## 2.0 TITLE

## Development of Concrete Admixtures from DGC's Catechols

Applicant:

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Dakota Gasification Company Bismarck, ND 75

Principal Investigator:

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Date:

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Amount of Request:

 Phase 1
 \$28,000

 Phase 2
 \_42,000

 Total
 \$70,000

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## 4.0 ABSTRACT

In late 1995 DGC became aware of the possibility of making an additive for concrete mixtures from compounds present in its crude phenol pitch stream. Such admixtures are referred to as superplasticizers, water reducers, or retarders for concrete setting.

Superplasticizers are used in the concrete industry to reduce the amount of water required in a concrete mix, while maintaining the necessary flow characteristics to allow the pouring of concrete. Superplasticizers have the inherent benefit of making a stronger concrete, due to both the more desirable way concrete cures, as well as the lower amount of water used. Similar but less potent materials are also used to act just as water reducers and/or as retarders, to provide more working time before concrete sets up.

Based on available literature and near-expiration-date patent information, last year DGC explored the possiblity of making superplasticizers from its potential catechols by-products derived from crude phenol pitch. With help from an outside consultant, DGC produced a number of different superplasticizer samples which show significant reduction of viscosity when added to cement mixtures, equal or better to commercially available superplasticizers. Subsequent in-house developments resulted in materials with even greater performance levels.

The objective of this product development program is to evaluate the efficacy of the DGC polymers, and to determine various use-characteristics and effects upon concrete properties. Starting materials for such superplasticizers are two grades of catechols, including the catechol homologues derived from DGC's crude phenol stream. In late December 1996, Braun Intertec, with headquarters located in Minneapolis, was chosen to perform the evaluations of the DGC resins. Braun Intertec has been established and recognized as a leader for unbiased evaluations in this field for many years.

Based on various discussions held with Braun Intertec and proposals received from them, a three phase program will be carried out. Phase One is a screening program in which five potential superplasticizer products are compared to each other, as well as to a blank, to determine which should be used for further testing. Depending on the findings, DGC may wish to repeat part of this work with additional superplasticizer samples, to evaluate differences such as spray dried polymer versus liquid, and the appropriate amount of synergist to be used, before Phase Two is launched.

During Phase Two, one or at most two preferred specimens of DGC superplasticizers will be used. Various properties of concrete will be studied in more detail, over a longer period of time. At the same time, performance comparisons will be carried out using different types of cement, and other concrete compositions.

The results from these evaluations will be recorded in a manner such that experts in the concrete industry can clearly recognize the benefits of DGC's superplasticizer, and appreciate the thoroughness of the work performed. At the same time, DGC will obtain data to perform better economic analysis (the dosage rate which is necessary impacts competitive pricing). Companies active in marketing superplasticizers will be contacted to ultimately reach an agreement how one of them can become a partner in a venture to produce and market a

superplasticizer. Braun Intertec has indicated that they can provide functions which will enhance DGC's entry into the market place.

DGC is aware that such a development program requires many steps before a commercial project is realized. In addition to the above outlined program, Braun Intertec expects a Phase Three program, which should include commercial scale testing as well as assessment of permeability and resistance to chloride ion penetration. The Phase Three program with Braun Intertec and the necessary interactions with companies dealing with admixtures are not part of this current grant application, but are mentioned to round out the description of the development program for a superplasticizer derived from DGC catechols feedstock.

## 5.0 PROJECT SUMMARY

#### 5.1 Project History

Development of a superplasticizer from DGC's catechols requires a number of project steps. In order to assess the suitability of producing a superplasticizer from DGC's liquid byproduct streams, a number of activities have already been carried out, as follows:

- 1. Development of manufacturing procedures to utilize DGC catechols for synthesis of resins, useful as superplasticizers;
- 2. Development of methods for bench scale testing of these resins with cement slurries, to measure the efficacy of these resins in reducing cement slurry viscosity;
- 3. Evaluation of a variety of feedstock combinations, assessment of these using the cement slurry testing protocols, and narrowing of the field of choices to five resins for further testing;
- 4. Development of a contract for testing services, with a firm having large scale test facilities and staff with expertise in the ASTM procedures for testing of concrete specimens with admixtures.

