING ORGANIZATION Belvoir Fuels and Lubricants Research Facility (SwRI)	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION	
6c. ADDRESS (City, State, and ZIP Code) Southwest Research Institute P.O. Drawer 28510 San Antonio, TX 78284		7b. ADDRESS (City, State, and ZIP Code)	
8a. NAME OF FUNDING/SPONSORING ORGANIZATION Belvoir Research and Development Center	8b. OFFICE SYMBOL (If applicable) STRBE-VF	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER DAAK70-82-C-0001; WD 6 DAAK70-85-C-0007; WD 6	
8c. ADDRESS (City, State, and ZIP Code) Fort Belvoir, VA 22060		10. SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO.	PROJECT NO.
		TASK NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) Fuel Component and Heteroatom Effects on Deposits and Wear (U)			
12. PERSONAL AUTHOR(S) Frame, E.A.			
13a. TYPE OF REPORT Interim	13b. TIME COVERED FROM 01 Oct 81 to 30 Sept 84	14. DATE OF REPORT (Year, Month, Day) 1985 December	15. PAGE COUNT 38
16. SUPPLEMENTARY NOTATION			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	
		Diesel fuel	
		Fuel sulfur	
		Diesel engine	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The objective of this project was to investigate the effects of fuel heteroatom compounds on diesel engine performance, particularly engine deposition and wear. This work involved operating a single-cylinder diesel research engine using a highly controlled base fuel (JP-7) which was extremely low in sulfur and nitrogen. The engine lubricant was a synthetic polyalphaolefin material, also free of sulfur and nitrogen. Thus, wear and deposit effects observed during the 60-hour single-cylinder laboratory engine test were directly related to the heteroatom compound added to the base fuel. Through these controlled laboratory engine tests, wear and deposit effects were studied using four different types of known fuel sulfur complexes, and two different fuels containing naturally occurring sulfur compounds. Fuel sulfur type did not appear to directly influence wear rate, whereas the disulfide-type sulfur was found to produce high piston deposit tendencies. No increase in engine wear was observed for three fuels each of which contain a different known nitro- (Continued)			
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL Mr. F.W. Schaeckel		22b. TELEPHONE (Include Area Code) (703) 664-3576	22c. OFFICE SYMBOL STRBE-VF

19. ABSTRACT - Continued

gen compound. Fuel injector deposits and fouling occurred with some of the nitrogen-containing fuels. A fuel containing a combination of both known sulfur and nitrogen heteroatoms was tested and no synergistic effects on wear or deposits were observed.

Keywords: Diesel Fuel

FOREWORD

This report was prepared at the Belvoir Fuels and Lubricants Research Facility (SwRI) (formerly U.S. Army Fuels and Lubricants Research Laboratory) located at Southwest Research Institute, San Antonio, TX, under Contract Nos. DAAK70-82-C-0001 and DAAK70-85-C-0007, for the period 1 October 1981 through 30 September 1984. Work was funded by the U.S. Army Belvoir Research and Development Center, Ft. Belvoir, VA, with Mr. F.W. Schaekel (STRBE-VF) serving as contracting officer's representative. Project technical monitor was Mr. M.E. LePera, STRBE-VF.

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ACKNOWLEDGMENTS

The author acknowledges the support of the Belvoir Fuels and Lubricants Research Facility (SwRI) engine and chemistry laboratory personnel, particularly Mr. R. Pena for his attention to detail in blending the test fuels and conducting the engine tests; and, to those staff members who made helpful suggestions in conduct of this work and provided review of this report.

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I. INTRODUCTION/BACKGROUND

Fuel for use in U.S. Military ground vehicle mobility operations is procured against Federal specification VV-F-800C, "Fuel Oil, Diesel".^{(1)*} This specification controls fuel sulfur content to 0.5 wt% maximum in the continental United States (CONUS) and allows fuel sulfur content of 0.7 wt% maximum outside CONUS (OCONUS). In the future, diesel fuel sulfur levels are expected to increase, especially in OCONUS areas. Increasing Department of Defense (DOD) emphasis on utilizing mobility fuels refined from high sulfur and nonconventional crude resources makes it mandatory to investigate the effects of heteroatom components on compression ignition engine deposits and wear. Heteroatom compounds which individually contain sulfur, nitrogen, and oxygen are of principal concern. Fuel sulfur, the primary heteroatom type found in finished petroleum fuels, has been identified as the main contributor to engine performance degradation, as illustrated in the following review of the literature.

During the 1940's, several researchers reported on the detrimental effects of sulfur compounds in diesel fuel. Cloud and Blackwood (1943) used both cyclic and steady-state 80-hour engine test procedures to determine the effects of diesel fuel sulfur content on deposits and wear.⁽²⁾ They reported that an increase in fuel sulfur from 0.2 to 1.0 wt% resulted in a two to sixfold increase in measured piston ring wear and a two to fourfold increase in cylinder bore wear. A 40 to 80 percent increase in ring zone deposits was observed, as well as increased ring sticking. Cloud and Blackwood concluded that fuel sulfur type was relatively unimportant as fuels containing naturally occurring and added sulfur (carbon disulfide and diamyl trisulfide) produced about the same level of engine distress. Increased wear and fouling were also caused by the addition of small amounts of SO_2 to the intake air of a fired engine. Addition of SO_3 to the intake air of a motored engine caused dramatic increases in ring wear and deposits. Finally, they reported that 60 to 90 percent of the fuel sulfur was converted to SO_3 during the combustion process. In 1947, Moore and Kent determined the effect of nitrogen, sulfur, and naphthenic acids on single-cylinder diesel engine (Caterpillar) wear by using crankcase iron content as an indication of wear.⁽³⁾

*Underscored numbers in parentheses refer to the list of references at the end of this report.

No increase in iron wear metals was observed for fuels containing 0.17 percent nitrogen or naphthenic acids (fuel TAN = 0.3). Fuels containing natural sulfur (0.7 wt%) and sulfur added as thiophene (0.7 percent S) produced a four to fivefold increase in iron wear metals. A fuel with 1.3 percent S present as thiophene gave a sevenfold increase in iron wear metals. They also reported that reducing engine coolant temperature from 160°F (71°C) to 100°F (37°C) caused a fourfold increase in wear. Also in 1947, Blanc of Caterpillar Tractor Co. reported that fuel nitrogen content (0.08 wt%) had no effect on either engine wear or deposits (4), but experiments in a single-cylinder Caterpillar engine showed that as fuel sulfur content increases, ring and cylinder bore wear (top) and piston deposits increase. Blanc used both naturally occurring sulfur and added sulfur as thiophene, dodecyl mercaptan, and diamyl sulfide. He concluded that different types of fuel sulfur compounds may show differences in wear and deposits; however, the differences are small. Finally, Blanc reported that distillation range of high sulfur fuels was found to affect deposits somewhat, but not to the extent that fuel sulfur content impacted on deposits.

In 1948, Gadebusch reported that fuel sulfur content alone is not satisfactory for predicting engine deposits. He found that a fuel blend of straight run and catalytically cracked material which contained 0.6 wt% S gave more deposits than a straight run fuel with a sulfur content of 1.15 wt%. (5) Cattaneo and Starkman (1948) reported that ring wear increased threefold in going from zero to 1.0 wt% fuel sulfur and that basic material in the engine oil significantly reduced the wear. (6) Furstoss (1949) investigated field experience involving small-bore medium-speed diesels using high-sulfur fuel and reported that operation on fuel with greater than 0.5 wt% S resulted in abnormal upper cylinder and ring wear with increased engine deposits. (7) Also in 1949, Broeze reported that cylinder bore wear increased twofold and ring wear increased threefold when fuel sulfur was increased from 0.08 to 1.5 wt%. (8) In experiments with a Pyrex window in the combustion chamber, Broeze observed that increased fuel sulfur content caused increased lacquer deposits. Because of anomalous wear and deposition behavior of some sulfur-containing compounds, Broeze questioned the theory that fuel sulfur type had no influence on engine wear and deposits.

