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June

CLIFFORD R. PORTER Director, Lignite Research & Development Program

State of North Dakota The Industrial Commission State Capitol Bismarck, ND 58505

Attn: Lignite Research Program

Dear Commission Member:

Dakota Gasification Company (DGC) submits the attached proposal (35 copies) for a lignite research grant. The proposal seeks partial funding and helps to accelerate the study of separation and purification techniques for naphthols from DGC's tar oil stream. Naphthols have a high market value but the DGC source of naphthols requires substantial upgrading to meet commercial specifications.

DGC has an excellent staff to perform this type of development work. For example, prior proprietary development work by DGC has led to construction of a process for recovering salable phenol from the crude stream at the gasification plant. A similar course is being pursued with catechols. This research effort clearly increases further the possibility of deriving commercially salable chemicals obtained from gasifying North Dakota Lignite. Some of the ideas to be pursued in the proposed bench scale work may lead to patentable processing concepts. Therefore, the scope description for the laboratory work tends to be generalized. DGC is prepared to present results from this project on a non-confidential basis, similar to work that had been performed under DOE ownership of the Great Plains facility.

DGC proposes to carry out the development work in two phases. The first phase explores the potential technologies and characterizes various streams containing the naphthols, while a second phase demonstrates the necessary technology on a pilot plant scale. The total project costs have been estimated at \$115,000 for Phase 1 and \$930,000 for Phase 2.

DGC is requesting \$50,000 for Phase 1 from the North Dakota Industrial Commission, with an additional \$200,000 for Phase 2. All other funding will be supplied by DGC.

The project will require about fifteen months to completion, at which time a final summary report will be issued.

Equal Employment Opportunity Employer M/F/H/V State of North Dakota June 30, 1993 Page 2

We ask that you give this proposal serious consideration. If there are any questions, please do not hesitate to contact me (221-4410) for additional information.

Sincerely,

Kent anssin

Kent E. Janssen Vice President and Chief Operating Officer

KEJ/AKK/jsd Enc. - Proposal

- \$100 Registration Fee

- Tax

2.0 <u>TITLE</u>

DEVELOPMENT OF COMMERCIAL GRADE NAPHTHOL

FROM DGC'S TAR OIL

Applicant:

Dakota Gasification Company Bismarck, ND

Principal Investigator:

Alfred K. Kuhn Process Development Manager Dakota Gasification Company P.O. Box 1149 Beulah, ND 58523

Date:

June 30, 1993

Amount of Request:

 Phase 1
 \$ 50,000

 Phase 2
 200,000

 Tota1
 \$250,000

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4.0 ABSTRACT

The objectives of this development project are twofold. During Phase 1, basic bench scale explorations of how to produce commercially acceptable naphthols (both alpha and beta isomers) from a tar oil fraction will be carried out. Based on the literature search and earlier work performed, it is anticipated that some of the investigations have to be treated as confidential until patent rights have been granted. The findings will be instrumental in forecasting preliminary economics of a commercial facility to produce naphthols at DGC in North Dakota. Naphthols are used to manufacture products such as dyes, lubricating oil additives, specialty polymers and antioxidants. During Phase 2, pilot plant runs will be performed to demonstrate the technology, obtain performance data for the design of a commercial plant, and to produce trade samples. This effort will allow a more accurate forecast of the economics. Trade samples will be used to establish acceptability of naphthol produced from coal in the market place.

Additional laboratory equipment is needed to accelerate DGC's program of developing naphthols derived from gasification of North Dakota Lignite. In particular, an additional high performance liquid chromatograph (HPLC) and a fume hood are required to permit working concurrently on the new by-product development programs for production of naphthols while other development work is in progress.

The majority of the proposed work could be performed between October 1993 and December 1994. The total cost of the project has been estimated at \$1,045,000. It can be broken down into \$115,000 for Phase 1 and \$930,000 for Phase 2. The amount which is requested to be funded by the North Dakota Industrial Commission is \$50,000 for Phase 1, and an additional \$200,000 for Phase 2.

The major participant in this project will be Dakota Gasification Company.