As a result of promising findings in steps two and three, a contract was established late last year with Braun Intertec (one of two laboratories which have been visited by DGC). Efforts to date with Braun Intertec have resulted in defining a test program which will demonstrate the performance of DGC's superplasticizer in a manner acceptable to the concrete industry.

#### 5.2 Project Plans for 1997

During 1997, DGC's R&D Group plans to further pursue the development of a superplasticizer product (or products), designed for use as an admixture for the concrete industry. The overall development program involves the following additional phases:

- Phase 1 Evaluation of five superplasticizer compositions by means of four ASTM procedures for testing of concrete specimens. This is the Phase One portion of the contract with Braun Intertec.
- Phase 2 Narrowing of the field of choices to one ot two compositions for in-depth, further testing during a Phase Two contract, in which more detailed studies will be done.

# Phase 3 Final study of candidate material(s), to conclude the testing necessary for a market-ready superplasticizer product.

**Phase One** represents a screening test to compare the candidate superplasticizers on a fast track basis. Compressive strength, flexural strength, slump, air content, shrinkage etc. will be measured and compared for one cement, which will be used in one specific concrete mixture. Depending on the findings, a few additional tests, similar to the tests just described, could be carried out to compare performance of an even more desirable superplasticizer formula (e.g. optimization of a certain resin for market entry).

During **Phase Two**, one or at most two admixtures will be studied, and performance comparisons will be made at several addition rates, as well as for different cements and concrete mixtures. This type of data is needed to satisfy the interests of batch concrete processors, as well as end-users of concrete, and potential marketing organizations for concrete admixture distribution.

**Phase Three** involves appropriate commercial test application studies to demonstrate large scale performance under field conditions (e.g. impact of late/early dosing, double dosing, as well as permeability, will be measured). This program is very much dependent on findings from the earlier work, and will not be implemented before late 1997. This step is mentioned here to complete the descriptions of the overall development program. Depending on the findings in Phases One and Two, and the projected scope of work in Phase Three, DGC may submit a grant application for Phase Three work to the North Dakota Industrial Commission at a later date.

The results expected from this program consist of technical findings and data needed to support the entry of DGC into the business of manufacturing cement admixture products, and forming an alliance with a suitable party for commercialization and marketing of such product(s).

The total cost to DGC for the proposed project is estimated to be \$144,000. This project will be managed by DGC. All of the large scale product evaluations for efficacy of the compositions in concrete specimens will be the responsibility of Braun Intertec, Inc., according to the contract established with Braun, by DGC. The timetable for the proposed project, now consisting of the remaining three steps, is estimated to be one year, concluding at the end of 1997.

## 6.0 PROJECT DESCRIPTION

#### 6.1 Early Project Developments

During 1995, a literature search of the Chemical Abstracts for information on catechol and methylcatechols resulted in learning that Diamond Shamrock had patented a procedure for manufacturing a superplasticizer from catechol (Diamond's chemical operations were sold in the late 80's, and since that time, the patent has not been maintained). Not long after the date of this patent, Czechoslovakian researchers developed a resin from a mixture of catechol and methylcatechols, derived from coal processing, and patented their procedures both in Czechoslovakia and in Canada (but not in the United States). Study of this patent literature indicated that the Czech product was more effective than the Diamond material. Based on

these references, DGC proceded to examine the Czech methods for polymer synthesis. The resins made in the DGC laboratory, using one of the two methods disclosed in the Czech patent, were found to have significant problems; they contained "clumps" of insoluble polymeric material.