After the excellent research of the 1940's, very little information was published on high-sulfur fuel usage effects in high-speed diesel engines. Not until 1974 did Perry and Anderson of the U.S. Navy investigate the effects of increasing the sulfur content of diesel fuel marine (DFM).(9) They found during 1000-hour tests that in going from 1.0 to 1.3 wt% fuel S (all naturally occurring), top compression ring wear increased by a factor of 2.5, and more ring sticking occurred in both two- and four-cycle diesel engines. U.S. Army research on high-sulfur fuel utilization was reported by Lestz, LePera, and Bowen in 1976.(10) Using a cyclic operating procedure in an aluminum block two-cycle diesel engine, they found severe increases in fire ring (1.4 to sixfold) and bore wear (zero to threefold) when comparing reference fuel (0.4 percent S) with fuels containing 0.64 and 1.2 wt% naturally occurring sulfur. Higher lubricant ash content helped in controlling fire ring and bore wear; however, more ring sticking occurred with the higher ash oil. In this work, greater engine distress was consistently observed with the 0.64 wt% S fuel than with the 1.2 wt% S fuel. This greater distress led the authors to speculate that other fuel components present such as olefinic compounds, oxygenated compounds, naphthenic acids, and pyrrole nitrogen were contributing to the increased wear. In 1978, Frame reported that in going from 0.4 to 1.0 wt% natural occurring fuel sulfur, fire ring wear increased fourfold and liner scuffing increased five to tenfold in a two-cycle diesel engine.(11) No change in engine deposits accompanied the fuel sulfur increase in this work.

Gergel (1980) reported on modified Cat 1-G2 tests run with fuel containing 1.4 wt% sulfur, with the additional sulfur added as tertiary butyl disulfide.(12) In these tests, which were run without the standard oil drains, top piston groove deposit filling remained the same while weighted total piston deposit rating (WTD) increased threefold, and top ring wear, as determined by weight loss, increased twenty-four fold when the high sulfur fuel was used. Recent work on fuel sulfur effects has been reported by McGeehan. In 1982 McGeehan found, as Gergel had earlier, that fuel sulfur content had very little effect on high temperatures (200° to 260°C) piston top groove deposits in a single-cylinder turbocharged Caterpillar engine.(13) In these experiments, fuel sulfur content was increased by adding tertiary butyl

disulfide to the base fuel. Total piston deposits increased overall with the higher sulfur fuel due to increased lower area piston deposits at temperatures of 120° to 190°C. Finally, in 1983 McGeehan published results of research covering the effects of fuel sulfur content on diesel engine bore polishing.⁽¹⁴⁾ He found in going from 0.2 to 1.0 percent fuel sulfur, bore polishing increased two to threefold in the Mack T-6 600-hour test, and sixfold in the 200-hour Ford Tornado test. This completes the literature review relating to the effects of fuel heteroatom compounds on high-speed diesel engine performance.

In summary, fuel sulfur content has been shown to be directly related to engine wear. While many factors such as operating temperature and lubricant quality impact on engine wear, the effect of fuel sulfur content on engine wear is very large. In general, for each additional one percent of fuel sulfur content (e.g., 0.3 to 1.3 percent S), ring and cylinder bore wear increased approximately eightfold and fourfold, respectively. Also, increased fuel sulfur content generally led to additional piston deposits and often even to ring-sticking. There is disagreement in the literature as to whether the type of sulfur compound (i.e. natural occurring or added) has an impact on wear and deposits.

The effects of nitrogen heteroatom compounds in the fuel on engine wear and deposits have not been as thoroughly documented. At relatively low fuel nitrogen contents (<0.2 wt%), no effects were observed.

II. OBJECTIVES

The objectives of this research project were to provide a clear definition of the effects of fuel heteroatom compounds on diesel engine condition and to investigate the mechanisms through which known fuel heteroatom compounds affect engine deposition and wear. An additional objective was to resolve areas of disagreement concerning heteroatom effects on engine performance which were found in the literature.

III. EXPERIMENTAL APPROACH

An engine-based approach was used in this research. It involved operating a single-cylinder research diesel engine on fuel containing a known heteroatom compound and determining the effects of the known compound on engine wear and deposits. To isolate the effect of the heteroatom compound added to the base fuel, it was necessary to use a base fuel and engine lubricant which were free of sulfur and nitrogen. A highly controlled base fuel which met U.S. Air Force specification MIL-T-38219, JP-7, (15) and was extremely low in nitrogen and sulfur content was used throughout the program. Typical analyses of the JP-7 base fuel, as shown in Table 1, reveal that it is a highly refined, low aromatic fuel with a fairly narrow boiling range. The engine lubricant used throughout the program was a 6 cSt polyalphaolefin base stock which contained no nitrogen or sulfur. The properties of this additive-free lubricant are given in Table 2.

TABLE 1. BASE FUEL PROPERTIES
JP-7, AL-11417

Property	Test Method	Value	Requirements MIL-T-38219
Gravity, API°	D 287	44.9	44-50
Viscosity, cSt, at 40°C	D 445	1.65	NR***
Flash Point, °C	D 93	72	60 min
Distillation, °C	D 86		
IBP		196	182 min
10%		206	196 min
50%		215	Report
90%		237	260 max
EP		269	288 max
Sulfur, wt%	D 1266	0.004	0.1 max
Nitrogen, wt%	CLM*	<0.001	NR
Cetane Number	D 613	54	NR
Hydrocarbon Type, vol%	D 1319		
Saturates		96	NR
Olefins		0	NR
Aromatics		4	5 max
Aromaticity, wt% Ring Carbon	UV**		NR
mono-		1.12	
di-		0.11	
tri-		0.01	

* CLM = Chemiluminescent Method

** UV = Ultraviolet Spectroscopy

***NR = No Requirement

TABLE 2. LUBRICANT PROPERTIES
6 cSt PAO, AL-11474

Property	Test Method	Value
Gravity, API°	D 287	40.6
Viscosity, cSt, at 40°C	D 445	30.89
at 100°C	D 445	5.84
Viscosity Index	D 2270	135
TAN, mg KOH/g	D 664	0.0
TBN, mg KOH/g	D 664	0.0
Flash Point, °C	D 92	240
Elemental Content, wt%	XRF*	
Ca		<0.01
Zn		<0.01
P		<0.01
S		<0.01
N	CLM**	<0.001

* XRF = X-Ray Fluorescence

** CLM - Chemiluminescent Method

It was desired to use a small diesel engine in this program. Petter model PH1W was selected based on its availability at BFLRF and because it had the following properties: single-cylinder, four-cycle, direct injection, water-cooled diesel. Petter PH1W engine characteristics are given in Table 3, and a photograph of the engine is presented in Figure 1. Prior to each experiment, the liner was honed and the piston cleaned to remove deposits and the

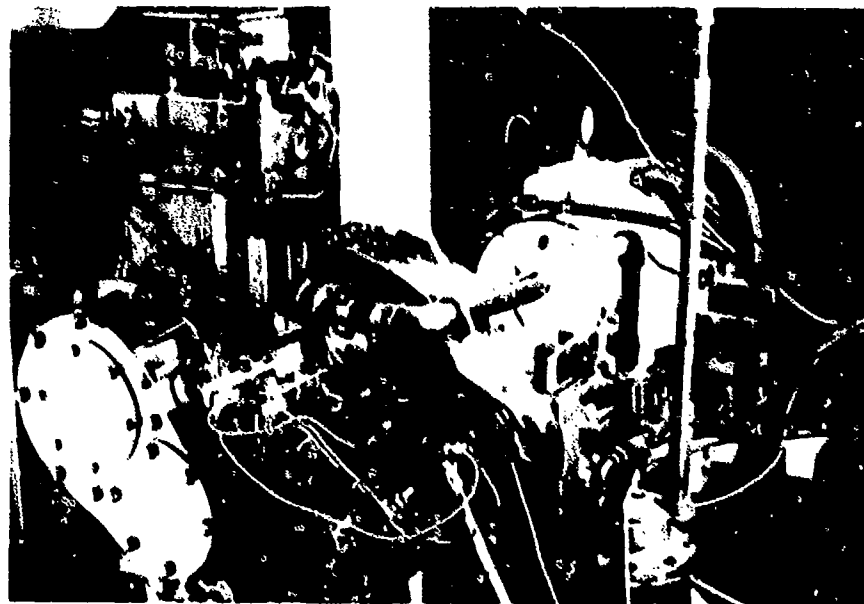


FIGURE 1. PETTER MODEL PH1W ENGINE

TABLE 3. PETTER PH1W ENGINE CHARACTERISTICS

Displacement	659 cm ³ (40.2 CID)
Bore and Stroke	87.3 mm x 110 mm
Compression Ratio	16.5:1
Piston	Aluminum
Piston Rings	3 Rectangular Compression Rings, 1 oil control
Oil Capacity	2.84 L (no oil filter used)

engine was rebuilt with new piston rings. A new piston and liner were installed after every five to eight tests. Following a short break-in on a test fuel, the engine oil was changed and the test was started. The test procedure consisted of 60 hours of steady-state operation accumulated 8 hours per day for 7.5 days with a 16-hour overnight shutdown each day. Typical engine operating conditions are given in Table 4. Engine oil level was adjusted to full at the end of each day, and a small used oil sample was drawn for wear metals analysis.