5.0 PROJECT SUMMARY

Dakota Gasification Company (DGC) plans to pursue the development of commercial grade alpha- and beta-naphthol, derived from the tar oil stream produced from gasifying North Dakota Lignite at DGC's coal gasification facility. The first study phase consists of bench scale investigations on how to quantify, separate, and purify naphthols from tar oil. Appropriate analytical procedures to identify and quantify naphthols, as well as other polar, neutral oil, and tar base compounds will be developed both for gas chromatography (GC) and high performance liquid chromatograph (HPLC) equipment.

The expected results from this study phase include a determination of potential product quality for naphthols derived from DGC's tar oil stream, a definition of the basic processing scheme for a naphthols production facility at DGC, and a basic appreciation of the residual impurities in the naphthols. The equipment purchased and analytical methods developed during this phase will also be used for subsequent pilot plant scale work on naphthols.

During the second phase of the project, the above developed information will be used to demonstrate the process technology on a pilot plant scale. Operating data will be collected during the various tasks of the pilot plant operation, including drying, topping, depitching, and fractionating the tar oil feed stream, to arrive at the cut which contains the naphthols. These separations will be achieved in a column configured for processing tar oil.

Removal of impurities to achieve commercial grade alpha- and beta-naphthol will be carried out in other pilot plant equipment such as the dual solvent extractor and/or the 6" fractionator. Depending on the findings in phase one, decisions will be made about which equipment will be used. At the end of this second phase sufficient data will have been developed to define the necessary design parameters for a commercial plant facility, and enough product will be available for extensive identification of any remaining impurities and for use as trade samples.

Ultimately, sales of naphthols in the order of \$12 MM/Yr. may be realized. Development of naphthols as salable by-products will also be beneficial in connection with marketing efforts to sell a lighter boiling tar oil fraction which contains cresylic acid. Marketing these additional liquid by-products will enhance the economic stability of DGC's coal gasification facility. In addition, the employment of a construction work force to build such a liquid by-product facility will benefit North Dakota directly.

The total cost for the proposed project is estimated at \$1,045,000. This project will be managed solely by DGC. The duration of the proposed project is anticipated to be about 15 months. A. Kuhn, manager of the Process Development Department, has been associated in various capacities with the Great Plains Coal Gasification Project for over 17 years. He will assume the responsibility for the program.

6.0 BACKGROUND

6.1 Introduction

Tar oil derived from gasification of North Dakota Lignite is a complex mixture of organic components which distill over a wide boiling range. The mixture contains a naphtha fraction, a cresylic acid fraction, a neutral oil fraction, a NAPHTHOL fraction, and pitch. 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 two fractions have already been investigated to a limited extent by DGC and efforts to purify and market these materials have been ongoing. However, the naphthol fraction has yet to be explored.

DGC has always been committed to the development of new liquid by-products derived from gasification of North Dakota Lignite for marketing as chemical feedstocks or intermediates. An example of an earlier successful project is the commercialization of phenol and cresylic acid production. This was based on bench scale studies, as well as confirmation runs in a pilot plant. Currently, separation and upgrading of divalent phenols such as catechol and methylcatechols from crude phenol pitch is being studied.

6.2 By-product Development Approach

Each by-product development program requires at least three progressive phases of investigations:

- 1. Bench Scale Exploration
- 2. Pilot Plant Demonstration
- 3. Commercialization

During the first phase, concepts for upgrading liquid by-products are explored. For DGC's crude phenol and tar oil liquids, processing steps such as distillation, solvent extraction, and chemical treatment may be applied to obtain marketable products from the liquid by-products of the gasification process. A significant amount of analytical work is required to determine the composition of feed material and products, to evaluate the benefits of various treatment steps. The basic data obtained from this effort is necessary to design the Phase 2 program.

The second development phase, pilot plant operation, more closely simulates commercial operation. As an example, most bench scale distillations are performed batch wise, while most pilot plant testing is done on a continuous basis. Consequently, pilot plant tests allow collection of better data to design a commercial production plant. Pilot plant demonstration runs also permit production of trade samples to ascertain the technical acceptability of a new product in the market place.

At the end of each phase, economic evaluations are made to establish the profitability of producing a new product. With each step, the accuracy of the information increases.

6.3 Literature and Consultant Search

The methodology required to develop marketable naphthols from tar oil is similar to the approach taken for past and current by-product development efforts at DGC. To prepare for this project, a search of the Chemical Abstracts has been completed for the topic of alpha- and beta-naphthols. This search revealed all entries for these two substances which were included in the Abstracts from 1982 to 1992, and from these entries, dozens of abstracts were obtained online via STN International (The Scientific & Technical Information Network). Copies of the most promising of the papers and patents (in full) are currently being ordered.

