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PROCESSING OF STEVIO

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THE DEMOCRATIC PEOPLE'S REPUBLIC OF KOREA

Technical report: Process selection and optimization*

Prepared for the Government of
the Democratic People's Republic of Korea
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

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ABSTRACT
STEVIA PROCESSING
DP/DRK/88/008

This report covers activities at the Foodstuff Research Institute (FRI) in Pyongyang from 5th. September to 27th October, 1992. The main objectives of the mission were; analytical method development and testing, evaluation of process alternatives and selection of a conducive one, optimization of the selected process and definition of the overall process for design of a pilot plant.

Two methodologies based on TLC and HPLC were tested and adopted for the research activities on stevia at FRI. Evaluation of three process alternatives identified electrochemical treatment to be the most conducive one for FRI pilot plant. Unit operations in the process were optimized using mulfifactor optimization designs. The overall process was defined giving through-put for each unit operation. The daily production was perched at 120 kg of leaves per 24 h with anticipated yield of 14-17 kg. Due to safety concerns the termination point of the process was changed to production of partially purified stevia extract rather than sweetener crystals. The following were recommended:

1.The next task for the project be performed off-site.

2.Funds be provided by host to keep laboratory equipment functional and to refinish the pilot plant floor.

3.Project budget be reviewed with the view of incresing it.

4.A 4-6 month fellowship be provided for training on enzymology of transglucosylation processes.

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INTRODUCTION.

Compelled to depend on the non-caloric stevia sweetener to augment both industrial and domestic needs for sweeteners, the stevia processing project is of socio-economic importance to DPR-Korea. The project which is under the mandate of the Foodstuff Research Institute (FRI) has been on-going for some years now. In a previous UNIDO experts mission report (DP/ID/SER.A/1358, 15th. June 1990) it was recommended that FRI should test other process alternatives which could reduce the level of consumables that are dependent on foreign exchange. It was further recommended that UNIDO/UNDP provides the laboratory with minimum analytical and labware capabilities in order to facilitate their research and development efforts and for proper quality control and assurance of the process and products. The project is almost over one year behind schedule based on the revised project schedule (Appendix V of the 1990 report). The schedule clearly allocated the tasks for subsequent work and provided targeted completion dates.

This report, written by Dr.Yaw J Owusu-Ansah, an expert in food processing and process development, covers activities at the FRI in Pyongyang, dating from 5th. September to 27th. October, 1992. The report provides responses to tasks 3 and 4 (i.e.Analytical methods development, equipment testing and process optimization (3) and process alternatives selection(4)), as expounded in Appendix V of the 1990 report. The two months (1st. September-31st. October, 1992) mission of which the activities discussed in this report form a part was a follow-up to the 1990 mission.

The project job description and duties as sent by UNIDO is presented in Appendix I. This description does not seem to take into account the work done in previous missions as it was essentially the same job description for the first mission. During the briefing session in Vienna the mandate of the mission was verbally and succinctly put as; to define the pilot plant

process for design purposes and equipment acquisition.The main objective of this mission was, therefore,to define the process for the stevia pilot plant and identify the key equipment for purchase. The specific goals to attain are expanded as follow:

A.Review equipment and glassware sent by UNIDO and assess their adequacy and limitations (if any) and potential contribution to the progress and completion of the stevia processing project.

B.Evaluate the merits of the training given by the equipment manufacturer and during the training mission in China.

C.Set-up equipment for the various analyses that would be used during the mission and prepare the necessary calibration standards and curves for quantitation.

D.Test and critically examine the three processing options described in the 1990 mission report and select the most appropriate one for optimization.

E.Optimize the pertinent unit operations in the chosen process.

F.Define the process based on the optimized data and specify throughputs for design and/or equipment selection.

G.Provide a listing of the main equipment needed for the process and possibly cost estimates. Clearly indicate which items would be produced in-house and which would have to be provided by UNIDO/UNDP.

H.Evaluate any potential environmental consequences of the process and assess its overall safety.

I.EXAMINATION AND REVIEW OF CURRENT SITUATION.

Parts of the recommendations made in the 1990 mission report have been carried out. As such it became important to evaluate the impact of these on the overall progress of the project. The recommendations carried out so far were: a study tour of senior officials to India and China to select an institute for subsequent training, a six week training mission to China for four personnel associated with the project, purchase and installation of pertinent analytical equipment and training of personnel on how to run the equipment by a representative of the equipment manufacturer.

A.Review of the overall project.

The positive impact of meeting the recommendations cited above on the overall progress of the project was not apparent. The selection of China for the training by the study team might be a good one. However, my examination of the trip report indicates that proper hands-on training was not attained. This might be due to problems with material acquisition and handling. China and DPR-Korea seem to be equally crippled when it comes to supply of essential, foreign dependent, scientific items. Several operations which require few hours to complete took days (unproductive) to complete. For example it took nine days for ion exchange and adsorption resins to be regenerated and made ready for use. The work schedule was planned in such a way that, other activities were not or could not be performed during this waiting period. As such the time spent on unit operations and on analytical equipment for which training was needed was minimal. Few days were spent on HPLC analysis and possibly limited training, and on melting point determination. As a result of this apparent inadequate training and exposure to prototype equipment supplied by UNIDO/UNDP, most of the supplied equipment remained

unused until the commencement of this mission.

The one week training offered by the equipment manufacturer during installation was, at best, very rudimentary. Except for the fermentor and the spectrophotometer, which could be operated because of prior familiarity, the information gained during the training could not be used to operate the equipment. Inadequate understanding of the english language hindered the use of the manuals for self training.

It was anticipated that with the installation of the laboratory equipment and other materials, process alternatives B and C described in the previous report (DP/ID/SER./1358, 15th. June.1990) would be fully evaluated before the commencement of this mission. This was not done due to the factors discussed in this and preceding paragraphs. The personnel in the institute were, therefore, counting on the consultant to provide the necessary guidance for operating the equipment under expected conditions, so that data obtained from them could be dependable. Consequently developmental work (R&D) on the stevia project has essentially been stagnant; at the same status quo since 1990. Any activities have been to repeat what had already been established using the same methods used before. This mission, therefore, had to fulfill other tasks previously not allocated to it (i.e.testing of process alternatives B and C, evaluation and selection of an acceptable process) as presented in Appendix V of the 1990 report.

B. Examination and assessment of materials and equipment supplied by UNIDO/UNDP.

Glassware and other materials.

Materials, including glassware, filter papers etc. recommended were supplied,except for few items especially, cnromarods SII, which are needed for the existing Iatroscan to be functional. Other important items missing were; glassware and filters for filtering HPLC solvents, syringes (disposable or otherwise) and sample filters for filtration of samples before HPLC analysis.

There was an over supply of some items above the quantities recommended in the 1990 report. This was obviously due to minimum order limit requirements normally imposed by suppliers of scientific materials. Also due to misreading of the list of recommended list by the suppliers, some odd specifications of glassware were supplied. For example, the listing of 1, 5, 10, and 25 mL pipettes in the recommended list was read as 1.5 mL, 10.0 mL and 25.0 mL. As a result 1.5 mL pipettes were supplied but non for 1.0 and 5.0 mL. Overall if the chromorods SII are supplied, it is my view that there are enough supplies for the institute to carry out the project to fruition without much problems with respect to laboratory equipment and materials.

Equipment.

1.Fermentor.

This equipment is needed for the transglucosylation experiments. The equipment was supplied with all needed accessories. This was in use during the mission.

2.**TLC plate scanner.**

The supplied scanner was an up to-date computerized version capable of handling both TLC and gel electrophoresis. This equipment would be needed for research on the stevia process, especially for identifying decomposition products due to variable processing conditions. The low amount of solvent needed for TLC coupled with the minimum amount of accessories and materials needed for such test make TLC the obvious choice in situations of scarcity as experienced in the institute. The scanner would complement the TLC accessories and allow quantitative determinations of the stevia sweetener components. The equipment was not functional during the early parts of the mission. This was diagnosed to be the result of poor electrical contact on the slit plates within the equipment. It has been repaired by an electrical engineer and is now fully operational.

3.**UV/VIS spectrophotometer.**

The equipment is functional and is currently used routinely to follow the decolorization operation in the stevia process.

There was obviously an over supply of unrecommended accessories for this equipment. Kinetics software, 160 L sipper, GSC-3A gel scanner, TSC-240A thermal cell holder and ASC-5 auto sample changer, supplied with the equipment (not recommended) are NOT needed for the project. The cost of these items (equivalent to $18,407) becomes an unwarranted over budget expenditure.

4.**HPLC system and accessories.**

The HPLC is an indispensable equipment for proper quality control of the stevia sweeteners according to industrial standards for the product. It is, therefore, a necessary equipment for any research and development (R & D) efforts on these sweeteners. The

model supplied is good and up-to-date. The equipment was not functional during the early parts of the mission due to a minor "VALVE HP ERROR", disenabling the automatic injector. The lack of an appropriate (correct) manual for the controller of the injector delayed any efforts to correct the error.The equipment is now functional and used routinely.

There was an over supply problem with this equipment.A system with a manual injector and a UV/VIS detector was recommended.However, a system with automatic injector, a UV/VIS detector and a fluorescence detector were supplied. Automatic injectors are normally more expensive to purchase and maintain than manual injectors. Secondly, conditions that necessitates the use of automatic injectors are not the prevailing conditions in the Foodstuff Research Institute of DPR-Korea. The supply of an additional fluorescence detector (not applicable to the stevia project, cost not listed in purchase order but estimated to be $6,000) again represents an unusable over expenditure. A simple manual injector plus the UV/VIS detector with the HPLC pump would have adequately met the needs of the project.

General remarks.

The basic equipment and materials needs for this project have been met.It would be up to the management of the institute to provide sustainable materials (i.e., parts, solvents etc.) to support the equipment and keep them functioning. It is,however,obvious that probably an over zealous salesman took undue advantage of the situation and supplied unneeded and unrecommended accessories for some of the equipment, notably some items for the HPLC and the UV/VIS spectrophotometer. The additional cost would obviously affect the overall budgetary requirement of the project irrespective of its magnitude.

II. EVALUATION OF PROCESS ALTERNATIVES.

To accurately evaluate the three processing options recommended in the 1990 report, the analytical equipment had to be standardized and methods established. The HPLC system was set-up and used to determine the sweetener content. TLC plate analyses was correlated with the HPLC data. Mindful of the problem with replenishing sustainable supplies dependent on foreign resources at the institute, this correlation was crucial in case the HPLC system cannot be used for lack of HPLC grade solvents or a breakdown. The methodology used for the TLC is routinely used by the institute. The HPLC method was based on protocols in the literature (Kitada et al., 1989. J. of Chromatography 474:447-451). and used in my laboratory for determining stevia sweeteners in foods.

Sweetener standards; stevioside and rebaudioside A were prepared on site to over 95% purity through repeated methanol recrystallization and preparative TLC. Other sweetener components for which standards were not available were not analyzed. However, since stevioside and rebaudioside A form the bulk of the sweeteners in stevia, their combined use as yardstick for evaluating the three process alternatives can be said to be technically sound and dependable. The regression equations and correlation coefficients for the two standards (co-determined in a mixture) for the HPLC and scanned TLC plates are shown below:

HPLC

Stevioside:- y= 5320.31 +117289.24X r=0.9996

Rebaudioside A:- y= 1405.21 + 73720.71X r=0.9997

where X is the peak area count from the integrator.

TLC Plate

Stevioside:- y= 17466.76X - 1380.20 r=0.997 (1-3ug)

Rebaudioside A:- y= 15238.64X - 1749.74 r=0.995 (1-3ug)

where X is the peak area of the scanned TLC spot of the sweetener.

All responses on yield in this report are based HPLC analysis. Percent decolorization was calculated as follows:

$$\frac{(Ai420-Ai671)-(At420-At671)}{(Ai420-Ai671)} \times 100$$

where Ai420 and At420 are the respective absorbances of the input solution and the solution after treatment at 420 nm. Ai671 and At671 are the corresponding absorbances at 671 nm.

The evaluation of the three processes was based on the following criteria in order of priority;

1. Processing yield and purity of the sweetener and efficiency of the unit operations involved.

2.Availability of consumables at affordable prices.

3.Infrastructure support availability and compatibility to the process.

4.Cost of operation of the process.

5.Potential of pollution and compatibility with sound environmental protection practices.

Throughout the evaluation due cognizance was given to potential cost of capital equipment and accessories involved in each process.

A. **Electrolysis plus ion exchange treatment.**

The mass balance, efficiency (based on yield and percent decolorization) of the unit operations within the electrolysis process are shown in Table II.1. An overall process yield of 67.71% was obtained for the two replicate tests conducted. The major losses of the process occurred during the ion exchange treatment. The loss due to the electrolysis treatment could almost quantitatively be recovered by resuspension of the sludge produced in the reaction and refiltration. Discussions with the scientist involved in the project and who developed the electrolysis process currently in use at the institute indicated that the losses due to the ion exchange treatment are dependent on the type and extent of usage of the resins. He further indicated that the resins used in the tests; Levettit MP- 64,(the only type available) seem to have greater retention properties for the sweeteners.According to him, almost total recovery can only be obtained if the column is repeatedly washed with water. Repeated washing was not carried out in the test and may be responsible for the high losses observed. He also indicated that from his experience with the process and the many resins he has tried, the best

TABLE II.1. COMPARISON OF YIELD AND EFFICIENCIES OF PROCESS ALTERNATIVES.

