

EFFECTS OF SULFUR COMPOUNDS ON A JP-8 SURROGATE FUEL

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INTRODUCTION

The thermal stability of military jet fuels is of increasing concern as the fuels must withstand ever increasing loads as the coolant/heat sink for the system of advanced aircraft⁽¹⁾. Fuels exposed to elevated temperatures and oxygen form soluble and insoluble oxidation products. The formation of insoluble deposits in fuel systems and the resulting possibility of system failure is of particular concern.

Hazlett pointed out the role of phenols and oxidative phenolic coupling in the formation of insoluble deposits under accelerated storage conditions in 1986⁽²⁾. We have observed phenols as the only reactive species in engine augments fuel tube and spray ring deposits from a military engine⁽³⁾. From the real engine deposits we postulate deposit initiation by oxidative phenolic coupling, followed by condensation to three dimensional aromatic networks as the initial phenolic dimers and oligomers are exposed to more heat energy. In experiments with fuel samples, solid phase extraction of polar compounds were predominately phenolic for non-hydrated fuels but not with hydrated fuels⁽⁴⁾. The hydrated fuels oxidized rapidly and extensively to give alcohols and carbonyl products, including the homologous series of 5-alkylidihydrofuranones. Hydrated fuels did not form significant amounts of insoluble solid deposit. Non-hydrated fuels oxidized slowly and formed large amounts of insoluble deposits. Heneghan has observed this inverse relationship of deposit formation and "oxidizability" for 20 different fuels as measured by eight separate techniques⁽⁵⁾.

Non-hydrated fuels are generally high in sulfur relative to hydrated fuels. The catalytic effect of sulfuric and sulfonic acids in the decomposition of α -arylhydroperoxides to phenols is well known⁽⁶⁾. Hardy, et. al., observed the effect of several sulfur compounds as anti-peroxidants⁽⁷⁾ in fuels and in model systems⁽⁸⁾.

If oxidative phenolic coupling is a major mechanism for deposit formation, the effect of sulfur compounds in the production of phenols by decomposition of arylhydroperoxide precursors should significantly increase deposit formation. The relative effects of different classes of sulfur compounds is probably predictable, but the effect of these compounds on phenolic coupling is not known. A previously prepared, 12 component, surrogate fuel⁽⁹⁾ presents a test media for doping experiments with representative sulfur compounds from which reaction products of fuel components and sulfur compounds should be easy to follow, compared to complex real fuels.

Aliphatic thiols, thiophenols, thiophenes, benzothiophenes and disulfides are represented by dopant compounds. Gravimetric results of deposit formation, extraction and analysis of polar products by GC-MS and analysis of insoluble solids by step wise thermal desorption/pyrolysis-GC-MS gives a fairly complete picture of the oxidation and deposit processes. The information should establish a relationship of structure to deposit tendency for these classes of sulfur compounds. To investigate possible effects of the sulfur compounds in phenolic coupling, 2-propylphenol was added to all samples.

EXPERIMENTAL

Surrogate fuel: Isooctane, 5.0 %, wt./wt., methylcyclohexane, 5.0 %, m-xylene, 5.0 %, cyclooctane, 5.0 %, tetramethylbenzene, 5.0 %, tetralin, 5.0 %, dodecane, 20.0 %, methylnaphthalene, 5.0 %, tetradecane, 15.0 % and hexadecane, 10.0 %, were 99% grade purchased from Aldrich.

Dopant compounds: 2-propylphenol, 3,4-dimethylthiophenol, phenylethylmercaptan, dibenzothiophene, phenylsulfide, 2-ethylthiophenol, 2-ethylthiophene and hexanethiol were Aldrich reagent grade. Solvents: acetone, methanol, methylene chloride and toluene were Aldrich reagent or HPLC grade. High purity helium, nitrogen and oxygen gases were used as supplied by Central Kentucky Welding Supply.