DGC knew a specialist in phenolic polymers who could serve as a consultant. A contract was developed, and laboratory facilities were made available to the consultant, here at DGC, for a month during the summer of 1996. The consultant explored the alternate method disclosed in the Czech patent, yet this, too, did not provide a material having desirable properties. The consultant went on to develop his own method of synthesis, as well as experimental protocols for assessing efficacy of the resins in mixture with cement slurries. The final work product was a batch recipe, optimized for a number of parameters such as pH, formaldehyde to phenolics (f/p) ratio, degree of sulphonation, etc. The consultant's viscosity data for cement slurries, with aliquots of DGC resins added, showed these resins to be very similar to, or better than competing products, in their ability to reduce slurry viscosities. For these tests, increasingly greater amounts of polymer were added to cement slurries of constant composition. The viscosities dropped substantially in these tests, as greater amounts of resin were added. In a second test format, wherein a constant amount of resin was added to cement slurries containing varying (diminishing) amounts of water in the slurry mix, the DGC resins were found to be superior to existing products.

Our consultant's findings confirmed that, as recited in the Czech patent, sodium silicate is a strong synergist with the catechols polymers, to enhance their viscosity-lowering effect upon concrete. Polymers containing sulfonic acid groups, which most superplasticizers are, have a tendency to retard the setting of concrete. Sodium silicate is known to accelerate the setting of concrete, but the amounts required to achieve this effect are significantly greater than the amount appropriate to achieve the synergistic effect with the catechols polymers. Future testing will reveal if the use of silicate as a synergist for the DGC resins will have an effect upon the set-retarding properties of the DGC resin.

After the outside consultant completed his work, DGC continued to apply the batch recipe he developed to various other catechols mixtures in much lower state of purity (refinement), and has found that feedstocks of much lower purity result in product performance greater than when high purity materials are used in the resin-making process. This finding shows that a better product can be made from a less expensive feedstock.

All these efforts have been necessary to develop and identify the most effective resins, and to prepare the necessary sample quantities for Braun Intertec to perform the proposed work. The work has led to five candidate polymers which will be evaluated in large scale testing, using batches of concrete mixed in two cubic yard lots, prepared in ready mix trucks. The in-house screening tests were very simplified, using only cement and water to evaluate the impact on viscosity achieved by addition of superplasticizer samples. Braun Intertec's test program will provide the necessary sophistication demanded by the concrete industry to evaluate polymer performance as a superplasticizer.

#### 6.2 Superplasticizer Test Program

#### 6.2.1 The Phase One Testing Program with Braun Intertec

In the first phase of the Braun Intertec evaluation, five different DGC superplasticizer formulations will be investigated, as well as control concrete, to which no admixture will be added. This evaluation will be carried out in accordance with ASTM C 494 (Standard Specification for Chemical Admixtures for Concrete). It is anticipated that the resins will be properly classified as *Type F* -- *Water -reducing, high range admixtures*. This is one of the seven types of admixture categories for which this testing method is appropriate. Alternately, *Type G* -- *Water-reducing, high range, and retarding admixtures* may prove from the testing to be the appropriate category. In the terminology section of the ASTM Standard, *Type F* is given the following definition: "an admixture that reduces the quantity of mixing water required to produce concrete of a given consistency by 12% or greater." The *Type G* definition is: "an admixture that reduces the setting of concrete." In contrast, the requirement for a water reducer (other than high range) is only to "reduce the quantity of mixing water required..." (a percentage requirement is not stipulated). Based on this, we know the candidate materials are indeed high range.

An exception to the tests outlined in ASTM C 494 is that only three beams will be evaluated for ASTM C 666 "Resistance of Concrete to Rapid Freezing and Thawing." For the purposes of this study, concrete will be batched at a ready mix plant, and the admixture added to the load of concrete prior to testing. In order to maintain some between-batch consistency, Braun will be batching approximately two cubic yards per truck. In order to perform this phase of the work, Braun will use sufficient admixture to dose approximately 1,400 lbs of cement. Sufficient superplasticizer will be used to achieve a slump of eight inches.

