Great Plains SYNFUELS
DAKOTA GASIFICATION COMPANY
1600 East Interstate Avenue
Bismarck, ND 58506-5540 (701)221-4400

July 3, 1995

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

Attn: Lignite Research Program

Dear Commission Members:

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 development of additional chemical by-products at DGC. Currently, pitch from the phenol/cresylic acid upgrade facility is being burned as fuel. This pitch contains catechol, 3-methylcatechol, and 4-methylcatechol. Based on feedback from the marketplace, these intermediate chemicals can be valued at approximately $2/lb. In the case of 3-methylcatechol, however, a marketplace needs to be developed, for DGC's anticipated volume of about 2 MM lbs/yr. .

DGC is pursuing a new application for 3-methylcatechol as feedstock to produce pyrogallol acetonide, a precursor to the manufacture of bendiocarb, a pesticide. This application can consume a large portion of DGC's potential yearly production of 3-methylcatechol. However, it is necessary to find additional uses for 3-methylcatechol to assure sale of the total production. The economics of building a catechol/3-methylcatechol facility will only be favorable if both products can be sold.

Some months ago DGC learned that in Europe a resin manufacturer is using 3-methylcatechol to modify properties of phenolic resins. Due to the sophisticated nature of the resin manufacturing industry, DGC, after having explored making some resin of its own, recognized the need to have an experienced resin research organization participate in this effort. After a search and after evaluation of two Forest Products Laboratories, it was concluded that the USDA Forest Products Laboratory in Madison, WI has excellent background and experience in evaluating resorcinol resins. A study program as discussed with them will provide information on the behavior of various ingredients in making phenolic resins, as well as document the impact on resin properties such as flexibility. DGC expects that this information will be suitable to attract commercial resin manufacturers to confirm the findings and ultimately, make a commitment to purchase 3-methylcatechol from DGC.

The total cost for this development effort has been estimated at over $185,000 of which about two-thirds is the cost for work to be performed at the Forest Products Laboratory. DGC is

requesting $75,000, approximately 40% of the project cost and slightly more than 50% of the external contract cost. DGC is committed to this program since it is imperative that additional liquid by-products be developed at DGC in support of future operations.

The risk of this study lies in the unknown impact which 3-methylcatechol may have on resin properties. Resin development work requires input from experienced chemists having worked in the field of polymers. DGC is confident that a most suitable program has been put together using the appropriate talent. The program requires about 18 months to complete but within the first year, major discoveries will be made how to best utilize 3-methylcatechol in the field of resin manufacturing. It is proposed that two intermediate progress reports and a final report provide the North Dakota Industrial Commission with valuable updates on this development program.

We ask that you give this proposal serious consideration. If there are any questions, please do not hesitate to contact me (701-221-4410) for additional information.

Sincerely,

Kent E. Janssen

Kent E. Janssen
Vice President & Chief Operating Officer

KEJ/AKK/jsd
Enclosures: Proposal
$100 Registration Fee
Tax Affidavit

6/29/95

2.0 TITLE

DEVELOPMENT OF RESINS

FROM DGC's 3-METHYLCATECHOL

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, 1995
Amount of Request:	Total $75,000

3.0 INDEX

4.0 ABSTRACT

The objective of this product and market development program is to determine if resol resins of the resorcinol-type, can be produced using 3-methylcatechol as one of the phenolic ingredients to the resin manufacturing process, resulting in improved resin properties. Resorcinol/ formaldehyde (RF) resin will be the principal type of resin to be explored, although some study will also be directed toward phenol-resorcinol/formaldehyde resins (PRF resins). Based on exploratory work performed by DGC last year, it has become evident that for this investigation, it would be more appropriate to use an outside research and development organization which is well versed in this type of development work . Such an organization, with expertise in the field of resorcinol resins, would be expected to produce results that are both convincing and credible to the resin manufacturing industry.