None of the abstracts revealed unusual new processing technologies (crystallization seemed to be a focus), but this is not perceived as a drawback, since DGC has, since 1988, perfected several technological approaches (physical methods) for the isolation and separation of polar phenolic substances from neutral hydrocarbon matrices, rich in heterocyclics and other classes of impurities which are slightly less polar than the sought-after phenolics.

Much useful information was found on the subject of analytical methods, such as gas chromatography, GC/MS, and high performance liquid chromatography (all of which are useful, each in a different way). Numerous citations were found which describe work that has been done to identify and quantitate the naphthols in coal derived by-product liquids.

One of the more valuable outcomes of this literature search was the finding that naphthols are used in the manufacture of a broad range of products. This information, which has been passed along to DGC's Marketing Department, shows naphthols are now being used to manufacture products beyond the spectrum of traditional end uses (diazo coupling reaction products, e.g., dyes, etc.), such as heat sensitive coatings, specialty acrylic resin-based adhesives, surfactants and swelling agents, photosensitizers, beta-blockers and antidepressants (pharmaceuticals), and others too numerous to list.

D. Duncan interviewed Dr. Paul Berner, a consultant who has a lifetime of experience in naphthol chemistry, diazo chemistry, diazo reprochemicals, and photoresist sensitizers (for silicon chips). He presently is a faculty member of the Department of Chemistry, University of Rhode Island. While employed at Pfister, he developed synthetic methods to convert naphthols, via Kolbe reactions, to carboxylic acid derivatives. These can be esterified to the methyl esters, from which anilides are manufactured. These are used to manufacture the AS series of dyes.

Dr. Berner was especially interested to learn that our raw material is rich in methylnaphthol isomers, since these, too, would be useful to manufacture a wide range of anilides. He characterized this opportunity for DGC as the sort of arena in which we could "carve our own niche". He recommended Hoechst-Celanese as a likely customer for methylnaphthols. This information is regarded as particularly valuable, since the amount of methylnaphthols may well exceed the volume of alpha- and beta-naphthols available from DGC's tar oil stream. There is considerable economic incentive to explore these, also. Dr. Berner has offered to work with DGC when we have reached the point where trade samples are available, to recommend (based on product analyses) the marketability of the grades DGC would produce, and to assist in approaching his many contacts in this industry. This will be of much value to open doors.

6.4 Laboratory Needs

A second high performance liquid chromatograph (HPLC) is needed to expedite the pace at which this program can be accomplished. At this time, insufficient HPLC instrument time is available for supporting both bench scale studies of naphthols and ongoing pilot plant operation.

A second HPLC could also be beneficial for other pilot plant studies, such as development of catechols or indanols. The number of pilot plant samples which can be analyzed with one existing instrument is limited to obtaining a very basic amount of information to track the performance of the pilot plant distillation columns. We are presently able to analyze only three sets of samples collected from the pilot plant each day, spaced eight (8) hours apart. With a second HPLC, a set of samples could be analyzed every four (4) hours, thereby improving the operation of the pilot plant. Long term, additional by-products which should be investigated, such as indanols from DGC's tar oil stream, and follow up studies and requests from the market place regarding all of these potential by-products, would also benefit from this second HPLC.

Some of the bench scale explorations for by-product development, including naphthol purification, require distillation in a fairly tall laboratory column. Due to space limitation, permanent installation of a second, special purpose distillation column is delayed until adequate room in the existing 10 ft. high stainless steel fume hood is available. This tall fume hood has been a valuable asset for the bench scale studies which require tall equipment, such as the glass fractionation column, which is about 9 ft. in height. Due to the space limitations in other fume hoods, the tall fume hood is also currently used for the corrosion testing apparatus and for one-plate distillation studies, neither of which requires the height that the full height fume hood offers. The addition of a medium height fume hood would allow the tall fume hood to be used for this additional distillation column.

