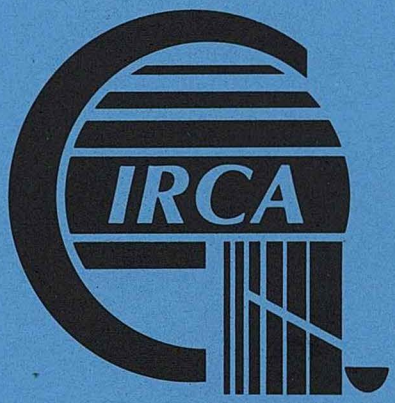
PROJECT DP VIE 86/036/32-01

CHLORINATED RUBBER

REPORT OF MISSION IN VIETNAM

H. de Livonnière - December 1988



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ABSTRACT

Project DP/VIE/86/036
Chlorinated rubber

The project involves construction of a pilot plant for natural rubber chlorination, capacity 50 kg per operation, so as to transfer know-how and produce chlorinated rubber, which is an essential ingredient in corrosive-proof paints. An assessment report was drawn up after a 5-day visit to Vietnam at the Institute of Chemical Industry in Hanoï, which will accommodate the project, and after a visit by the Vietnamese experts in charge of the project to the Carl Scholemmer University in Leuna Merseburg, German Democratic Republic, the IRCA/CIRAD, an organization specialized in the research and development of natural rubber in France and Côte d'Ivoire and French industries specialized in chemical engineering and polymer chlorination. For each country visited, the report presents the visit schedule, a description and evaluation of the organizations proposed to participate in the project. The report also proposes a project document complete with annexes.

RECOMMENDATIONS

Subsequent to the mission in Vietnam and the study tours undertaken by Vietnamese experts from the Institute of Chemical Industry in GDR, France and Côte d'Ivoire, the recommendations and proposals put forward to UNDP and the Government of Vietnam are as follows :

- Construction of a pilot plant for natural rubber chlorination is necessary to enable the Institute of Chemical Industry to meet the demands of local paint industries and to acquire technical know-how in the field of rubber and polymer chemical modification.

- The size of the pilot plant should correspond to a production capacity of 50 kg per operation.

- The pilot plant should be delivered turnkey, with prior testing before shipment to Vietnam.

- The project should provide training in Europe for the team in charge of assembling and running the pilot plant in Vietnam.

- Additional analysis and laboratory equipment should be funded by the project to guarantee raw and finished product quality and enable adaptive experimentation during and after the project.

- The wide experience acquired by IRCA/CIRAD and the De Dietrich company in the fields of natural rubber, chemical modification of rubber and chemical engineering constitutes a technical guarantee as to the design, assembly and start-up of the pilot plant.

- A mission by the expert charged with writing this report will be necessary to draw up the final Project Document with the Vietnamese experts.

GENERAL SCOPE

General information on natural rubber chlorination

Chlorination of natural rubber is a well known technique and has been used for almost 50 years. Recent documentary literature on this technique is not readily available, as improvements in the process are confidential, known only to the 5 or 6 producers who currently share the world market of this product, which is essentially used in sea-worthy paints. The production phases are as follows :

- Transformation of rubber to solution form, tests and adjustment of its molecular weight (the choice of initial molecular weight depends on final grade desired).

- Chlorination of the solution with gaseous chlorine (the chlorine rate has to be at least 65 % to obtain a stable product).

- De-gasification of the solution to eliminate residual chlorine.

- Precipitation of the chlorinated rubber in distillation flasks with steam or hot water (elimination of residual hydrochloric acid).

- Drying of the chlorinated rubber (elimination of carbon tetrachloride), and, simultaneously :

    - Treatment of liquid or gaseous effluents
    - Recycling of the solvent.

The sensitive aspects of each phase have been placed in parentheses and require either adjustment of the molecular weight or the final chlorine rate and elimination of the residual carbon tetrachloride, or require great care due to the presence of toxic products (CL_2, HCl, or highly corrosive HCl). The design, construction, starting up and running of a pilot natural rubber chlorination plant requires a maximum of guarantees to provide good quality corrosion resistant equipment, adequate design to avoid the risk of leakage or dangerous operations and treatment of liquid or gaseous effluents.