PROCESS UNIT OPERATIONS	SODIUM ALUMINATE				ELECTROLYSIS				HYDROGEN PEROXIDE			
	% D.M.	% Decolor	Sweeten er (g)	% Loss	% D.M.	% Decolor	Sweeten er (g)	% Loss	% D.M.	% Decolor	Sweete ner (g)	% Loss
Starting Extract	2.45	0	2.49	0	2.45	0	9.94	0	2.45	0	2.49	0
Electrolysis Treatment	N.A.	N.A.	N.A.	N.A.	1.45	98.58	7.20	27.57	N.A.	N.A.	N.A.	N.A.
Cation Exchange Treatment	N.A.	N.A.	N.A.	N.A.	1.11	99.05	6.30	12.50	N.A.	N.A.	N.A.	N.A.
Anion Exchange Treatment	N.A.	N.A.	N.A.	N.A.	1.00	99.78	3.73	40.79	N.A.	N.A.	N.A.	N.A.
Sodium Aluminate	2.67	98.31	0.038	97.4	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
Hydrogen Peroxide	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	1.54	92.12	1.47	40.96
Activated Carbon Treatment	3.01	98.86	0.038	97.40	N.A.	N.A.	N.A.	N.A.	1.33	96.96	0.35	76.19
Sludge Filtration	0.76	99.77	1.00	-	0.48	99.81	3.00	N.A.	N.A.	N.A.	N.A.	N.A.
Final Filtrate	3.01	98.86	0.038	98.47	1.00	99.78	3.73	34.41	1.89	98.82	0.214	38.86
Overall Yield(g)	1.038				6.73				0.214			
Overall Yield(%)	41.69				67.71				8.59			

resins for the process seem to be Amberlite IR-120 and Amberlite IRA-45, cation and anion exchange resins respectively. When these resins are used the losses were said to be 2 and 3% respectively after washing the columns with water.

The combined electrolysis and ion exchange treatment was very effective in removing the contaminating coloring components. Over 99% decolorization was achieved by this process. The high losses was, however, of concern but the observations of the resident scientist could not be verified due to lack of those particular resins he claims to function better.

The key consumable materials and utilities for the process are; aluminum plates, ion exchange resins and electricity. Aluminum plates were said to be locally available; costing approximately 130 won ($62.50) per ton. The consumption of aluminum plates per ton of stevia leaves processed has been estimated to be 30 kg. Thus it costs approximately $1.90 in aluminum consumption to process one ton of leaves. Electricity cost was said to be similarly minimal; perched at 25 won ($12.02) per 1000 kWh. The most expensive and locally unavailable consumable is the ion exchange resins.

The process is essentially simple, environmentally compatible since it does not generate any toxic or undesirable effluents. The precipitated,probably proteinaceous sludge could conveniently be incinerated or developed into a composite for plants.

B.Sodium aluminate plus activated carbon treatment.

The sodium aluminate treatment was simple and as effective as the electrolysis in removing the color pigments from the extract (Table II.1). The immediate treatment yield (i.e. after the aluminate treatment but before carbon treatment) was 58.63%. The

loss could be quantitatively recovered by reslurrying the sludge resulting from the filtration and refiltering.
The subsequent activated charcoal treatment operation did not substantially improve the color (Table.II.1), but there was a loss of 97.38% of the input sweetener to this operation. The loss could not be recovered by resuspending the retentate in water and filtering. From my personal experience with activated carbon retention of stevia sweeteners, they can only be effectively removed with alcohols or aqueous alcohols but not only water. The overall yield of the process was 41.69% (Table.II.1).

This treatment offered a very simple and effective way for decolorization of the extract. The contact time was relatively short and only stirred tanks would be needed for the process. The overall yield was, however, low apparently due to the activated carbon treatment. It is conceivable and probably more prudent to link the aluminate treatment with the ion exchange treatment instead of activated carbon in order to improve the yield.

The major consumables needed for the process are sodium aluminate, hydrochloric acid and activated carbon. For some unexplained reasons, aluminum plates are said to be available to the general consumer but aluminate from which the aluminum plates are made is said to be unavailable. The personnel at the institute doubted whether a sustainable supply of this chemical would be available for their needs. Activated carbon which is not produced in the country cannot be obtained readily. The aluminate treatment when coupled with ion exchange treatment could be equally as effective as the electrolysis process. When coupled with activated carbon treatment, the overall process is inferior to the electrolysis plus ion exchange treatment in terms of sweetener yield.

Similar to the electrolysis treatment, the aluminate treatment process was safe and equally compatible with the environment.

C.Hydrogen peroxide plus activated carbon treatment.

The process was satisfactory in reducing the color of the extract but this was achieved at the expense of the sweetener content. There was approximately an unrecoverable loss of 41% of the input sweetener after the hydrogen peroxide treatment alone. The subsequent activated carbon treatment further reduced the yield by 76.19% (Table.II.1), bringing the cumulative loss to 85.94%. Another 38.86% loss of sweetener was observed in the final treatment with Actisil activated clay (Table II.1). The overall percent decolorization of the extract by this process was 98.82% with a sweetener yield of 8.59%. Comparing the hydrogen peroxide treatment alone with that of the sodium aluminate, approximately the same amount of sweetener was lost (Table II.1), but the aluminate was more effective in color removal than the hydrogen peroxide. The hydrogen peroxide treatment could also be linked with ion exchange treatment rather than activated carbon into an overall acceptable process.

Hydrogen peroxide is considered a rare item locally. Infact obtaining a small sample for this test posed considerable difficulties. It would, therefore, not be possible to obtain it in larger quantities to sustain the needs of the pilot plant. The additional aluminum chloride needed for the process was equally difficult to obtain. The process, therefore, seems unfavorable because it cannot be supported by the local infrastructure resources.

The process was simple, requiring relatively short contact time and was as environmentally compatible as the others.

D. General discussions and conclusions.

Based on the results from the replicate tests of each process and the assessment of the unit operations and materials involved, the electrolysis process was considered more conducive. The choice was mainly due to the higher yield than the other processes tested and the ready availability of the needed materials and resources. With respect to capital equipment cost, the process seems more expensive than any of the other two tested since an electrolyser has to be designed, probably from scratch, for the process. However, it would be an unpardonable mistake to set up a process plant for which the key consumables are not readily available or can be obtained within reasonable cost and time.

The use of ion exchange resins seems almost unavoidable since each of the processes evaluated introduce foreign ions into the product. Other chelators can only be considered if they form physically removable precipitates with the ions. Such a chelator has not been identified for the process and would take considerable amount of time to develop any possible protocol for such an operation. The best choice at the moment is to use ion exchange resins which happens to be universally used in the stevia industry.

The choice of the electrolysis process also surreptitiously provides an added benefit of versatility. If sodium aluminate becomes available to warrant its use, the same equipment in place at the time could be used. Such flexibility could be of immense benefit towards the R&D efforts of the institute on stevia sweeteners.

III. OPTIMIZATION OF PROCESS UNIT OPERATIONS.

The selection of a workable process required that the process be optimized for final definition and designing. The electrolysis process development has been going on since 1979, but the parameters have been empirically selected or chosen based on one factor at a time experimentation. Such experiments neither provide the best use of time and resources nor yield the optimum conditions. As such the pertinent unit operations, notably; extraction, electrolysis and ion exchange treatments were optimized using appropriate multiple factors optimization designs.

A. Extraction operation.

In the mission report of 1990, extraction at the pilot plant was considered redundant due to the existence of the Sam Suk plant. A suggestion was made to collect extracts from the Sam Suk factory for use in the pilot plant. Upon further examination of the situation, during this mission, and analyzing the ultimate objective for setting up the pilot plant, it is concluded that such an exercise would neither be feasible nor beneficial to the institute for the following reasons:

1. The main objective for setting up the pilot plant is to enable the institute to carry out independent research. The findings of such research efforts would then be extended to the industry. Depending on the Sam Suk plant for extract would restrict the institute to working with one type of extract. Such an imposition would hinder the development of novel extraction methods and conditions and prevent their being tested.

2. The Sam Suk plant and the research institute function under

different organizations. Negotiations could take a long time and would adversely affect any progress on the stevia R&D efforts.

3. Transportation would be needed to bring the extract from the Sam Suk plant. A vehicle and appropriate tanks to do this task are currently not available to the institute.

4. Sam Suk might not be receptive to preparing small amounts of extracts to the specifications of the research needs of the institute. Such requests might not be congenial to their production practices.

Due to these factors it was decided that the extraction be performed in the pilot plant, therefore, the operation needed to be optimized.

Important variables that could influence yield in the extraction operation are; temperature, solid to water ratio and time of extraction. Three factors optimization designs based on Box-Behnken design (Box, G.E.P. and Behnken, D.W., 1960. Technometrics, 2: 455-475) and uniform precision rotatable central composite designs (Joglekar, A.M., 1991; Optimization Software) were used to optimize this operation.
The optimum yields were obtained at high water to solids ratio, 20:1 (the maximum level used in the designs). A yield of 101% based on stevioside and rebaudioside A relative to the amounts of these sweeteners extracted by Soxhlet extraction with methanol was obtained at conditions of 85°C, 20:1 water to solids ratio and extraction time of 60 mins. Due to volume limitations, this water to solid ratio was not considered practically feasible. The practical optimal conditions were set at 65°C, water/solid ratio of 11:1 and extraction time of 60 mins. These conditions were predicted by model equations to yield 99.5% recovery for combined stevioside and rebaudioside A relative to corresponding yields from

Soxhlet methanol extraction. Confirmatory tests in the laboratory consistently produced recoveries of 98.2-99.8% with mean of 99.0%. These conditions were modeled with leaves milled to pass 20 mesh screens. When unmilled leaves were used, as currently practiced, the mean yield was 83.1%. This indicates that the current practice of extracting the leaves with 8:1 water/ratio may be grossly suboptimal. The new conditions were selected for the extraction operation in the pilot plant using milled leaves between 15 to 20 mesh. The residue after the first extraction may be extracted once more and the solution used as part of the liquid feed for the next extraction in an attempt to recover residual sweeteners.

B. Electrolysis operation.

The electrolysis operation is probably the most crucial of all the operations in the DPR-Korea process, therefore, particular attention was paid to its optimization. Discussions with the resident scientist before setting up the experimental designs indicated that the modulating factors were; pH, temperature of the reaction, voltage, type of electrode material, distance between the electrodes, time for the reaction and mode of connecting the electrodes.Volume of extract used seemed not to be significant based on experiments previously conducted by the resident scientist with 0.1 L and 40 L extracts using the same conditions. From previous experiments, aluminum plate electrodes at distances of 25 mm apart have been found to produce the best results. The 25 mm was the lowest limit in the experiments. Mathematical extrapolations of the data obtained by the scientist suggested that the true optimum might be 20 mm. Also parallel connection rather than series had been identified as the best. The electrolyser available has, therefore, been designed using aluminum plates at 25 mm apart and a parallel connection. The remaining factors; pH, temperature, voltage and reaction time were optimized using a rotatable central composite optimization design. To stabilize the variances, the responses were squared and analyzed.

Analysis of the percent decolorization response data indicated that the full model equation was significant but to improve this the less significant variables were removed. The linear, quadratic and interaction effects of the factors were all found to be significant (Table III.1) The lack of fit was highly significant, indicating that there were other important factor(s) not considered in modeling the experiment. Possibly the plate distances could be the/or one of those factor(s). In my view protein content of the extract could be an important factor. Future model experiments (during pilot plant optimization) would have to take these factors into consideration. The model equation generated (Table III.2) was highly significant and can be used in a predictive manner to estimate this response.

Response surface plots for the model equation at various levels of the factors are represented in Appendix II. The plots show that the optimum regions for the tested factors are; pH, 2.0-3.3, temperature, 20-50°C, voltage, 6.8-11.2 and time 20-45 mins. Various combinations of these levels would yield different results. For example at a temperature of 45°C and time of 25 mins, the optimum pH and voltage are 2.64 and 8.0 respectively.

The electrolysis process has a yield response. Overlay plots of the yield and the percent decolorization model equations showed that the compromised optimal conditions are: temperature; 45°C, time; 20 mins voltage; 10 and pH;2.4.

TABLE III.1
ANALYSIS OF VARIANCE TABLE FOR ELECTROLYSIS OPTIMIZATION RESPONSE.