The program described herein will be a Phase I program. In this Phase I program, various resorcinol resins, some incorporating 3-methylcatechol into the polymer structure, will be made and screened for valuable property improvements such as cured resin flexibility. Additionally, measurements will also be made of cure time and resin strength, to assure that no deleterious effects are found as a consequence of including 3-methylcatechol into the polymer structure. If the results of this Phase I program are sufficiently encouraging, then a second phase of R&D, leading to commercialization, may later be initiated by DGC. A Phase II program would expand upon promising findings from Phase I, may include commercial resin manufacturers and/or continue with the outside R&D facility, and would be implemented in order to develop sufficient data on the performance of optimized resin formulations to attract commercial resin manufacturers to endorse the benefits of the new resin. This Phase II program is beyond the scope of this application and if it consists of working with commercial resin manufacturers as it is anticipated at this time, it will not be considered for further grant application with the North Dakota Industrial Commission.

DGC is currently in the process of negotiating with the U.S.D.A. National Forest Products Laboratory (FPL) in Madison, Wisconsin. It is planned to have a development program in place by August, 1995, and to have all Phase I results and a final report available by about March 1, 1997. The total cost for this effort has been estimated to be $185,614. Nearly 70%, or over $127,000 is cost incurred by the FPL to perform the basic research work.

The major participants in this program will be DGC, as project coordinator, and the U.S.D.A. National Forest Products Laboratory in Madison, WI

5.0 PROJECT SUMMARY

The objective of this product and market development program is to determine if resol resins of the resorcinol-type can be produced using 3-methylcatechol as one of the phenolic ingredients to the resin manufacturing process, resulting in improved resin properties. Resorcinol/ formaldehyde (RF) resin will be the principal type of resin to be explored, although some study will also be directed toward phenol-resorcinol/formaldehyde resins (PRF resins). In addition, a comparison of the benefits of 3-methylcatechol will be made substituting it in a mixed resin formula with m-cresol. Several variables are being studied, including pH, formaldehyde to phenolic resin, and two different methods of incorporating the 3-methylcatechol into the resorcinol polymer structure. During these program steps, analytical information from differential scanning calorimeter (DSC) and nuclear magnetic resonance spectroscopy (NMR) will be obtained. The DSC reveals information about the rate at which polymerization reactions are proceeding, and NMR provides information on the substitution patterns on the aromatic ring that are occurring (e.g., cross linking versus more linear in structure). Gelation time tests will be used on some samples to determine time to gelation slightly above room temperature. Promising resins will be evaluated for cured resin flexibility using Dynamic Mechanical Analyses (DMA). Once optimization is achieved for copolymers from resorcinol and 3-methylcatechol, then resin properties are determined with samples of glued laminates made from samples of wood and several resins, including commercially available resorcinol resin samples for comparison purposes.

This project is needed by DGC to support a decision whether or not to build a catechol recovery facility at DGC's Great Plains facility. The technology to recover and purify catechol, 3-methyl catechol, and 4-methylcatechol from crude phenol pitch has been developed in DGC's pilot plant facilities. Marketing of over 2 MM lbs/yr of 3-methylcatechol is considered to be the biggest challenge, since no large scale uses are known to exist in the USA. DGC is currently pursuing development work to produce pyrogallol acetonide from 3-methylcatechol (a precursor to manufacture bendiocarb, a pesticide). In addition, DGC has been informed that 3-methylcatechol has been used in Europe to improve resin flexibility. If both of these applications can be penetrated, DGC will have sufficient assurance to proceed with the project.

The results of this study, if favorable, will be made available to one or several resin manufacturers (depending on proprietary restrictions imposed by resin manufacturers) for their evaluation before DGC will decide on the go-ahead for a catechols recovery project. Because of the tight control of resin making know-how by manufacturers, as well as research institutes such as the FPL, DGC is limited in providing specific figures for resin recipes to be tested. A confidentiality agreement is being negotiated between DGC and the FPL. However, DGC will be able to provide progress reports and a final study report which describe the findings in such a manner that the success of the program can be assessed.