Project context

The request for funding corresponding to project DP/VIE/86/036 "chlorinated rubber" was made by the Institute of Industrial Chemistry. This organization provides technical assistance to the Vietnamese chemical industry : analyses, development of processes, training. After preliminary research and development, this Institute is therefore in a very good position to ensure the transfer of new technology to this country. Chlorine and natural rubber are locally produced raw materials which would be considerably valorized through the production of chlorinated rubber, which is an essential ingredient in the manufacture of sea-resistant paints. A pilot plant would enable researchers, engineers and technicians at the Institute to become familiar with the rubber chlorination technique and to have enough chlorinated rubber at their disposal to develop corrosion-proof paint formulas adapted to the local environment. The pilot plant should be designed to meet the technical and security requirements for this type of reaction and, given the aims of the Institute, respond to the need for training in chemical engineering. Hence, it should not be too specific. The infrastructures exist locally (buildings, utilities), but the supply of spare parts is currently a problem. It is therefore most desirable that the pilot be delivered turnkey, after being assembled, tested and partially dismantled for shipment by the designer, in the presence of the Vietnamese team who will be responsible for it on site.

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1. INTRODUCTION

Under an agreement signed with the United Nations Development Program (UNDP), the Vietnamese Government made a request for funding to construct a pilot plant designed to produce chlorinated rubber (Project DP/VIE/86/036 "chlorinated rubber"). In 1986, the Institute of Chemical Industry, affiliated with the General Department of Chemistry, thus proposed a project to UNDP. A a preliminary expertise mission was requested, so as to assess this project.

The aims of the mission carried out in connection with contract DP/VIE/86/036/11-01/J313420 are given in the "Job Description" in Annex I. For administrative reasons, the inital plan envisaged was modified as follows :

1) Mission to Vietnam.
2) and 3) Study tour in GDR, France and Côte d'Ivoire and drawing up of the project document with Vietnamese experts.

The mission to Vietnam took place from 27th May to 2nd June 1988. Its aims were twofold : to contact Vietnamese experts directly in charge of the project, as well as their Superiors and the UNDP representatives and to visit the installations and infrastructures designed to house the future pilot plant, so as to acquire a better idea of the means required to implement the project.

Subsequent to this mission, a study tour was organized which enabled Vietnamese experts to see the rubber chlorination work carried out in GDR and France. A mission to Côte d'Ivoire completed this study tour, so as to acquaint the Vietnamese experts with the work undertaken in connection with UNIDO contracts on liquid rubber.

2. MISSION TO VIETNAM

2.1 Mission schedule

Friday, 27th May

Arrival in Vietnam. Welcomed at the airport by the Vietnamese experts involved in the project : Mr. Le Van Nguyen, Director, Mr. Nguyen Tien Hüng, Chemical Engineer, Mrs. Ngo Thi Lien, Engineer.

Saturday, 28th May

Visit to the Back Jamg Shipping factory.

Sunday, 29th May

Discussion of Project documents and study tour program.

Monday, 30th May

Visit to the Institute of Chemical Industry : project site and laboratory.

Tuesday, 31st May

Visit to the Viet Tri soda and chlorine factory.

Wednesday, 1st June

Visit to UNIDO. Discussion of Project documents.

Thursday, 2nd June

Departure for Ho Chi Minh City.

2.2 Progress made in the project up to June 1988

2.2.1 Situating the problem

The problem is clearly defined on page 2 of the "Job Description" in Annex I; the broad outlines are summarized below :

- Chlorinated rubber accounts for 20 % of the weight of corrosion-proof paint for the navy, as it is very resistant to sea fog.

- Current demands are estimated at 30 to 50 thousand tons/year for this type of paint.

- National production of chlorine, which is a by-product of soda manufacture, is currently estimated at 17,000 tons and is likely to double by 1995. National production of natural rubber was 48,000 tons/year in 1988, with forecasts of 237,000 tons in the year 2000. Chlorinated rubber, for which demand on the international market reaches between 60 and 100 thousand tons/year, is currently produced using synthetic rubber (Polyisoprene), but could be produced from natural rubber, as it was in the past.