ANALYSIS OF VARIANCE

Experiment: steex Electrolysis | INT'L QUAL-TECH,Ltd. | Response: Decolor Power | Transform: Power

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Value	Percent Confidence
Model	12	2.62E+08	2.18E+07	9.057	99.9%
Linear	4	1.62E+08	4.06E+07	16.827	99.9%
Quadratic	2	3.64E+07	1.82E+07	7.541	99.4%
Interactions	6	6.34E+07	1.06E+07	4.381	98.9%
Residual	14	3.38E+07	2.41E+06		
Replication	2	2.58E+04	1.29E+04		
Lack of Fit	12	3.37E+07	2.81E+06	217.867	99.6%
TOTAL	26	2.96E+08	1.14E+07		
Full Model	14	2.62E+08	1.87E+07	6.729	99.9%
Deleted Terms	2	3.36E+05	1.68E+05	6.03E-02	5.8%

Variation Accounted For By The Model:	88.588%
Standard Error of Estimate:	1553.068
Coefficient of Variation:	17.155%

TABLE III.2

THE COEFFICIENTS AND SIGNIFICANCE OF MODEL EQUATIONS FOR THE ELECTROLYSIS RESPONSE.

SELECT MODEL PARAMETERS

Experiment: steex Electrolysis — INT'L QUAL-TECH,Ltd. — Response: Decolor — Transform: Power

Expected Mid Point Value = 9053.063

Linear Terms

	#	Parameter	Coefficient	%Conf
*	1	A-PH	-2007.470	99.9
*	2	B-Temp	1212.041	99.8
*	3	C-Volt	1021.102	99.4
*	4	D-Time	472.208	84.4

Interaction Terms

	#	Param.	Coefficient	%Conf
*	9	A x B	1773.501	99.9
*	10	A x C	313.806	56.0
*	11	A x D	368.803	62.7
*	12	B x C	-398.590	67.7
*	13	B x D	-490.749	77.5
*	14	C x D	428.341	71.1

Quadratic Terms

	#	Parameter	Coefficient	%Conf
*	5	A Squared	-1060.639	99.6
	6	B Squared		
*	7	C Squared	-745.299	97.2
	8	D Squared		

The predicted percent decolorization at these conditions was 97.18. Actual mean value obtained from confirmatory laboratory trials was 97.24%, indicating that the model could adequately predict the response. The yield obtained at these conditions was 72% after the first filtration. When the pH and time levels were changed to 3.0 and 40 mins. respectively, the percent decolorization obtained was 98.56 against a predicted value of 99.18. The yield obtained was, however, 63.98%, which was lower than that at the compromised optimal conditions.

The suggested optimal, operational conditions for the electrolysis process are; temperature, 45°C, pH,2.4, time, 20 mins. and voltage, 10.0. These conditions are optimal for an electrolyser made of aluminum plate electrodes at 25 mm apart.

C. Ion exchange operation.

The ion exchange treatment was the next important operation in the process and was optimized using a Box-Behnken optimization design. The important variables were; % dry matter of the feed, the feed pH, and the permeate rate(SV), assuming standardized, well packed columns. Due to non availability of the preferred ion exchange resins (i.e. Amberlite IR-120 and IRA-45) the cation exchange resin used was SK-111 and the anion exchange was Levettit MP-64 (both strong ion exchange resins).

The responses were separated into the contribution made by the cation exchange treatment alone and the overall contribution of the whole operation (i.e. cation and anion exchange treatments).

Analysis of the cation exchange response and the significant factors affecting this treatment are shown in Tables III.3 and III.4 respectively. The model equation was significant and

TABLE III.3.
ANALYSIS OF VARIANCE TABLE FOR CATION EXCHANGE OPTIMIZATION RESPONSE.

ANALYSIS OF VARIANCE

Experiment: SteA Cation Optimization — INT'L QUAL-TECH,Ltd. — Response: DECOLOR — Transform: None

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Value	Percent Confidence
Model	7	3232.484	461.783	4.039	95.7%
Linear	3	1229.361	409.787	3.584	92.5%
Quadratic	2	647.953	323.976	2.834	87.5%
Interactions	2	1355.170	677.585	5.926	96.9%
Residual	7	800.323	114.332		
Replication	2	132.629	66.314		
Lack of Fit	5	667.694	133.539	2.014	63.9%
TOTAL	14	4032.807	288.058		
Full Model	9	3241.986	360.221	2.278	81.2%
Deleted Terms	2	9.502	4.751	3.00E-02	2.8%

Variation Accounted For By The Model:	80.155%
Standard Error of Estimate:	10.693
Coefficient of Variation:	21.572%

TABLE III.4.

THE COEFFICIENTS AND SIGNIFICANCE OF MODEL EQUATION FOR THE CATION EXCHANGE RESPONSE.

SELECT MODEL PARAMETERS

Experiment: SteA INT'L QUAL-TECH,Ltd. Response: DECOLOR
Cation Optimization Transform: None

Expected Mid Point Value = 49.567

Linear Terms

	#	Parameter	Coefficient	%Conf
*	1	A-pH	3.599	61.6
*	2	B-SV	-2.202	41.7
*	3	C-DM	-11.656	98.3

Interaction Terms

	#	Param.	Coefficient	%Conf
*	7	A x B	17.190	98.5
	8	A x C		
*	9	B x C	6.580	74.2

Quadratic Terms

	#	Parameter	Coefficient	%Conf
	4	A Squared		
*	5	B Squared	11.083	91.6
*	6	C Squared	-6.375	71.2

accounted for 81.2% of the total variation (Table III.3) It is worthwhile to note that insignificant factors were removed from the equation and that the response was not transformed.

Response surface plots of the decolorization response showed that optimum results at practically reasonable rates can be attained at lower dry matter content of the permeate (Appendix III). The practically reasonable dry matter regions for this treatment was 0.5-1.8. At these ranges the optimal pH of the feed was from 3-5 at a flow rate (SV) of 0.5-1.3. For a reasonably economical operation, a compromised permeate rate could be perched at 2.0 and the pH around 4.

Decolorization response for the overall ion exchange treatment was transformed into its logarithmic derivative and analyzed. The model was highly significant (Table III.5). The linear, quadratic and interaction effects of the factors were all significant (Table.III.6). The model equation accounted for 81.98% of the total variation.

Response surface plots of the decolorization response (Appendix IV a-e) identified the optimal dry matter level of the permeate to be 0.88% (Appendix IV b). Above this level, but below a dry matter content of 2.5%, optimum decolorization can be attained at relatively low permeate rates (Appendix IV e) Between dry matter levels of 0.5 to 1.5%, optimal conditions can be attained at practically reasonable flow rates of 1.0 and pH ranges of 4.2-6.0 (Appendix IV a-d).

The suggested optimal conditions for the anion exchange treatment are; percent dry matter of 0.5-1.5, pH range of 4.2-6.0, at a flow rate of 1.0. Using an extract with 1.4% dry matter, pH of 4.3, a cation permeate rate of 2.0 and an anion permeate rate of 1.0, the predicted response for decolorization was 98.6%. The actual value obtained at these conditions was 98.96%.

TABLE III.5.

ANALYSIS OF VARIANCE TABLE FOR THE OVERALL ION EXCHANGE OPTIMIZATION RESPONSE.

ANALYSIS OF VARIANCE

Experiment: ANION Anion Optimization | INT'L QUAL-TECH,Ltd. | Response: Decolor-T Transform: Log 10

Source	Degrees of Freedom	Sum of Squares	Mean Square	F Value	Percent Confidence
Model	6	0.843	0.140	6.031	98.8%
Linear	3	0.288	9.62E-02	4.129	95.2%
Quadratic	2	0.127	6.33E-02	2.719	87.5%
Interactions	1	0.428	0.428	18.361	99.7%
Residual	8	0.186	2.33E-02		
Replication	2	5.56E-02	2.78E-02		
Lack of Fit	6	0.131	2.18E-02	0.783	34.6%
TOTAL	14	1.029	7.35E-02		
Full Model	9	0.864	9.60E-02	2.914	87.4%
Deleted Terms	3	2.16E-02	7.18E-03	0.218	12.0%

Variation Accounted For By The Model:	81.894%
Standard Error of Estimate:	0.153
Coefficient of Variation:	8.485%

TABLE III.6.

THE COEFFICIENTS AND SIGNIFICANCE OF MODEL EQUATION FOR THE OVERALL ION EXCHANGE OPTIMIZATION RESPONSES.

SELECT MODEL PARAMETERS

Experiment: ANION Anion Optimization — INT'L QUAL-TECH,Ltd. — Response: Decolor-T — Transform: Log 10

Expected Mid Point Value = 1.799

Linear Terms

	#	Parameter	Coefficient	%Conf
*	1	A-pH	0.133	96.2
*	2	B-SV	-0.106	91.8
*	3	C-DM	-8.43E-02	84.6

Interaction Terms

	#	Param.	Coefficient	%Conf
*	7	A x B	0.327	99.7
	8	A x C		
	9	B x C		

Quadratic Terms

	#	Parameter	Coefficient	%Conf
*	4	A Squared	-0.131	86.6
	5	B Squared		
*	6	C Squared	0.120	83.5

IV. PROCESS DEFINITION AND REQUIREMENTS

A. Process definition.

The original product target of the pilot plant was a crystalline stevioside and rebaudioside A sweeteners produced through methanol crystallization of partially purified sweetener extract (PPSE). As previously indicated in the 1990 consultants' report, this would require major renovations of the existing building and supplies in order to meet the safety standards for handling flammable solvents. Such renovations, though recommended in that report, have not been done yet and it is not foreseeable that this would be done, at least not within the time span for completion of this project. As such, for purely safety reasons, the target product and termination point of the project should be the production of dry PPSE. The process definition has been made with this product as the target. Based on the results and discussions in the preceding chapter, the process for the pilot plant is defined as follows:

Extraction.

The plant would process 120 kg of milled (15-20 mesh) stevia leaves (@ 7% moisture) with an anticipated yield of 14-17 kg powder as final product depending on the purity, per 24 h. The 120 kg leaves would be extracted using a water to leaves ratio of 11:1, at 65+1°C with mild agitation for 60 mins. The slurry would be filtered, and the sediment recycled for an additional extraction. The filtered or drained extract from the second extraction would be used as part of the feed water for the next extraction. The expected yield from the filtered first extraction would be 1130-1180 L, with a total soluble solids of 50-52 kg and percent dry matter of 4.2-4.6 and would serve as the feed to the electrolysis operation.

Electrolysis.

The electrolyser would be charged with 100-120 L of extract in a batch mode, or should be capable of processing 100-120 L of extract in 20 mins in a continuous mode. The electrodes for the unit would be made of aluminum plates at distances of 20-25 mm, preferably 20 mm at an operational voltage of 10 volts. The pH of the extract would be between 2.3-2.5, adjusted with hydrochloric acid. The reaction time or residence time would be 20 mins. Anticipated time for completion of this operation is 4 h. The electrolyzed extract would be filtered to yield 790-825 L of filtrate and a sludge of 320-330 kg. The sludge would be slurred with water three times its weight and mixed for 20 mins and filtered in order to remove entrained sweeteners.Volume yield of this second filtration would be 950-1000 L. The two filtrates (1740-1825 L) would be the feed for the ion exchange treatment. The expected percent dry matter of a pooled filtrate would be 0.7-0.9%. The duration of the filtration operation would depend on the capacity of the filter and pumps used but should be sized such that a minimum filtration rate of 300 L/h is achieved.

Ion exchange treatment.

The pH of the filtrate would be adjusted if necessary to bring it within the optimal range of 4.2-6.0. The cation exchange treatment would operate at SV of 2 and the anion at an SV of 1. To further improve the purity of the eluate, a third mixed bed resin (40:60 cation:anion) would be connected in series with the preceding two and would operate at an SV of 2. Using a cation resin of 150 L bed volume and an anion resin of 300 L bed volume and a mixed bed resin of 150 L bed volume, the anticipated completion time of this treatment is 6 h. However, to remove entrained sweeteners from the beds,water 2.5 times the total bed volumes would be pumped at the same flow rates as the feed through the beds.

The expected yield volume of the first permeate would be 1300-1360 L and that for the second washing operation would be 960-1040 L. A total volume of 2260-2400 L with anticipated dry matter content of 0.66-0.71% would be the total feed to the evaporator. The total operation time for the ion exchange treatment would be 9 h.

Evaporation.

Since the solids content of the feed is relatively small, it can be safely assumed, for evaporation time purposes, that the entire volume of the feed is being evaporated. With an evaporator of 150-200 L/h (reasonably sized pilot plant evaporator) the expected completion time for this operation would be 16 h.

Spray drying.

The minimum target solids content for the feed to the spray dryer is 25%. This means that a feed of approximately 70 L would be entering the dryer. With a dryer capable of operating with nominal water removal capacity of 30 L/h, this operation would take 2.5 h to complete.

General comments.

A conceptual flow diagram of the process is shown in Appendix V. The combined total hours for the operations in the process as defined is over 24 h. As such some of the operations would have to be overlapped with each other whenever possible in order to complete it within 24 h. A schematic diagram for scheduling the operations in order to achieve this is provided in Appendix VI. To further buy time for the operations and also free the plate and frame filter and a pump for the electrolyser, the extraction and filtering is scheduled to be done ahead of time. The pH of the filtered extract would be adjusted and the extract kept in a surge tank for feeding to the electrolyser. Appendix VI was prepared with this in mind and, therefore, does not include the extraction operation.

