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<p>A successful pilot-plant campaign produced 100 barrels of prototype JP-900 coal-based jet fuel. Most of this fuel was used by Williams International in a long test of their FJ44-3 aviation gas turbine engine. Results of this test program were that coal-based JP-900 is essentially a drop-in replacement for Jet A, except that the higher volumetric energy density of JP-900 would give the aircraft extended range. Laboratory-scale combustion tests showed that JP-900 produced a lower soot volume fraction than JP-8. This fuel, with an added cetane enhancer, was also successfully tested in over-the-road tests in a diesel-engine pickup truck. Blending studies of JP-900 with surrogate paraffinic fuels showed no incompatibility issues in terms of blend properties or stability. In most cases the blend properties were linear averages of the neat components. Continued laboratory-scale development of solvent extraction processing of coal showed that Pittsburgh seam bituminous coal provided 50% conversion to desired feedstock for processing into JP-900. Fuel stability studies showed correlations among the soluble macromolecular oxidatively reactive species, extraction-induced precipitates, and smoke point.</p>					
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FINAL REPORT FOR:

“Advanced Thermally Stable Coal-Based Jet Fuels”

Contract No. FA9550-07-1-0451

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Introduction

This is the final project report for Contract No. FA9550-07-1-0451. This contract was funded for July 1, 2007 through June 30, 2008, but was awarded a no-cost extension to carry the work through June 30, 2009. However, the work performed under this contract was the culmination of a twenty-year project that began in 1989 and that was funded, at various times, by Sandia National Laboratory; the former Pittsburgh Energy Technology Center (now National Energy Technology Laboratory) of the Department of Energy; the Air Force Research Laboratory, Wright-Patterson Air Force Base; and the Air Force Office of Scientific Research. Documentation of that prior work exists in the public domain in the forms of annual and final project reports, publications in the peer-reviewed and conference literature, and student theses. While the content of this particular report focuses on work performed under contract FA9550-07-1-0451, some very brief discussion of work prior to 2007 will be given, to provide context for the present results. For many years, the overall project has been divided (for internal management, not contractually) into three major research thrust areas: fuel production, fuel stability, and fuel combustion. That division will be used to structure the summary of work accomplished.

Fuel Production

Background prior to the present contract

During the lifespan of the entire advanced thermally stable jet fuel project at Penn State University, the original focus for fuel production was on direct coal liquefaction. While that early work was successful from a technical standpoint, it reached completion in the early to mid-1990s, when there was no interest in direct liquefaction at the federal level, and, insofar as we could learn, no plans in the private sector for constructing a liquefaction plant. Consequently the focus was shifted to examine strategies for introducing coal, or coal-derived liquids, into the operations and process streams of oil refineries. Three strategies were identified: adding coal to the feed to delayed cokers, blending coal liquids with a suitable petroleum process stream, or the co-processing of coal with petroleum residua. The last choice was eliminated on the basis that co-processing is simply a tactical variation of direct liquefaction, so would not really represent a major advance.

At the time these decisions were being made, the only coal-derived liquids that satisfied the criteria of retaining some of the molecular “fingerprint” of the parent coal and of being commercially available so that tests could be run through pilot-plant scale were derivatives of coal tar from the metallurgical coke industry. The specific coal tar product selected was refined chemical oil, a distillate rich in two-ring compounds (naphthalenes and indanes) and their derivatives.

However, the noteworthy feature of the coal-based jet fuel process development is its remarkable versatility. What is required as the coal portion of the feedstock is a material rich in two-ring aromatics. *Any coal conversion process* whatsoever that

provides such a material can be used as the “front end” for the coal-based jet fuel production. Options include direct liquefaction, tars from coal carbonization (coking), solvent extracts, coker liquids from coking coal/petroleum blends, or by-product tars from fixed-bed coal gasification. Provided that any of these materials is suitably fractionated to give the desired two-ring aromatic product stream, they can be used interchangeably or in combination as the source of coal-derived feedstock.

