LRC-II-10

ANG Coal Gasification Company
Administrator of the Great Plains Coal Gasification Project
for the United States Department of Energy
P.O. Box 1149
Beulah, ND 58523
(701) 873-2345

October 5, 1988 9440-AKK-88-143

North Dakota Industrial Commission
1016 East Owens Avenue, Suite 200
Bismarck, ND 58502

Gentlemen:

ANG Coal Gasification Company is interested in submitting a proposal for the construction and subsequent operation of a multi-purpose 6-inch distillation facility to produce trade sample quantities of cresylic acid and individual isomers of cresol or xylenol under Article 43-03, Contracts for Lignite Development and Land Reclamation Research and Hydroelectricity Impact Studies. Due to the ongoing efforts to transfer ownership of the Great Plains facilities from DOE to Basin Electric, ANG has been requested and authorized only a few days ago to proceed with such a proposal. The following summary provides an overview of the potential benefits and the scope of the project.

The objectives of this study will be to install a high efficiency 6-inch diameter fractionation column and to use this column to prepare cresylic acid distillate trade samples from lignite gasification by-product liquids. Yet another objective will be to characterize the composition of the distillate fractions. Long-range objectives include scaleup of laboratory studies accomplished on relatively small glass distillation columns.

The expected results will be to satisfy the above objectives, that is, to install and operate a 6-inch column to prepare phenolic trade samples and to study the composition of the cresylic acid distillate fractions.

This 6-inch column will be designed, procured and installed at Great Plains over a 6 to 8 month period starting January 1, 1989. The total cost of this project is currently projected to be in the $300,000 range excluding costs to operate the facility. Since operation will be on a part-time basis, these costs will be minimal and will be absorbed by other cost centers for the Great Plains facility.

Attached please find a project description which is still preliminary, partly due to time constraints to prepare this application and partly due to the current status of engineering efforts to construct the system.

ANG considers this system a vital part of the effort to promote marketing of by-products derived from lignite. This request should qualify per chapters 43-03-02-02, item F and is instrumental in Dakota Gasification Company's interest to sell by-products from Great Plains. Based on the total project cost, ANG requests funding of $50,000. This will be less than 20 percent of the total project cost considering operating costs. A check for $100 covering the registration fee is enclosed.

We trust you will give this proposal serious consideration. If there are any questions, please do not hesitate to contact myself for contractual considerations or D. Pollock, 701-873-6650, for technical questions.

Very truly yours

D. L. Imler
Executive Vice President and Chief Operating Officer

by: M J Mujadin
M. J. Mujadin, Vice President, Operations

AKK/wp

CC: K. Janssen
A. Kuhn
M. Mujadin
D. Pollock
D. Schmitz
Records Management
correspondence file

PROJECT TITLE: 6-INCH DIAMETER FRACTIONATION SYSTEM FOR PRODUCTION OF TRADE SAMPLES DERIVED FROM LIGNITE VIA COAL GASIFICATION

ORGANIZATION: ANG Coal Gasification Company/Dakota Gasification Company

INVESTIGATOR: D. C. Pollock

DATE OF SUBMISSION: October 3, 1988

AMOUNT OF REQUEST: $50,000.00

1. SUMMARY

The objectives of this study will be to install a high efficiency 6-inch diameter fractionation column and to use this column to prepare cresylic acid distillate trade samples from lignite gasification by-product liquids. Yet another objective will be to characterize the composition of the distillate fractions. Long-range objectives include scaleup of laboratory studies accomplished on relatively small glass distillation columns.

The expected results will be to satisfy the above objectives, that is, to install and operate a 6-inch column, to prepare phenolic trade samples and to study the composition of the cresylic acid distillate fractions.

This 6-inch column will be designed, specified, procured and installed during the first 6 to 8 months of 1988 and operated thereafter to satisfy the above objectives.

The total cost of this project will be $300,000, and the portion of that cost, which is requested from the funding of the North Dakota Industrial Commission shall be $50,000.

The major participants in this project will be Dakota Gasification Company (after acquisition of ANG, expected to be accomplished in October 1988) and vendors supplying equipment for the facility. ANG is currently performing project engineering services, but will transfer all activities to Dakota Gasification Company as the sale of the Great Plains' facilities is being completed.

2. OBJECTIVES

The objectives of this project will be to construct a fractionation pilot plant for a) the study of cresylic acid and distillation and for b) the preparation of trade samples.

This project will be an important element of the Cresylic Acid Development Program at the Great Plains Gasification Project. Cresylic acid products will be the most lucrative of the hydrocarbon by-products of the lignite gasification process and successful manufacture and marketing of these materials will strongly improve the overall economics of the gasification process. This will strengthen the long-range viability of coal gasification at the Great Plains Project in North Dakota.

3. BACKGROUND

The cresylic acid industry is one in which fractional distillation plays a major role. Fractional distillation is used for nearly all of the isomer separations, such as phenol from ortho-cresol, ortho-cresol from meta/para-cresol, 2,4-2,5- xylenol from mixed xylenol fractions, etc. Isomers are also separated by way of butylation and fractional distillation to separate the butylated isomers.

Several of the cresylic acid isomers have boiling points fairly close to one another. Separation of these substances requires columns with great ability to separate substances, eg., quite long columns or multiple (side-by-side) columns.

Variations of fractional distillation such as rectified steam distillation or extractive distillation also play an important role in phenolic isomer purification process technologies.

All of the above fractional distillation techniques are operated in a continuous mode rather than as batch operations in the cresylic acid industry. Cresylic acid feedstock liquids are continuously pumped to such columns and distillates are continuously removed from such columns as products.