B. Process requirements.

The following equipment and accessories would be required for the process as defined. It is probably premature to provide cost estimates for the equipment, however, to support the discussion on the project budget (next chapter), tentative cost estimates are provided based on used, refurbished equipment prices where applicable. It should be noted that these are for discussion purposes only. The prices could significantly vary depending on the make of the equipment, source of acquisition and options needed, as would be specified by the design and specification engineer(s) for the project.

Mill:-A hammer mill for coarse milling (15-20 mesh) of the leaves. A mill with feed auger and compatible motor and screens capable of producing 150-200 kg product/h would be required. The mill would be manually fed. (UNDP,$8-10,000).

Extractor:- Two jacketed tanks of 2000 L capacity each, with motorized agitators would be required for the extraction operation. The tanks would serve as both batch extractors and surge tanks for the extraction and feeding of the electrolyser. Each tank would have a bottom spout with compatible flanges to be connected to flexible hoses by clamps to pumps to and from the filter. (UNDP, $15-20,000)

Filter:-A plate and frame filter of probably adequate capacity is already available at the institute and would not need to be purchased (FRI)

Air compressor:- An air compressor for blowing the plate and frame filter would be required. (UNDP, $6000).

Electrolyser:-A custom designed jacketed batch electrolyser with temperature readout and controller, aluminum plates and leads to a AC/DC converter would be required. An optional design with heating elements on the outside of the inner chamber may prove useful. It would have a spout with a compatible flange for connecting to pumps via flexible hoses to and from the unit. Although the rated input feed is 100-120 L, a foam of 4-5 times this volume is generated during the reaction. Adequate headspace would have to be provided to hold this foam. The inside of the unit would be stainless steel or food grade heavy duty epoxy resin based material resistent to acidic conditions and should be contoured to facilitate easy cleaning. (UNDP, $10-12,000).

AC/DC converter:- An AC/DC converter with capacity to provide the needed voltage and variable controllers would be required. (UNDP, $8-10,000)

Ion-exchange columns:- Two columns each of strong ion exchange resins (Amberlite IR-120, cation and Amberlite IRA-45, anion); cation (150 L), anion (300 L) and mixed (40:60 cation:anion, 150 L) would be required for a streamlined operation. One set would be in operational mode while the other is in regeneration mode. Additional resins of one column volume each would be required as a support item. Two tanks for alkali and acid respectively would be required for regeneration of the columns. The columns would be made of stainless steel with inlet and outlet flanges compatible for connecting to flexible hosings leading to pumps feeding each unit connected in series. Totalizing counters to monitor flow rates would also be needed.(UNDP, $10-12,000).

Evaporator:- As indicated in the 1990 report, a forced film evaporator with stainless steel (food grade finish) construction, capable of evaporating 150-200 L/h water, with feed/vacuum condenser system would be required. (UNDP,$35-45,000).

Spray dryer:- A stainless steel spray dryer (food grade finish) capable of removing a minimum of 30 L/h water would be needed. the dryer should have exchangeable rotary atomizer and may be steam or electrically heated. Since electrically heated models are cheaper this may be the option to consider. It should be a complete system with its cyclones, motors, dust collecting or retaining systems and product collecting chambers. (UNDP,$45-55,000).

Packaging:- A small form-fill-seal packaging equipment with built in scaling device might be needed to package the prototype products in 10 g polyethylene sachets. (UNDP, $10-12,000).

Auxiliary supplies:- Supplies such as; surge tanks of 600 L capacity each, pumps of various pumping capacities, flexible (food grade) hosing with compatible stainless steel flange fittings, valves, filter cloths for the plate and frame filter and washers would be needed (UNDP, $40-55,000).

It should be noted that the list provided is by no means exhaustive. The actual specifications of the required equipment would have to be provided by the design engineer for the process. However, as a preliminary measure the estimates provided could be used to forecast possible cost scenario.

V. PROJECT BUDGET AND SCHEDULING.

A. Project budget.

A copy of a project budget document given to me at the UNDP office in Pyongyang showed the projected total budget of the project for 1992 is $260,535 and $59,000 for 1993. Of these amounts $59,000 each has been allocated to personnel (item 11-99) for 1992 and 1993. A total of $126,612 was the remaining amount for equipment (item 049) and allocated to 1992. Since there was no and would not be a major equipment purchase in 1992, this amount may be transferred to 1993.

The preliminary estimates presented in the preceding chapter for discussion purposes indicate that a projected amount of $237,000 would be needed for equipment purchase to equip the pilot plant if used refurbished equipment are considered. Thus the total equipment budget (item 049 of $128,612) would be insufficient to equip the pilot plant. Major revision of the budget, with the view of increasing it, would be needed if the project is to be carried to fruition. Streamlining the personnel budget might free a relatively small amount to augment the equipment budget.

According to the budget document 1.0 m/m is allocated for personnel item 011-001 for 1992. Since this amount would not be used this year it would be transferred to 1993 and consolidated into a 3 m/m activity for that year. If used equipment are considered then the only equipment requiring design work would be the electrolyser. Since a batch operation is contemplated as the preferred option the designing of the unit would become less complicated. However, extensive planning would be needed in sizing, selecting and designing the equipment placement in the pilot plant. The three month activities of this item could be divided into a 1.5 m/m off

site activity for equipment design, sizing, selection and placement designs and 1.5 m/m for supervision of installation and start-up.

A 3.0 m/m activity had been projected for item 011-002 in the document for 1992. Since 1.0 m/m would not be utilized this year this can be transferred to 1993 and the activities consolidated into a 3.0 m/m activities. Activities for this item can be divided into 0.5 m/m off site to liaison with 011-001 in the equipment selection, sizing and the designing in order to minimize potential errors due to unqualified assumptions. This may be crucial since the designs are being prepared off-site. 2.0 m/m would be spent on quality assurance and control and transglucosylation process testing and optimization. Since the budget for items 011-001 and 011-002 seem low for today's consulting costs, the amount for the remaining 0.5 m/m allocated to 011-002 could be used to augment the total budget for items 011-001 and 011-002.

Budget items 011-003 and 011-050 do not seem to have any clearly defined purpose. Due to the gross under budget of the equipment component (041 and 042) the combined budget for 011-003 and 011-050 could be transferred to generally item 049 (equipment).The remaining amount in the equipment (item 049) budget is $113,252. By transferring budget items 011-003 and 011-050 to 049 the new amount would be $136,852. Based on the tentative cost estimate provided in the preceding chapter, this amount is still below what is required to equip the pilot plant into a viably operational facility.

The original total budget amount for equipment on this project was $285,000. This amount would have been able to equip the pilot plant if equipment refurbished to original specifications were considered. The budget was probably made with the assumption that basic laboratory equipment to support the project were available. When this erroneous assumption was corrected in 1991 by providing the necessary laboratory equipment, this was not compensated for in

the original budget. As such the equipment component of the budget is currently grossly inadequate. The over supply of some laboratory equipment accessories not needed in the project,and not recommended by the consulting experts, also puts a dent in the amount available for this component. Changes in the dollar value over the years and inflation have also affected the original budget. It seems that a revision of the budget is necessary in order to reflect current situations and to compensate for the cost of the supplied laboratory hardware not originally considered in the budget. The exact amount needed would only be known after the design and equipment specifications have been made but for budget purposes an increase of about $100,000 would suffice.

B. **Scheduling of activities.**

A revised project activity schedule is provided in Appendix VII. This was made with the assumption that all the tasks for the project would be completed in 1993. It is essential that this target is achieved since any delays could adversely affect the budget requirements of the project. In order to meet this target, it is essential that all tasks are executed promptly and adequate preplanning given to implementation of each task.

VI. CONCLUSION

The stevia project is a proposition of socio-economic importance to DPR-Korea and needs to be fully supported both domestically and by UNIDO/UNDP to carry it to its successful completion. The overall success of the project (short and long term) may depend on the cooperative efforts of the administrative body of the project. The project has steadily progressed and would culminate in success if the additional funding needed to purchase the essential pilot plant equipment is provided.

Three processes have been evaluated and the most conducive one (electrolysis process) selected and optimized. The process has been defined and the equipment needed have been identified. Preliminary cost estimates, based on used equipment prices have been provided as a budgetary guide.

The defined process is efficient, environmentally compatible and comparatively sustainable by the current infrastructure than other contemplated alternatives. Adequate laboratory equipment and hardware are now in place for further research and for quality control/assurance of the process and product.
Due to safety factors the original goal of producing sweetener crystals cannot be realized at the pilot plant level. In pursuance of this goal research efforts could be carried out in the laboratory. The projected transglucosylation process does not seem ready for pilot plant. Further laboratory development should be encouraged and supported.

The UNIDO/UNDP component equipment budget seems inadequate to achieve the contemplated task. A revision of the budget with the view of increasing it is necessary.

RECOMMENDATIONS

The following recommendations, if carried out, would help in attaining the overall project objective.

A. Execution of next task in the project.

The next tasks for the project is equipment design equipment specification, selection and equipment placement drawings. These tasks are to be carried out by a design engineer. It is recommended that they are done off-site in order to reduce cost and for fast execution. The designs, specifications and drawings could be sent by fax or courier through the UNDP office when completed for rectification by the local authorities. The tasks should take 1.5 m/m to complete. It is further recommended that such designs and selections be made in consultations with the process development specialist who defined the process in order to minimize design errors due to unsubstantiated assumptions. This activity should take 0.5 m/m.

B. Laboratory.

Sophisticated equipment have been provided by UNDP to augment the R&D efforts of FRI on the project. The equipment would require proper maintenance and supplies dependent on foreign exchange. Funds should be made available from the national administrative body of FRI to maintain and keep the equipment functional. An annual allocation of funds in hard currency, from the government is recommended for this purpose.

Future research in improving the unit operations involved in the process would be beneficial. A more fruitful approach would be for the institute to set up project teams involving specialists from various related disciplines. Such concerted efforts yield better and speedy results.

C. Pilot plant processing.

The process as currently defined requires a key item (ion exchange resins) dependent on foreign exchange. To keep the plant running successfully and yield products of consistent quality, this item would have to be replenished when exhausted. Foreign exchange allocation towards purchase of this item is strongly recommended. The R&D efforts of the institute should be directed towards finding local substitutes or locally developed resins for the process.

The pilot plant facility must be renovated and the utilities upgraded to improve its safety. The anticipated termination point of the project as conceived by FRI is not compatible with the currently available service resources in the facility. To accommodate this plan in future, renovations such as upgrade of electrical work, refrigeration plant and upgraded boiler would be needed. To meet the quality assurance standards for the process as currently defined, the floor should be refinished and pest control systems put in place.

D. Budget and scheduling.

The remaining equipment component of the UNDP project budget is insufficient to meet the demands of the process. A revision of the budget is recommended. Personnel budget components 011-003 and 011-050 do not seem to have defined purposes. It is recommended that these be transferred to the equipment component of the budget to augment it.

Adherence to the scheduling plan as presented in Appendix VII is recommended in order to prevent further budgetary problems.

E. Training.

Transglucosylation is a beneficial technique for stevia sweetener

development. A fellowship is recommended for a food scientist or biochemist to study the enzymology aspect of this process and expertly develop and implement it in the institute's stevia research programme. Much work has been done on the microbiology aspect of this process in the institute but the practical enzymology aspect is lacking. A 4-6 month fellowship is recommended to be funded by UNDP.

APPENDIX I. JOB DESCRIPTION

T. De Silva/la

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

DP/DRK/88/008/11-02(J13422)

JOB DESCRIPTION

Post title:	Chemical Technologist
Duration:	3m/m (split mission)
Date required:	July 1992
Duty Station:	Pyongyang
Purpose of project:	Enhancement of the efforts of the Foodstuffs Research Institute's Stevio Research Project to develop improved commerfcially viable processes for the production of sweetening agents from the plant Stevia rebaudiana, based on the laboratory research already carried out and ongoing commercial methods.

