2.0 TITLE

DEVELOPMENT OF ANODE BINDER & ASPHALT SUBSTITUTE MATERIALS FROM DGC'S TAR OIL PITCH

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3.0 INDEX

4.0. Abstract

Tar oil produced from lignite in a Lurgi gasifier or other low temperature gasification process has the potential to be upgraded into a number of useful and more valuable products than being consumed as a fuel. DGC has and continues to study upgrading technologies to produce cresylic acid from one fraction of tar oil. DGC has also recognized that a resulting tar oil pitch might be upgradable into anode binder blendstock and asphalt substitutes for roofing systems and driveway sealers. DGC's potential volume of about 200 MM lbs/year of pitch could fit well into the North American market place for anode binder feedstock of 450 MM lbs/year. In the spring of 1995 DGC started working with AlliedSignal who are well recognized in the tar oil industry.

AlliedSignal has over 130 years of experience how to upgrade tar oil pitch. Because of a declining supply of tar oil pitch from coke oven plants, they are looking into new sources of feedstock. Earlier this year they tested tar oil pitch samples from DGC and found that using their proprietary technology they could raise the softening point of the material into the range of 220°F to 240°F which is required to make it a suitable material as anode binder. As a result of this encouraging finding, pilot plant scale development work has been recommended.

Both AlliedSignal and DGC are very much interested in demonstrating that AlliedSignal's technology works on a larger scale to produce anode binder blend stock from DGC's tar oil stream. We arrived at the following development program:

a) Construct a batch tar oil pitch oxidizer which is sized to produce barrel quantities of product.

b) Produce approximately 10 batches of product. Each batch will provide more information on the most suitable process conditions to achieve the necessary polymerization and rearrangement reactions via oxidation to produce a blending stock for anode binder material and possibly other applications.

A three week campaign of tar oil pitch oxidation runs is the heart of this development program. AlliedSignal will provide on site expert advise how to interpret the data and how to modify the heat-up rates, oxidation rates, length of operation, reflux of overhead material, etc. to produce a most desirable product. Softening point determinations will provide on the spot feedback while other analyses such as coking value and TGA residue are used to better characterize the final product from a batch.

It is planned to carry out as much as possible of this program in the first quarter of 1996. The total cost for this project has been estimated at $150,000, not including a potential contingency of $37,500 for additional days of pilot plant operation and an estimated cost of $25,000 for AlliedSignal's manpower and site expenses in support of the program.

The development program includes two types of risks. The first risk is of a technical nature. Because of the successful completion of bench scale testing, this risk is considered relatively low. The second risk is of an economic nature. Profit margins for these type of products are small. The product is a mixture of tar oil compounds and therefore does not compare with high priced single compound chemical intermediates. Part of this development work will result in process information that will be necessary for the economics evaluation for batch and/or continuous operation. Another major need

for these pilot plant runs is production of trade samples which lets others evaluate how they can make anodes, etc.

The major participants in this program will be DGC, acting as project coordinator and provider of facilities, and AlliedSignal, acting as technology adviser.

5.0. Project Summary

In 1996, DGC's R&D group plans to pursue (on the pilot plant level) the conversion of tar oil pitch to an anode binder blending stock and an asphalt substitute using thermal oxidation and polymerization. The overall development program is expected to involve the following steps:

1) pilot-scale batch tests and parametric studies;
2) preliminary economic analysis;
3) additional pilot-scale tests using continuous operation;
4) establishment of a firm process scheme; and
5) detailed economic analysis.

Initial bench scale studies for upgrading of DGC's tar pitch have been carried out by AlliedSignal in 1995. The results were most encouraging and efforts are now underway at DGC to proceed with step (1) of the remaining development plan. This step includes pilot-scale batch testing and parametric studies, along with production of trade samples. Step (1) consists of several tasks, some of which have already been completed:

A) the atmospheric drying of tar oil to remove water and naphtha;
B) the vacuum distillation of dry tar oil to remove the fraction containing cresylic acid and to produce pitch;
C) the design and construction of a pilot-scale oxidation reactor;
D) thermal oxidation of the pitch at several processing conditions to yield asphalt and/or anode binder pitch.

Tasks (A) and (B) have already been performed by DGC. This work was done in anticipation of this need, as well as to obtain tar oil distillate for another ongoing research program. The pilot-scale distillations produced approximately 4500 pounds of tar oil pitch, almost all of which is available to DGC for thermal oxidation studies.

The work being proposed for partial funding by the Lignite Council is that identified as Tasks (C) and (D) of the pilot plant program (see above). The work includes the design and construction of a pilot-scale oxidation reactor, followed by the parametric testing of process variables to yield different grades of pitch products. The primary product property that will be studied is how the softening point is impacted by the oxidation conditions.