6.0 PROJECT DESCRIPTION

6.1 INTRODUCTION

The overall objective of this project is to develop new resorcinol resin formulations offering enhanced glueline (cured bond) flexibility, which is expected to result from incorporating 3-methylcatechol into the resorcinol polymer structure. The program aims to determine the effect on resin properties of varying amounts of 3-methylcatechol added to a series of experimental resin batches, along with resorcinol (which will remain the major phenolic component used in these experimental batches of resol resins). Two different techniques of incorporating the 3-methylcatechol into the polymer structure will be explored. Due to the proprietary nature of the resin making industry, DGC is not in a position to make specific resin recipe information available. However, this development work will be carried out by means of the following steps:

1. A computer-based literature search is conducted to locate what literature exists on the topic of resorcinol resin preparations, and to gather the information that is available on various resorcinol-containing copolymers.

2. The second step consists of evaluating the reactivity of various phenols, including resorcinol, phenol, meta-cresol and 3-methylcatechol, with formaldehyde. Various instrumental techniques will be used to measure the reactivity of these species. Several variables are being studied, including pH, formaldehyde-to-phenolic ratios, etc. Two different methods of incorporating the 3-methylcatechol into the resorcinol polymer structure are to be explored.

3. During the third step, various resins that have been prepared will be studied by way of instrumental techniques, in order to evaluate the effect of added amounts of 3-methylcatechol upon the properties of resorcinol resins. In particular, the property of interest will be finished, cured resin flexibility (glueline flexibility).

4. The fourth step consists of adhesive resin composition trials to refine the techniques of preparing resorcinol resins modified with 3-methylcatechol for the specific purpose of achieving modification of cured resin (glueline) flexibility. The primary tool of evaluation for step four consists of the testing of laminates made from wood specimens, that have been glued with candidate resin samples, including a control (a commercial resin sample). Testing will be done to evaluate bond strength, in both compression and shear, and to evaluate the ability of the specimens to resist delamination.

Once the results from this project have been found to be favorable, DGC and the FPL may decide to pursue a Phase II program, the purpose of which would be to accomplish further testing to support the commercialization of a resin product (or resin products). DGC may also decide to initiate a Phase II program with one or more resin manufacturers to obtain their concurrence and confidence that resins containing 3-methylcatechol enhance certain properties of the resin.

6.2 RESIN PROPERTIES MODIFICATION WITH 3-METHYLCATECHOL

Task 1.1: Literature Search for Information on Resorcinol Resins, and Resorcinol Copolymers

The first step of the resin properties modification study is to locate by way of computer literature searching whatever information is available on the topic of resorcinol resins, especially resorcinol resins that are copolymers with other phenolic substances, formulated specifically for the purpose of achieving new and different resin properties. Literature of interest will address copolymers that might be similar to or compete with a copolymer that could be made with 3-methylcatechol and resorcinol. This literature search will also be performed for the purpose of gaining information about resorcinol resin and copolymer synthesis procedures.

Task 1.2: Assessment of Reactivity, Evaluation of Variables and Development of Procedures for Resins Made From Resorcinol, 3-Methylcatechol, m-Cresol, and Phenol

This task is subdivided into three subparts, as shown in the following:

subpart a) This first subpart results in the development of data regarding relative reactivity with formaldehyde of pure phenolic monomers derived from resorcinol, 3-methylcatechol, m-cresol, and phenol in a first series of tests, and then in a second series, subpart b) mixed phenolic monomers. Instrumental techniques that will be used to analyze the results of the various batches of materials made in this subpart will be DSC (differential scanning calorimetry) and C_{13} - NMR (carbon-13 nuclear magnetic resonance spectroscopy). The DSC reveals information about the rate at which polymerization reactions are proceeding (how vigorously they are occurring), and NMR provides data on the substitution patterns on the aromatic ring that are occurring, given various phenolic monomers (e.g., whether the polymers being formed are crosslinked, or if they are more linear in structure). The monomers to be investigated will be resorcinol, 3-methylcatechol, meta-cresol and phenol. The results from the latter two will serve as reference benchmarks.