- The Institute of Chemical Industry has been working on rubber chlorination and corrosion-proof paint formulation since 1962. Backed by the General Department of Chemistry, it would like to construct a pilot plant large enough to produce 100 to 200 kg of chlorinated rubber per operation and has made an official request to UNDP for the corresponding funds.

- This pilot plant would enable Vietnamese chemical engineers to become familiar with the process and make it possible to determine certain production parameters according to local conditions and requirements : increase in chlorine content, solvent recuperation, processing of gaseous effluents.

2.2.2 Basic data

A project proposal was submitted to UNDP (see Annex II) for a total of US $ 1,000,000 with the following breakdown :

♦ Detachment of experts to Vietnam :	US $ 250,000
♦ Overseas training for Vietnamese engineers and technicians (study tour)	US $ 260,000
♦ Pilot plant equipment	US $ 420,000
♦ Laboratory equipment	US $ 70,000

The project is scheduled to last 3 years.

2.3 Local infrastructures and personnel

2.3.1 General description

The Institute of Industrial Chemistry has two establishments, one in Hanoi where the Head Office and certain Laboratories are located, including the analysis laboratory, and another about 15 km from Hanoi with laboratories and buildings, where pilot plants have been constructed, mainly for mineral chemistry operations (permanganate and polyphosphates). Annex III lists the various sectors and activities of this Institute with a summary diagram. Four hundred and fifty people are employed there, with the following breakdown :

- 3 Professors
- 2 Ph.D Doctors
- 40 Doctors in Science
- 200 Engineers
- 150 Workers and technicians

2.3.2 Resources envisaged for the project

In all, 44 people habe been allocated to the project (see Annex II), including 1 Project leader, 18 engineers, 7 technicians, 15 laborers and 4 odd-job men. The following personnel were introduced to us during the mission to Vietnam :

- Mr. Le Van Nguyen, Project Leader, a chemical engineer educated in China;
- Mrs. Ngo Thi Lien, Assistant Project Leader, Ph.D. in Science from the University of Dresden (GDR), specialized in polymers used in paints;
- Mr. Nguyen Tien Hung, Chemical Engineer from the University of Magdeburg (GDR), Head of construction for the future pilot plant;
- 2 Ph.D Doctors in Science from the University of Merseburg (East Germany) specialized in polymers;
- 2 engineers specialized in rubber and educated in Vietnam;
- 2 engineers specialized in rubber modifications and educated in the USSR;
- 1 specialized technician in charge of rubber chlorination;
- The Head of the latex laboratory educated in Vietnam.

The laboratories in Hanoi have a team in charge of analyses and use 20 to 30 year-old but operational equipment, including :

- 1 diffraction and X-fluorescence apparatus (GDR, 1960);
- 1 SPECORD V 15 UV spectrophotometer (GDR, 1970);
- 1 SPECORD 75 II IR spectrophotometer (GDR, 1970);
- 1 photocolorimeter (origin and age unkown);
- 1 AAS1N atomic dissolution spectrophotometer (GDR, 1970);
- 1 differential thermal analysis apparatus (Hungary, 1970);

- 1 Universal polarograph (URSS, age unknown);
- Common laboratory glassware
- Ovens, scales, etc.

The Center located 15 km from Hanoi includes :

- 2 floors of well lighted and well designed laboratories, one of which, the "latex laboratory", is reserved for the manufacture of small articles of latex (capsule seals), while the other is reserved for carrying out or implementing chemical modifications to rubber by epoxidization, chlorination and maleic anhydride treatment. What little equipment there is is very old and is limited to scales, oven, laboratory glassware and a few makeshift installations made on site.
- 1 building where the pilot plants already mentioned are located;
- 1 new building (1,000 m^2 x 10 m h) divided into two sections intended to house new pilot plants, for chlorination in particular. This building has yet to be equipped with water, steam, electricity and compressed air; it is located near a railroad linked to a national network.
- 1 maintenance and repair workshop with lathes, milling machines and a bending machine, all of them rather decrepit.
- The pilot chlorination plant requires more equipment, since the main reactor was destroyed in 1984 after a faulty manoeuver; the other elements in this plant were re-used elsewhere.