The fuel production focused on blending refined chemical oil with light cycle oil (one of the products from a catalytic cracking operation in a refinery), followed by hydrotreating to remove sulfur and then by hydrogenation for partial or complete ring saturation. Although this approach leads to a fuel of excellent quality, the Penn State team recognized in 2002 that eventual commercialization of this process would be severely limited by available supplies of refined chemical oil. This assessment was verified in a follow-on study published by RAND in 2007. It was clearly apparent that an alternative process route was needed to obtain good yields of a product having the same chemical characteristics as refined chemical oil, but one not requiring a coke-making facility as its source. This assessment shifted attention to the use of solvent extraction of coal as a route to “substitute refined chemical oil.” Since the process strategy would involve blending this substitute material with light cycle oil for further refining, the obvious choice of extraction solvent was light cycle oil itself.

In order not to slow down the overall pace of the project while the solvent extraction process was being worked out at laboratory scale and then scaled up, refined chemical oil continued to be used in the pilot plant work to make fuel for engine testing.

Laboratory testing of solvent extraction

In the present work, a suite of five bituminous coals, from Pennsylvania, Utah, Illinois, and West Virginia, were extracted with light cycle oil (LCO). The extractions were done in a 165-mL stirred batch reactor with a fitted impeller. Conditions were 350 °C, 0.7 MPa nitrogen, and 1 hour. The extracted LCO:coal slurry was filtered using a Millipore filter (fine porosity) with a previously weighed PTFE filter. The reactor and the solid were washed with dichloromethane. The resulting solid, i.e., the residue, was transferred to a Petri dish and dried in vacuum at 110 °C and 4 kPa for at least 4 hours, cooled to room temperature and weighed. This was repeated until a constant weight was obtained. The resulting solution, which is called the extract hereafter, was rotary-evaporated in a water bath at 60 °C until all the visible dichloromethane was separated. To eliminate remaining dichloromethane, the recovered material was held overnight in a vacuum oven without heating and then weighed. This was repeated until the loss of weight was less than 200 mg. Conversions were calculated from the weight of initial amount of coal and residue on a dry, ash-free basis. Extract yields were calculated with respect to the initial amount of coal.

To characterize the material extracted from coal, the original LCO and the coal extract obtained at 350 °C with LCO:coal 10:1 were fractionated using preparative liquid chromatography (PLC). Ten-mL fractions were collected in vials that were previously

weighed. The solvents were evaporated to constant weight in a vacuum oven and the weighed to determine the mass of each fraction. Gas chromatography/mass spectrometry (GC/MS) analyses were conducted on the extracts.

At 350 °C at a solvent to coal ratio of 10:1, the conversions were 30–50 wt% and extract yields 28–40 wt% when testing five different coals. When using lower LCO:coal ratios, conversions and extract yields were much lower; lower LCO:coal ratios also caused mechanical issues. LCO is thought to behave similarly to a non-polar, non-hydrogen solvent, which would facilitate heat-induced structural relaxation of the coal followed by solubilization. The main components contributed from the coal to the extract when using Pittsburgh coal are di- and triaromatic compounds.

Extraction of coals with LCO at 350°C, 100 psi N₂, 1 h reaction time, at a 10:1 LCO:coal ratio, can provide conversions of up to ~50 wt%. These results are comparable to those of other researchers using LCO. At lower LCO:coal ratios (5:1 and 3:1), lower conversions were obtained. The higher solids loading at these LCO:coal ratios also contributed to mechanical problems. Based on the LCO:coal ratios tested here, 10:1 would be the preferred. Extraction of Pittsburgh coal generated the types of precursors desired for production of thermally stable jet fuel. The compounds contributed by this coal were mainly two- and three-ring compounds. With hydrotreating to reduce sulfur and nitrogen and saturation of the aromatics, this extract can be expected to produce high-quality jet fuel. Since LCO is already produced in refineries (from the fluid catalytic cracker unit), it could be a useful solvent for process configurations that couple coal conversion upstream with standard downstream hydrotreating, aromatics saturation, and fractionation in a refinery. This offers a route to the production of coal-petroleum blended liquid fuels at lower capital cost than construction of a grass-roots synthetic fuel plant.