Fuel stressing: The flask test apparatus and operation have been described previously⁽⁹⁾. 30.0 mL of surrogate fuel and dopants were stressed in a 50 mL round bottom flask, equipped with a Friedrichs condenser with coolant at 0-5 °C and heated with mantles controlled to 175 +/- 2 °C. The fuel was nitrogen purged until temperature equilibrated and oxygen was then sparged into the fuel at 100 mL/min. via 0.53 mm fused quartz capillary. Samples were stressed for 24 hours, filtered and washed upon reaching room temperature. Stressed fuel was vacuum filtered with Osmonics[®] 0.45 silver membrane 47 mm filter. Glassware and filters were washed with heptane. Filtered deposits were washed with acetone. Filters, with acetone insoluble material were dried in vacuum (20-30 torr, 80 °C) 36-48 hours and reweighed. Acetone soluble gums were collected as filtrate, the solvent evaporated with dry nitrogen and weighed.

Solid phase extraction: 1.0 g J & W silica gel SPE cartridges were conditioned with 2 x 5 mL portions of methanol, methanol/acetone, acetone, acetone/heptane and heptane. A 5.0 mL portion of filtered, stressed fuel was extracted and the cartridge washed with 3 x 3 mL of heptane, lightly air dried (2 x 10 mL) and the extract eluted with 3.0 mL methanol. Methanol was evaporated with dry nitrogen, the extract weighed and immediately rediluted to 1.0 mL with methanol.

Analysis: Hewlett Packard 5890 series II GC/5890 MS with 7673A autosampler. MS scanned 35-550 m/z, six minute solvent delay. 1.0 μ L splitless injection, 280 °C injection port and transfer line. 50 meter x 0.25 mm x 0.5 μ m J & W DB-5 MS column at 30 psi head pressure. Purge time 0.5 min., 2 min. at 60 °C, 2 °C/min. to 250 °C,

10 °C/min. to 280 and 10 min. final hold. Sample samples by thermal desorption/pyrolysis with CDS model 1000 Pyroprobe®. Coil probe, 2 x 16 min quartz tube. Approximately 10 mg of dry sample for sequential thermal desorption and pyrolysis at 200, 280, 450, 750 and 1100 °C. Interface at 200 and 280 °C for first two samples, probe fired immediately upon attaining interface temperature. Interface at 325-330 °C for final three pyrolysis runs. Probe fired 99.9 seconds in all cases and GC run started immediately after probe firing. MS scanned 15-550 m/z. GC purge time four minutes. Initial temperature @ -50 °C for six minutes, 10 °C/min. to 50 °C, then three °C/min. to 280 °C and 17.33 min. hold for 110 min. Total run time with the same column as soluble sample runs.

RESULTS & DISCUSSION

The conditions of the flask test in this work (175 °C, 24 hr., 100 mL/min. O₂) represent extremes, short of pyrolytic conditions, for the thermal-oxidative stress of fuel and must be understood to represent no possible real case. If anything, it is a hyper-accelerated storage model. The advantage of the conditions is the production of oxidation products and deposits in quantities amenable to the analytical methods used in a relatively short time.

We have subjected nine samples to thermal oxidative stress under identical conditions: The surrogate fuel, undoped; surrogate + 1.0 % 2-propylphenol; and surrogate + 1.0 % 2-propylphenol with 0.5 % sulfur compound (3,4-dimethylthiophenol, 2-ethylthiophenol, phenylethylmercaptan, hexanethiol, 2-ethylthiophene, dibenzothiophene and phenylsulfide). Soluble oxidation products are represented as silica gel extracts. Fuel insoluble, but acetone soluble and acetone insoluble material is called solids. Table 3 is a compilation of the gravimetric data obtained. Relative comparison of these data to that obtained from real fuels (4) suggest that solid formation is the most valid indicator of thermal stability.

Fuels stable to deposit formation and thermally stable by JFTOT, etc., such as JP-7, JPTS, POSF 2747 and the surrogate fuel form substantial amounts of gum but insignificant solids in the flask test. Less stable fuels form less gum but dramatically more solids. The fuels with high gum and extract concentrations can be characterized as "oxidizable" fuels and those with high solids but low gums and extracts as "non-oxidizable". Table 3 shows dibenzothiophene, phenylsulfide and 2-ethylthiophene as dopants that produce "oxidizable" fuels relative to undoped surrogate (JP-8S) or surrogate doped only with 2-propylphenol. The thiophenols and alkyl thiols, 3,4-dimethylthiophenol, 2-ethylthiophenol, phenylethylmercaptan and hexanethiol doped fuels are "non-oxidizable" and form larger amounts of solid deposit. 2-ethylthiophene is somewhat anomalous by gravimetric data as well as chromatographic analysis. This is probably due to extreme oxidative conditions, cleaving the ring to form a sulfonic acid.