2.4 State of knowledge in the field of chlorination at the Institute of Chemical Industry in Hanoi

The documents passed on to us by the Vietnamese experts during our mission are found in Annex IV :

- Summary of the trials already carried out;
- Description of the pilot plant operating from 1980 to 1984.
- Details of the "solvent recovery system".

These documents make it possible to assess the difficulties encountered by the Institute in their attempts to carry out rubber chlorination successfully :

- Corrosion of the equipment
- Insufficient treatment of the gaseous or liquid effluents;
- Insufficient degasification of the chlorinated rubber solution;
- Insufficient hydrochloric acid removal from the finished product;
- High carbon tetrachloride content in the finished product;

Most of these difficulties result from the use of poorly adapted equipment which had never been treated for corrosion. The critical problems were those concerning the transport of liquids or gases due to the lack of efficient pumps.

The engineers in charge of the project, whom we met during the mission are qualified in their respective fields : chemistry, chemistry of paints and chemical engineering, and are very experienced and knowledgeable in the areas they work in. As regards the other personnel, senior staff or technicians, it was difficult to judge their qualifications given the linguistic problems encountered. Nonetheless, should this project go ahead, it is necessary to envisage solid training for the personnel involved with accepting and running the future pilot plant.

3. STUDY TOUR IN GDR

3.1 Mission schedule

Monday, 5th September 1988

Arrival in Leipzig; welcomed by Dr. Udo Schumann, from the University of Merseburg, and Mr. Wolfgang Rietzschel, GDR diplomat (International Projects Office) in Dresden. Customs formalities, search for lodgings, visit to the Leipzig fair, mission preparation.

Tuesday, 6th September 1988

Morning — Visit to the chemical engineering and chemistry departments at the Carl Schorlemmer University in Leuna Merseburg. Welcome and introductions made by Dr. S. Weiss, Dr. J. Schumman, Dr. Timpe, Dr. Heine and several of their assistants.

Afternoon — Discussion of the chlorinated rubber project proposal presented by Dr. Günter Weickert. The Vietnamese delegation, led by Mr. Le Van Nguyen, was present.

Evening — Kind invitation to a cocktail given by Dr. Hans Joachim Böhme, Minister of Education, in connection with the Leipzig fair.

Wednesday, 7th September 1988

Morning — Visit to the paint factory VEB FARBEN UND LACKFABRIK in Leipzig. Received by Mr. Ralph Schmidt, Director, and accompanied by Dr. Heinz and the Vietnamese delegation.

Afternoon — Visit to the Leipzig fair and discussion at the BUHAWERKE stand with Dr. Detlef Krüger from VEB AMMENDORFER PLASTWERK, which produced chlorinated rubber under the name "CHLOROPHANE". Accompanied by Dr. U. Schumann, Dr. G. Weickert, Dr. Heinz and the Vietnamese delegation.

Thursday, 8th September 1988

Morning — Meeting for the project with the Vietnamese delegation and the principal people in charge of the chemical engineering sector at the University.

Afternoon — Departure for Paris with the Vietnamese delegation.

3.2 Introduction

Subsequent to a request made by the Vietnamese Government to UNIDO, a study tour was organized from 22nd August to 8th September 1988 at the Carl Schorlemmer University in Leuna Merseburg for the Vietnamese delegation in charge of preparing project DP/VIE/86/036/32-01 "Chlorinated Rubber". The UNIDO expert joined the delegation on 5th September. The aims of this study tour were to :

- visit the university, a paint factory and a chlorinated rubber manufacturer;
- present and discuss the proposals put foward by the chemical engineering sector to construct a pilot rubber chlorination plant.