The expected results from this study include 1) the generation of sufficient engineering data and process definition to proceed with a preliminary economic analysis, and 2) the production of several "drum-size" trade samples for testing and evaluation by potential customers.

The total cost to DGC for the proposed project is estimated at $150,250. This project will be managed by DGC. Input regarding design parameters for the equipment and pilot plant run conditions as well as product evaluation will be obtained from AlliedSignal. The duration of the proposed project is anticipated to be about 3 months.

6.0. Project Description

6.1. Introduction

Coal tar pitch is an essential ingredient in the manufacture of electric arc furnace electrodes and reduction anodes used in the production f steel and aluminum. The high carbon content and binding characteristics provides electrodes with high density and conductive properties vital to these industries.

Coal tar pitch is also custom produced to suit individual process demands for application in built up roofing and waterproofing systems. Additional uses include the manufacture of activated carbon for pollution control systems, refractory brick and blast furnace supplies.

Pitch is a component of high temperature coke oven tar which is a by-product from the carbonization of coal to produce coke. Domestic coke making has declined in recent years as older coke batteries reach obsolescence or fail to meet stricter pollution control requirements mandated by the Clean Air Act amendments of 1990. Faced with the prospect of a diminished raw material supply, AlliedSignal has conducted extensive investigations to identify alternate feedstocks in order to meet the supply needs of an expanding aluminum industry. One promising feedstock is tar oil derived from North Dakota lignite coal.

AlliedSignal has developed a technology which converts tar oil components to pitch by increasing the size of low molecular weight fractions present in this material. The process involves a combination of polymerization and rearrangement reactions forming a pitch extender which can be blended with AlliedSignal's standard high temperature coal tar pitch to product an acceptable binder material.

The softening point is the primary product property to characterize material needed in various applications. As an example anode binder pitch should have a softening point in the 220°F to 240°F, driveway sealers 100°F and roofing compounds 130°F to 140°F, oxidation of tar oil pitch at elevated temperatures increases the softening point over time.

Based on bench scale exploratory testing of DGC tar oil pitch, AlliedSignal concluded that a next phase of pilot plant scale production of air oxidized tar oil pitch was warranted. Since two suitable tar oil fractions have been received from an earlier pilot plant distillation, the proposed program consists of two major tasks: (numbering is same as in project summary) C) the design and construction of a pilot-scale, batch, thermal oxidation reactor and D) operation of the reactor to study the effects of various operating parameters. As a result of discussions with AlliedSignal, it was agreed that a total of ten "55 gallon drum" size batches should be sufficient to explore the various production run scenarios for effective air blowing of DGC's tar oil pitch. These samples will be supplied to potential customers for testing and evaluation. As mentioned previously, other tasks which involved the preparation of the tar oil pitch have already been completed. A description of the remaining tasks is given below:

6.2. Task (C) - Design and construction of a batch thermal oxidation reactor.

Under this task, DGC will design and build a small batch thermal oxidation reactor. The reactor will be sized to hold approximately 55 gallons of feed material and will be electrically heated using two 7.5 kilowatt electric immersion heaters. The reaction vessel will be designed to withstand pressures of up to 15 psig at temperatures of 700°F, and will include provisions to condense volatile organics. The non-condensable vent gases will be scrubbed using a dilute sodium hydroxide solution before release to the atmosphere. Oxidation of the tar oil pitch will be accomplished by sparging the reaction vessel with compressed air. Sufficient instrumentation will be included to provide temperature, pressure and flow control. Scales will be included to generate material balance data and to monitor devolatilization rates. Piping and valving will include provisions for real time sampling. The entire unit will be skid mounted with the exception of the scales.

Estimated cost for design and construction of the unit is **$25,000.**

6.3. Task (D) - Operation of the reactor to study the effects of various operating parameters.

In this task, the oxidation reactor will be used to study the effects of several operating parameters on the quality of the finished product. The variables to be studied include the reaction temperature range, ramp up rate for reaction temperature, residence time, aeration rate, refluxing of condensate and feed composition. DGC has two tar oil pitch samples available for the study, differing from one another by the severity of the vacuum distillation used in their production. One pitch sample represents about 60% of the raw tar oil, while the other sample represents about 50%. Each sample is about 250 gallons in volume.

On site expertise for the air blowing experiments of tar oil pitch will be provided by AlliedSignal. AlliedSignal brings to the program a vast amount of experience that will be used in this program to make operating adjustments as needed. Benefits of these process adjustments during each batch can be monitored quickly by performing a softening point analysis. Ultimately, if a certain increase in softening temperature has not been reached in a given time, the oxidation may be continued until the softening temperature has risen sufficiently. Other properties which are also impacted by this operation such as residual volatiles, coking value, etc., will be determined as needed.