After each test, the engine was disassembled. Then ring wear was measured, and a standard CRC weighted total deposit piston rating was performed. Used engine oil was analyzed for degradation and contamination buildup using standard ASTM tests. Deposits were washed from the piston and intake valve using heptane and then dimethylformamide (DMF). Heptane and DMF soluble deposits were recovered using a vacuum rotary evaporator to remove the sol-

TABLE 4. TYPICAL OPERATING CONDITIONS
FOR 60-HOUR PETTER TEST

Engine Speed, RPM	1800
Load, lb-ft	28 to 29
Bhp, observed	9.6 to 9.8
Oil Temp, °C	74 to 78
Coolant In, Temp. °C	80
Coolant Out, Temp. °C	82 to 83
Exhaust Temp. °C	550 to 575
BSFC, lb/BHp hr	0.387±0.005
Piston Temperatures*	
Top Land (thrust and antithrust)	182°C
Skirt Top (thrust and antithrust)	131°C

* Determined by temperature-sensitive plugs

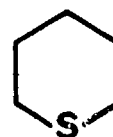
vent. The collected deposits were then analyzed for composition to determine if the heteroatom compound added to the fuel was affecting piston deposits.

IV. FUEL SULFUR EFFECTS

The first issue investigated was to determine if fuel sulfur type impacts on compression ignition engine wear and deposits. Sulfur may be present in petroleum-based fuels in a variety of chemical compound types.

Some representative sulfur compound type structures are shown below:

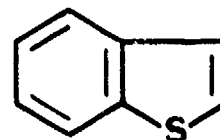
- Alkyl thiol (mercaptan) R-SH
- Aryl thiol (mercaptan) Ar-SH
- Alkyl sulfide R-S-R
- Aliphatic cyclic sulfide



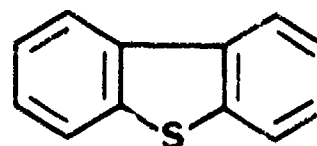
- Alkyl disulfide R-S-S-R
- Thiophene



- Benzothiophene



- Dibenzothiophene



Bailey surveyed the literature to determine the types and concentrations of sulfur compounds which have been identified in crude oil and the diesel fuel boiling range.(16) Bailey concluded that predominant species were cyclic aliphatic sulfides, aromatic sulfides, including thiophenes, benzothiophenes, and dibenzothiophenes. Thiols, straight-chain sulfides, and disulfides are probably more prevalent in the lower end of the diesel boiling range.

Model test fuels were blended to contain 1 wt% sulfur as a known compound. The number of sulfur compound types which could be investigated was somewhat limited because the 60-hour test procedure used approximately 300 pounds of

fuel blend. To obtain 1 wt% fuel sulfur required from 8 to 20 pounds of sulfur compound. Thus, each sulfur compound for investigation had to be available in fairly large quantities at reasonable cost.

A. Sulfur Types at 1 Wt% Fuel S

Each of the following sulfur types was used to raise the sulfur content of JP-7 to 1 percent:

- tert butyldisulfide (TBDS)
- n-butyldisulfide (C4S)
- n-octylmercaptan (C8SH)
- n-dodecylmercaptan (C11SH)
- thiophene (TPH)

These fuels were evaluated in the 60-hour Petter test procedure, with the results summarized in Tables 5, 6, and 7. Cylinder liner wear was negligible for all tests. Thus engine wear was evaluated based on used oil iron content and measured piston ring end gap change. The baseline JP-7 tests (average of 3) resulted in low measured engine ring wear and low used oil iron content (56 ppm, Std dev = 16). Piston deposits were fairly light with a WTD of 151 (Std dev = 18) and a piston lacquer demerit of 3.2 (10 = Dirty). The TBDS fuel (1 wt% S) was used as a high sulfur fuel baseline (7 tests). Overall test repeatability was only fair for used oil iron content (Avg = 130, Std dev = 37), and piston WTD (Avg = 216, Std dev = 55). Each of the fuels which contained a known sulfur compound (1 wt% S) produced at least twice as much overall engine wear as compared to neat JP-7. Within the test repeatability, wear severity of individual fuel sulfur compounds was judged as approximately equivalent. Piston cleanliness, as determined by WTD, tended to be degraded for the fuels containing TBDS and C8SH, while the other sulfur compounds investigated had no major effect on piston cleanliness.

The analyses of the recovered piston deposits for this series of tests are presented in Table 6. Based on carbon/hydrogen ratio, the heptane-soluble fraction was primarily residual lube oil. A slight amount of fuel-related deposit was detected in the heptane-soluble fraction as determined by sulfur content.

TABLE 5. PETER ENGINE TESTS - 1% FUEL S

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	JP-7+1% S as n-butyl sulfide	JP-7+1% S as n-octyl mercaptan	JP-7+1% S as n-dodecyl mercaptan	JP-7+1% S as thiophene
<u>Avg. Operating Conditions</u>						
Test Hours	60	60	60	60	60	60
RPM	1800	1805	1805	1803	1803	1804
Load, lb-ft	29	28	29	29	30	28
Bhp	9.8	9.6	9.8	9.8	10.1	9.7
Oil Temp, °C	79.1	76.3	75.0	77.2	77.2	75.6
Exhaust Temp, °C	605	565	539	612	576	611
Oil Consumption, lb/hr	0.228	0.226	0.346	0.237	0.245	0.278
<u>Results</u>						
Used Oil Fe, ppm	56	130	104	142	110	106
Ring End Gap Change, in.						
Top	0.001	0.005	0.002	0.001	0.003	0.006
2	0.001	0.003	0.002	0.006	0.001	0.002
3	0.003	0.006	0.002	0.009	0.003	0.008
Piston WTD Rating	151	216	167	210	152	169
Piston Lacquer Demerit	3.2	5.5	5.2	3.3	3.5	5.2
Ring Sticking	None	None	None	None	None	None

TABLE 6. PETTER PISTON DEPOSIT COMPOSITION - 1% FUEL S

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	JP-7+1% S as n-butyl sulfide	JP-7+1% S as n-octyl mercaptan	JP-7+1% S as n-dodecyl mercaptan	JP-7+1% S as thiophene
Piston/Int Valve Deposit Analyses						
Heptane-Soluble Deposit, wt %	1.75	2.32	2.78	1.81	2.11	2.31
Composition, wt%						
C	85.4	85.1	85.0	85.0	85.0	85.1
H	14.1	14.3	14.2	14.1	14.3	14.2
N	0.11	0.13	0.12	0.14	0.12	0.51
S	0.12	0.50	0.21	0.30	1.23	0.38
Dimethylformamide (DMF)- Soluble Deposit, wt %	0.74	0.99	1.06	0.57	0.78	0.59
Composition, wt%						
C	67.1	66.7	66.6	57.6	57.1	67.2
H	5.0	5.0	5.2	2.1	6.2	4.0
N	3.9	2.3	3.1	5.7	2.8	1.7
S	0.6	6.1	0.8	3.4	3.9	2.1

TABLE 7. USED OIL PROPERTIES - PETTER ENGINE TESTS - 1% FUEL S

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	JP-7+1% S as n-butyl sulfide	JP-7+1% S as n-octyl mercaptan	JP-7+1% S as n-dodecyl mercaptan	JP-7+1% S as thiophene
Used Oil Analyses						
K. Vis. cSt. at 40°C	34.32	34.73	50.2	34.11	34.50	34.70
TAN	6.67	7.32	12.0	7.54	7.58	7.22
	0.2	0.8	0.9	0.8	0.7	0.6
Insolubles, wt%						
Pentane "A"	0.71	0.85	2.35	0.60	1.18	0.54
Toluene "A"	0.53	0.79	1.08	0.51	1.12	0.49
Pentane "B"	0.69	0.85	2.39	0.59	1.17	0.53
Toluene "B"	0.28	0.48	0.98	0.47	0.58	0.41
Elements						
Fe, ppm	56	130	104	142	110	106
Cu, ppm	15	17	<10	18	<10	24
Pb, ppm	<60	<60	<60	<60	<60	<60
S, wt%	<0.01	0.01	0.03	0.02	0.08	0.01
N, wt%	0.004	0.006	0.010	0.005	0.008	0.004
Differential IR, Oxidation	NIL	NIL	NIL	NIL	NIL	NIL
Absorbance @ 1710 cm ⁻¹						

For tests using 1 percent S fuel, sulfur tended to be concentrated in the DMF-soluble portion of the deposit. The DMF-soluble deposit from tests using fuel with TBDS had the greatest sulfur concentration (6.1 wt%) which was consistent with the most heavily deposited pistons.