Space permits only representative chromatograms and product identification for polar extracts and thermal desorption/pyrolysis of solids. The more extreme cases are used. Chromatograms of polar extracts of "oxidizable" fuels are Figure 1a, surrogate, no dopant, 1b, phenylsulfide dopant and Figure 1c, dibenzothiophene dopant. Tables 1a, 1b and 1c are product identification for the corresponding chromatograms. Figures 2a and 2b are chromatograms of "non-oxidizable" fuels, doped with phenylethylmercaptan and 3,4-dimethylthiophenol. Tables 2a and 2b are product identification for these chromatograms.

Comparison of the chromatograms shows a dearth of oxidation products from the fuels doped with the thiol and thiophenol and a proliferation of oxidized species in undoped and fuels doped with phenylsulfide and dibenzothiophene. 2-propylphenol and 2,4,5-trimethylphenol are the most abundant compounds in the extracts of phenylethylmercaptan and 3,4-dimethylthiophenol doped surrogate. The trimethylphenol has to be the product of hydroperoxide decomposition from the 1,2,4,5-tetramethylbenzene (durene), present in the surrogate fuel. The phenols are minor components in the chromatograms of the "oxidizable" fuel extracts. The sulfones of dibenzothiophene and phenylsulfide are found in the respective chromatograms (at about 94 and 88 min. R.T.). The most reasonable interpretation of these observations is that the alkyl and aryl thiols both quickly oxidize to sulfonic acids that catalyze phenol production from α -arylhydroperoxides. Oxidation of aliphatic fuel components is inhibited. The sulfide and thiophene are oxidized to sulfones but essentially do not affect oxidation of fuel components.

Sequential thermal desorption/pyrolysis with GC-MS analysis has proven a valuable technique for characterization of insoluble solids, but care must be exercised in interpretation of data. Pyrolysis onset temperature and the result of thermo-synthesis degradation and rearrangements can not be exactly known. Total ion chromatograms for the technique applied to solids formed by the surrogate fuel doped with 2-propylphenol and 3,4-dimethylthiophenol are presented in Figure 3. The temperature sequence the sample is subjected to for separate chromatograms is 200, 280, 450, 750 and 1100 °C. Experience has shown that solid deposits from jet fuels contain high concentrations of absorbed fuel components. These are essentially all desorbed at 200 °C. 280 and 450 °C provide the most useful information for characterizing the solid matrix. At 280 °C, it is assumed that strongly absorbed compounds are desorbed and weak chemical bonds are broken. At 450 °C, we assume pyrolytic decomposition of the polymeric matrix without extensive rearrangement or pyrosynthesis. This is supported by model studies with "Noryl" (poly-2,6-dimethylphenol) polymer (3). Pyrolysis at 450 °C produced monomeric C₁-C₃ phenols in high abundance and several -O- and -CH₂- linked dimers. The major product was 2,6-dimethylphenol. We assume that this step condenses remaining solid sample and subsequent chromatograms are essentially all from aromatic hydrocarbons.

Figures 4a and 4b through 7a and 7b, are the respective total ion chromatograms obtained from solids of fuels doped with phenylsulfide, dibenzothiophene, phenylethylmercaptan and 3,4-dimethylthiophenol at 280 and 450 °C. These chromatograms support the oxidative phenolic coupling as the mechanism for formation of extensive solid deposits. The chromatograms from the "non-oxidizing" thiophenol and mercaptan doped surrogate are essentially all phenolic and aromatic while the chromatograms of solids from sulfide and thiophene doped fuel show predominately alkenes, alcohols and ketones. The sulfonic acids of phenylethylmercaptan and 3,4-dimethylthiophenol are present in the respective chromatograms, as are the dibenzosulfone and phenylsulfoxide.

The data from this work is consistent with sulfonic acid formation and catalysis of phenol production to result in insoluble solid deposits formed by oxidative phenolic coupling. A consequence of these observations is that it should be relatively simple to devise a method to predict fuel stability (to solid deposit formation) that would be

based on caustic extraction followed by further separation and analysis. Such a method would need to include removal of extractable interferences such as deicer.