3.3 The Carl Schorlemmer University in Leuna Merseburg

Created in 1954 with 12 different departments, the Carl Scholemmer University is a 45-minute drive from Liepzig, in the heart of the heavy industry zone in southern GDR. There are 3,500 students enrolled; the five-year program leads to a diploma equivalent to the Master of Science. After 3 or 4 years of further research, certain students obtain the equivalent of a Ph.D. Under the terms of this project, the departments of chemistry and chemical engineering have proposed to :

- provide training for engineers and technicians
- design the pilot chlorination plant in the facilities available, e.g. 4 equipped workshops approximately 20 m x 40 m x 15 m.
- offer chemical engineering courses in thermodynamics, miscellaneous separations, evaporation, drying, catalysis, synthesis, etc.
- carry out research in these same fields.
- Application through collaboration with the polymer industry, among others.

Within this framework, and working with the chlorinated rubber manufacturer VEB AMMENDORFER, a pilot polymerization chlorination plant is to be constructed, due to be operational in September 1989 with a production capacity of 40 kg per operation.

The Carl Schorlemmer University will thus have the opportunity to acquire know-how in the field of chlorination.

It is worthwhile noting that there is a polymer physics and rheology study laboratory with modern equipment such as :

- MONSANTO and GOETFERT Rheometers, ZWICH capillary rheometer
- GABO QUALIMETER TESTANLAGEN rheovibron (FRG)

This laboratory also has experienced data processing personnal at all levels.

3.4 Chlorinated rubber

Subsequent to visits to VEB AMMENDORFER and VEBFARBEN UND LACKFABRIK, the technical and economic parameters governing chlorinated rubber were determined as follows :

- Stable Market estimated at 45 to 50 thousand tons/year, possibly with a slight rise in the years to come due to increased industrialization in developing Asian countries.

- Production capacity of 60,000 t shared among 6 producers, the leader being the Institute of Chemical Industry.

- For the past 10-15 years, due to economic reasons (price fluctuations, etc.), natural rubber has been gradually replaced by synthetic polyisoprene.

- Competition from chlorinated polyethylene and chlorinated PVC, nonetheless judged less effective in anti-rust paints.

- Applications in the corrosion-proof paint industry for grades with the lowest molecular weight (15 to 25 % dry weight in paint formulas) and in the adhesive industry for grades with a higher molecular weight.

3.5 Project proposals put forward by the Carl Scholemmer University

Aim Construction of a pilot plant with a capacity of 40-50 kg based on the technique used at PLASTICWERK AMMENDORFER. Estimated production at full capacity : 40 tons/year with 4 operations/day, 5 hours/operation = 5,000 hours/year. Initially, the plant will not function continuously, but will eventually do so through additional investments.

Description of the "discontinuous" pilot plant

Three phases :

1) Solution phase : Parallel depolymerization in 3 250-l tanks equipped with stirrers.

2) Chlorination of the reactional mass : rubber solution in a glass bubble column (diameter 300 mm, height 3 m) with a hot zone for chlorination in the strict sense (introduction of chlorine at the base of the column, steam and electric heating), and a cold zone for tetrachlorine condensation and the evacuation of hydrochloric acid and chlorine which did not react.

3) Precipitation of chlorinated rubber with steam; the texture of the finished product depends on the stirring system; the finished product thus obtained is filtered and dried.

Treatment of gaseous effluents, HCl and Cl2 is envisaged as well as the recuperation and storage of CCl4.

Continuous process

It should be possible to transform the discontinuous unit described above into a continuous unit by adding a degasification column, increasing the capacity for rubber solution preparation and by adding a few buffer tanks.

The complete list of equipment proposed as well as plans for the pilot plant are given in Annex V.

Timetable

0 - 5 months :	Design and theoretical finalization of the pilot plant.
6 - 8 months :	Checks and verification.
9 - 13 months :	Final technical project
13 - 19 months :	Construction of equipment
20 - 22 months :	Equipment checks
23 - 29 months :	Shipment and assembly on site.

Technical assistance and training

6 months : Project leader (construction on site).