The used oil analyses for this series of tests are presented in Table 7. In all tests, except for the one which used fuel containing C4S, viscosity increase was very minimal. The used oil from the C4S test had high insolubles content (2.4 wt%) which contributed to the observed viscosity increase. Since the combustion of fuel sulfur compounds produces sulfur oxides which can combine with water to form sulfur-containing acids, the TAN of the used oil was closely monitored. While the TAN increase for all tests was less than 1.0 during the relatively short 60-hour test procedure, the sulfur-containing fuels still produced a three to fourfold TAN increase as compared to neat JP-7. Over longer duration engine operation, this trend toward higher used oil TAN with high-sulfur fuels would be expected to cause more frequent oil drain requirements. In all tests, the used oil was lightly stressed as negligible oil oxidation and nitration were determined by differential infrared analysis.

B. Sulfur Types at 2 Wt% Fuel S

A limited number of 60-hour Petter engine tests were conducted using model fuels containing 2 wt% fuel sulfur. TBDS, C8SH, and TPH were each used to raise the sulfur content of JP-7 to 2 wt%. The test results are summarized in Tables 8, 9, and 10. Compared to neat JP-7, the fuels with 2 wt% S had approximately a fourfold increase in engine wear as indicated by used oil iron content, see Figure 2. In going from 1 to 2 wt% fuel sulfur, the used oil iron content was slightly less than doubled for TBDS and C8SH and slightly more than doubled for thiophene. The effect of fuel sulfur content on piston WTD is shown in Figure 3. Only a slight increase in WTD (dirtier piston) was observed in going from 1 to 2 wt% fuel S. The used oil analyses for the tests inducted with 2 wt% fuel sulfur showed slightly increased TAN's compared to tests at 1 wt% fuel S. Overall, at 2 wt% fuel S, each of the three different sulfur compounds investigated caused about the same level of engine distress.