Pilot-plant production of test quantities of fuel

As noted, the pilot-scale production of fuel continued to rely on refined chemical oil as the coal-derived input, as a surrogate feedstock until solvent extraction was ready for scale-up.

The pilot-plant work was performed at Intertek-PARC in Harmarville, Pennsylvania. Refined chemical oil was obtained from Koppers, also of Harmarville. It was fractionated to remove components boiling above 300°C, since they have been implicated in having a negative impact on catalyst performance and lifetime in both hydrotreating and hydrogenation. Light cycle oil was obtained from United Refining Co., Warren, Pennsylvania, distilled to a “95% over” point at 310°C. These preliminary fractionations of feedstocks insure that methylnaphthalenes, which are desirable in the feed, are in the still overheads, while undesirable nitrogen-containing compounds and heavier aromatics are in the still bottoms. The refined chemical oil and light cycle oil were blended in 1:1 ratio.

Hydrotreating was performed over commercially available Ni-Mo catalysts at 4.2 MPa pressure, 0.5 space velocity, and 85 m³/bbl inlet hydrogen. The hydrotreated product contained 27–30 wt% tetralin, <5 ppm each of nitrogen and sulfur, and –180°C content of 6%. This hydrotreated product was then hydrogenated over commercially available Pt-Pd catalyst. Hydrogen was introduced at a flow rate of 1.33 m³/hr at 4.2 MPa.

This procedure was followed to produce 100 drums of coal-based jet fuel. Most of this fuel was shipped to Williams International for an engine test, described below in the section on fuel combustion.

Fuel Stability

Background prior to the present contract

The original thrust of this project involved the development of a jet fuel that would be stable to 480°C (900°F) for two hours. Extensive work was done in both batch and flow reactors in evaluating the stability of numerous pure compounds, surrogate mixtures, and actual fuels. This work established the importance of having high concentrations of cycloalkanes (decalin and its derivatives) and of hydroaromatics (tetralin and its derivatives) in the fuel. Such compounds possess both intrinsically high resistance to thermal decomposition and the ability to act as hydrogen donors to shut down free radical chain processes in the decomposition of other fuel constituents, such as alkanes. The outcome of this work was the recognition that a liquid rich in decalins and tetralins would be an ideal base composition for an advanced thermally stable fuel.

Studies of fuel additives had two motivations. One was to develop additives that would extend the thermal stability even beyond what could be achieved with a decalin/tetralin base fuel alone. The other was the investigation of the oxidative stability of these coal-based fuels, and possible additives for enhancing oxidative stability. Although the primary focus in the early years of this program was the stability of the fuel at $\geq 400^\circ\text{C}$, even a fuel intended as a heat-sink for thermal management must still pass through the lower-temperature regime of oxidative degradation, so that the stability of the fuel in that regime is also important.

The principal experimental techniques used in this work were sealed microautoclave reactors, which are especially useful for quick and inexpensive scouting studies, and simple flow reactor rigs, based on a design obtained from colleagues at the Air Force Research Laboratory.

Soluble macromolecular oxidatively reactive species

The formation of high-molecular weight materials called soluble macromolecular oxidatively reactive species (SMORS) can increase both the thermal oxidative deposit formation and the fuel soot propensity, as measured by smoke point. Microautoclave testing has shown that thermal oxidative stressing significantly increases the mass of extraction-induced precipitate (EIP) while SMORS volumes remain constant. It appears

that oxidative stress transforms reactive polar compounds in the fuel into low-molecular-weight SMORS; this is followed by their conversion into higher-molecular-weight SMORS, which can be isolated as EIP. Polar compounds indigenous to the fuel likely produce SMORS of various molecular weights. Minimal EIP mass was isolated from three different unstressed fuels, which suggests that long periods of ambient storage are needed to generate the higher-molecular-weight SMORS. EIP mass appears to be limited by fuel solubility. Similarly, high-molecular-weight SMORS have limited solubility in jet fuels.