The dilemma of stability to peroxidation versus deposition remains. Hydrotreated fuels would seem to solve the deposition problems if engines and fuel transfer systems were "peroxide proof". At present, it seems the solution requires antioxidants that are themselves not deposit promoters.

ACKNOWLEDGMENTS

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Figure 1a. Total Ion Chromatogram of Solid Phase Extract of JP-8S + 1.0 % 2-propylphenol. 24 Hour Flask Test at 175 °C With Flowing Oxygen.

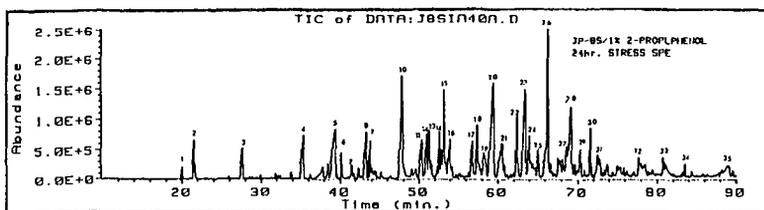


Figure 1b. Total Ion Chromatogram of Solid Phase Extract of JP-8S + 1.0 % 2-propylphenol and 0.5 % Phenylsulfide. 24 Hour Flask Test at 175 °C With Flowing Oxygen.

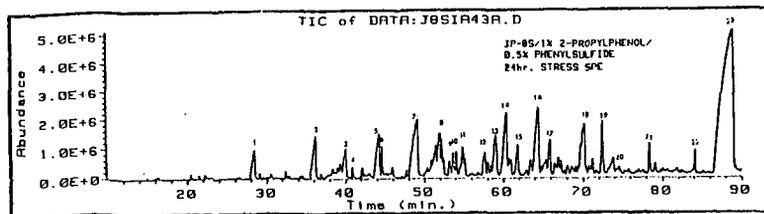


Figure 1c. Total Ion Chromatogram of Solid Phase Extract of JP-8S + 1.0 % 2-propylphenol and 0.5 % Dibenzothiophene. 24 Hour Flask Test at 175 °C With Flowing Oxygen.

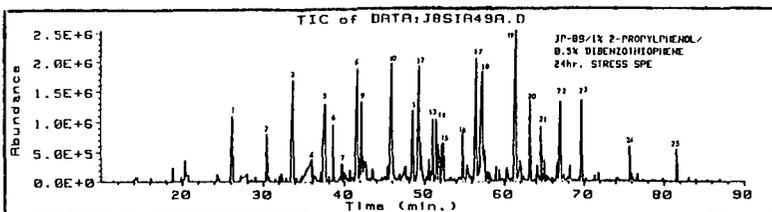


Figure 2a. Total Ion Chromatogram of Solid Phase Extract of JP-8S + 1.0 % 2-propylphenol and 0.5 % Phenylethylmercaptan. 24 Hour Flask Test at 175 °C with Flowing Oxygen.

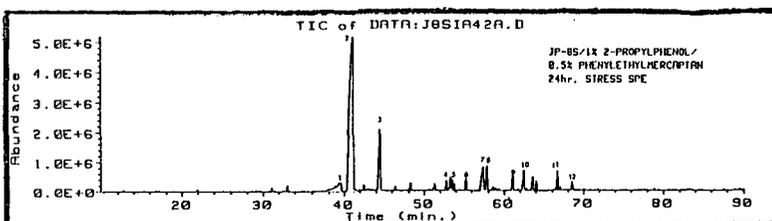


Figure 2b. Total Ion chromatogram of Solid Phase Extract of JP-8S + 1.0 % 2-propylphenol and 0.5 % 3,4-dimethylthiophenol. 24 Hour Flask Test at 175 °C With Flowing Oxygen.

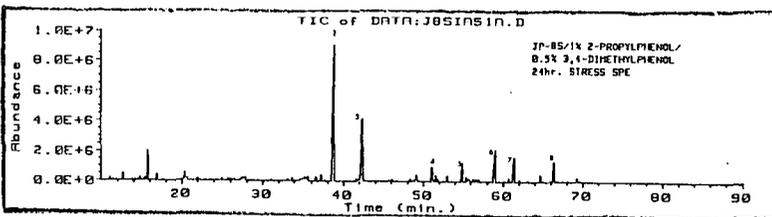


Table 1a. Peak Identification for Figure 1a.