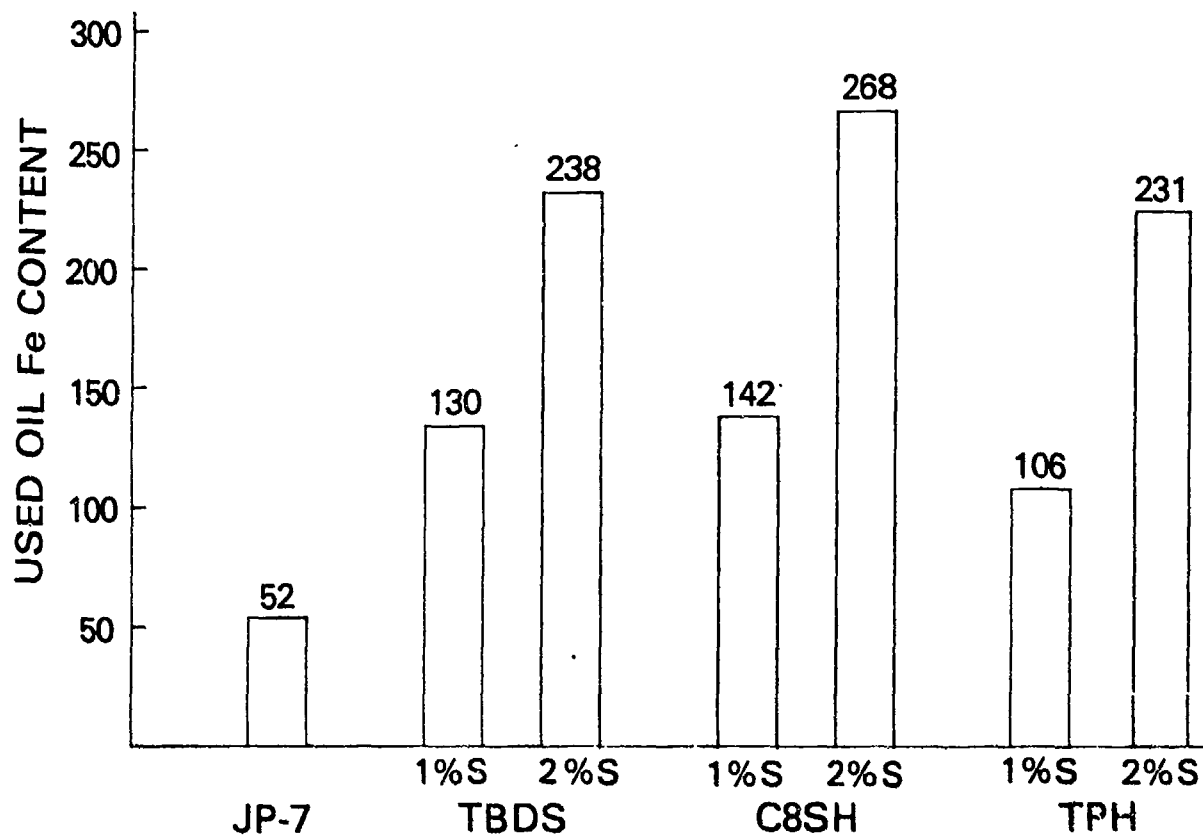


FIGURE 2. EFFECT OF FUEL SULFUR CONTENT ON USED OIL IRON

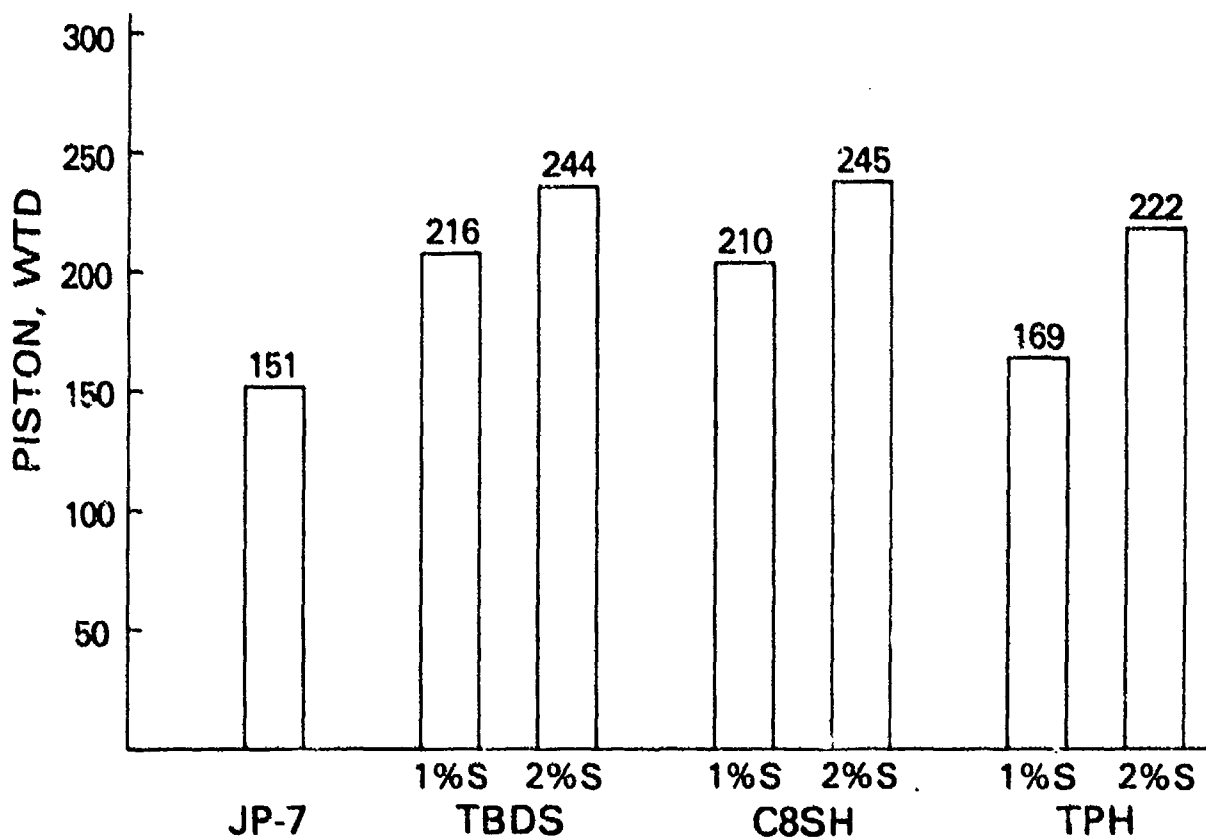


FIGURE 3. EFFECT OF FUEL SULFUR CONTENT ON PISTON WTD

TABLE 8. PETER ENGINE TESTS - 2% FUEL S

Fuel Heteroatom Type	JP-7 None	JP-7+2% S as t-butyl disulfide	JP-7+2% S as n-octyl mercaptan	JP-7+2% S as thiophene
<u>Avg. Operating Conditions</u>				
Test Hours	60	60	60	60
RPM	1800	1806	1804	1805
Load, lb-ft	29	29	30	30
Bhp	9.8	10.1	10.1	
Oil Temp, °C	79.1	77.4	76.7	74.4
Exhaust Temp, °C	605	584	624	621
Oil Consumption, lb/hr	0.228	0.256	0.302	0.346
<u>Results</u>				
Used Oil Fe, ppm	52	238	268	231
Ring End Gap Change, in.				
Top	0.001	0.006	0.002	0.001
2	0.001	0.006	0.008	0.016
3	0.003	0.008	0.009	0.013
Piston WTD Rating	151	244	245	222
Piston Lacquer Demerit	3.2	5.5	4.3	3.5
Ring Sticking	None	None	None	None

TABLE 9. PETER PISTON DEPOSIT COMPOSITION - 2% FUEL S

Fuel Heteroatom Type	JP-7 None	JP-7+2% S as t-butyl disulfide	JP-7+2% S as n-octyl mercaptan	JP-7+2% S as thiophene
<u>Piston/Int Valve Deposit Analyses</u>				
Heptane-Soluble Deposit, wt g	1.75	2.52	2.13	1.63
Composition, wt%				
C	85.4	85.2	85.3	85.1
H	14.1	14.3	14.4	14.4
N	0.11	0.14	0.14	0.42
S	0.12	0.56	1.0	0.17
Dimethylformamide (DMF)- Soluble Deposit, wt g	0.74	1.49	0.92	1.60
Composition, wt%				
C	67.1	58.0	59.7	53.0
H	5.0	5.0	5.9	8.4
N	3.9	5.6	1.1	3.3
S	0.6	3.8	4.1	3.1

TABLE 10. USED OIL PROPERTIES - PETTER ENGINE - 2% FUEL S

Fuel Heteroatom Type	JP-7 None	JP-7+2% S as t-butyl disulfide	JP-7+2% S as n-octyl mercaptan	JP-7+2% S as thiophene
<u>Used Oil Analyses</u>				
K. Vis, cSt, at 40°C	34.32	34.95	37.00	34.95
at 100°C	6.67	7.39	9.30	8.43
TAN	0.2	0.74	1.1	1.4
 Insolubles, wt%				
Pentane "A"	0.71	1.11	1.02	0.81
Toluene "A"	0.53	0.90	0.94	0.73
Pentane "B"	0.69	1.10	1.02	0.81
Toluene "B"	0.28	0.94	0.83	0.61
 Elements				
Fe, ppm	52	238	268	231
Cu, ppm	15	20	23	23
Pb, ppm	<60	<60	<60	<60
S, wt%	<0.01	0.03	0.04	0.02
N, wt%	0.004	0.005	0.007	0.006
Differential IR, Oxidation	NIL	NIL	NIL	NIL
Absorbance @ 1710 cm ⁻¹				

C. Naturally Occurring Versus Added Sulfur Compounds

Two 60-hour Petter engine tests were run using fuels containing 1 wt% naturally occurring sulfur compounds. The properties of AL-12157, a full boiling range DF-2 fuel, are shown in Table 11. The other test fuel was a 50/50 vol% blend of JP-7 and light cycle oil. The properties of light cycle oil (AL-12174) are shown in Table 12.

The results of these tests are summarized in Tables 13, 14, and 15. Compared to tests with TBDS at 1 wt% fuel S, the tests using AL-12157 and the blend (JP-7/cycle oil) had approximately the same engine wear as indicated by used oil iron content. However, piston deposits (WTD) with the natural sulfur

TABLE 11. TEST FUEL PROPERTIES AL-12157

Gravity, API°	30.3
K. Vis, cSt, 100°F, (38°C)*	3.12
Sulfur, wt%	0.997
Flash Point, °F (°C)	174 (79)
Pour Point, °F (°C)	15 (-9)
Cloud Point, °F (°C)	18 (-8)
Calc. Cetane No.	42.5
Distillation, °F (°C)	
IBP	386 (197)
5%	422 (217)
10%	438 (226)
20%	466 (241)
50%	536 (280)
70%	573 (301)
90%	625 (329)
95%	652 (344)
EP	680 (360)

*Viscosity data from fuel supplier was determined at 100°F

TABLE 12. LIGHT CYCLE OIL PROPERTIES AL-12174

Sulfur, wt%	1.95
K. Vis, cSt, 40°C	3.44
Nitrogen, wt%	0.050
Flash Point, °C	93
Cetane No.	28
Distillation, °F (°C)	
IBP	415 (213)
20%	484 (251)
50%	547 (286)
70%	585 (307)
90%	644 (340)
EP	690 (366)

fuels were not as severe as those obtained with added TBDS. Both fuels which contained naturally occurring sulfur produced more used oil insolubles due to the increased heavy ends of these fuels as compared to the model test fuels which were based on narrow boiling range JP-7. Overall, fuel sulfur type (full boiling range natural vs. low boiling added) did not influence engine wear.

TABLE 13. PETTER ENGINE TESTS - NATURAL VERSUS ADDED SULFUR

Fuel	JP-7	JP-7+1% S	AL-12157	JP-7/Light Cycle
Heteroatom Type	None	as t-butyl disulfide	as 1% Nat. S	Oil as 1% Nat. S
<u>Avg. Operating Conditions</u>				
Test Hours	60	60	60	60
RPM	1800	1805	1803	1803
Load, lb-ft	29	28	29	28
Bhp	9.8	9.6	9.8	9.6
Oil Temp, °C	79.1	76.3	78.3	75.0
Exhaust Temp, °C	605	565	544	557
Oil Consumption, lb/hr	0.228	0.226	0.156	0.332
<u>Results</u>				
Used Oil Fe, ppm	52	130	142	150
Ring End Gap Change, in.				
Top	0.001	0.005	0.006	0.003
2	0.001	0.003	0.001	0.001
3	0.003	0.006	0.004	0.001
Piston WTD Rating	151	216	148	152
Piston Lacquer Demerit	3.2	5.5	4.5	5.0
Ring Sticking	None	None	None	None

TABLE 14. USED OIL PROPERTIES - PETTER ENGINE TESTS -
NATURAL VERSUS ADDED S

Fuel	JP-7	JP-7+1% S	AL-12157	JP-7/Cycle
Heteroatom Type	None	as t-butyl disulfide	1% Nat. S	Oil 1% Nat. S
<u>Used Oil Analyses</u>				
K. Vis, cSt, at 40°C	34.32	34.73	36.85	38.28
at 100°C	6.67	7.32	9.27	9.02
TAN	0.2	0.8	0.5	1.0
<u>Insolubles, wt%</u>				
Pentane "A"	0.71	0.85	1.24	1.64
Toluene "A"	0.53	0.79	1.23	1.56
Pentane "B"	0.69	0.85	1.24	1.63
Toluene "B"	0.28	0.48	0.52	0.90
<u>Elements</u>				
Fe, ppm	52	130	142	150
Cu, ppm	15	17	17	25
Pb, ppm	<60	<60	<60	<60
S, wt%	<0.01	0.01	0.06	0.07
N, wt%	0.004	0.006	0.007	0.009
Differential IR, Oxidation Absorbance @ 1710 cm ⁻¹	NIL	NIL	NIL	NIL

TABLE 15. PETTER PISTON DEPOSIT COMPOSITION -
NATURAL VERSUS ADDED S

Fuel S AL-12157 Heteroatom Type	JP-7 JP-7/Cycle None	JP-7+1% as t-butyl disulfide	1% Nat. S	Oil 1% Nat. S
Piston/Int Valve Deposit Analyses				
Heptane-Soluble Deposit, wt g	1.75	2.32	1.96	3.38
Composition, wt%				
C	85.4	85.1	85.4	85.3
H	14.1	14.3	14.0	14.3
N	0.11	0.13	0.11	0.12
S	0.12	0.50	0.19	0.16
Dimethylformamide (DMF)- Soluble Deposit, wt g	0.74	0.99	1.50	2.59
Composition, wt%				
C	67.1	66.7	63.6	67.2
H	5.0	5.0	6.6	7.2
N	3.9	2.3	2.1	2.5
S	0.6	6.1	1.3	1.2