7.0 PROJECT DESCRIPTION

7.1 INTRODUCTION

The objective of this project is to develop and demonstrate an appropriate technology for production of marketable alpha- and beta-naphthol derived from gasification of North Dakota Lignite. This development work will be carried out in two phases:

PHASE 1: Bench Scale Study of Naphthol Purification

- Perform bench scale distillations and analyses to determine naphthol concentrations in various fractions derived from DGC's tar oil stream. This effort will be geared to establish desirable and achievable yields for naphthols.
- 2. Explore process options to purify and separate naphthols (alpha- and beta-naphthol) which would provide product purity compatible with the market place. This bench scale work includes investigation of potentially patentable processing schemes. Therefore, the reporting of results to the North Dakota Industrial Commission will document the achievements without divulging confidential information on the processing schemes investigated.
- Develop basic design data, such as vapor-liquid equilibrium data, to provide a design basis for the pilot plant demonstration runs, and to perform preliminary economic evaluations.

PHASE 2: Pilot Plant Scale Demonstration Run

- 1. Obtain engineering and material balance data which can be used to design a commercial facility for the processing of gasification tar oil, and more specifically for the separation and purification of the alpha- and beta-naphthol fractions.
- 2. Obtain continuous processing data in confirmation of laboratory determinations of vapor liquid equilibria, and any other properties needed to design a commercial production facility.
- 3. Produce sufficient and representative quantities of the naphthols for extensive identification of any remaining impurities, and for use as trade samples. The goal is to produce about one barrel of alpha-naphthol and about four barrels of beta-naphthol. To the extent that bench scale study proves this practical, up to four barrels of methylnaphthols may also be obtained.

7.2 PHASE 1: BENCH SCALE STUDY OF NAPHTHOL PURIFICATION

Task 1.1: Batch Distillation

The first step of the bench scale program will be to perform a very careful fractionation, at high reflux ratio (50:1) and moderate vacuum (20 mm Hg), of a distillate cut (a "concentrate") previously isolated from tar oil, which

contains the homologous series of substances known as naphthols. The charge to this fractionation will, in this way, not contain pitch (high boiling tar) substances having boiling points higher than those of the alkyl-naphthols. By this means, the stillpot temperature will be maintained at cooler temperatures during the fractionation, which will help minimize product degradation.

A number of distillate cuts will be obtained, and a portion of each of these will be separated via preparative scale techniques into neutral, acidic and basic fractions (tar base nitrogen compounds). These fractions will be investigated via GC/MS, to identify as much as possible the substances in each fraction of each distillate cut. This analytical information will then be used to construct graphic output, to describe the incidence and abundance of these substances during the progress of the distillation (e.g., as a function of overhead temperature). Abundance of critical substances will be double-checked by way of HPLC and GC techniques.

The identification of the highly polar substances which are present, other than the target compounds, will enable us to develop fractionation criteria which will be used in the distillation pilot plant for preparation of larger volumes of material. Highly polar substances are known to be the most difficult to separate via physical methods, and if these substances are recognized in advance, and if their distillation behavior has been characterized (such as by one-plate VLE studies, using both GC and HPLC analytical methods), then fractionation methods may often be chosen which can exclude them from the pilot plant distillates, which will later be used in the studies of physical methods of separation.

Additionally, GC/MS studies will provide an opportunity to identify other species of potential economic value, such as indanols.

Task 1.2: Concentration Measurements for Critical Components

Following the fractionation/GC/MS study, the second step of the bench scale study will be to develop the analytical methodologies, both GC and HPLC, for accurate analysis of substances in the distillate fractions. These techniques will be carefully developed, both to optimize the chromatographic resolution of the numerous substances identified, and to establish detector response factors (using, as much as possible, samples of pure substances from Aldrich Chemical or the like). The development of these analytical methods will be critical to the success of the next two projects.

Task 1.3: Screening of Purification Technologies

In parallel to these analytical efforts, a number of exploratory single stage experiments will be performed to determine the effectiveness of various proposed, most likely proprietary technologies including physical and/or chemical separation. This information will be reviewed both from performance as well as engineering considerations. Recommendations will be made to decide on the preferred purification technology for the pilot plant operation.