Several pH targets will be studied in the various polymerization reactions of these monomers with formaldehyde. Additionally, various mole ratios of formaldehyde to phenolics will be studied. For all variables to be investigated, the two above-mentioned instrumental techniques will be applied, as well as gelation time experiments for a number of the more promising reaction products provided by this study of variables. These techniques will also be applied to commercial samples of resorcinol/formaldehyde (RF) and phenol-resorcinol/formaldehyde resins, to develop data for comparison purposes.

subpart b) Once the above work is completed, two avenues of investigation will be open to pursue. To explore one approach, the work will proceed at this point to studies of copolymerization of 3-methylcatechol with resorcinol, e.g., the production of polymers from mixtures of these two phenolic species, with both species present in the reaction

vessel (resin kettle), to be reacted together at the same time with formaldehyde. This approach is the copolymerization strategy. To be studied at this stage of the investigation will be pH, mole ratio of phenolics to formaldehyde, and mole ratio of 3-methylcatechol to resorcinol. The effect of these variables on resin properties, as measured by both instrumental techniques and gelation tests, will be determined. Comparison resins will also be made using m-cresol instead of 3-methylcatechol. Again, the impact on resin properties will be measured the same as for the resin mixtures containing 3-methylcatechol.

If these studies reveal that 3-methylcatechol shows a significantly lower tendency to react with formaldehyde than the resorcinol (e.g., if the resorcinol tends to self-polymerize to the exclusion of the 3-methylcatechol), then the direction the work will take would be to investigate the reaction of 3-methylcatechol with formaldehyde (in the absence of resorcinol). The reaction product from this approach would consist of hydroxy-methylated 3-methylcatechol, which is the reaction product that would occur from reacting 3-methylcatechol with formaldehyde. This material, hydroxymethylated 3-methylcatechol, would then be added to a reactor along with some resorcinol, and these materials would then be reacted together to form a copolymer (this technique can be thought of as providing a "head start" for the 3-methylcatechol, to allow it to proceed to an intermediate degree of polymerization, prior to it having to compete with resorcinol for available formaldehyde). It is expected that the hydroxymethylation product from 3-methylcatechol would more readily participate along with resorcinol in the reaction with formaldehyde, when added to resorcinol, than would raw 3-methylcatechol. In this way, the propensity of the two phenolic species to react with each other to form useful polymers could be enhanced.

For this strategy also, the variables to be studied are mole ratio of formaldehyde to phenolic substance, pH, and mole ratio of 3-methylcatechol (or meta-cresol) to resorcinol. The phenolic substance primarily focused upon in this stage of the study is 3-methylcatechol, although meta-cresol will also be studied, to define how well it compares to 3-methylcatechol. The same instrumental and experimental approaches as indicated in a) above will again be employed, to track the properties of the polymers being created at this stage of the study. The intent of the study will shift, toward the end of subpart b), to the goal of creating curable polymers, e.g., polymers that are capable of being cured into a glueline (a bond) with good adhesive properties, given appropriate temperatures (expected to be room temperature, or perhaps a little above) and amounts of catalyst (paraformaldehyde) required for such curing.

subpart c) Comparative studies are included to assess the results being obtained from adding unreacted (virgin) 3-methylcatechol, in one set of experiments, and in another, adding hydroxymethylated 3-methylcatechol, to a finished RF resin (or PRF resin), to observe the effect of this approach upon resin properties. Mole ratio of 3-methylcatechol to resorcinol, as well as the phenolics to formaldehyde ratio, will be explored. The effect this approach has upon the molecular structure of the resin will be determined.