D. Fuel Sulfur Volatility

To determine the effect of naturally occurring fuel sulfur volatility (boiling range) on engine deposits and wear, Petter diesel engine tests were run on both the high and low boiling fractions of a light cycle oil. It should be noted that in all probability, the high and low boiling fractions contained different amounts of fuel sulfur types; however, volatility of the naturally occurring sulfur compounds was the primary variable under consideration. One drum of AL-12174 light cycle oil from Howell Hydrocarbons which was previously tested in the Petter engine was separated by fractional distillation under vacuum at its 50 percent point. An illustration of the batch fractionation apparatus is presented in Figure 4. Six batches, 26 to 38 liters each, of whole light cycle oil were charged to the 50L kettle of the

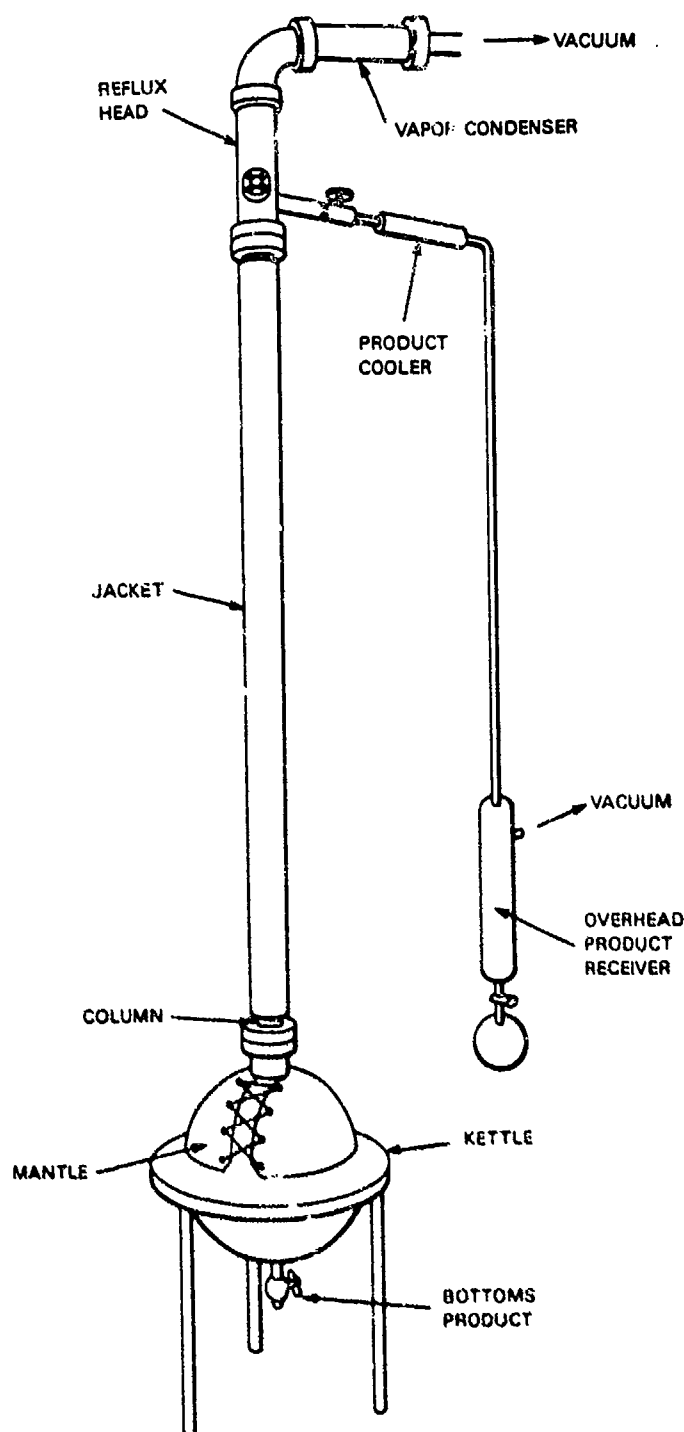


FIGURE 4. BATCH FRACTIONATION APPARATUS

distillation column. A charge of 40L is considered maximum. Smaller than maximum batches were selected to allow for turbulence should bumping occur during heating. In addition to the reduced charge volume, 15 compressed carbon boiling chips were placed in the kettle.

The vacuum-jacketed column above the kettle is 2 inches in diameter by 6 feet high and packed with stainless steel Helicoil packing to a depth of 60 inches. The column provides roughly 60 theoretical plates for separation. The rest of the equipment is stainless steel except the product receiver. The glass product receiver can hold one liter and permits removing the product while the system is under vacuum.

The pressure in the system was held below 1 mm/Hg during distillation. This allowed distillation to begin at a kettle temperature of 270°F (132°C) [head @ 102°F (38.9°C)]. At atmospheric pressure, the initial boiling point (D 86) was 415°F (214°C) [head @ 230°F (110°C)] and the 50 percent temperature was 547°F (286°C). The lower temperature was needed to prevent degradation of

the higher boiling fraction left in the kettle. During the distillation, the product was eluted initially at the rate of 15 mL/min. By the end of the distillation after 50 percent of the feed had been recovered, the production rate was 120 mL/min.

Table 16 contains the inspection properties for the light cycle oil (AL-12174) and the light (AL-12410) and heavy (AL-12409) fractions. The desired fractionation at the 50 percent point was achieved.

A Petter engine test was conducted using fuel AL-12950, which was a blend of high boiling fraction (HBF) of the cycle oil and JP-7. The blend had a sulfur content of 1 wt%. A second Petter test was conducted on a blend (AL-13110) of light boiling fraction of the cycle oil and JP-7, which had a sulfur content of 1 wt%. Fuel AL-13110 was treated with 1 vol% cetane improver additive (DII-3) to prevent diesel knock in the Petter engine. The results of these two Petter tests are summarized in Tables 17, 18, and 19 along with results for neat JP-7 and JP-7+TBDS (1 wt% S). The used oil iron content from both tests was contaminated with a sludge from the oil screen which increased the iron content. The sludge accumulated during an extended duration (480 hr) Petter test. Complete rebuild and cleaning of the engine eliminated the problem in subsequent tests. The blend containing HBF produced approximately the same measured ring wear and deposits as the JP-7/TBDS

TABLE 16. FUEL PROPERTIES

	AL-12174 Light Cycle Oil	AL-12410 Light Fraction	AL-12409 Heavy Fraction
Sulfur, wt%	1.95	1.68	2.35
K. Vis, cSt, at 40°C	3.44	2.16	6.36
Nitrogen, wt%	0.050	0.019	0.085
Flash point, °C	93	91	151
Cetane No.	28	26	30
Distillation, °F (°C)			
IBP	415 (213)	414 (212)	544 (284)
20%	484 (251)	458 (237)	573 (301)
50%	547 (286)	488 (253)	603 (317)
70%	585 (307)	507 (234)	628 (331)
90%	644 (340)	544 (284)	667 (353)
EP	690 (366)	612 (322)	705 (374)

TABLE 17. PETTER ENGINE - SULFUR VOLATILITY

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	AL-12950 JP-7/HBF	AL-13110 +1% DII3 JP-7/LBF
<u>Avg. Operating Conditions</u>				
Test Hours	60	60	60	60
RPM	1800	1805	1805	1806
Load, lb-ft	29	28	28	28
Bhp	9.8	9.6	9.6	9.6
Oil Temp, °C	79.1	76.3	76.1	76.1
Exhaust Temp, °C	605	565	573	576
Oil Consumption, lb/hr	0.228	0.226	0.178	0.03
<u>Results</u>				
Used Oil Fe, ppm	52	130	NM*	NM
Ring End Gap Change, in				
Top	0.001	0.005	0.005	0.003
2	0.001	0.003	0.002	0.001
3	0.003	0.006	0.005	0.002
Piston WTD Rating	151	216	184	206
Piston Lacquer Demerit	3.2	5.5	5.4	4.5
Ring Sticking	None	None	None	None