3 x 3 months : 3 experts (mechanical engineer, chemical engineer, chemist); the chemical engineer will be from PLASTWERK AMMENODORFER

2 months : management

Training : 4 x 2 months during equipment checks for 4 Vietnamese technicians or engineers and completed by a study tour whose aims and program remain to be defined.

4. STUDY TOUR IN FRANCE AND IN COTE D'IVOIRE

4.1 Mission schedule

Thursday, 8th September

Arrival in Paris, welcomed by ACTIM, Mrs.Le Vigoureux.

Friday, 9th September

Visit to IRCA, welcomed by Mr. Campaignolle, Director.

Monday, 12th and Tuesday, 13th September

Visit to IRAP and the University of Maine. Welcomed by Mr. Pautrat, IRAP Director, Professor Brosse, University of Maine, Department of Chemistry.

Wednesday, 14th September

Departure for Côte d'Ivoire.

Thursday, 15th to Sunday, 18th September

Mission to Côte d'Ivoire : exhaustive visit of the pilot LNR plant, the chemistry, analysis, technical specifications and plantation laboratories and, an Ivorian rubber processing factory. Detailed presentation of the two UNIDO projects on the production and development of modified and unmodified LNR.

Monday, 19th September

Return to France.

Tuesday, 20th, Wednesday, 21st and Thursday 22nd September

Visit to the De Dietrich company, chemical engineering department, welcomed by Messrs. Macchioni and Wacrenier.

Friday, 23rd September

IRCA : presentation of a pilot chlorination plant project by Messrs. Gignier and Delassus, Chimie Développement International (CDI), in the company of Mr. Wacrenier from De Dietrich.

Monday, 22nd September

IRCA : Finalization of the project

Tuesday, 27th September

Visit to the ATOCHEM chlorination factory in Mouren, near Pau. Accompanied by Mr. Delassus, CDI, and welcomed by Mr. Quet, ATOCHEM.

Wednesday, 28th September

IRCA : Meeting and conclusions.

Thursday, 29th September

Departure for Bangkok.

4.2 Introduction

The aims of this mission were to enable the Vietnamese delegation of experts :

1) Le Van Nguyen : National Project Director, Director of the Institute of Chemical Industry

2) Ngo Thi Lien : Project Assistant (ICI)

3) Nguyen Tien Hung : Engineer of chemical equipment construction (ICI)

4) Luong Van Cau : Engineer of rubber technology, Head of the technical department, General Department of Chemistry.

to become familiar with research carried out by IRCA (Institut de Recherches sur le Caoutchouc) in France and in Côte d'Ivoire, and by its closely collaborating partners such as IRAP (Institut de Recherches et d'Application sur les Polymères), the University of Maine, and engineering companies e.g. De Dietrich and CDI (Chimie Développement International) in the fields of natural rubber chemistry and technology, and polymer chlorination. Another organization, IFP (Institut Français du Petrole), which works with De Dietrich in the fields of chemical engineering and polymer halogenization, was unfortunately unable to receive the Vietnamese delegation, but has the acquired know-how to construct and test the pilot plant before it is shipped to Vietnam.

4.3 Research and Development organizations : IRCA/CIRAD and IRAP

The mission was oriented towards problems dealing with laboratory equipment, natural rubber chemistry and technology, and the results obtained from the following UNIDO contracts :

- UF/GLO/81/059 : Production and development of liquid natural rubber
- US/GLO/85/151 : Development of the applications of liquid natural rubber

Chemistry, processing and technology of natural rubber

IRCA/CIRAD - IRAP in France and IRCA in Côte d'Ivoire have the scientific and technical resources available and the equipment required to ensure follow-up of research and technical assistance programmes for growers and manufacturers with respect to collection, processing, technical specifications, quality control and improvement, starting up and chemical modification of natural rubber.

Liquid rubber

The pilot liquid natural rubber plant and details of the processes involved in manufacturing LNR have already been discussed. So as to avoid assembly and starting up problems, it should be noted that the design and construction of this pilot plant have been entrusted to a single engineering company, which, before the mission on site, assembled and carried out 2 operations in Côte d'Ivoire to verify the proper running of this unit.