The relationship of SMORS with smoke point was established by, first, isolating a composite SMORS sample by oxidative stressing of fuels. The fuels used in this work were four different samples of petroleum-derived JP-8 provided by our colleagues at the Air Force Research Laboratory. Adding this prepared SMORS to four samples of actual jet fuels reduced the smoke point (i.e., increased the sooting tendency) in every case. The practical implication of these findings is that there is likely a significant drop in jet fuel smoke point prior to combustion, due to thermal oxidative degradation. This observation is consistent with the poor air quality, in terms of particulate matter, generally observed at busy commercial airports. However, we have also developed an interesting possible solution to this problem.

A significant increase in smoke point is observed when the petroleum-derived JP-8 samples are blended with a Fischer-Tropsch (FT) jet fuel to make a 1:1 volume blend. This observation is consistent with dilution of the high-molecular-weight SMORS by the FT fuel. In fact, it appears that the FT-diluted fuel is losing high-molecular-weight SMORS faster than they can be produced by stressing. The blend is a poorer solvent for the high-molecular-weight SMORS.

Fuel blending studies

The study on SMORS, EIP, and smoke point just described highlights possible benefits to be obtained from blending of fuels. Although that work involved conventional petroleum-derived JP-8, it has also seemed that the coal-based JP-900 merits consideration not only as a fuel in its own right, but as a blend stock for petroleum, FT, or bio-based fuels. The JP-900 is clearly superior in terms of high-temperature thermal stability and volumetric energy density. Blending with one of these other fuels could take advantage, in principle, of the best properties of both components of the blend, thereby producing what might be considered as a "super-jet" fuel. Further, this approach could allow JP-900 to transition into production and into use in a gradual, evolutionary manner, requiring much less capital investment in new production plant, and potentially easier transition into the fleet.

A blending study is now in progress, aimed at evaluating the effects of blending JP-900 into two surrogate models of an FT fuel, neat dodecane and Norpar-13. The latter material is a commercially available solvent that is a mixture of linear alkanes of about C₁₁ to C₁₅, with C₁₃ being dominant. This study has involved the preparation of blends of coal-based JP-900 with dodecane and, separately, with Norpar-13 in 10% increments,

ranging from 10% JP-900 to 90% JP-900 in the blend. Various physical and chemical properties of the blends were determined using the relevant ASTM procedures. Specifically, these included the density, API gravity, flash point, freeze point, kinematic viscosity, heat of combustion, smoke point, hydrogen content, and sulfur content. For the most part, properties of the blends showed a linear dependence on the percentages of the two blend stocks (i.e., JP-900 and either dodecane or Norpar-13), with very good linear regression fits. Furthermore, there was no visual evidence of incompatibility, such as formation of precipitates or phase separation. The high-temperature thermal stability of the blends, whether measured by cracking to gas or deposition of solids, was directly dependent on the amount of JP-900 in the blend; the more JP-900, the less cracking and less deposition.

This study is still a work in progress, even though the relevant AFOSR contract has expired. It is expected that the student performing this work will complete her thesis in the 2009/10 academic year. Therefore, the results reported here should still be regarded as preliminary, and may be subject to change. As has been done in the past, once the completed thesis has been vetted by appropriate Penn State faculty, a approved copy will be provided to our colleagues at the Air Force Research Laboratory.

These results show that JP-900 could be freely mixed with paraffinic fuels. The properties of the blend generally reflect the weighted linear average of the properties of the two blend stocks, i.e., JP-900 and either dodecane or Norpar-13. Addition of JP-900 enhances high-temperature thermal stability. These results strongly suggest that JP-900 could be an ideal candidate for blending with FT fuels. In fact, Penn State University now has a strategic alliance with the world's largest producer of FT fuels, for comparative studies of JP-900, FT jet fuel, and blends.