Task 1.3: DMA Testing for Cured Resin Flexibility

Polymers made in Task 1.2, alongside resins made from resorcinol (alone) and commercial resorcinol polymers, will be tested by way of DMA (Dynamic Mechanical Analysis). This equipment tests cured resin films in order to establish resin properties, such as flexibility and strength. Resin samples are cured on a substrate of glass filter material, and the cured samples are then tested via DMA. Data will be developed for lab-synthesized RF and the commercial RF samples, as well as PRF resin, and these data will be of value as benchmarks (controls) for comparison purposes. DMA data developed for copolymers of resorcinol and 3-methylcatechol, made from either strategy, will be compared to the benchmark samples. In this way, resin property enhancements will be quantified.

Task 1.4: Adhesive Composition Trials

Once optimization is achieved for copolymer resins from resorcinol and 3-methylcatechol, then resin properties are determined with actual samples of glued laminates, made from samples of wood. It is possible that several formulations will be studied, in order to provide varying degrees of impact upon resin properties, such as impact upon cured resin flexibility. Again, tests will be done with both a commercial resorcinol resin sample, and a lab-produced sample of the same, in order to obtain control data. Test results from trial resins will be compared to the benchmark data. The various glued wood specimens that will be made will be subjected to the kinds of tests specified in ASTM D-2559 (compression, shear and delamination tests). These demonstration resin test results will be made available to resin manufacturers, as an incentive to the use of 3-methylcatechol in resorcinol resin formulations.

6.3 DISCUSSION

The result which is anticipated from this study is the confirmation of what DGC has learned about the use of 3-methylcatechol to achieve resin property modifications, namely, that the flexibility of resins, both P-F and resorcinol, is improved by incorporation of 3-methylcatechol into resin recipes. Of these two kinds of resins, DGC expects that the greatest opportunity will be in the area of resorcinol resins, since the value of these resins will support the cost of additional ingredients such as 3-methylcatechol.

In contrast, the value of phenol/formaldehyde resin is fairly low, and it may be more difficult to justify the addition of a relatively costly material such as 3-methylcatechol. An exception to this would be specialty markets which could support the additional economic burden, based on the value of a resin property modification to a niche market. For phenol/formaldehyde resin, an example of a niche market would be a resin specially tailored for making plywood to be used by industries such as window frame manufacturers. For this end use, resin flexibility would be a property of great value during the machining process, where breakage of the plywood tends to occur. If the glueline were to have sufficient flexibility to withstand the forces generated during machining, the resin would support an increased cost of manufacture. (Further study of

phenol/formaldehyde resin property modification, beyond the scope of the work done in Phase I, could become a part of the scope of a Phase II study.)

The facility that has been chosen for this contract is the Forest Products Laboratory in Madison, Wisconsin, which is owned and administered by the U.S.D.A. Forest Service. This facility is located adjacent to the University of Wisconsin, Madison Campus. Key scientists employed at this laboratory, specializing in forest products adhesives, have been interviewed by DGC's Principal Investigator, and a Chemist. A proposal for research has been prepared by them, and this work plan is presently being finalized along the lines described above.

It is expected there will be no environmental or economic impacts of this project of any significance, while it is underway. The ultimate technological and economic impacts will be reviewed in Section 10, Value to North Dakota.

This project is needed by Dakota Gasification Company to support a decision whether or not to construct and operate a catechols refining facility in North Dakota. Such a chemical plant would manufacture catechol, 3-methylcatechol, and 4-methylcatechol from crude phenol pitch via distillation and subsequent purification. The process technology to make these three chemical intermediates has been developed and demonstrated in DGC's pilot plant. Marketing of these products, particularly 3-methylcatechol, appears to be the greatest challenge.

For catechol, the markets are well established, and for a material with sufficient purity such as DGC would be able to manufacture, it appears reasonable to forecast that all would be sold without much difficulty.

With regard to 4-methylcatechol, the recent marketing study revealed there are dozens of substances which could be manufactured, including pharmaceutical intermediates, flavors and fragrances, and other fine chemicals of considerable value. Laboratory studies are planned to define commercial synthetic routes to many of these products, anticipating that Dakota Gasification could create a considerable amount of industry to manufacture these in chemical manufacturing process areas which would be above and beyond those areas required to manufacture the catechols themselves.