TABLE 18. USED OIL PROPERTIES - PETTER ENGINE TESTS - SULFUR VOLATILITY

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	AL-12950 JP-7/HBF	AL-13110 +1% DII3 JP-7/LBF
<u>Used Oil Analyses</u>				
K. Vis, cSt, at 40°C	34.32	34.73	41.41	30.04
at 100°C	6.67	7.32	9.87	5.90
TAN	0.2	0.8	0.9	0.9
<u>Insolubles, wt%</u>				
Pentane "A"	0.71	0.85	1.95	0.94
Toluene "A"	0.53	0.79	1.89	0.90
Pentane "B"	0.69	0.85	1.94	0.91
Toluene "B"	0.28	0.48	0.86	0.66
<u>Elements</u>				
Fe, ppm	52	130	NM*	NM
Cu, ppm	15	17	<10	16
Pb, ppm	<60	<60	<60	<60
S, wt%	<0.01	0.01	0.04	0.11
N, wt%	0.004	0.006	0.008	0.004
Differential IR, Oxidation Absorbance @ 1710 cm ⁻¹	NIL	NIL	V. Slight	V. Slight

*NM = Not Meaningful, used oil contaminated with carry-over Fe from a previous test

TABLE 19. PETTER DEPOSIT COMPOSITION -
SULFUR VOLATILITY

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	AL-12950 JP-7/HBF	AL-13110+ 1% DII3 JP-7/LBF
Piston/Int Valve Deposit Analyses				
Heptane-Soluble Deposit, wt g	1.75	2.32	3.02	1.54
Composition, wt%				
C	85.4	85.1	85.4	8.47
H	14.1	14.3	14.1	14.1
N	0.11	0.13	0.10	0.14
S	0.12	0.50	0.70	1.01
Dimethylformamide (DMF) Soluble Deposit, wt g	0.74	0.99	2.05	1.07
Composition, wt%				
C	67.1	66.7	63.7	56.1
H	5.0	5.0	5.8	6.1
N	3.9	2.3	3.2	2.8
S	0.6	6.1	4.8	6.6

fuel. Used oil viscosity was increased slightly due to higher insolubles content with fuel blend AL-12950. The blend containing LBF cycle oil produced slightly less measured ring wear and approximately the same deposits as the HBF blend. Used oil viscosity was slightly decreased in this test apparently due to slight fuel dilution as evidenced by the sulfur content of the used oil. Despite the similar WTD piston ratings, approximately twice the DMF-soluble piston deposit was recovered from the HBF test. As in previous tests, the fuel-related sulfur collected in the DMF-soluble piston deposits. Overall, the volatility of the natural occurring sulfur compounds present in the cycle oil did not have a major impact on measured ring wear or piston deposit ratings.

V. FUEL NITROGEN EFFECTS

The objective of this series of Petter engine tests was to determine if fuel nitrogen type impacts on diesel engine wear and deposits. The following

three nitrogen compounds were used to raise the nitrogen content of JP-7 to one percent:

- Pyridine
- N-methylpyrrole
- Quinoline

These nitrogen-containing test fuels were evaluated in the 60-hour Petter test procedure. The results of the tests are summarized in Tables 20, 21, and 22. In general, the addition of the nitrogen compounds to JP-7 did not cause increased engine wear. Piston deposits were substantially increased by the addition of the N-methylpyrrole, while fuels containing the other nitrogen compounds produced only a slight increase in piston WTD rating compared to neat JP-7.

Several problems with fuel system components were observed with the nitrogen-containing JP-7 test fuels. In the test which used pyridine to raise the JP-7 nitrogen content to 1 wt%, the fuel injector stuck at 30 hours and the

TABLE 20. PETTER ENGINE TESTS - 1% FUEL N

Fuel Heteroatom Type Avg. Operating Conditions	JP-7 None	JP-7+1% N as pyridine	JP-7+1% as N-methyl- pyrrole	JP-7+1% N as quinoline
Test Hours	60	60	60	60
RPM	1800	1806	1803	1803
Load, lb-ft	29	29	28	29
Bhp	9.8	9.8	9.6	9.8
Oil Temp, °C	79.1	75.0	75.0	73.3
Exhaust Temp, °C	605	587	543	574
Oil Consumption, lb/hr	0.228	0.311	0.324	0.162
Results				
Used Oil Fe, ppm	52	29	41	53
Ring End Gap Change, in.				
Top	0.001	0.001	0.001	0.001
2	0.001	0.002	0.001	0.001
3	0.003	0.003	0.002	0.003
Piston WTD Rating	151	190	245	185
Piston Lacquer Demerit	3.2	4.2	6.0	7.3
Ring Sticking	None	None	None	None

TABLE 21. USED OIL PROPERTIES - PETTER ENGINE
TESTS - 1% FUEL N

Fuel Heteroatom Type	None	JP-7+1% N as pyridine	JP-7+1% N as N-methyl- pyrrole	JP-7+1% N as quinoline
<u>Used Oil Analyses</u>				
K. Vis cSt, at 40°C	34.32	33.69	32.97	36.92
at 100°C	6.67	6.27	7.95	8.22
TAN	0.2	0.3	0.3	0.3
<u>Insolubles, wt%</u>				
Pentane "A"	0.71	0.35	0.71	1.19
Toluene "A"	0.53	0.16	0.30	1.12
Pentane "B"	0.69	0.33	0.71	1.17
Toluene "B"	0.28	0.16	0.27	0.70
<u>Elements</u>				
Fe, ppm	52	29	41	53
Cu, ppm	15	<10	16	11
Pb, ppm	<60	<60	<60	<60
S, wt%	<0.01	<0.01	<0.01	<0.01
N, wt%	0.004	0.006	0.007	0.016
Differential IR, Oxidation Absorbance @ 1710 cm ⁻¹	NIL	NIL	V. Slight	NIL

TABLE 22. PETTER PISTON DEPOSIT COMPOSITION -
1% FUEL N

Fuel Heteroatom Type	JP-7 None	JP-7+1% N as pyridine	JP-7+1% N as N-methyl- pyrrole	JP-7+1% N as quinoline
<u>Piston/Int Valve Deposit Analyses</u>				
Heptane-Soluble Deposit, wt g	1.75	2.15	2.68	1.47
Composition, wt%				
C	85.4	85.0	85.3	85.2
H	14.1	14.0	14.3	14.2
N	0.11	0.29	0.23	0.20
S	0.12	0.15	0.04	0.14
Dimethylformamide (DMF)- Soluble Deposit, wt g	0.74	1.87	1.50	0.92
Composition, wt%				
C	67.1	56.1	61.3	72.3
H	5.0	8.2	7.8	14.5
N	3.9	2.6	1.4	3.1
S	0.6	0.4	0.3	1.5

fuel pump also needed to be replaced at 30 hours. The fuel pump plunger was stuck in the barrel due to a slight deposit which had closed the machine fit tolerance of the pump. In the test which used N-methylpyrrole in the JP-7, the fuel pump stuck at 27 hours, again due to slight deposit buildup in the plunger/barrel area. Finally, a fuel injector stuck at 21 hours, when using JP-7 spiked with quinoline. Overall, the nitrogen compound types added to the fuel had very little or no effect on engine wear. Piston WTD substantially increased with the N-methylpyrrole, and all three nitrogen types caused deposit problems in close tolerance areas of the fuel system components.

VI. MISCELLANEOUS EFFECTS

In this section, miscellaneous heteroatom effects on diesel engine wear and deposits are examined. The first issue addressed was to determine if the presence of both nitrogen- and sulfur-containing heteroatom compounds would synergistically increase engine wear and deposits. A 60-hour Petter engine test was conducted using JP-7 fuel which contained 1.2 wt% nitrogen present as pyridine and 1 wt% sulfur present as TBDS. The summarized results of this test, the neat JP-7, and the 1 wt% fuel S as TBDS tests are presented in Tables 23, 24, and 25. Engine wear determined by piston ring and bore measurements and iron content in the oil was approximately the same for the N+S and 1% S as TBDS tests, while the piston deposit rating for the N+S test was similar to that with neat JP-7. The used oil from the N+S test had a slightly increased TAN and sulfur content compared to the TBDS test. The DMF-soluble deposit from the N+S test was unique in that it had a much higher sulfur content than any other DF-recovered deposit. The fuel pump problems described in previous tests when using pyridine in the fuel were also observed in this test as the fuel pump needed to be replaced at 9.5 and 60 hours. In conclusion, there appeared to be no synergism with respect to wear between nitrogen (pyridine) and sulfur (TBDS) when added to the fuel. The presence of both N+S might have had a deposit-reducing synergism as a cleaner piston WTD rating was observed in this test.