Fuel combustion

Background prior to the present contract

Fundamental combustion research on JP-900 and its early prototypes utilized the state-of-the-art model gas turbine facilities in Penn State's Propulsion Engineering Research Center. In addition to the contributions to combustion science gained by studying this unique coal-based fuel very important practical aspect of this work was the continuous feedback to the fuel production and fuel stability portions of this project. This interdisciplinary collaboration among combustion research, fuel formulation, and production teams was especially helpful in determining the necessary hydrotreating protocols for making a prototype fuel that would meet smoke-point specifications.

The most important combustion work in actual engines was conducted in a very successful test in a T-63 turboshaft engine at the Air Force Research Laboratory. The engine was started on JP-8, run through idle and cruise cycles, switched to JP-900 and run through the same cycles, then switched back to JP-8 and the cycles repeated once more. Although small differences in emissions and performance were noted, there were no practical, major differences between operation on the two fuels. This test

demonstrated the potential for considering JP-900 to be a potential coal-based drop-in replacement for JP-8 (albeit with substantially superior thermal stability and higher volumetric energy density), rather than relegating JP-900 to the status of a “boutique fuel” only for aircraft needing a high-heat-sink fuel for thermal management.

Sooting tendency

Prototype JP-900 meets the smoke point specification for JP-8, with different batches having measured smoke points of 19 and 20.3 mm. However, the smoke point measurement alone does not provide an indication of the amount of soot that would be formed when a fuel is burned under conditions typical of a jet engine. Direct measurements of soot formation were made in a model gas turbine combustor. Comparison studies were made with a sample of JP-8 provided by colleagues at the Air Force Research Laboratory, and with an 80:20 blend of heptane and toluene. The heptane:toluene blend has a smoke point similar to that of JP-8.

JP-900 shows a steep increase in soot production as the equivalence ratio increases above $\phi = 1.0$. Both JP-900 and FT fuel tend to show the lowest NO_x formation, among a suite of fuels that included JP-8 and a 50:50 blend of JP-8 with FT jet fuel. Among these fuels, FT had the lowest soot formation, with correlates with its very low concentration of aromatics. Overall, however, JP-900 could be a good candidate fuel to meet aviation purposes without requiring changes to existing engines.

The key finding of this work is that both batches of prototype JP-900 had soot volume fractions equivalent to, or slightly *lower* than, JP-8. As an interesting sidelight, the JP-900 batch with the lower smoke point (19 mm), also had a lower soot volume fraction than the batch having a 20.3 mm smoke point, contrary to what might have been predicted. However, the very important result of this work is its demonstration that coal-based fuels can be refined to fuels equivalent or *superior* to JP-8 in terms of formation of soot, notwithstanding much earlier research by others that suggested that coal-based fuels will produce high concentrations of soot. We envision future work, under an agreement with a major producer of FT fuels, to involve combustion studies on blends of JP-900 with FT fuel.

Single battlefield fuel

For diesel-engine applications, a direct relationship has been established between tetralin or decalin content of the fuel and injection timing. As tetralin or decalin content increased, injection timing also increased. This work also revealed a strong effect of engine timing on NO_x emissions. Particulate matter (PM) emissions decreased as tetralin content increased from 0 to 10%, but thereafter increased with further increases of tetralin content. Except for the initial stage, the trend corresponds with what would be expected from comparable behavior in sooting tendency.

The derived cetane number of JP-900 was 33, although it is important to recognize that this batch of JP-900 was cut to provide optimum jet fuel behavior, not

diesel, nor some compromise between the two. A large concentration of monocyclic aliphatic compounds and tetralins contributed to the low cetane number, along with very low concentration of linear alkanes. Addition of 5000 ppm ethylhexyl nitrate raised the cetane number to 43, which is comparable with commercially available ultra-low sulfur diesel fuel. (The sulfur content of the JP-900 as tested as 1 ppm, well below the 15 ppm maximum specification for ultra-low sulfur diesel.)