Peak #	Rt (min.)	Compound
1.	20.06	Methyldihydrofuran
2.	21.60	Mixed phenol/furanmethanol
3.	27.65	Ethyldihydrofuran
4.	35.37	Propyldihydrofuran
5.	39.43	Mixed acid
6.	40.20	2-propylphenol
7.	41.56	Aliphatic alcohol
8.	43.36	Butyldihydrofuran
9.	43.87	2,4,5-trimethylphenol
10.	47.62	Mixed isobenzofurandione/methyl naphthalene
11.	50.37	Isobenzofuranone
12.	51.02	Pentyldihydrofuran
13.	51.29	Substituted benzene
14.	52.60	C ₆ phenol
15.	53.21	Tetradecane
16.	53.99	Methylisobenzofurandione
17.	56.80	Benzopyranone
18.	57.40	Substituted benzopyran
19.	58.29	Hexyldihydrofuran
20.	59.47	Naphthalenone
21.	60.45	Indole-diol
22.	64.02	C ₆ phenol?
23.	63.53	Propenylbenzodioxole
24.	64.02	C ₆ phenol
25.	65.15	Heptyldihydrofuran
26.	66.43	Hexadecane/C ₇ dihydrofuran.mix
27.	68.66	Methylnaphthalenol
28.	69.19	Phenylketone
29.	70.39	Substituted cycloketone
30.	71.62	C ₈ dihydrofuran
31.	72.64	Substituted naphthalene
32.	77.64	C ₉ dihydrofuran
33.	80.78	Substituted aromatic
34.	83.47	C ₁₀ dihydrofuran
35.	89.03	Substituted aromatic

Figure 1c. Total Ion Chromatogram of Solid Phase Extract of JP-8S + 1.0 % 2-propylphenol and 0.5 % Dibenzothiophene. 24 Hour Flask Test at 175 °C With Flowing Oxygen.

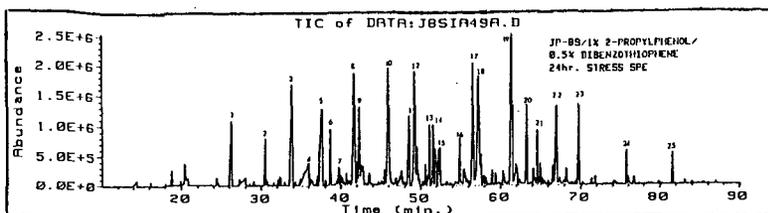


Figure 2a. Total Ion Chromatogram of Solid Phase Extract of JP-8S + 1.0 % 2-propylphenol and 0.5 % Phenylethylmercaptan. 24 Hour Flask Test at 175 °C with Flowing Oxygen.

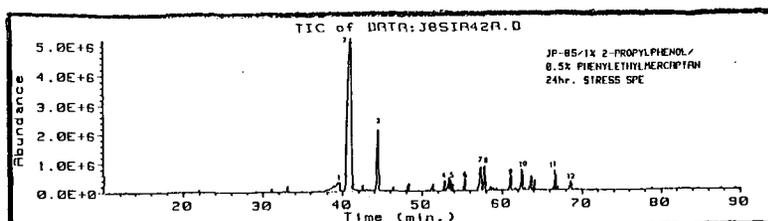


Figure 2b. Total Ion chromatogram of Solid Phase Extract of JP-8S + 1.0 % 2-propylphenol and 0.5 % 3,4-dimethylthiophenol. 24 Hour Flask Test at 175 °C With Flowing Oxygen.

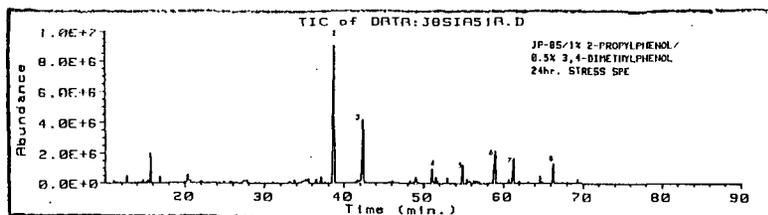


Table 1a. Peak Identification for Figure 1a.