As many as ten (10) batch oxidation tests may be performed depending upon the results. During each test, samples will be taken and analyzed on a routine basis. Hourly readings of field and panel instruments will be collected and entered into a computer data base, which will be used for material balance and yield calculations, as well as determining potential correlations for product properties versus treatment conditions.

Product quality will be gauged by several measurable parameters, including softening point, sulfur and ash content, coking value, residual volatiles, flash

point and residue after thermogravimetric analysis (TGA). In general, the analytical work will be closely coordinated with the recommendations by AlliedSignal.

The estimated cost to DGC for this task is **$150,250 (+/- $39,125 contingency**).

6.4. Deliverables

The deliverables from this project include on a "non-proprietary" basis one progress report and one final report. These reports will summarize the status of the work along with a discussion of the major achievements and/or difficulties. Proprietary scientific and operating data will not be included in the reports. However, appropriate product characteristics and analytical results will be provided. In addition, the reports will offer sufficient information to define the success of the program.

7.0. Standards of Success

The standard of success for this project will be the production of tar oil pitch trade samples which meet with a favorable market response. The samples will be either an anode binder pitch or an asphalt substitute or both. The technical feedback from this project will help DGC to perform a preliminary economic analysis of the process and will provide guidance as to whether further continuous testing is warranted. Ultimately, success of the project should lead to the commercialization of tar oil processing at the Great Plains Synfuels Plant and the sale of up to 200 MM pounds per year of upgraded tar oil pitch.

8.0. Background

Tar oil derived from gasification of North Dakota lignite is a complex mixture of organic compounds which distill over a wide boiling range. The mixture contains a naphtha fraction, a cresylic acid/neutral oil fraction, a naphthols fraction, and a pitch fraction. In addition, the tar oil also contains a small amount of water.

All of these fractions have profit potential, if they can be separated and purified to meet market requirements. The first three fractions have been investigated to varying extents, although last year's studies on the naphthols fraction, as reported to the Industrial Commission under the grant program FY 94-XIV-48, have proved to be discouraging. DGC is now focusing its attention to further develop the purification steps for the cresylic acid fraction and to develop uses for the pitch from tar oil.

Since marketing of a finished tar pitch product into various markets such as anode binder blend stock and roofing and water-proofing applications requires commercial experience, recognition in the field and know-how of the needs for down-stream customers, DGC decided to work with AlliedSignal in developing this product.

AlliedSignal has been a leading producer of coal tar based products for over 130 years. In that time Allied's products have been used in many industries including transportation, construction, aluminum, steel and chemicals. In 1892, the first by-product recovery coke ovens were built at Syracuse NY using an AlliedSignal design. While today's modern plants bear little resemblance to these early designs, the evolution of significant change began to occur in the 1950's. Improved efficiency coupled with recession and reduced demand for steel resulted in a steady decline in US tar production from 1950 to 1980. Furthermore since 1980, domestic coal tar production has dropped by half. During the past fifteen years several 30-40 year old coke ovens, which needed costly upgrades to meet tighter pollution controls, were shut down. While these steel makers now rely on outside suppliers for their coke, producers of coal tar products have been forced to develop alternative feedstock sources or identify other replacements for the diminished supply of coal tar.

Projections for the future are that additional coke ovens will shut down although at a slower rate than the early 1990's as even stricter pollution control regulations under the Clean Air Act amendments of 1990 go into effect.

The North American market for anode binder pitch is in the order of 450,000 tons per year of which AlliedSignal has more than a one-third share. DGC's potential supply of tar oil pitch into this market is in the order of 100,000 tons per year. Considering a market value of $280 per ton, the potential revenues for this material could approach $28MM per year.

These considerations are reasons why both DGC and AlliedSignal are interested to determine if tar oil pitch from North Dakota lignite can be converted to a pitch which is suitable as anode binder feedstock or as an asphalt substitute.

9.0. Qualifications

Dakota Gasification Company (DGC), a wholly owned subsidiary of Basin Electric Power Cooperative (BEPC) has owned and operated the Great Plains Gasification Facility since it was acquired from DOE in October 1988. DGC has and continues to show a great interest in developing by-products from the gasification complex. Aside from building commercial facilities for production of krypton/xenon, phenol, cresylic acid, ammonia, and ammonium sulfate, DGC continues to invest heavily in facilities and people to enhance development work of new technologies to separate, purify, and synthesize coal derived chemical by-products.

DGC maintains excellent plant and by-product laboratory facilities to support these R&D efforts. DGC's Process Development Department is highly esteemed by outsiders, and has performed contract research work for outside clients. The same staff of engineers and chemists will be dedicated to the proposed program. Attachment A provides resumes for the Principal Investigator and a few of the professionals involved in the project.