An effort must be made to provide assurance that a catechols facility would be supported by sales of 3-methylcatechol. There appear to be several possibilities for sales of derivatives of 3-methylcatechol as pharmaceutical intermediates, but these are fairly small in annual product volume. DGC is presently working on the development of a route to pyrogallol acetonide, a substance used to manufacture bendiocarb, a pesticide. This would offer opportunity for considerably larger volumes of 3-methylcatechol to be utilized. It is also possible to manufacture pyrogallol, an important industrial chemical. However, additional opportunities for the sale of 3-methylcatechol are yet required to sell all of DGC's production rate.. If 3-methylcatechol were to be beneficial as a resin additive, this would contribute greatly to the soundness of a decision to construct a catechols facility in North Dakota, since DGC would have assurance that the total volume of 3-methylcatechol could be sold into the market place.

Such a facility, and additionally other facilities to manufacture intermediates from the catechols, would provide a valuable addition to the industrial base of North Dakota, and could ultimately provide employment opportunities for perhaps as many as dozens of North Dakotans. DGC's studies have revealed the gross revenues possible from the three catechols could approach the $30 MM/yr mark, and development of niche products beyond the scope of the major intermediates would raise this to an even greater value. This would represent a significant addition to the economy of North Dakota.

6.4 DELIVERABLES

The deliverables of this project consist of two progress reports and a final report to summarize the findings with regard to property modifications of resorcinol resins. The final report will be of use to provide potential customers with an appreciation of what 3-methylcatechol is capable of accomplishing, with regard to resorcinol resin property modification. Due to confidentiality restrictions with the USDA FPL, as well as commercial practices in the resin manufacturing industry, these reports will be edited to provide appropriate information on the findings without violating the secrecy restrictions.

The final Phase I report will include the following:

- The results of the computerized literature search, for information on resorcinol resins and copolymers of various kinds;

- The results from the baseline studies of both single monomer and mixed monomer resins, which will include all measurements of resin properties pertinent to DGC's interests;

- The results of the studies wherein increments of 3-methylcatechol or hydroxymethylated 3-methylcatechol are added in lieu of a portion of the phenolic component of the resin charge, in a series of batch tests, which will include all measurements of resin properties of interest to DGC;

- The results of all resin optimization studies, wherein resin recipes for resorcinol copolymers are optimized to best exploit any property benefits revealed by step 2, which will include all measurements of resin properties or interest to DGC, and all recipes developed (detailed instructions, sufficient to permit replication of the work); and

- The results of the laminated wood specimen studies, wherein test data are developed to quantitate the strength of cured gluelines made by way of the candidate copolymer recipes developed, and which will include all data relevant to DGC's interests.

The interim reports will describe the progress achieved so far, highlighting any findings of success and/or difficulties and report on cost and schedule considerations. The technical topics will be addressed as outlined above for the final report.

7.0 STANDARD OF SUCCESS

The standard of success for the resorcinol resin properties modification project will be the production of commercially superior or more economic resins for the lamination of wood products, such as laminate beams and other glued wood products which require resorcinol based resins that cure at room temperature, and which would benefit from increased glueline flexibility.

The results from this project will provide sufficient information for DGC to decide if and how to proceed with resin manufacturers and possibly wood handling factories to convince them of the benefits of resins having improved properties. Acceptance of resins which incorporate 3-methylcatechol as a raw material, and which have favorably modified properties and sound economics, would be a final standard of success.

8.0 BACKGROUND

DGC, late in 1994, employed a consultant, Mike Herrington of Cascade Resins, to work with the Process Development Laboratory staff to investigate the possibility that a room temperature curing resin could be made from 3-methylcatechol. The intent of this laboratory investigation was to determine if 3-methylcatechol could be substituted for resorcinol, to prepare a resol resin which would cure at room temperature, using paraformaldehyde as a catalyst. To accomplish this study, a proprietary recipe provided by Cascade Resins, intended for making a commercial resorcinol resin, was used as a starting point. This recipe was used, with 3-methylcatechol substituted for resorcinol. A number of the variables were explored, but in all cases, the product resin failed to cross link as it ought to have done.