Peak #	Rt (min.)	Compound
1.	20.06	Methyldihydrofuran
2.	21.60	Mixed phenol/furanmethanol
3.	27.65	Ethyldihydrofuran
4.	35.37	Propyldihydrofuran
5.	39.43	Mixed acid
6.	40.20	2-propylphenol
7.	41.56	Aliphatic alcohol
8.	43.36	Butyldihydrofuran
9.	43.87	2,4,5-trimethylphenol
10.	47.62	Mixed isobenzofurandione/methyl naphthalene
11.	50.37	Isobenzofurawone
12.	51.02	Pentyldihydrofuran
13.	51.29	Substituted benzene
14.	52.60	C ₆ phenol
15.	53.21	Tetradecane
16.	53.99	Methylisobenzofurandione
17.	56.80	Benzopyranone
18.	57.40	Substituted benzopyran
19.	58.29	Hexyldihydrofuran
20.	59.47	Naphthalenone
21.	60.45	Indole-diole
22.	64.02	C ₆ phenol?
23.	63.53	Propenylbenzodioxole
24.	64.02	C ₆ phenol
25.	65.15	Heptyldihydrofuran
26.	66.43	Hexadecane/C ₇ dihydrofuran mix
27.	68.66	Methylnaphthalenol
28.	69.19	Phenylketone
29.	70.39	Substituted cycloketone
30.	71.62	C ₈ dihydrofuran
31.	72.64	Substituted naphthalene
32.	77.64	C ₉ dihydrofuran
33.	80.78	Substituted aromatic
34.	83.47	C ₁₀ dihydrofuran
35.	89.03	Substituted aromatic

Table 1b. Peak Identification for Figure 1b.

Peak #	Rt (min.)	Compound
1.	28.52	Ethylidihydrofuran
2.	36.34	Propylidihydrofuran
3.	40.12	Hexanoic acid
4.	40.97	2-propylphenol
5.	44.37	Butylidihydrofuran
6.	44.72	C ₆ dihydrofuran
7.	49.27	Isobenzofurandione
8.	52.13	C ₅ dihydrofuran
9.	53.83	Methylbenzofurandione
10.	54.23	Alkanol
11.	54.99	Methylisobenzofurandione
12.	57.78	Benzopyranone
13.	59.12	Hexylidihydrofuran
14.	60.53	Dihydronaphthalenone
15.	61.94	Phthalate
16.	64.56	Phenylpropenal
17.	66.07	C ₇ dihydrofuran
18.	70.27	Substituted cycloketone
19.	72.52	C ₈ dihydrofuran
20.	73.91	Naphthoic acid
21.	78.50	C ₉ dihydrofuran
22.	84.25	C ₁₀ dihydrofuran
23.	88.94	Phenylsulfone

Table 1c. Peak Identification for Figure 1c.

Peak #	Rt (min.)	Compound
1.	26.22	Ethylidihydrofuran
2.	30.49	Dione ?
3.	33.82	Propylidihydrofuran
4.	35.95	Phenylethylethanone
5.	37.55	Carboxylic acid
6.	38.50	2-propylphenol
7.	39.69	Substituted cyclohexanone
8.	41.72	Butylidihydrofuran
9.	42.25	2,4,5-trimethylphenol
10.	46.03	Isobenzofurandione
11.	48.52	Isobenzofurandione
12.	49.29	Butylidihydrofuranone
13.	51.11	Substituted benzofuran
14.	51.52	Substituted alkane
15.	52.42	Pentylidihydrofuran
16.	54.86	Benzopyranone
17.	56.62	Hexylidihydrofuran
18.	57.34	Substituted benzoic acid
19.	61.45	Substituted benzene
20.	63.27	Heptylidihydrofuran
21.	64.57	Alkane
22.	67.02	Substituted ketone
23.	69.73	Octylidihydrofuran
24.	75.77	Nonylidihydrofuran
25.	81.54	Decylidihydrofuran