A significant contribution to this development program is provided by AlliedSignal. Their vast historical experience extending over more than 130 years as well as their modern up-to-date technical and analytical capabilities are a great asset to the execution of this program. AlliedSignal is a major producer of anode binder material providing about 1/3 of the US market and a similar figure for the Canadian market.

10.0. Value to North Dakota

Development of marketable tar oil pitch products is beneficial to North Dakota in several ways. First of all, DGC's tar oil stream may be able to produce in the order of 200 MM lb/yr of anode binder pitch and/or asphalt substitute. At market prices of $0.14/lb., the potential yearly revenues are estimated at over $20 MM/year. In addition, commercialization of tar oil processing would also encourage production of over 40 MM lb/year of cresylic acid which would also be sold into the marketplace.

Although the revenues generated by the tar oil pitch and the tar oil cresylic acid sales would primarily benefit DGC and AlliedSignal, the necessary plant addition will create temporary construction jobs in North Dakota and contribute to the financial stability of DGC. Together with other plant expansion projects, tar oil processing may also contribute to an increase in the plant labor force, thus creating additional tax revenues from these plant employees as well as from their impact on the North Dakota economy.

11.0. Management

This project is managed by Dakota Gasification Company. It will be executed under the direction of Mr. Alfred Kuhn, Manager of the Process Development Department. Mr. Kuhn will be responsible for the reporting of progress to the North Dakota Industrial Commission. He will also direct the efforts within his department. Mr. David Duncan will have responsibility for the analytical support given the project.

Four engineers will provide supervision to the pilot plant operations group as needed on a 7 day/week basis. Mr. Duncan is assisted by two additional chemists and one laboratory technician. Senior management input from AlliedSignal will be obtained from Howard Simon, Project Manager, Binder Pitch. AlliedSignal also will have at least one representative present in Beulah when the test runs are being carried out.

12.0. Timetable

Dakota Gasification Company is prepared to initiate some of the proposed work (Task 6.2) in January of 1996. it is anticipated that the batch oxidation reactor can be designed and built within four to six weeks and that the pilot scale exploration studies can be completed eight weeks after the equipment is available. The following potential schedule lists key events:

February 29, 1996 -	Fabrication and Commissioning of Pilot Scale Oxidation Reactor is complete.
March 20, 1996 -	Interim Report
April 20, 1996 -	Thermal Oxidation Parametric Studies are complete.
May 15, 1996 -	Final Report to ND Industrial Commission.

13.0. Budget

The following information represents the current best estimate relating to the project cost. Necessary R & D man-hours are difficult to predict. It is common that additional efforts are required to explore other options when the first results are not conclusive or do not adequately answer all questions. This estimate assumes that 10 separate batch thermal oxidation reactions will be studied and that a total of three weeks of operating time will be used.

Design, materials and fabrication of pilot-scale, batch, thermal oxidation reactor:	$ 25,000
Operation of pilot thermal oxidation reactor and completion of parametric study (10 batch tests) (20 days @ $3,500/day)	$ 70,000
Laboratory Services, 10 days @ $825	$ 8,250
Data Evaluation, Report Writing:	$ 12,000
Value of Feedstock (1)	$ 35,000
DGC Subtotal	$ 150,250
Allied Signal Support (2)	$ 25,000
Project Total	$ 175,250
Contingency (10 days operation, 5 days laboratory support)	$ 39,125
Maximum Potential Project Cost:	$ 214,375

Note: 1) Preparation of the feedstock required 20 days of pilot plant distillation work. Splitting this cost between tar oil distillate and pitch products results in a value of the tar pitch feedstock for this program of $35,000.

2) AlliedSignal's expenditures include manhour charges for test preparations, test participation, evaluations and consultations, as well as management supervision and travel expenses for test program.

14. Matching Funds

DGC is prepared to provide the time, material, expertise and financing to carry out this project. However, DGC would like to request matching funds as follows:

North Dakota Lignite Research Fund	$ 60,000
Dakota Gasification Company	90,250
	$150,250

15.0 Tax Liability

I, Rod J. Kuhn, certify that Basin Electric Power Cooperative and its wholly owned subsidiary Dakota Gasification Company, are not delinquent in any tax liability owed to the State of North Dakota.

Rod J. Kuhn, CPA
Tax and Insurance Manager
Basin Electric Power Cooperative

12/22/95

16. Confidential Information

No confidential information is contained in this proposal. However, any specific process descriptions and requests for detailed study reports would most likely have to be handled as confidential information. DGC will issue the interim and the final report on a non-confidential basis by eliminating some of the specific operating data for the batch reactions. Product characterizations will be provided in sufficient detail to size up the viability of the products for their intended application.