Duties: The expert will be required to review the present processes at laboratory level and the commercial process for production of purified extract of stevia and determine the optimum process parameters for designing of a pilot plant, and assist in its construction. Once the pilot plant has been designed and fabricated the expert will:-

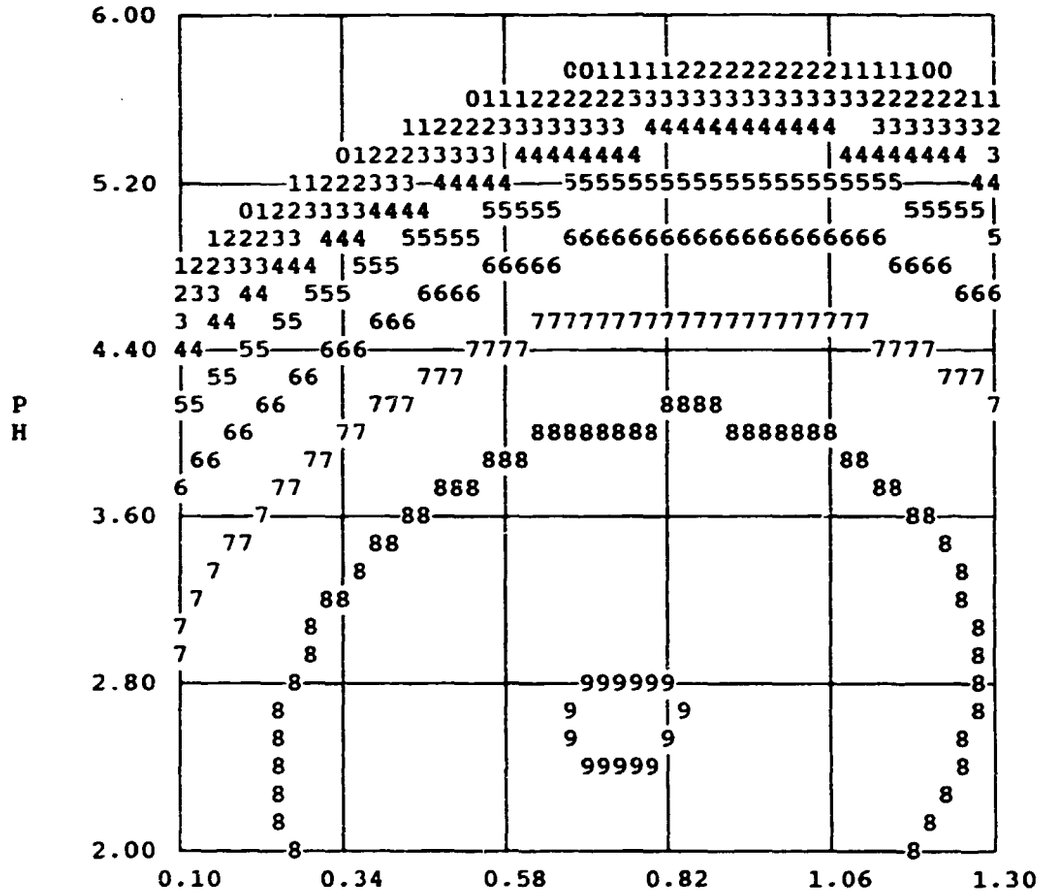
(i) Conduct test-runs utilising the plant
(ii) Work out the process parameters and optimise the process.
(iii) Train local staff in process technology and particularly in the up-scaling of laboratory-scale processes.
(iv) Obtain standardized products and determine the viability for commercial scale production.

The expert will be required to submit a Final Report embodying his final findings and describing the entire work done during the mission and recommendations.

APPENDIX II. RESPONSE SURFACE DIAGRAMS FOR ELECTROLYSIS OPTIMIZATION (a)

RESPONSE SURFACE PLOT

Experiment: steex — INT'L QUAL-TECH,Ltd. — Response: Decolor
Electrolysis — Transform: Power



Variable	Level	Variable	Level
Temp	45	Time	25

Contour Values			
0 =	1.310	5 =	56.310
1 =	12.310	6 =	67.310
2 =	23.310	7 =	78.310
3 =	34.310	8 =	89.310
4 =	45.310	9 =	100.310

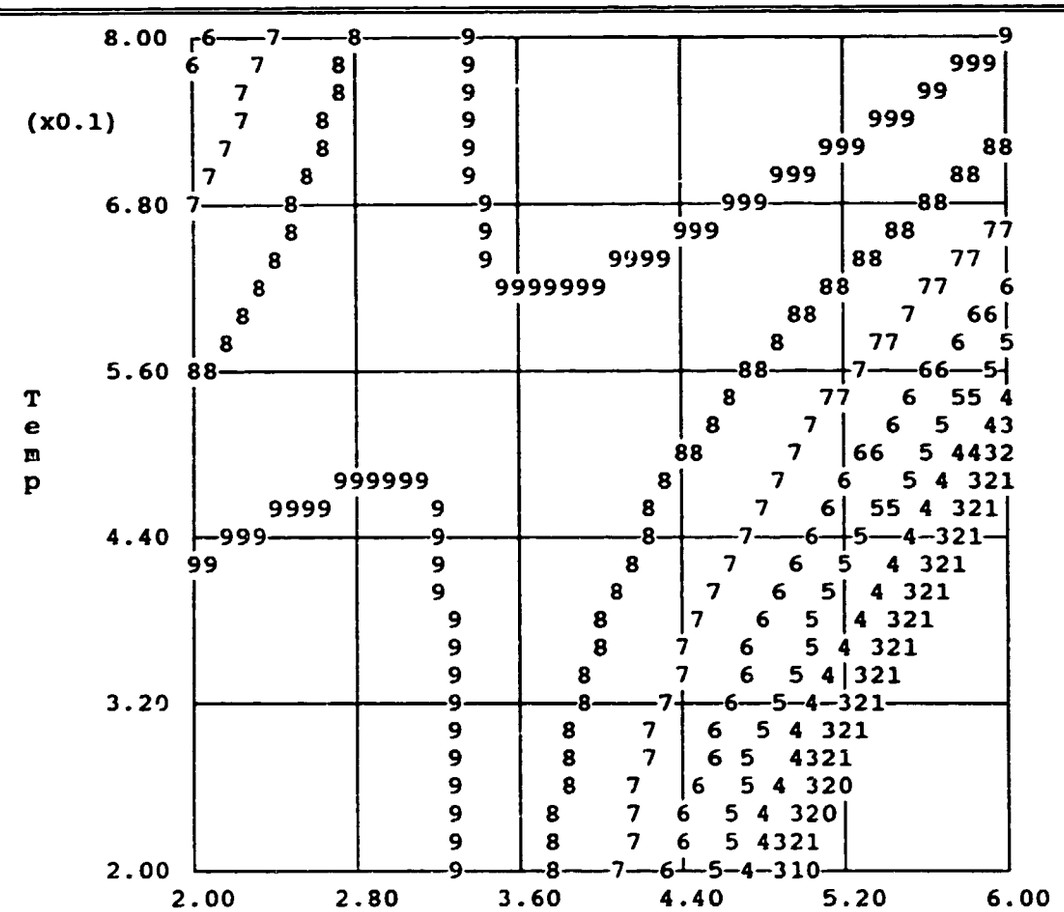
APPENDIX II (b)

RESPONSE SURFACE PLOT

Experiment: steex
Electrolysis

INT'L QUAL-TECH,Ltd.

Response: Decolor
Transform: Power



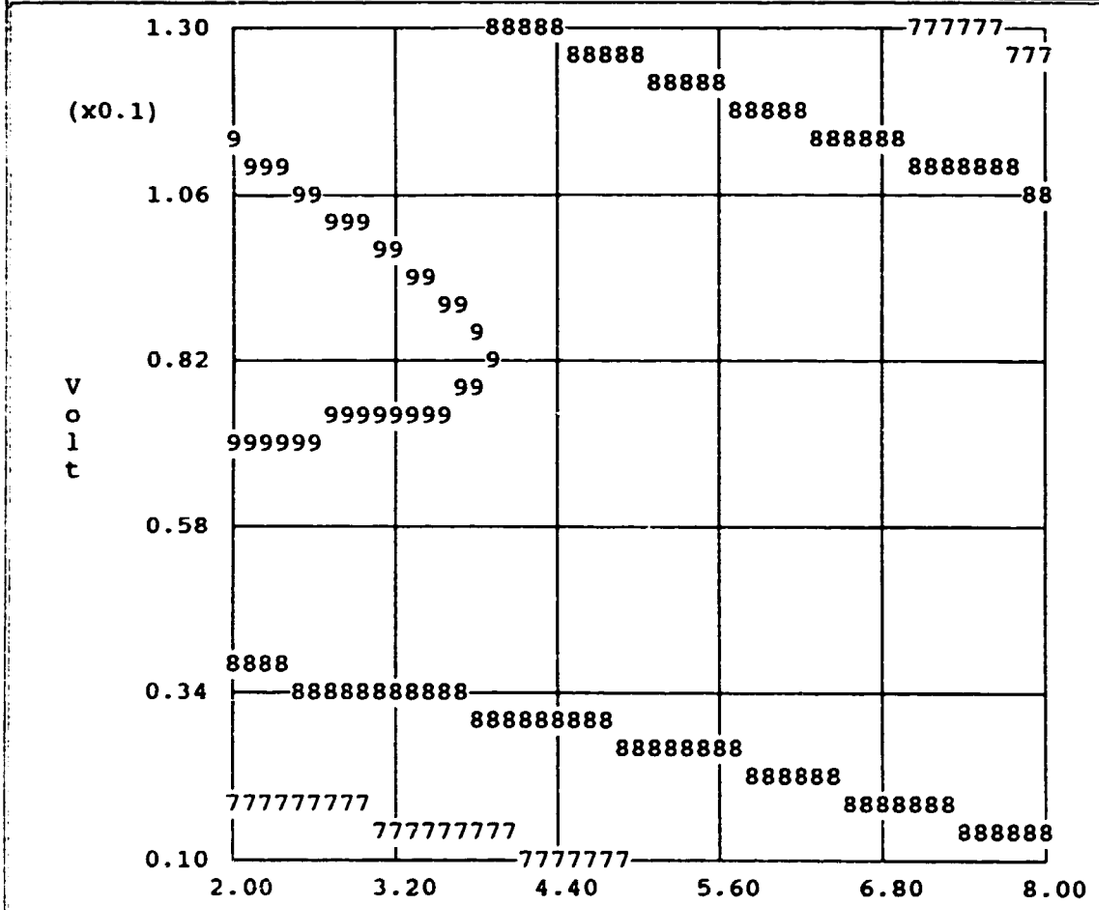
Variable	Level	Variable	Level
Volt	7	Time	40

Contour Values			
0 =	1.310	5 =	56.310
1 =	12.310	6 =	67.310
2 =	23.310	7 =	78.310
3 =	34.310	8 =	89.310
4 =	45.310	9 =	100.310

APPENDIX II (c)

RESPONSE SURFACE PLOT

Experiment: steex Electrolysis | INT'L QUAL-TECH,Ltd. | Response: Decolor Transform: Power



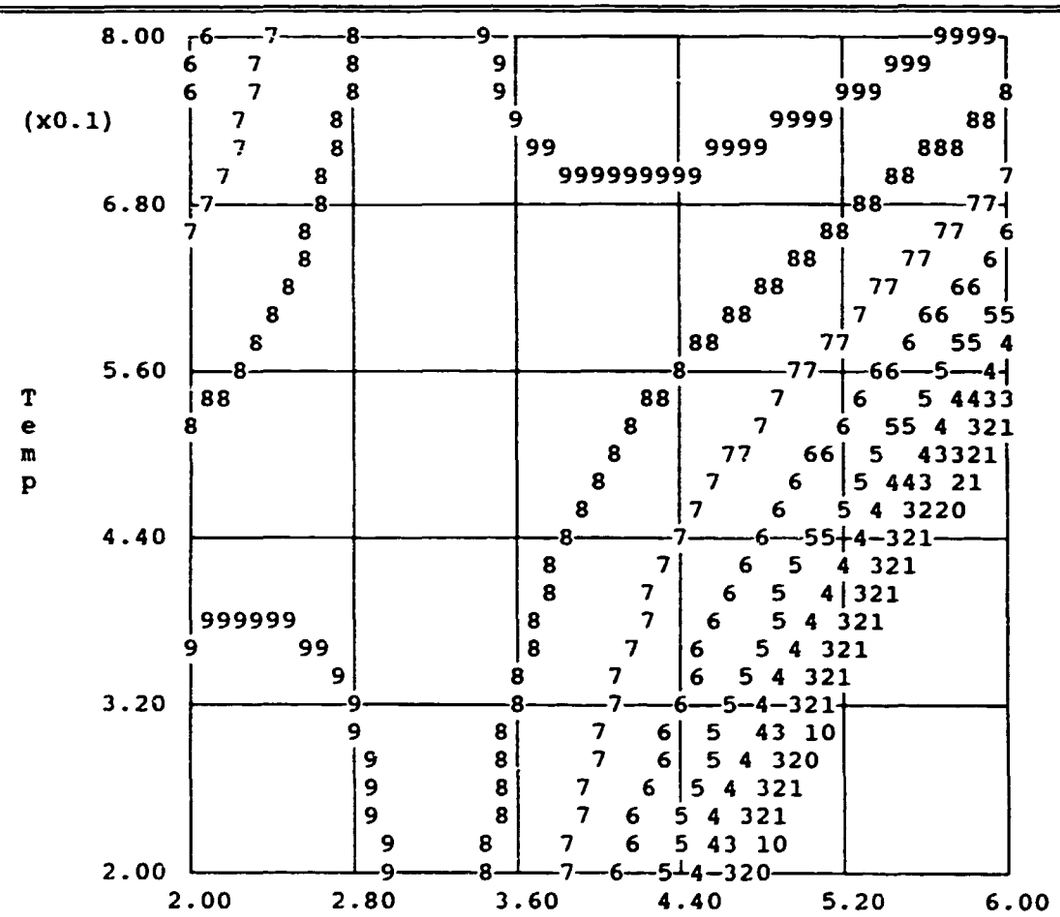
Variable	Level	Variable	Level
PH	3	Time	25