A 60-hour Petter test was run using a fuel blend of 90 vol% JP-7 and 10 vol% naphthenic acids (AV-220). The summarized results are also shown in Tables

TABLE 23. MISCELLANEOUS PETTER ENGINE TESTS

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	JP+1% S as TBDS +1.2% N as pyridine	JP-7+10% AV 220
<u>Avg. Operating Conditions</u>				
Test Hours	60	60	60	60
RPM	1800	1805	1804	1806
Load, lb-ft	29	28	26	28
Bhp	9.8	9.6	8.9	9.6
Oil Temp, °C	79.1	76.3	77.5	75.0
Exhaust Temp, °C	605	565	570	534
Oil Consumption, lb/hr	0.228	0.226	0.03	0.213
<u>Results</u>				
Used Oil Fe, ppm	56	130	145	1046
Ring End Gap Change, in.				
Top	0.001	0.005	0.004	0.009
2	0.001	0.003	0.001	0.001
3	0.003	0.006	0.003	0.005
Piston WTD Rating	151	216	132	147
Piston Lacquer Demerit	3.2	5.5	6.8	3.6
Ring Sticking	None	None	None	None

TABLE 24. USED OIL PROPERTIES - MISCELLANEOUS PETTER ENGINE TESTS

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	JP-7+1% S as TBDS +1.2% N as pyridine	JP-7+10% AV 220
<u>Used Oil Analyses</u>				
K. Vis, cSt, at 40°C	34.32	34.73	35.70	33.26
at 100°C	6.67	7.32	8.07	7.28
TAN	0.2	0.8	1.3	0.9
<u>Insolubles, wt%</u>				
Pentane "A"	0.71	0.85	1.10	0.73
Toluene "A"	0.53	0.79	0.90	0.66
Pentane "B"	0.69	0.85	1.05	0.70
Toluene "B"	0.28	0.48	0.88	0.52
<u>Elements</u>				
Fe, ppm	56	130	145	1046
Cu, ppm	15	17	18	31
Pb, ppm	<60	<60	<60	<60
S, wt%	<0.01	0.01	0.04	<0.01
N, wt%	0.004	0.006	0.010	0.004
Differential IR, Oxidation Absorbance @ 1710 cm ⁻¹	NIL	NIL	NIL	NIL

TABLE 25. PETER PISTON DEPOSIT COMPOSITION -
MISCELLANEOUS TESTS

Fuel Heteroatom Type	JP-7 None	JP-7+1% S as t-butyl disulfide	JP-7+1% S as TBDS +1.2% N as pyridine	JP-7+10% AV 220
Piston/Int Valve Deposit Analyses				
Heptane-Soluble Deposit, wt g	1.75	2.32	1.52	1.58
Composition, wt%				
C	85.4	85.1	84.7	84.0
H	14.1	14.3	14.1	14.1
N	0.11	0.13	0.28	0.11
S	0.12	0.50	0.58	0.39
Dimethylformamide (DMF)- Soluble Deposit, wt g	0.74	0.99	1.02	0.90
Composition, wt%				
C	67.1	66.7	59.0	48.8
H	5.0	5.0	5.5	5.0
N	3.9	2.3	3.8	2.6
S	0.6	6.1	11.5	1.4

23, 24, 25. The experiment demonstrated the detrimental effects of oxygen-containing compounds present as naphthenic acids in the fuel on engine condition. The top compression ring experienced very high measured wear and the used oil contained 1046 ppm iron. The naphthenic acids did not impact on piston deposits, as the piston WTD was virtually the same as when using neat JP-7. The fuel injector was sticking at 55 hours due to deposits and was replaced. Overall, the naphthenic acids were very detrimental to engine wear, while not affecting piston deposits, but did cause injector deposits.

A test was conducted using a referee diesel fuel (MIL-F-46162B) which contained 1 wt% sulfur as naturally occurring compounds. The analysis results of this fuel (AL-12624) are presented in Table 26. Fuel AL-12624 did not meet all requirements of MIL-F-46162B, as it had poor accelerated stability

and excessive particulate contaminates. This test was continued beyond the normal 60 hours to 480 hours to determine the effects of extended use of this fuel, and to determine if engine wear and deposit buildup occurred linearly with test time. The engine completed the scheduled 480 hours without problems.

TABLE 26. FUEL ANALYSIS
Fuel: AL-12624-F (HSF)

Property	ASTM Method	Value
Density at 20°C, kg/L	D 1298	0.8730
Gravity at 60°F, API°	D 287	30.5
Distillation, °F (°C)	D 86	
IBP		379 (193)
10%		434 (223)
20%		468 (242)
30%		498 (259)
40%		525 (274)
50%		543 (284)
60%		558 (292)
70%		573 (301)
80%		592 (311)
90%		617 (325)
95%		637 (336)
End Point		657 (347)
Residue, vol%		1.0
Flash Point, °F (°C)	D 93	175 (79)
Cloud Point, °F (°C)	D 2500	12* (-11)
Pour Point, °F (°C)	D 97	10* (-12)
Cetane Number	D 613	42.7
Cetane Index	D 976	43.4
K. Vis, cSt, at 40°C	D 445	2.97
Carbon Residue on 10% Bottoms, mass%	D 524	0.21
Cu Strip Corrosion, 3 hours at 50°C	D 130	1a
TAN	D 974	0.02
Accelerated Stability,		
Total Insolubles, mg/100 mL	D 2274	4.30
Particulate Contamination, mg/240 mL	D 2276	3.6**
Saturates, vol%	D 1319	46.5
Olefins, vol%	D 1319	1.1
Aromatics, vol%	D 1319	52.4
Net Heat of Combustion, Btu/lb (MJ/kg)	D 1405	17968
(41.794)		
Ash, wt%	D 482	<0.01
Sulfur, wt%	D 2622	1.01
Karl Fisher H ₂ O, ppm	D 1744	607

* Sample was centrifuged and the supernatant was used for these tests.

**Fitter plugged at 240 mL.

A comparison of engine wear and deposits at 60 hours and 480 hours was made. The extended duration test (eightfold increase in time) had approximately a fivefold increase in measured ring wear and a twofold increase in piston deposits. These results indicate that engine wear and deposit buildup are occurring at a less than linear rate with time.

VII. CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

The following conclusions are made based on the Petter engine tests reported herein:

Wear

- At 1.0 wt% fuel sulfur, wear severity was 2 to 2.7 times that of the neat base fuel (JP-7).
- At 2.0 wt% fuel sulfur, a fourfold increase in wear was observed as compared to the base fuel.
- Naturally occurring fuel sulfur produced approximately the same engine wear as added sulfur compounds.
- Fuel sulfur chemical type did not influence observed engine wear levels.
- Volatility of the fuel sulfur did not influence observed engine wear levels.
- At 1 wt% fuel nitrogen, there was no increase in engine wear.
- Naphthenic acids in the fuel caused a large increase in top compression ring wear.

- No synergistic increase in engine wear was observed when both nitrogen and sulfur were present in the fuel.
- An eightfold increase in test duration caused a fivefold increase in engine wear; thus, wear was occurring at a less than linear rate with test duration.

Deposits

- At 1 wt% fuel sulfur, substantially increased piston deposits were observed with TBDS and C8SH
- 2 wt% fuel sulfur, piston deposits were only slightly greater than at 1 wt% S.
- At 1 wt% fuel sulfur, TBDS caused greater piston deposits than the naturally occurring fuel sulfur.
- Volatility of naturally occurring fuel sulfur compounds had no effect on piston deposit ratings. A greater weight of collected deposits was obtained with the HBF fuel.
- Substantially increased piston deposits were observed with N-methylpyrrole in the fuel.
- All the nitrogen compounds caused fuel system deposit problems in close tolerance areas such as the fuel pump plunger.
- The naphthenic acids had no effect on piston deposits, but did cause fuel injector sticking.
- An eightfold increase in test duration resulted in a twofold increase in piston deposits; thus, piston deposit accumulation occurred at a much less than linear rate with test duration.

B. Recommendations

The following recommendations for additional work are offered:

- Additional, more basic research which involves combustion experiments should be conducted to determine the relative roles of SO_2 and SO_3 in causing corrosive engine wear.
- Since SO_2 appears to be the primary sulfur oxide formed in the combustion chamber, the mechanism of $\text{SO}_2 \rightarrow \text{SO}_3$ conversion in a compression ignition engine should be examined.
- The mechanism of piston deposit formation should be determined, especially for TBDS which causes increased deposits.
- The mechanisms by which engine oil additives counteract the deleterious effects of fuel heteroatom compounds should be investigated.

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