The following properties of concrete will be determined during this study:

- Compressive strength at 1, 3, 5, 7 and 28 days. This differs from the requirements of ASTM C 494, which also requires strength at 90 days and one year (however, specimens will be prepared and kept so that these tests could be run, at a later date, at the discretion of DGC).
- Flexural strength. Beams for flexural strength will be tested at 3, 7, and 28 days.
- The properties of plastic concrete will be tested, including air content, slump, slump loss and time of setting. Time of setting and slump loss are important properties to test, as they are the reason for using high-range water reducing admixtures. High-range water reducing admixtures generally fall into two classes, retarding and non-retarding.
- Resistance to freezing and thawing. The concrete will be cast into beams and tested in accordance with the method outlined in ASTM C 666, Method B, for resistance to freezing and thawing.
- Length Change. The effect of the admixture on shrinkage/expansion of concrete is required by ASTM C 494. The concrete will be cast into beams and tested in accordance with ASTM C 157, "Length Change of Hardened Hydraulic-Cement Mortar and Concrete."

No petrographic or microscopic studies of the microstructure of the concrete will be performed during the Phase One study. At the completion of Phase One, the results of Braun's tests will be reported to DGC. The formal report will include a comparison with the control concrete, data for the other requirements of ASTM C 494, and general observations of the performance of the admixture samples and their relative efficacy. A meeting will be held between DGC and Braun's scientific and engineering staff, to discuss the feasibility of proceeding to Phase Two, as well as specifics for Phase Two, of the evaluation studies.

Depending on these exploratory results, DGC may elect to screen a few additional admixture batches in the same manner as described above, e.g. to explore the impact of spray dried versus liquid superplasticizer, changes in composition of plasticizer, etc., before proceeding to Phase Two.

#### 6.2.2 The Phase Two Testing Program with Braun Intertec

If at the conclusion of the Phase One program it is determined that one or more admixtures are worth exploring further for use as a high-range water reducing agent, a more extensive study including variation of the composition of the cement and concrete, timing and quantity of admixture addition, and interference with other commercially available admixtures, is to be carried out. In this phase, a more complete battery of tests will be run at each composition and each rate of addition, including the later age strength measurements required by ASTM C 494, and petrographic analyses to assess if microstructure development of the cementitious phase of the concrete is not inhibited. Based on the findings in the 6.2.1 step of the program, and joint discussions between DGC and Braun Intertec, a detailed work plan will be prepared and agreed upon by both parties.

#### 6.2.3 The Phase Three Testing Program

This program step is listed here to indicate what additional research and development work is anticipated after completion of tasks 6.2.1 and 6.2.2. Because the definition of this portion of the program, including cost, is too vague at this time, and because no work is expected to start before the last quarter of 1997, there is sufficient lead time for DGC to evaluate the situation later this year. If deemed appropriate, DGC may prepare a follow up application to the North Dakota Industrial Commission later this year.

If at the completion of the Phase Two program, it is determined that product development should proceed for a selected admixture, a further study of the durability and long-term use characteristics of concrete prepared with the candidate superplasticizer(s) will be undertaken. This phase will consist of measuring properties of concrete made with the high-range water reducing agent, such as permeability, resistance to chloride ion penetration and microstructural changes. In addition, work will be carried out in conjunction with several ready mix concrete suppliers to evaluate the performance of the admixture under actual field conditions, including late dosing, double dosing and early dosing. The details, timing and scope of Phase Three depend on the findings in the earlier phases of the evaluation. After discussions between the two parties, Braun Intertec will provide a proposal to DGC outlining the scope of work and projected cost.

#### 6.3 Deliverables

The deliverables of this project include, on a "non-proprietary basis," at least one report for each of the two phases (per sections 6.2.1. and 6.2.2.). Each report will describe the findings and major achievements, as well as any difficulties encountered. Proprietary scientific data will not be included in the reports. However, appropriate product performance characteristics and analytical results will be provided. The reports will include sections on conclusions and recommendations, thereby offering sufficient information to define the success of the program.