Contour Values			
0 =	1.310	5 =	56.310
1 =	12.310	6 =	67.310
2 =	23.310	7 =	78.310
3 =	34.310	8 =	89.310
4 =	45.310	9 =	100.310

APPENDIX II (d)

RESPONSE SURFACE PLOT

Experiment: steex — INT'L QUAL-TECH,Ltd. — Response: Decolor
Electrolysis — Transform: Power



Variable	Level	Variable	Level
Volt	10	Time	20

Contour Values			
0 =	1.310	5 =	56.310
1 =	12.310	6 =	67.310
2 =	23.310	7 =	78.310
3 =	34.310	8 =	89.310
4 =	45.310	9 =	100.310

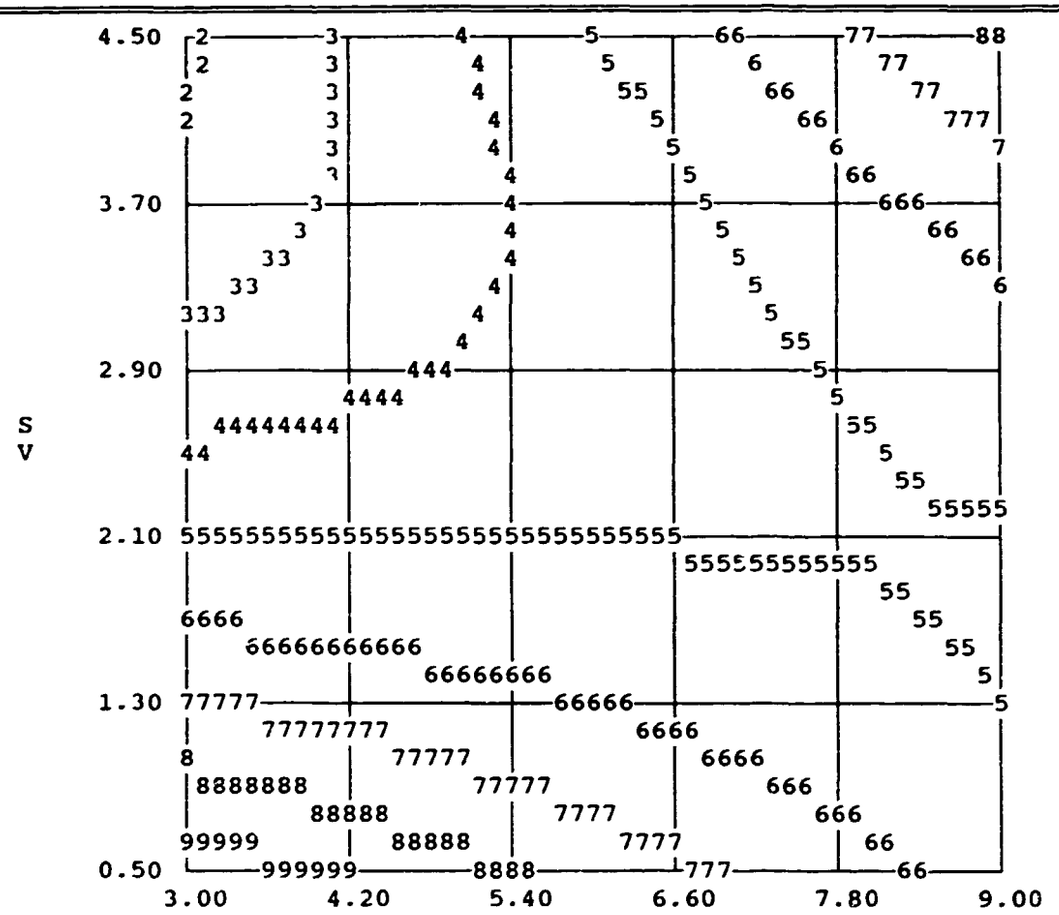
APPENDIX III PESPONSE SURFACE DIAGRAMS FOR CATION EXCHANGE OPTIMIZATION (a)

RESPONSE SURFACE PLOT

Experiment: SteA
Cation Optimization

INT'L QUAL-TECH,Ltd.

Response: DECOLOR
Transform: None



Variable	Level	Variable	Level
DM	.50		

Contour Values			
0 =	23.910	5 =	57.410
1 =	30.610	6 =	64.110
2 =	37.310	7 =	70.810
3 =	44.010	8 =	77.510
4 =	50.710	9 =	84.210

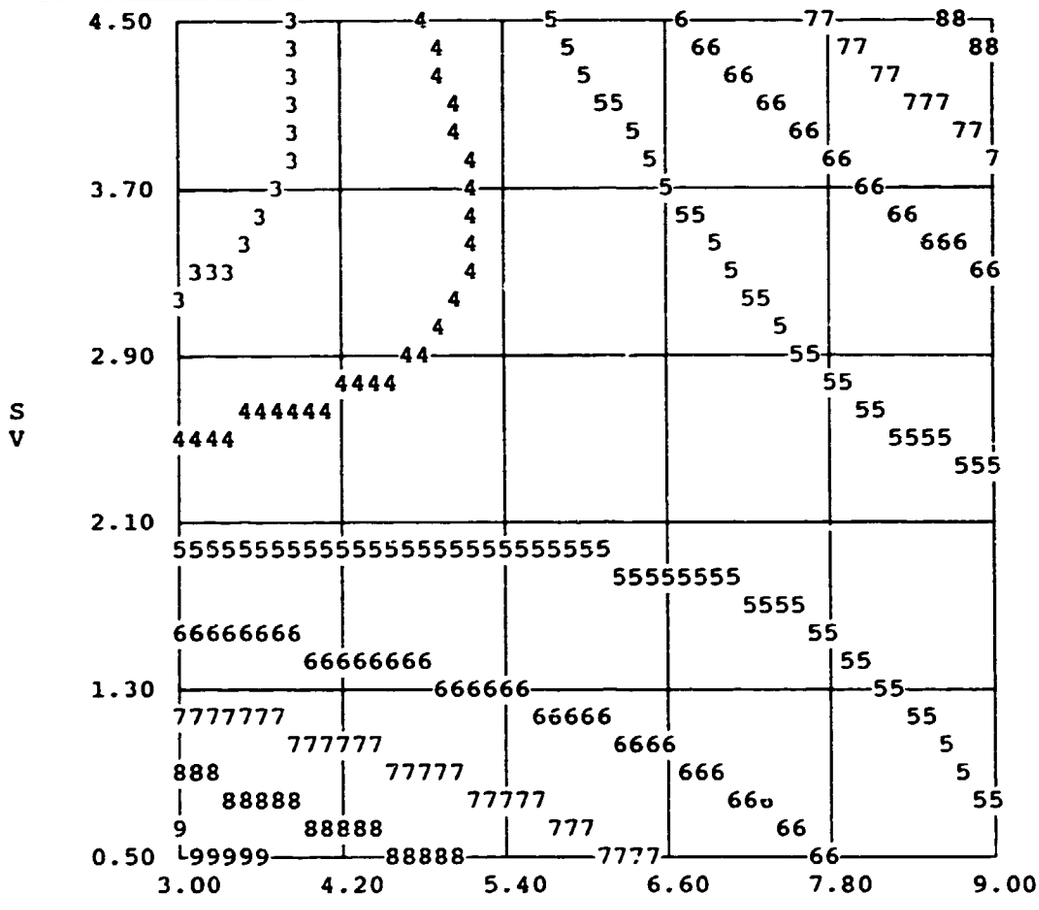
APPENDIX III (Cont, b)

RESPONSE SURFACE PLOT

Experiment: SteA
Cation Optimization

INT'L QUAL-TECH,Ltd.

Response: DECOLOR
Transform: None



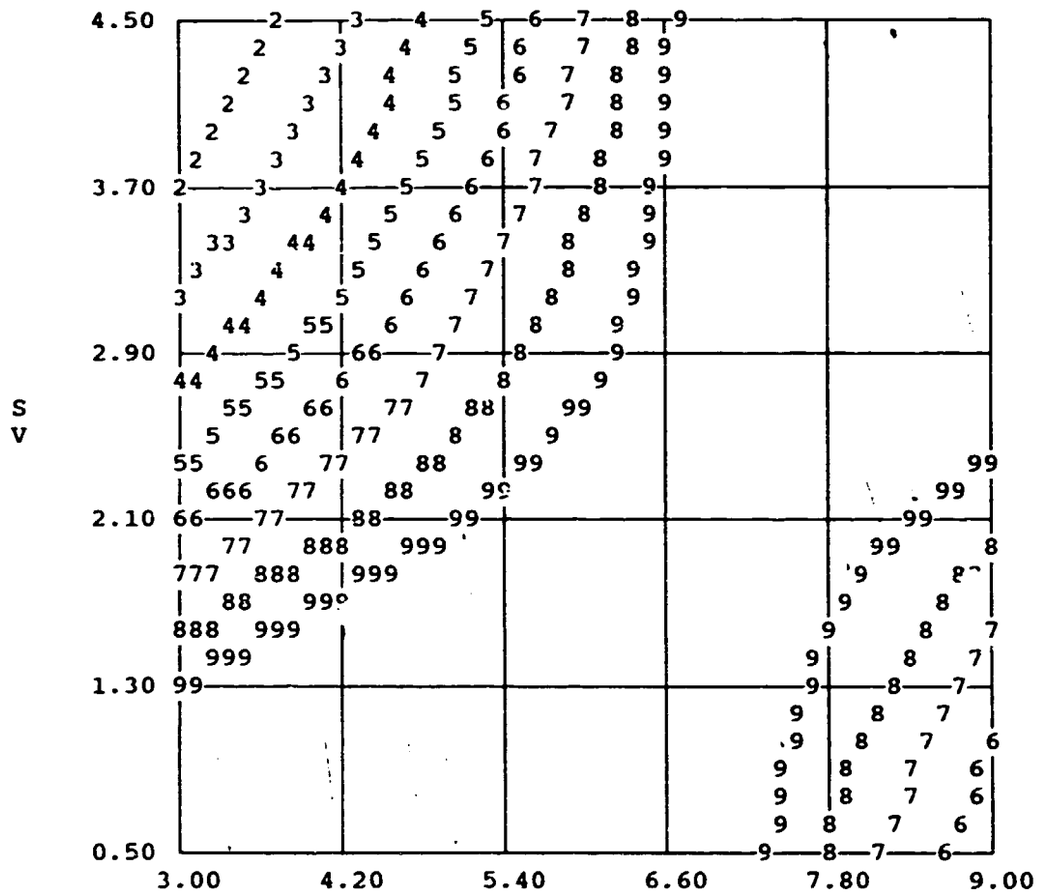
Variable	Level	Variable	Level
DM	1.5		

Contour Values			
0 =	23.910	5 =	57.410
1 =	30.610	6 =	64.110
2 =	37.310	7 =	70.810
3 =	44.010	8 =	77.510
4 =	50.710	9 =	84.210

APPENDIX IV. RESPONSE SURFACE DIAGRAMS FOR OVERALL ION EXCHANGE TREATMENT OPTIMIZATION (a)

RESPONSE SURFACE PLOT

Experiment: ANION — INT'L QUAL-TECH,Ltd. — Response: Decolor-T
Anion Optimization — Transform: Log 10