Task 1.4: Performance Coefficients

The fourth step of the bench scale study will serve to develop the above-mentioned VLE data for the critical substances which have been identified. It is critical to obtain VLE data by both HPLC and GC methods, since coeluting substances for one method are quickly revealed by comparison of data. By means of the data acquired (relative volatility data, relative to the substances of interest, the naphthols), computer simulations can be used to predict the distillate and bottoms product compositions which will be obtained, given various configurations of the pilot plant fractionation equipment. Once this work is completed, the pilot plant can be configured, and processing of larger volumes of material can commence. These larger volumes will be later used in the investigations of separation and purification methods (for which DGC also has a considerable investment in pilot plant equipment).

Task 1.5: Performance Coefficients for Proprietary Technology

Once the pilot plant feedstock preparation steps are underway, the Process Development laboratory staff will begin the fifth step of the bench scale program: measurement of various coefficients by way of both HPLC and GC methods, which will describe the behavior of the numerous impurity substances in what we call "physical separations technologies". For instance, coefficients for complex liquid phase systems will be developed. Likewise, coefficients to quantitate behaviors of impurities in other more proprietary technologies will also be generated. These data will be used for computer modeling to provide a reasonably accurate picture of the behaviors of substances, and therefore the product purities to be expected.

7.3 PHASE 2: PILOT PLANT SCALE DEMONSTRATION RUN

Following the laboratory studies on the tar oil naphthol fractions outlined in Phase 1 (batch distillation, analytical characterization, vapor-liquid equilibria measurement, and other explorations), efforts will focus on developing the engineering data base required for commercialization. This work will be conducted using DGC's continuous pilot plant facilities after appropriate modifications are made. The scope of the proposed work is outlined below.

Task 2.1: Drying and Light Ends Removal.

The first step in the recovery of tar oil naphthols is "Drying", which removes both water and light naphtha from the feed. This step is required so that the remaining fraction can be vacuum distilled at low pressure. The work will be accomplished using DGC's 6" diameter glass still. This still is designed for continuous operation, and when in use runs 24-hours/day. Since the tar oil contains only 2 wt% naphthols, it is expected that approximately 14,000 gallons of tar oil will have to be processed. This is expected to take about 2 weeks. Approximately 8 to 10 wt% of the feed is expected to be taken overhead with about two-thirds being light naphtha.

During the test, samples will be taken of each fraction and analyses performed on a daily 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.

Task 2.2: Topping.

The second step in obtaining tar oil naphthols is the vacuum fractionation of the "dry" tar oil to remove heavy naphtha, cresylic acid, and neutral oil. this distillation will again be done using DGC's 6" glass still but at reduced pressure (100 to 300 mm Hg absolute). Approximately 40 wt% of the original feed is expected to be taken overhead leaving the naphthols behind in a heavy residue fraction. This step in the distillation process is expected to take about 10 weeks. The increase in processing time is due to the greatly diminished column throughput which results from the vacuum operation.

As during the drying task, samples will be taken of each fraction and analyses performed on a daily 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.

Task 2.3: Depitching.

The third step in the recovery of the tar oil naphthols is the separation of a naphthol enriched fraction from pitch. This distillation will again be done using DGC's 6" glass still but at a further reduced pressure (less than 100 mm Hg abs). This step in the distillation process is expected to take about 3 weeks.

As with the previous two tasks, samples will be taken of each fraction and analyses performed on a daily 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.

Task 2.4: Fractionation.

Once a naphthol-rich distillate is obtained from the 6" glass still, that material will be used as feed for DGC's 6-inch by 75-foot fractionator. Like the 6-inch glass still, the fractionator is a continuous distillation column which runs 24 hours/day. However, unlike the glass still, the fractionator has sufficient number of stages to make very fine boiling point separations. Because of its separating ability, the fractionator will be able to separate the naphthols into a concentrated fraction.

Prior to operation, the laboratory VLE data will be used to perform computer simulations. The results of the simulations will be used to define the starting operating conditions such as feed rate, reflux ratio, and approximate operating temperatures for the fractionator.

During the test, samples will be taken of the overhead and bottom fractions and analyses performed every two to four hours. The results of the analyses will be used to make necessary changes in boilup rate and reflux, along with processing temperatures and pressures. 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. Column performance data will be used to verify the laboratory VLE results. Product samples will be thoroughly characterized and additional purification steps will be defined. These steps may involve proprietary separation steps.

Fractionation of the naphthols is expected to take about 4 weeks.

Task 2.5: Purification.