## 7.0 STANDARDS OF SUCCESS

The standard of success for this project will be the development of data supporting the market entry of a DGC superplasticizer product(s). This data will provide the necessary product performance information to attract firms having both an established position in the admixtures field, as well as a marketing and technical support organization. It will strengthen DGC's position in negotiations with such firms. At the same time, it will allow DGC to better establish a competitive market value for its product, which is needed for a final economic analysis of such a market entry. Successful development of a product will greatly enhance the prospects of constructing a facility for production of catechols, given that a superplasticizer product would increase the likelihood of selling a substantial portion of the plant capacity.

## 8.0 BACKGROUND

#### 8.1. DGC's Interest in Developing a Market for Superplasticizers from Catechols

Pitch from the phenol/cresylic acid processing area at DGC is presently being combusted as boiler fuel. This 35 to 40 MM pounds per year stream contains about 2 MM pounds per year of catechol, 2 MM pounds per year of 3-methylcatechol, and 4 MM pounds per year of 4-methylcatechol. DGC has developed a process technology to separate and purify these substances to purities ranging from 99% to 99.8% purity. DGC has applied for U.S. patent coverage for this technology. Considering the various sources of catechols at DGC, the yearly production volume of resin could reach 26.5 MM lb. per year.

Of the three substances available, 3-methylcatechol will be the most difficult to market, due to a lack of chemical intermediates of sufficient market volume which could be derived from it as a synthesis starting point. The situation is quite different for 4-methyl catechol, in that many dozens of intermediates could be manufactured, but even for this material, the capacity of a full stream manufacturing facility at DGC would outstrip the demand for some years to come. DGC's 3-methylcatechol is presently the subject of a study partially funded by NDIC funds, to investigate its potential use in resins for adhesive applications. The basis of this study had been word that 3-methylcatechol is being used in Europe for such uses, and late last year this indication was confirmed via another source of information.

Due to a number of product and market uncertainties, and due to the large volume of total potential output from DGC, it is necessary to investigate several potential applications for catechols in parallel. Sufficient end uses for 3- and 4-methylcatechol are essential to the economics of plant construction, given the amounts that could be produced by DGC.

#### 8.2 Use of Superplasticizers in the U.S. Concrete Market

The total amount of ready mix concrete poured in the United States, in 1996, was 264 million cubic yards (this is a 7.32% increase over 1995). The total market for admixture products of various kinds, such as water-reducers, set controllers, air entrainers and superplasticizers, amounted to over \$280 million in 1995. Superplasticizers account for a little less than 20% of this market, and water-reducers, similar to superplasticizers but less potent in effect and less expensive, amount to nearly another 40% of this market volume, altogether representing about \$150 MM in revenues during 1995.

Projections for the future growth of superplasticizers are excellent. The superplasticizer market is projected to have considerable growth by the year 2000 (a 60% increase over the 1995 market volume). Estimates have been made by a knowledgeable firm that the growth from 2000 to 2005 will be almost as good (about 44%). The large growth figures are in part related to the projections showing the need for rebuilding a significant fraction of the infrastructure of the United States. Presently there is a shortage of cement in the construction industry. Superplasticizers permit using less cement, while concrete strength specifications are met. Economics dictate that the more rural ready mix operators become more technically astute, learning to substitute superplasticizers for cement to meet market demands and competition from the larger population centers.

The dose of superplasticizer (on a contained dry polymer-solids basis) which is typically used, is in the range of 0.1% to 0.4% by weight of the concrete (superplasticizers are usually sold as a 27% to 38% solution in water). The total market for superplasticizers is expected to grow from the \$50 MM per year range to the \$80 MM per year mark by the end of this decade. This translates to 15 million gallons, and if on the average, the materials are 33% solids content, and average 10 lb./gallon, then roughly 50 million pounds of resin solids will be sold per year by the year 2000. In addition, there is even a greater market for water reducers.