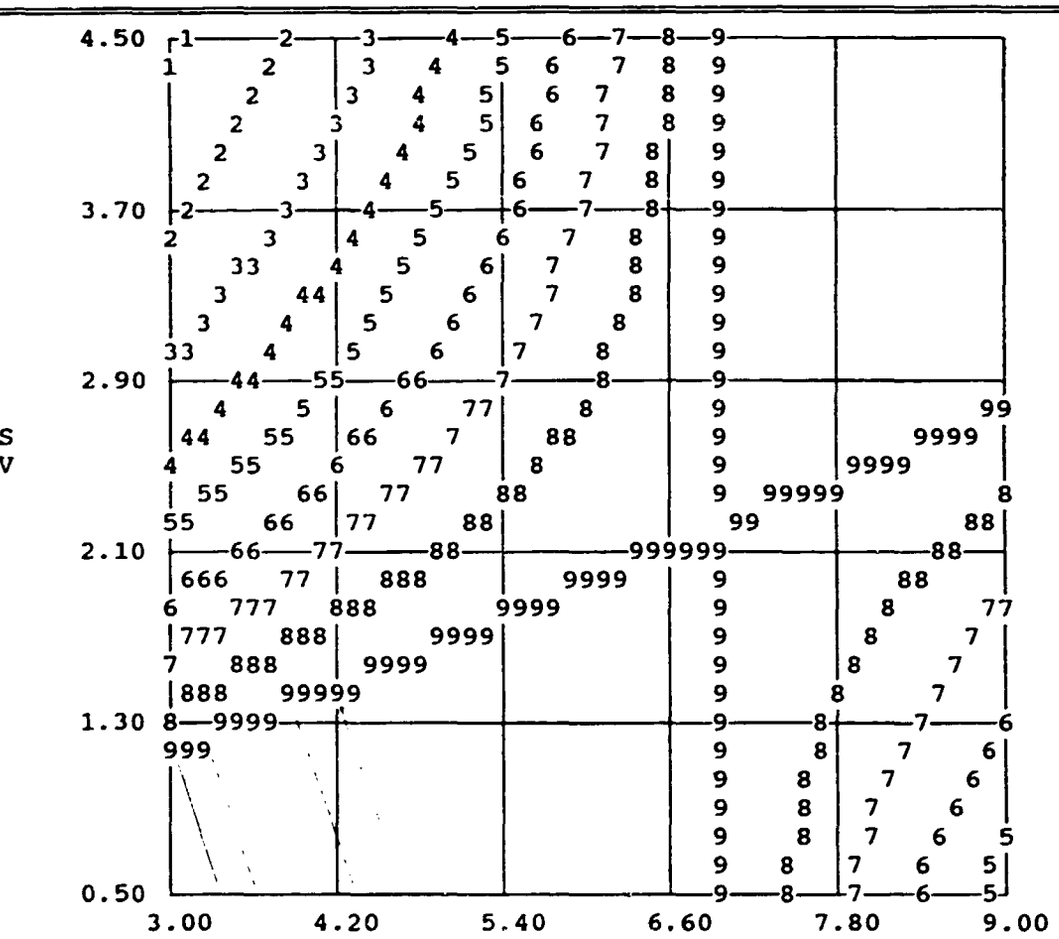
Variable	Level	Variable	Level
DM	.50		

Contour Values			
0 =	9.410	5 =	58.910
1 =	19.310	6 =	68.810
2 =	29.210	7 =	78.710
3 =	39.110	8 =	88.610
4 =	49.010	9 =	98.510

APPENDIX IV (Cont., (b))

RESPONSE SURFACE PLOT

Experiment: ANION — INT'L QUAL-TECH,Ltd. — Response: Decolor-T
Anion Optimization — Transform: Log 10



Variable	Level	Variable	Level
DM	.88		

Contour Values			
0 =	9.410	5 =	58.910
1 =	19.310	6 =	68.810
2 =	29.210	7 =	78.710
3 =	39.110	8 =	88.610
4 =	49.010	9 =	98.510

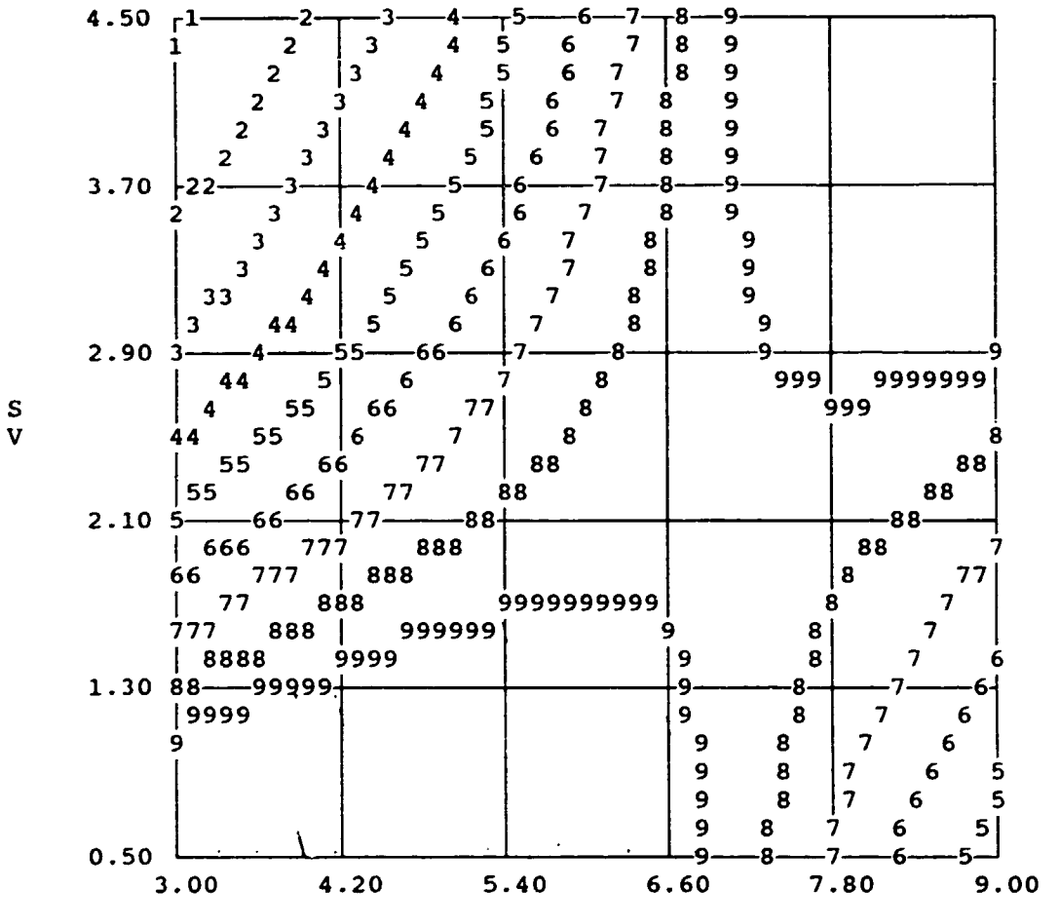
APPENDIX IV (Cont., (d))

RESPONSE SURFACE PLOT

Experiment: ANION
Anion Optimization

INT'L QUAL-TECH,Ltd.

Response: Decolor-T
Transform: Log 10



Variable	Level	Variable	Level
DM	1.0		

Contour Values			
0 =	9.410	5 =	58.910
1 =	19.310	6 =	68.810
2 =	29.210	7 =	78.710
3 =	39.110	8 =	88.610
4 =	49.010	9 =	98.510

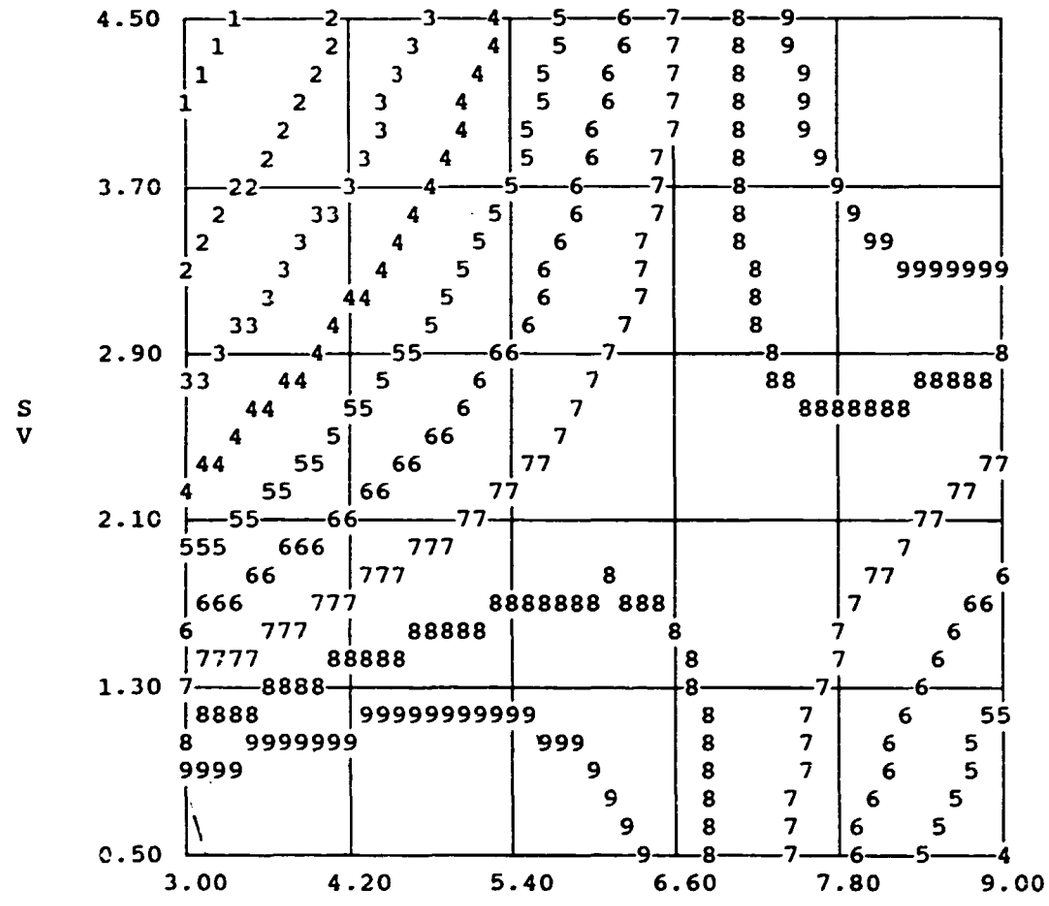
APPENDIX IV (Cont., (e))

RESPONSE SURFACE PLOT

Experiment: ANION
Anion Optimization

INT'L QUAL-TECH,Ltd.

Response: Decolor-T
Transform: Log 10



Variable	Level	Variable	Level
DM	1.5		

Contour Values			
0 =	9.410	5 =	58.910
1 =	19.310	6 =	68.810
2 =	29.210	7 =	78.710
3 =	39.110	8 =	88.610
4 =	49.010	9 =	98.510

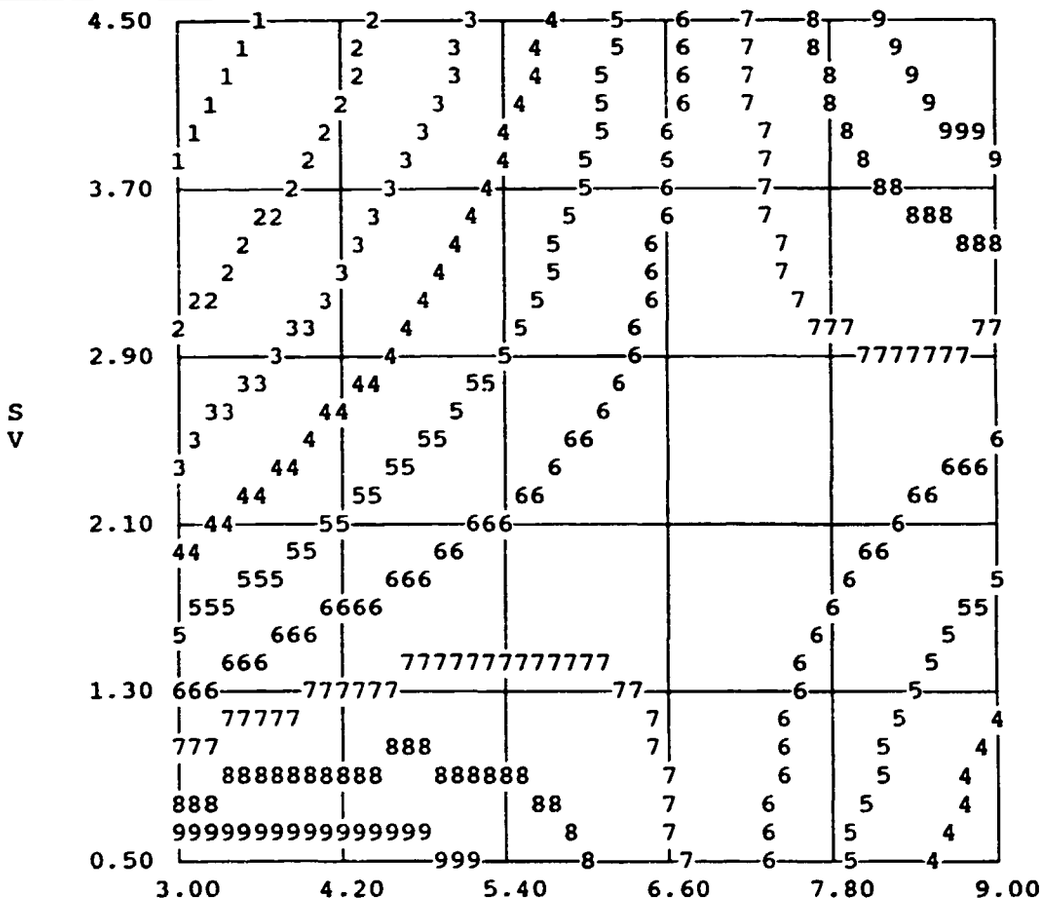
APPENDIX IV (Cont., (f))

RESPONSE SURFACE PLOT

Experiment: ANION
Anion Optimization

INT'L QUAL-TECH,Ltd.