Based on the results of this study, DGC has concluded that 3-methylcatechol is unlikely to be appropriate for manufacturing resins, when it is used as the principal phenolic specie (as a substitute for resorcinol), to make formaldehyde resins of the resol type. Apparently, the methyl group in the 3- position (on the aromatic ring) inhibits the ability of this kind of resin to properly cross link.

One feature of the resins produced at DGC that attracted our attention was the fact that they exhibited a high degree of flexibility. Indeed, it was the extreme flexibility of the resins that contributed to the downfall of the project. This finding was realized to be of importance, when near the end of this 1994 study, DGC learned that 3-methylcatechol has been successfully used, in Europe, not as a major component of the phenolic charge to resin batches, but as a minor component, in order to achieve modification of resin properties. DGC has learned that 3-methylcatechol has been used in this way, to modify the properties of both phenol-formaldehyde (PF) resins, and resorcinol-formaldehyde (RF) resins of the room temperature curing type. The amount of 3-methylcatechol required to achieve modification of resin properties would be fairly small, relative to the amount of the principal phenolic specie used in the charge to a resin batch kettle. The resin property to have been improved by the use of 3-methylcatechol was cured glueline flexibility.

Our consultant, Mike Herrington, upon learning of the beneficial use of 3-methylcatechol to achieve improved resin flexibility, shared with DGC that resin flexibility is an important property for a number of reasons. For one, glue laminate beams in some applications can be subjected to repeated bending stresses, and the flexibility of the glue line to a large degree dictates whether or not, over the years, the glue lines of a beam will retain their integrity, or start to delaminate. The glue line will start to crack (delaminate) after such a beam has been in service for a number of years, if the glue does not have sufficient flexibility. Furniture manufacturers consider flexibility to be an important issue, since glue lines in materials used for seats in such as classroom chairs, or park or church benches, are repeatedly subjected to bending stresses, and delamination can be a serious issue as such furniture ages. Also, a number of window manufacturers machine plywood (made with P-F glue), and the machinability of such plywood is a critical property, and is dictated by glue line flexibility.

Based on our information that 3-methylcatechol has been used to modify glue properties, and the suggestions made by our consultant, Mike Herrington, DGC intends to establish a contract with the USDA Forest Service Forest Products Laboratory, in Madison, Wisconsin. This Laboratory has considerable experience with resol-type phenolic glues, and is well equipped to investigate the impact of 3-methylcatechol on the properties of resorcinol glues.

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 the production of krypton/xenon, phenol, and cresylic acid, DGC has also invested heavily in facilities to enhance work to develop process technologies for the separation and purification of coal-derived chemical by-products. 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. Attachment A provides resumes for the Principal Investigator and a Chemist.

Since the major activity of developing a new resin lies with the FPL which was chosen for this work, the following background information and qualifications are provided:

U.S. Forest Products Laboratory
U.S. Department of Agriculture
P.O. Box 5130
Madison, WI 53705

This laboratory can draw upon over 80 years of experience, and offers service in many fields of wood product development. The attached information (Attachment B) contains a listing of publications on resin development prepared by the FPL, as well as some general information on the capabilities and history of the FPL. This organization is well prepared to tackle these issues. Wood adhesive studies are under the direction of A. H. Conner, whose background is ideal for this type of project.

10.0 VALUE TO NORTH DAKOTA

Development of a viable resin made with 3-methylcatechol is beneficial to North Dakota in several ways. At this time, DGC is nearing the completion of a development program that has resulted in a viable scheme to produce catechols from crude phenol pitch. Although DGC's technology is expected to arrive at reasonable processing costs, the economics of this project appear to have an element of risk, since the markets found so far for 3-methylcatechol would not permit the sale of the entire volume that could be produced. Development of a resin from 3-methylcatechol will result in opportunities to sell bulk volumes, and this will make it feasible to produce catechol and 3-methylcatechol. Building such a facility, estimated in the $20 - $30 MM range, will create over 100 construction jobs in North Dakota. Thereafter, the facility will provide permanent employment to at least 12 people associated with the operation of the plant. Additional maintenance and various support services (laboratory, marketing, etc.) will also be needed, creating yet other jobs.