The combustion performance of JP-900 with 5000 ppm of added ethylhexyl nitrate was evaluated in an on-road test. A stock Ford F-350 truck with no engine modifications was fueled with the JP-900 with additive and driven 560 km in mixed stop-and-go and open highway driving. At that point the truck was fueled with a 25:75 blend of the treated JP-900 and petroleum-derived ultra-low sulfur diesel, and driven another 560 km on the same route and same driving conditions. No differences could be discerned in engine performance, nor were any detrimental effects of the JP-900 observed.

Gas turbine engine test

Most of the JP-900 produced in the pilot-plant campaign described above was shipped to Williams International, Walled Lake, Michigan, for a test in one of their commercial gas turbine engines. An FJ44-3 engine, a small gas turbine widely used in the general aviation market, was powered by two thousand gallons of coal-based JP-900 for 118 cycles during 21 hours of running at Williams' Walled Lake facility. Every model in the FJ44 series is clean enough to incur no emissions landing surcharges (per Swiss regulations). The FJ44-3 engine powers the Cessna CJ2+ and CJ3 and the Beechcraft Premier II, and is used to re-engine many older aircraft to reduce their fuel consumption and emissions. The JP-900 fuel performed extremely well; the engine performance was identical to using Jet-A fuel and required no special engine or test cell modifications for the demonstration. The fact that the sulfur content was ≈ 1 ppm meant that there were essentially no SO_x emissions. The higher volumetric energy density, relative to Jet A or JP-8, was confirmed to extend aircraft range. This work validates and extends the earlier tests on a T-63 turboshaft engine at the Air Force Research Laboratory.

Williams International is promoting development and certification of alternative fuels for commercial aviation, and their specific plans include emissions and smoke measurement testing and engine testing of alternative fuels. Williams International also participates in the Commercial Aviation Alternative Fuels Initiative (CAAFI), an organization that seeks to enhance energy security and environmental sustainability for aviation through alternative jet fuels.

Coda

This is the last-ever report to be submitted as part of what has been a twenty-year project. The foregoing material has focused primarily on the achievements of the two years of contract FA 9550-07-1-0451, with some background material to place the present results in context. However, this section summarizes the highlights of the

accomplishments of the entire program, which are reported in detail in prior annual and final contract reports, publications in the peer-reviewed and conference literature, and student theses, all of which are in the public domain.

- Several processes have been developed to produce a coal-based jet fuel, JP-900, feeding coal or coal-derived liquids to the operations (hydrotreating, hydrogenation, and fractionation) of existing oil refineries.
- A start-up company, CoalStar Industries Inc., has been established in Johnstown, Pennsylvania for the purpose of commercializing this coal-based fuel technology. A second start-up, Integrated Synthetic Fuel, Inc., of Memphis, Tennessee, has also expressed interest in this technology. Therefore at least two groups are working to move this AFOSR-funded technology into commercial practice.
- The prototype JP-900 fuel meets or exceeds most of the specifications of JP-8 and Jet A. Among other things, it has ≈ 1 ppm sulfur and $< 2\%$ aromatics. However, it also meets the original target of high-temperature stability, 2 hours at 480°C (900°) set for us by the Air Force in 1989. This fuel could be a potential coal-based drop-in replacement for these current fuels, or could be used as a high-heat-sink fuel for thermal management.
- Prototype JP-900 fuel has been successfully tested in a T-63 turboshaft engine, a FJ44-3 gas turbine engine, and a Ford F350 light truck diesel engine. In all cases, differences in engine performance and emissions compared to the respective conventional petroleum-derived fuels were minimal. JP-900 has also been successfully tested in a solid oxide fuel cell. This work demonstrates clearly the potential application of coal-based JP-900 as a single battlefield fuel.
- Detailed briefings on JP-900 technology have been provided for management of a major American airline, a major national and international air freight company, and a major railroad that services 22 states, in regard to their interest in moving this coal-based fuel technology into the private sector.
- Penn State University has established a strategic alliance with the world's leading provider of Fischer-Tropsch fuels for research and development in coal-based fuels, including blending of JP-900 and FT liquids for improved performance.
- Penn State has also established an informal collaborative alliance with a South African university in the area of solvent extraction of coals for liquid fuel production.