Table 3. Gravimetric Analysis of Thermally Stressed Fuels

Stress Sample	Gums	Solids	Total	SiOH Extract*
				mg/5mL Fuel
Surrogate + Dopant	Mass % of Fuel			
1 % 2-propylphenol/0.5 % dibenzothiophene	4.70	0.46	5.00	110.20
1 % 2-propylphenol/0.5 % 2-ethylthiophene	4.13	0.58	4.71	210.20
JP-8S (No dopant)	3.32	0.06	3.38	122.70
1 % 2-propylphenol/0.5 % phenylsulfide	2.43	0.29	2.72	191.50
1 % 2-propylphenol	1.71	0.16	1.87	155.80
1 % 2-propylphenol/0.5 % 2-ethylthiophenol	0.57	0.91	1.48	29.00
1 % 2-propylphenol/0.5 % 3,4-dimethylthiophenol	0.38	1.30	1.68	32.00
1 % 2-propylphenol/0.5 % phenylethylmercaptan	0.35	0.46	0.81	34.10
1 % 2-propylphenol/0.5 % hexanethiol	0.29	0.43	0.72	21.60

*SiOH extract: Represents values for "soluble gums".

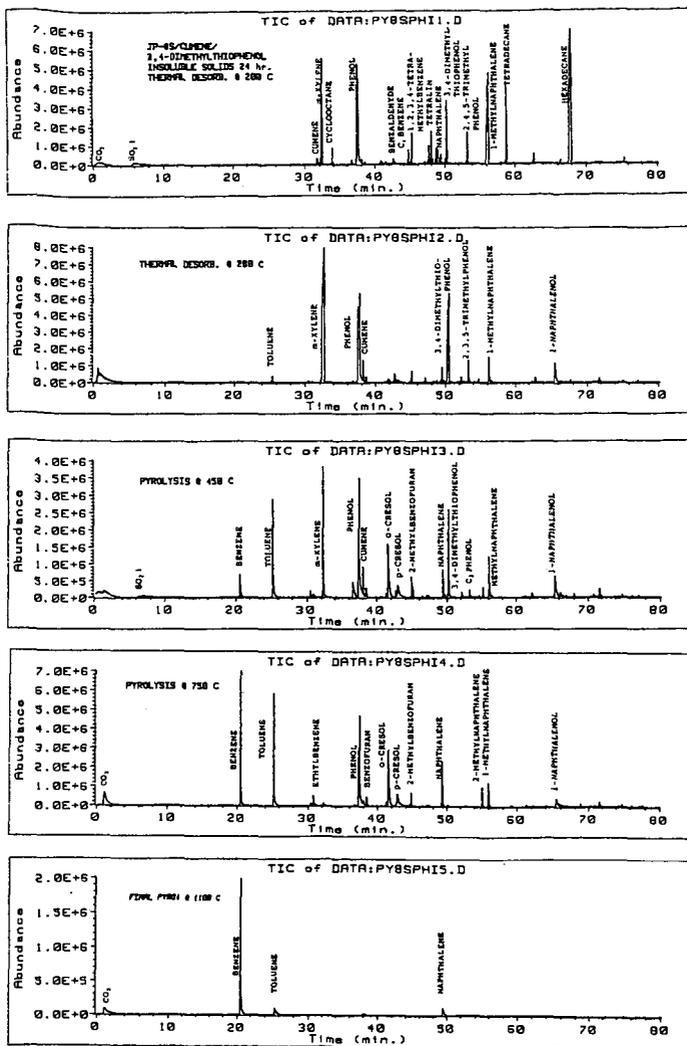
Table 2a. Peak Identification for Figure 2a.

Peak #	Rt (min.)	Compound
1.	39.42	Substituted benzoic acid
2.	41.18	2-propylphenol
3.	44.50	2,4,5-trimethylphenol
4.	52.75	Dihydronaphthalenone
5.	53.28	3,4-dihydrobenzopyran-2-one
6.	55.22	1,2-dihydroxypropylbenzene
7.	57.37	Benzopyran-2-one
8.	57.84	Substituted benzopyran
9.	61.04	Sub. dihydrobenzopyranone
10.	62.50	C ₅ phenol
11.	66.65	Pentadecane
12.	68.60	Methylnaphthalenol