The revenues projected from such a facility are projected to be in the $8 to $10 MM range. Although this revenue would be obtained by DGC, it would benefit the communities by the increased manpower needs, and would also have a trickle down benefit in local spending. Such a project is also desired to provide revenues to support the viability of DGC's plant operation. Although other projects are already under way, such as construction of an ammonia plant, DGC still needs other opportunities to offset the impact of low gas prices.

North Dakota will also enjoy less tangible benefits, such as the reputation of having highly qualified professionals who develop patentable technologies in the field of coal gasification, and chemical by-products.

11.0 MANAGEMENT

Dakota Gasification Company, as the project sponsor, will have the overall responsibility for the project. The USDA. Forest Products Laboratory in Madison, WI will be responsible for execution of the research plan and will make recommendations to DGC if major unexpected findings are uncovered. Mr. Kuhn, the principal investigator, serves as liaison between DGC and the FPL. He will also provide the progress reports to the Industrial Commission. D. Duncan will follow up on chemistry issues throughout the duration of this project.

12.0 TIME TABLE

The FPL has suggested an overall schedule of 18 months to complete this investigation. DGC anticipates that the schedule to complete Tasks 1.1 and 1.2, the assessment of reactivity, etc., of various pure phenolic, as well as mixed phenolic resins will require approximately 12 months from start of work (anticipated to start in August 1995).

Therefore, DGC proposes to prepare two interim reports and a final report having the following target dates (adjustments to be made as this grant is approved and the start date with the FPL are firmed up).

1st Interim Report	March 1, 1996
2nd Interim Report	September 1, 1996
Final Report	March 1, 1997

13.0 BUDGET

The following information represents the current best estimate relating to the project cost. At this time, DGC has received a proposal from the FPL stating that their to perform the work is $127,774. As these investigations progress, it is conceivable that scope changes may be introduced to enlarge the field of investigation. DGC is also in the process of negotiating cost deducts if the studies warrant being terminated after Task 1.1 and Task 1.2 are completed.

DGC has spent months of pilot plant work to fractionate catechols and to purify them. This development cost, prorated to the amount of 3-methylcatechol needed for this study (approximately 20 lbs.) amounts to $22,000. In addition, DGC has explored on a bench scale making phenolic and 3-methylcatechol resins. This effort contributes to this program in providing a mixed resin recipe and general guidelines for this program. DGC's cost for this effort, including the services of a resin consultant exceed $6,000. DGC support for the study program at the FPL will consist principally of man-hours by D. Duncan and A. Kuhn for consultations, monitoring of technical and project issues, as well as a number of trips to Madison, WI for review meetings.

On this basis, the following costs comprise this development program:

USDA Forest Products Laboratory Study	$127,774
DGC:	
Supply of 3-Methylcatechol	22,000
Preliminary Resin Studies	6,000
Support for FPL Program, e.g. Man-hours, Travel	29,840
Total	$185,614

14.0 MATCHING FUNDS

DGC is prepared to provide the time, material, and financing to carry out this project. DGC requests matching funds as follows:

North Dakota Lignite Research Fund	$ 75,000
Dakota Gasification Company	$110,614
TOTAL:	$185,614

15.0 TAX LIABILITY

See attached affidavit.

16.0 CONFIDENTIAL INFORMATION

No confidential information is contained in this proposal. However, any specific resin making descriptions and requests for detailed study reports would most likely have to be handled as confidential information. DGC plans to issue interim reports and the final report on a non-confidential basis, with the understanding that certain specific information about the resin making and possibly testing may be presented in a general manner.

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 owned to the State of North Dakota.

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