At present not enough is known about the types or levels of impurities that may be left in the naphthols after fractionation to adequately define what the next step in purification might be. Based on the literature search and DGC's prior experience, it is anticipated that proprietary technology, including some form of distillation, will result in substantial product improvements. Two potential processes have significantly different attributes and time requirements. Implementation of either process will require extensive parametric investigations to determine the appropriate processing conditions for commercialization.

At this time it is felt that a preliminary investigation of the most promising purification method (from task 1.3) can be completed in about 6 weeks. Included in the test work would be parametric studies using statistical methods and feedback from test results.

Task 2.6: Separation.

The final step in the processing of the naphthols is the separation of alpha-naphthol from beta-naphthol. This separation will be done using DGC's 6-inch fractionator, probably configured with a large number of stages. Starting conditions for this separation will be based on laboratory VLE data and computer simulations.

During the separation, samples will be taken of the overhead (alpha-naphthol) and bottom (beta-naphthol) fractions, and analyses performed every two to four hours. The results of the analyses will be used to make changes in processing conditions to bring both fractions to required specifications.

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. Column performance data will be used to verify the laboratory VLE results. Product samples will be thoroughly characterized, and may be used for trade sample purposes.

Separation of the two naphthols is expected to take about 4 weeks.

7.4 OTHER PROJECT CONSIDERATIONS

As indicated in the background information, additional laboratory equipment is needed to perform the naphthol development work on a timely basis. The following three tasks are part of this project:

Task 3.1: Acquire and commission an additional HPLC analyzer and necessary computer hardware and software to record and store analyzer results in the appropriate computer.

Task 3.2: Purchase and install a new, medium-sized fume hood, with a vacuum pump and necessary utilities in the byproducts laboratory. This will allow the relocation of corrosion testing and one-plate distillation equipment, and provide free space in the tall fume hood for final installation of the special purpose distillation column.

Task 3.3: The special purpose distillation column to be installed in the tall height fume hood will be used to explore proprietary processing schemes for purification of naphthols.

7.5 DELIVERABLES

The deliverables from this project will include "non-proprietary" progress reports. These reports will summarize the status of the work along with a discussion of major achievements and/or difficulties.

Proprietary scientific and operating data will not be included in the reports but may become available to the public should DGC file for patent protection on any resulting invention(s).

7.6 PROJECT NEED

This development program is needed to answer several questions. First it must be established that appropriate technology can be developed to upgrade naphthols from tar oil to become a marketable byproduct from a coal gasification plant.

Second, DGC must also obtain sufficient technical data to perform economic evaluations regarding the production of naphthols in Beulah, ND. This information is also necessary in conjunction with the evaluation of other potential tar oil cuts which can be marketed. Interest exists in a cut of tar oil which contains cresylic acid. The economics will be enhanced if additional byproducts can be marketed from this tar oil stream.

A third reason for this project is the production of trade samples. Due to concerns by buyers of coal derived chemical feedstocks, it is imperative that trade samples be made available to users for testing purposes. DGC must have assurance that the product it manufactures can be sold before commitments are made to construct a commercial facility.

8.0 STANDARDS OF SUCCESS

The standard of success for the project will be the ultimate production of commercially superior quality alpha- and beta-naphthol.

The results from this project will provide sufficient information for DGC to decide if and how to proceed with a program to produce over 8 million lbs/yr of commercial grade naphthols. Acceptance of trade samples in the naphthol market is another necessary sign of success.

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 shown a strong interest in developing by-products from the gasification complex. Aside from building commercial facilities for production of krypton/xenon, phenol, and cresylic acid, DGC has also invested heavily in facilities to enhance development work. The fractionator building, which has been an earlier project with North Dakota Industrial Commission participation, is now valued at over \$2 MM with all its additions, and is an example of DGC's dedication to by-product development.

DGC also maintains excellent plant laboratory 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 work for outside clients. This same staff of senior engineers and chemists will be dedicated to this proposed program.

10.0 VALUE TO NORTH DAKOTA

Development of marketable naphthol 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 1.3 MM lbs./yr of alpha-naphthol and 7.5 MM lbs./yr. of beta-naphthol. At market prices of \$1.85/lb and \$1.31/lb, respectively, the potential yearly revenues are estimated to be \$12.2 MM/year.