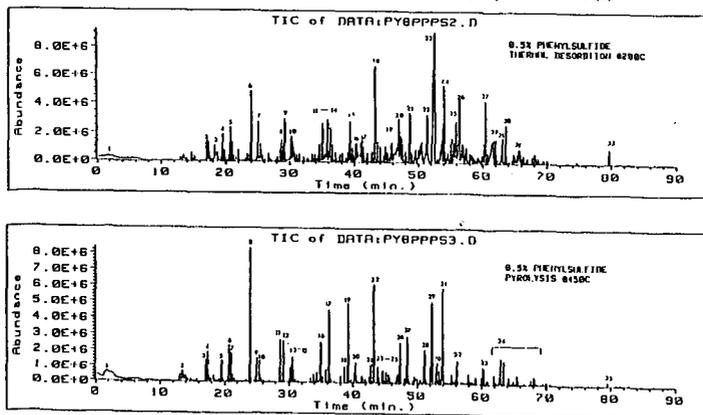
Table 2b. Peak Identification for Figure 2b

Peak #	Rt (min.)	Compound
1.	15.72	Ketone
2.	38.77	2-propylphenol
3.	42.35	2,4,5-trimethylphenol
4.	51.06	Methylbenzofuranone
5.	54.90	Benzopyran-2-one
6.	58.96	Substituted dimethylphenol
7.	61.29	Dimethyl-2,3-dihydroindene-1-one
8.	66.29	Substituted phenol

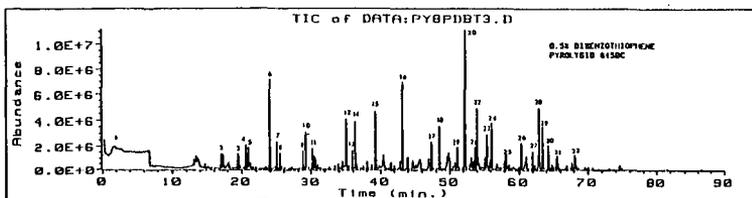
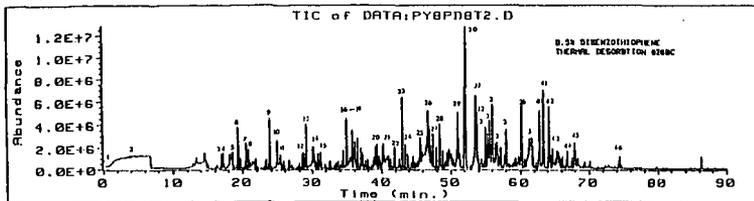
Figure 3. Sequential Thermal Desorption/Pyrolysis Total Ion Chromatograms of Insoluble Solids From JP-8S Doped With Cumene and 3,4-dimethylthiophenol.



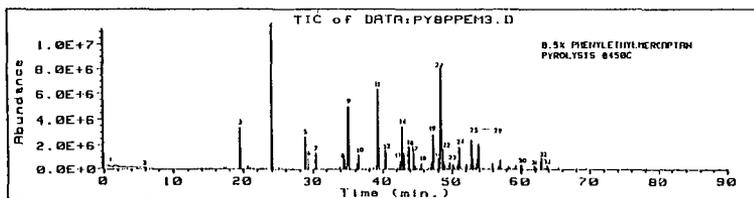
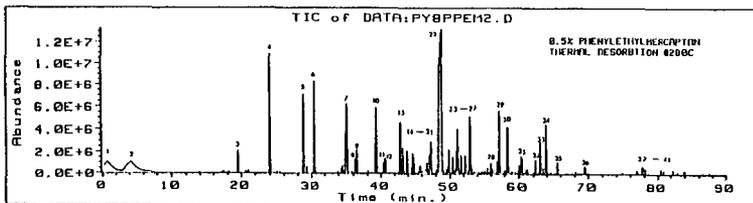
Figures 4a & b. Total Ion Chromatograms of Insoluble Solids From JP-8S + 1.0 % 2-propylphenol and 0.5 % Phenylsulfide. Thermal Desorption at 280 °C (a) and Pyrolysis at 450 °C (b).



Figures 5a & b. Total Ion Chromatograms of Insoluble Solids From JP-8S + 1.0 % 2-propylphenol and 0.5 % Dibenzothiophene



Figures 6a & b. Thermal Desorption @ 280 °C and Pyrolysis @ 450 °C of Solids Formed From JP-8S Doped With 1.0 % 2-propylphenol & 0.5 % Phenylethylmercaptan.



Figures 7a & b Thermal Desorption @ 280 °C and Pyrolysis @ 450 °C of Solids Formed From JP-8S Doped With 1.0 % 2-propylphenol & 0.5 % 3,4-dimethylthiophenol.