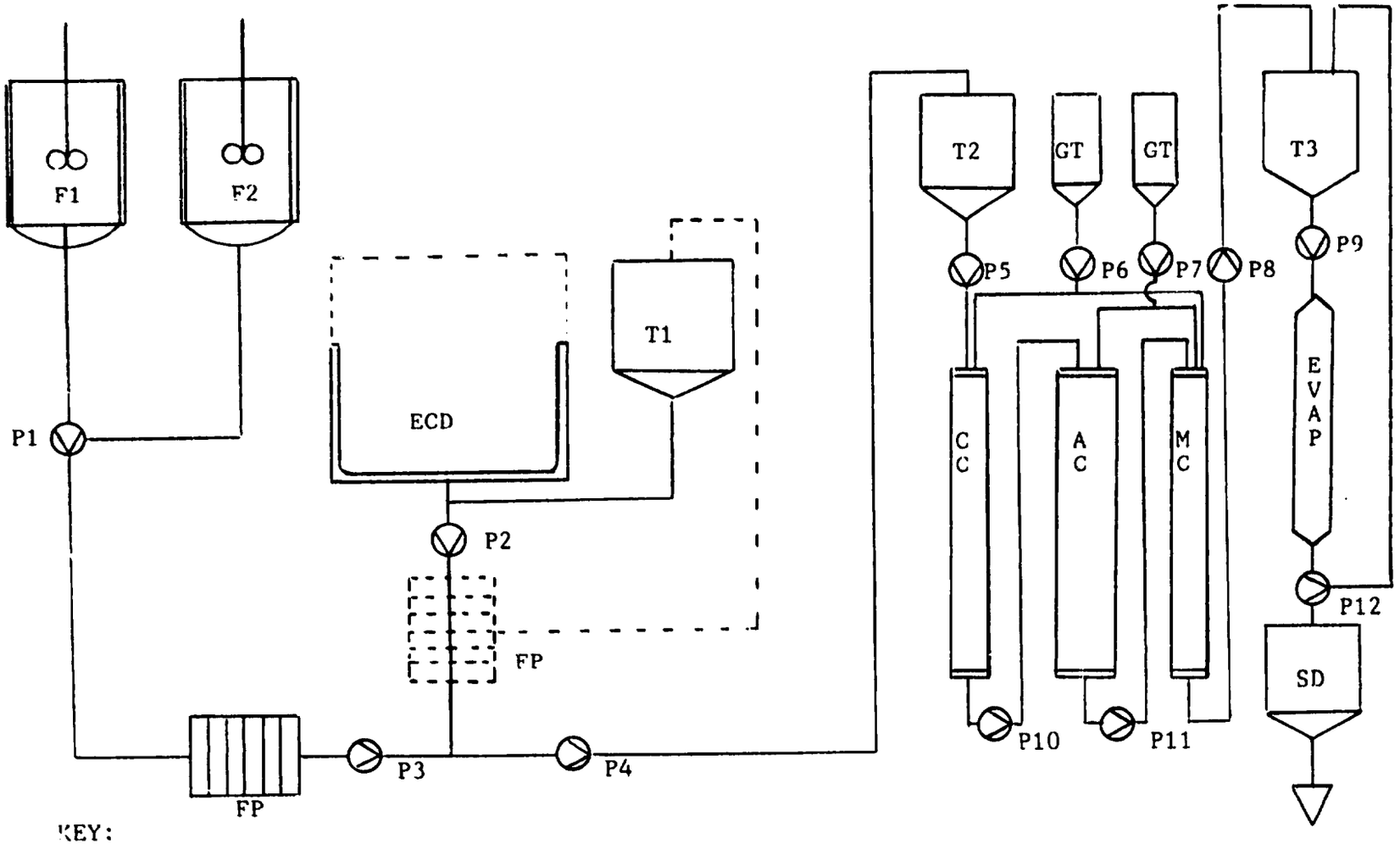
Response: Decolor-T
Transform: Log 10



Variable	Level	Variable	Level
DM	2.5		

Contour Values			
0 =	9.410	5 =	58.910
1 =	19.310	6 =	68.810
2 =	29.210	7 =	78.710
3 =	39.110	8 =	88.610
4 =	49.010	9 =	98.510

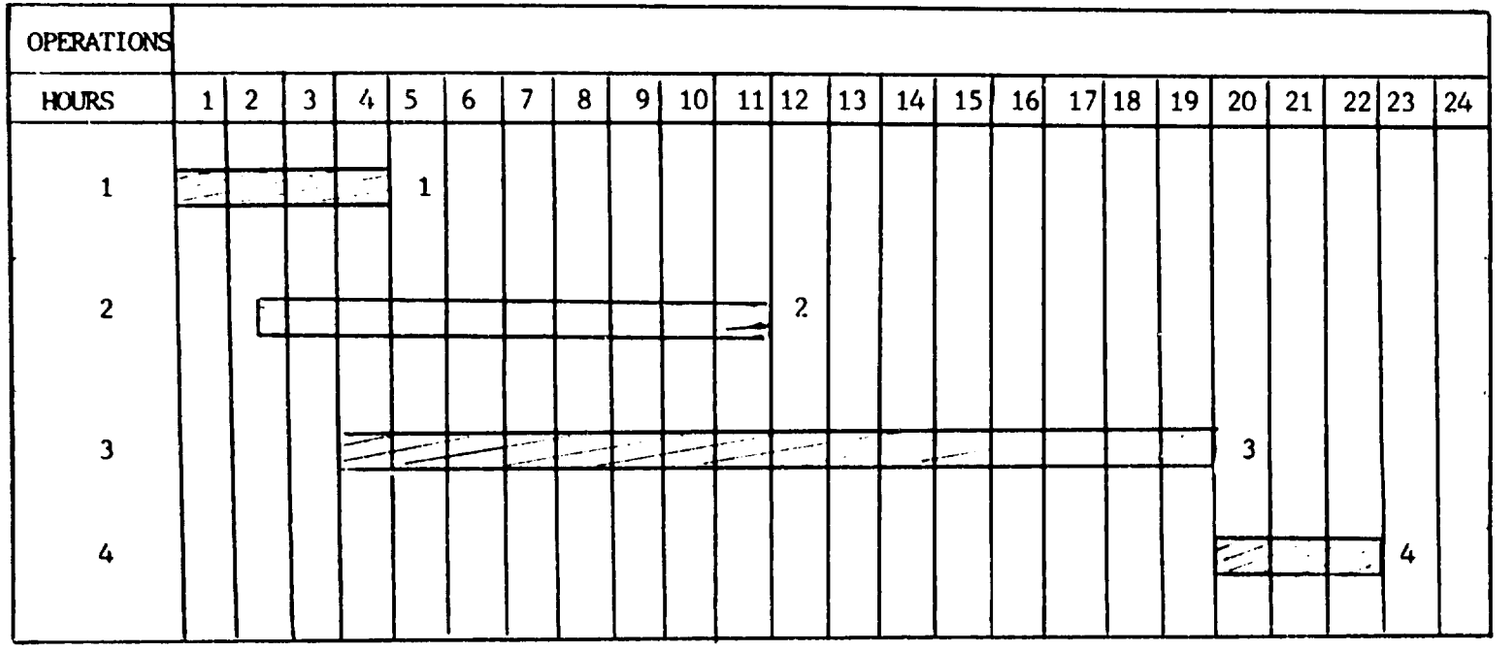
APPENDIX V. A CONCEPTUAL PROCESS FLOW FOR THE STEVIA PROCESS



KEY:

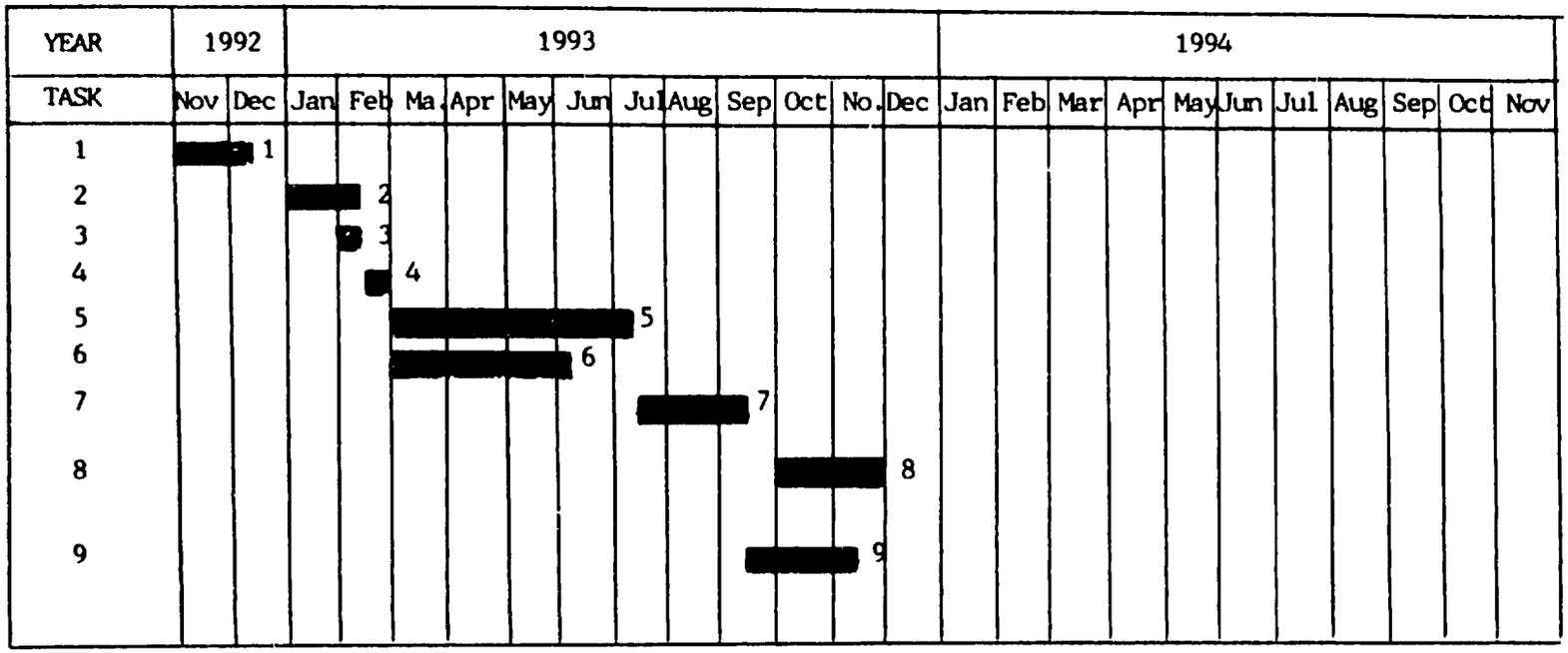
ECD : Electolyser, P: Pumps, T: Tanks, GT: Regeneration Tanks
CC : Cation Column AC: Anion Column MC: Mized Column
EVAP: Evaporator, SD: Spray Dryer

APPENDIX VI. SCHEDULE PLAN FOR DAILY OPERATION OF THE STEVIA PROCESSING PILOT PLANT



1. Electrochemical Treatment
2. Ion Exchange Treatment
3. Evaporation of Solution
4. Spray Drying

APPENDIX VII. A REVISED PROJECT SCHEDULE FOR THE STEVIA PROCESSING PROJECT



KEY TO TASKS

1. Selection of consultant and contracting next task (UNIDO).
2. Layout, installation drawings, find specs of materials and equipment off-site (UNIDO).
3. Laison of task 2 with current consultant (UNIDO).
4. Rectification of designs by FRI(UNIDO|FRI).
5. Order|deliver equipment, components (UNIDO).
6. Pilot plant upgrade and refinishing (FRI).
7. Installation, supervision, commissioning (UNIDO).
8. Pilot scale testing
9. Quality assurance and control, transglucosylation process testing and optimization (UNIDO).

Backstopping Officer's Technical Comments
based on the work of Mr. Y.J. Owusu-Ansah
DP/DRK/88/008/11-02

Mr. Owusu-Ansah has given a detailed account of his activities and made recommendations for follow-up action. He has also made recommendations about the work plan and the rephasing of the budget. The method recommended for pilot scale production of stevio and a conceptual flow diagram for designing the pilot plant equipment are given in the report. The expert has recommended purchase of second hand equipment which is not realistic as no guarantees could be obtained.

Furthermore, the cost estimate given by the expert using the reconditioned equipment is much in excess of the funds available. Hence, this aspect has to be discussed with the Government and the UNDP before any follow-up action.

There is a change in the physical form of the end product, as what is recommended now is powder instead of crystals envisaged in the project document. A cost analysis in terms of energy requirements has to be done as the process recommended has to use a higher water to leaves ratio than that used at present. Furthermore, quality specifications will have to be developed for the powder, taking into consideration residual metallic ions, pesticides and other contaminants.

The optimization of processes has to be further modified during process development using the pilot plant. The BSO needs to discuss in detail the recommendations of the expert with the counterpart staff and UNDP in order to take firm decisions on follow-up action.