Although this revenue would be obtained by DGC, the necessary plant addition will create temporary construction jobs in North Dakota, and contribute to the financial stability of DGC. Together with other liquid by-product projects, it may also contribute to an increase in plant labor force of up to six people.

North Dakota will also enjoy less tangible benefits that include the reputation of having highly qualified professionals that develop patentable technology in the field of coal gasification and its by-products.

11.0 MANAGEMENT

This project is controlled solely by Dakota Gasification Company. It will be executed under the direction of A. Kuhn, Manager of the Process Development Department. Mr. Kuhn will be responsible for the reporting of progress to the Commission. He will also direct the efforts within his department. D. Duncan will have technical responsibility for the by-product laboratory activities as outlined for Phase 1, and G. Baker will have technical responsibility for Phase 2, pilot plant operation.

Additional engineers and supervisors, D. Maas and J. Hughes will provide supervision to the pilot plant operation (two operators per shift), while D. Duncan is assisted by three laboratory technicians. Resumes of key people are attached.

12.0 TIMETABLE

Dakota Gasification Company is prepared to initiate this project as soon as the proposed contract is received from the North Dakota Industrial Commission.

Bench scale exploration of naphthol purification will be coordinated with other ongoing laboratory efforts. Based on the current shortage of HPLC equipment, the project will be fully released as soon as sufficient analyzer time is available. It is anticipated that six months of bench scale exploration will be needed to develop the basic information on naphthols separation and purification. Seven to nine months of pilot plant operation are anticipated to demonstrate the technology. The following schedule lists key dates:

Phase 1: Bench Scale Studies

October 1, 1993	North Dakota Industrial Commission Contract
December 1, 1993	Second HPLC operational, latest start date for bench scale studies
April 30, 1994	Bench Scale Results on Naphthol Purification
May 30, 1994	Final Report Phase 1 to ND Industrial Commission

Phase 2: Pilot Plant Operation

March 1, 1994	Ready Pilot Plant Facilities		
April 1, 1994	Start Tar Oil Distillations		
August 15, 1994	Complete Tar Oil Distillations		
September 30, 1994	Complete Exploration of Naphthol Purifications		
October 30, 1994	Complete alpha/beta Naphthol Separation		
December 31, 1994	Final Report Phase 2, also includes a 60 day contingency for Pilot Plant Operation		

13.0 BUDGET

The following information represents the current best estimate relating to the project cost. Necessary development 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 answer all questions. This estimate assumes that at least two approaches for purification of the naphthols will be studied on a bench scale basis (Phase 1). According to our previous experience, this should be sufficient to size up the commercial viability of producing alpha- and beta-naphthols at DGC. Phase 2 is less definable at this time. An estimate indicates that a total of about 29 weeks is required. A two months contingency is being added.

Phase 1: Bench Scale Studies

Laboratory Development Work	\$ 50,000
Analyzer/Equipment Purchases	50,000
Installation, Labor & Materials	15,000

Subtotal:

Phase 2: Pilot Plant Operation

\$710,000 29 Weeks Operation (203 days @ \$3500/day) 220,000 9 Weeks Contingency

Subtotal:

\$ 930,000

\$1,045,000

\$ 115,000

Total Project Cost:

DGC may decide to modify or add appropriate distillation Note: equipment to reduce the time required for distillation of the tar oil to separate the naphthol enriched fraction. Savings in operating costs would be offset by the initial investment. These evaluations are planned for the 4th guarter of 1993.

14.0 MATCHING FUNDS

DGC is prepared to provide the time, material, and financing to carry out this project. Due to the staged approach of work and a small risk that DGC may reach a conclusion that Phase 2 should not be pursued at that time due to economic evaluations, DGC requests matching funds as follows:

	Phase 1	Phase 2	Total
ND Lignite Research Fund	\$ 50,000	\$200,000	\$ 250,000
Dakota Gasification Co.	65,000	730,000	795,000
Total:	\$115,000	\$930,000	\$1,045,000

15.0 TAX LIABILITY

See attached affidavit

16.0 CONFIDENTIAL INFORMATION

No confidential information is contained in this proposal. However, any specific process treatment descriptions and requests for detailed study reports would most likely have to be handled as confidential information.

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.

Leh

Rod J. Kuhn, **EPA** Tax, Audit, and Insurance Manager Basin Electric Power Cooperative