Laboratory scale glass columns are very nearly always best operated in a batch mode rather than on a continuous basis. This difference in operating technique results in product compositions, which are unique to each process method. It is usually not possible to obtain phenolic isomer distillate products from a batch operation which have compositions identical to what would be obtained from an industrial continuous column. It will be necessary to be able to provide samples having compositions which accurately represent what will be obtained from a full-scale cresylic acid plant. As the cresylic acid development program matures, it will be come increasingly important to be capable of providing evaluation samples to prospective customers of such products. Such product evaluations are an important part of the foundation upon which a North Dakota cresylic acid industry must be built.

A 6-inch continuous column will enable characterization studies of impurities to accurately reflect what will be actually found in the distillates from full-scale industrial distillation equipment. Here, too, lab scale batch studies yield data which is skewed by the nature of a batch process.

Finally, studies of purification process technologies cannot be effectively carried out in a batch mode. Rectified steam and extractive distillation studies cannot be modeled effectively on a batch column. In order to optimize process parameters it is essential that a continuous column be used. It is not possible to scale up to industrial operations from small laboratory batch column data.

The laboratory scale glass column studies of the composition of crude phenols have all been completed during this past year. Several studies of distillate purification methods have been completed. By early to mid 1989, the remainder of the glass column phenolic purification studies will be concluded. At this point in time, the usefulness of laboratory scale equipment will become more limited.

The next logical step in the development of cresylic acid products at the Great Plains Gasification Project will be a pilot plant scale continuous 6-inch diameter fractionation column.

4. GOALS

The goals of this project will be:

a. To design, procure and install a high efficiency 6-inch diameter continuous fractionation column suitable for the separation of the alkyl-phenol isomers found in lignite gasification by-product liquids such as tar oil and crude phenols. Such a column will be equipped with a flash drum and dryer suitable for the preparation of such by-product liquids for the fractionation steps and all other ancillary equipment such as reboilers and condensers, feed pre-heaters, pumps, feed and storage tanks, steam ejector and necessary control instrumentation,

b. To use such equipment for distillation of the phenolic fractions of lignite gasification by-product liquids into cresylic acid distillate cuts such as phenol, ortho-cresol, meta/para-cresol and xylenol isomers and to determine the composition of such distillate costs. Such characterization studies will determine the levels of and where possible, the identity of impurities in the phenolic isomer fractions,

c. To make available such distillate products as trade samples for evaluation by potential end users (customers) for such materials, and

d. To evaluate the factors affecting successful scaleup of laboratory methods accomplished on lab sized glass distillation columns. This last goal is one which will be accomplished over a longer time frame than the former goals.

5. METHODS

The distillation system will consist of a tall 6-inch diameter fractionation tower with reboiler, overhead condenser, feed effluent exchangers, purger, storage tanks, piping and instrumentation. Specific details can be made available when sufficient design work has been completed. Some of this effort depends on further bench-scale investigations of purification methods to produce commercial grade products from lignite-derived crude phenols.

6. TIME TABLE

Dakota Gasification Company is prepared to initiate this project by January 1, 1989. Subject to further engineering and procurement efforts, it is expected that the total system can be constructed in a 6 to 8 month period. Since DGC is very much interested in providing trade samples as soon as possible, efforts will be made to keep the project on a very tight schedule.

7. PERSONNEL

Major contributors to this project will be senior engineers employed by ANG/DGC:

D. C. Pollock: Director Technical Services.

D. Pollock has many years of service with Chevron and ARCO before joining ANG in 1982. His background includes extensive experience in refinery operation, shale oil, underground coal gasification and pilot plant facilities.

A. K. Kuhn, Process Development Manager

A. Kuhn has many years of experience with engineering focus and after joining ANG in 1975, has directed the major process engineering efforts for the Great Plains Project. He has built and worked as development engineer on pilot plant facilities.

W. A. Sutthill, Plant Design Engineering Manager

W. Sutthill has had extensive experience in mechanical design of industrial facilities. After working with aerospace and electric utility companies, he supervised detailed engineering of the Great Plains' facilities at CE Lummus. In his current position, he is also experienced in monitoring cost and schedule for various capital projects at Great Plains.

G. G. Baker, Senior Process Development Engineer

G. Baker has had extensive experience at UNDEMRC building several pilot plant facilities. At Great Plains, he has built and supervised operation of the Stretford pilot plant and is currently providing the technical expertise in building a dual solvent continuous extractor facility (cresylic acid from tar oil).

D. H. Duncan, Senior R&D Chemist

D. Duncan has had over 15 years of experience in developing methods to upgrade cresylic acid from petroleum derived feedstocks. He has been instrumental at ANG to develop bench-scale information to be utilized in the further development of this project.

8. QUALIFICATIONS OF APPLICANT

ANG/DGC has the necessary staff to carry out such a project. A number of earlier capital projects in connection with solving start-up problems and odor projects were similar or larger in scope.

9. **BUDGET**

At this time, no detailed cost breakdown is available. Subject to further study, the total estimated cost of $300,000 could be broken down as follows:

	Dollars
Engineering	60,000.-
Materials	100,000.-
Construction	80,000.-
Contingency 25%	60,000.-
TOTAL	300,000.-

Operating Expense, 6 months, approximately $100,000.-

ANG/DGC funding for construction of the facility will be provided out of the internally approved R&D budget. Operating expense will be charged against other internal cost centers to be determined after DGC assumes operating responsibilities.

10. **APPENDICES**

None

Sidney —
Need a contract for this project.
Have a $5,000 upon execution of
Contract
then payments March 1 $10,000
May 1 $15,000
June 30 $20,000
with status reports Feb 15
April 15
June 15 OK

The contract should be to
Dakota Gasification Company
1600 East Interstate Ave.
Bismarck, ND 58501