#### 8.3 Benefits of Superplasticizers for Concrete Mixtures

Superplasticizers, as they are known in the concrete industry, are a class of materials which have a strong lowering effect upon the viscosity of freshly mixed concrete, and an enhancing effect upon the strength of concrete. In pouring concrete into forms, the benefit obtained consists of the fact that less water can be used in a concrete mix, without incurring an increase in viscosity (e.g. the slump of the concrete can be as good as if more water had been used). Freshly mixed concrete containing a relatively low amount of water, in the absence of a superplasticizer, is very difficult to place into forms. The less the amount of water used in the formulation of concrete, the greater the compressive strength the concrete will have. It is possible, using superplasticizers, to prepare concrete materials having compressive strengths as high as 20,000 psi. The gain in compressive strength of concrete is more than commensurate with the lessened amount of water; superplasticizers have the innate ability to affect strength above and beyond the degree obtained solely by reduction of the amount of water used for a concrete mix.

In a simplified manner, this phenomenon can be explained as follows. Superplasticizers are, chemically, polymers which contain a large number of sulfonic acid groups attached to the polymer chain (backbone). On a molecular level, the polymer chains orient to the cement particles in a way such that the sulfonic acid groups are attracted to the grain surface. The effect of this polymeric coating on the grains is to prevent the hydration of the grain for a time, until the effect dissipates, during which time the water in the mix can function as a slurrying agent, e.g. is able to act as a viscosity reducer, rather than become bound to the cement grains as water of hydration. According to the scientific literature on this topic, the function is not limited to this, as the polymers are said to ultimately result in greater degree of hydration at later ages of concrete, and this results in greater strength (compressive, flexural, tensile, shearing, etc.).

Concrete typically weighs 4050 lb. per cubic yard, and contains 700 lb. of cement, 3070 lb. of aggregate, and 280 lb. of water (a water : cement ratio of 0.4). When superplasticizers are used, the water : cement ratio can be as low as 0.28, and the slump, even at this low water ratio, can be sufficiently high to permit pumping and placement into forms (the "slump" of a concrete mix is measured as the loss of height incurred as concrete spreads out, when a form in the shape of a truncated cone is lifted away from a wet slurry sample of the freshly-mixed concrete).

In the case of water reducing and retarding admixtures, the emphasis is more on maintaining the flow characteristics of concrete while a truck is traveling or the construction site experiences delays before a pour can be made.

## 9.0 QUALIFICATIONS

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

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

A significant contribution to this development program is provided by Braun Intertec. Their engineering and scientific expertise, as well as state-of-the-art technical and analytical capabilities are greatly valued for this program. Braun Intertec has a number of locations in primarily the northern states, and has been involved in projects in nearly all fifty states. Concrete technologies are a strong point for Braun.

Braun Intertec is part of a family of Companies owned by COI, Incorporated. The other companies are Basys Technologies, which specializes in biofilters for biomass control, Braun Intertec Great Lakes, an environmental consulting firm, and Yucai-Braun Transportation, Consulting and Management, Ltd., a joint venture in China, with Chinese partners. Braun Intertec provides engineering, environmental and laboratory services, and is involved in a wide range of services, including the testing of concrete and asphalt for highway use. Background information on Braun Intertec is provided in Attachment B.

DGC is familiar with Braun Intertec, having employed them as site consultants for a number of years. The Braun representative on site referred us, last summer, to Kevin MacDonald, Braun's Senior Materials Engineer, who works at the Minneapolis testing laboratory. Mr. MacDonald will be Braun Intertec's project manager.

## **10.0 VALUE TO NORTH DAKOTA**

Development of admixture products is beneficial to North Dakota in a number of ways. DGC's Phenosolvan pitch stream could produce as much as 26.5 MM lb. per year of resin solids. Assuming a 15% U.S. market penetration, and some export sales, this would amount to 13 MM pounds per year of sales, and the value of the product could be in the order of \$20 MM per year. It should be noted that this project does not necessarily have to be able to absorb the total capacity of available catechols. The explorations with the U.S. Forest Products Laboratory (NDIC Contract FY-96-XX1-65) regarding use of 3-methylcatechol hopefully leads to another market for DGC's catechols.

The best of all benefits to DGC, and thus to North Dakota, would be that construction of a catechols processing facility becomes possible, with a secure product to assure the covering of the gap between the pounds of methylcatechols that could be produced, and the amount of the purified isomers that could be sold into the fine and specialty chemicals industry.

The necessary plant addition for manufacturing catechols products will create temporary construction jobs in North Dakota, and contribute to the financial stability of DGC. Along with other plant expansion projects, catechols processing may also contribute to an increase in the plant labor force, thus creating additional tax revenues from these plant employees as well as from their impact on the North Dakota economy.

## 11.0 MANAGEMENT

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

## **12.0 TIMETABLE**

As indicated in the project description, Dakota Gasification Company has already performed a number of tasks in preparation for this project, during 1996. It is anticipated that Phase One of the proposed program can be completed during the first four months of 1997. The following schedule lists key events:

#### Phase 1:

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Start of Phase 1 testing:	January 15, 1997	
Preliminary Phase 1 results ready for review by: (excepting ASTM C 666, which requires more time)	March 31, 1997	
Final Report for Phase 1:	April 30, 1997	
Phase 2:		
Start of Phase 2 Testing:	May 1, 1997	
Preliminary Phase 2 Results, ready for review:	September 30, 1997	
Final Report for Phase 2:	November 15, 1997	

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## 13.0 BUDGET

The following information represents the current best estimate relating to the project cost. Preparation of the superplasticizers required many trials and evaluations, including work done by the outside consultant. DGC has incurred costs exceeding \$30,000 for this effort, not counting the high cost of catechols feedstock produced at DGC's R&D facilities.

The projected cost for Braun Intertec Phase 1 work is \$21,000. The costs for Phase 2, according to Braun's experience, should be in the range of \$75,000. In addition, DGC will have to produce additional quantities of superplasticizer, and monitor as well as evaluate the program during both phases. Therefore, the following cost estimate for the first two phases is presented:

Phase 1

Development and Production of Feedstock Braun Intertec Scope of Work DGC Support, Evaluation	\$30,000 \$21,000 \$ 7,000	
Phase 1 Subtotal		\$ 58,000
Phase 2		
DGC Feedstock Production Braun Intertec Scope of Work DGC Support, Evaluation	\$ 4,000 \$75,000 \$ 7,000	
Phase 2 Subtotal		\$ 86,000
Total Project Cost (Phases 1 and 2)		\$144,000

#### 14.0 MATCHING FUNDS

DGC is prepared to provide the time, materials and financing to carry out this project. Due to the staged approach of work schedule considerations and the minimal risk that DGC may reach a conclusion that Phase 2 should not be pursued, DGC requests matching funds at this time for both phases, with the understanding that Phase 2 could be cancelled if findings from Phase 1 demonstrate unattractive performance or economics.

It should be noted that DGC may incur additional expenses if after Phase 1 it is deemed desirable to perform exploratory testing with additional resins (see Section 5.0). Furthermore, if both phases are successful, DGC will need to embark on a Phase 3 program (see Section 6.2.3). If the scope of Phase Three is small, DGC will absorb its cost. However, if the costs are found to exceed \$50,000, DGC might apply for an additional grant in the summer of 1997.

	Phase 1	Phase 2	Total
North Dakota Industrial Commission	\$28,000	\$42,000	\$ 70,000
Dakota Gasification Company	\$30,000	\$44,000	<u>\$ 74,000</u>
Total	\$58,000	\$86,000	\$144,000

## 15.0 Tax Liability

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

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

28/9

Date

## **16.0 CONFIDENTIAL INFORMATION**

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