Chlorinated rubber

Only the research work and results obtained on the chlorination of liquid natural rubber at the laboratory stage were presented :

- LNR is a prime material for obtaining chlorinated rubber. However, the product is darker than those obtained from synthetic polyisoprene.
- Ultraviolet catalysis makes it possible to obtain chlorine contents of around 70 %.
- The residual carbon tetrachloride content in the finished product can be reduced to less than 0.5 %.

Visits were organized to the chemical engineering pilot plant at the University of Maine's Technology Institute and to the University macromolecular laboratories. During the visits to

IRCA in Côte d'Ivoire and to IRCA/CIRAD-IRAP, problems concerning equipment, analysis and quality control of natural rubber, LNR and chlorinated rubber were reviewed.

With a view to project DP/VIE/86/036 "Chlorinated rubber", the following adaptive research was proposed :

- Reduction of the molecular weight of natural rubber;
- Chlorination reaction catalysis
- Effects of the initial concentration of rubber
- Elimination of carbon tetrachloride.

The equipment available at IRAP and at the University of Maine will make it possible to carry out analyses and controls on raw and chlorinated rubber as well as on the major chemical products and reagents playing a role in the reaction.

4.4. Proposal for the construction of a pilot unit

At the time of the mission by Vietnamese experts, a proposal was made for the construction of a pilot unit for natural rubber preparation by the De Dietrich Company - constructor of the liquid rubber pilot plant - and by Chimie Développement International - specialists in polymer chlorination. This proposal is based on the following principles:

- discontinuous chlorination process, which alone provides greater scope for varying reaction paramaters.

- capacity intentionally limited to 50 kg of chlorinated rubber per operation, so as to avoid the handling and processing of too great a volume of toxic gases and solvents at what is still the experimental stage.

- two-stage chlorination, so as to enable more in-depth study of catalyst effectiveness - chemical products or ultraviolet rays - coming into play either at the beginning or at the end of the reaction.

The schematic diagram for the pilot chlorination process is given in Annex VI. Rubber chlorination is carried out in two reactors equipped with stirrers whose profile enables more effective liquid-gas contact than in a bubble column. The equipment is divided up into 4 skids, each corresponding to a defined phase of the chlorination process, so as to enable prior assembly, firstly so that equipment can be tested before shipment and, secondly, so as to train the Vietnamese engineers and technicians who will subsequently be responsible for installation and operation of the pilot plant.

The composition of each skid is indicated below:

SKID 1 CHLORINATION

- 1 vitrified steel chlorination reactor,
- 1 vitrified steel finishing reactor,
- 1 distillation column,
- 1 finishing column,
- PVDF circulation and transfer pumps
- 3 graphite condensers,
- 1 fan,
- 1 suspension relay,
- 1 decanter.

SKID 2 FILTRATION

- 1 vitrified steel dryer,
- 1 filter,
- 1 filtration relay,
- 1 removable container,
- 1 vacuum pump with condenser.

SKID 3 EFFLUENT TREATMENT

- 1 absorption column,
- 1 knock-down relay,
- 1 vacuum ejector,
- 1 decanter,
- circulation pumps.

SKID 4 BUFFERS

- ♦ water buffer,
- ♦ recycled water buffer,
- ♦ crude CCL4 buffer,
- ♦ recycled CCL4 buffer,
- ♦ safety buffer,
- ♦ CCL4 and Chlorine buffer,
- ♦ transfer pump,

MISCELLANEOUS

- ♦ chlorine storage,
- ♦ hot water production.

The example described above has to be completed by a mixer and a guillotine for preparing the natural rubber before going into solution form, along with a device enabling photochlorination of the chlorinated rubber solution. The pilot unit will be assembled, tested and dismantled at the site belonging to the French Petrolium Institute at Sochaux near Lyons. Vietnamese experts will be welcome to come to the site and participate in all these operations.

Prior to construction of the pilot plant, the following aspects will be perfected: rubber solution production, degasification of the solution (elimination of HCl and of chlorine), precipitation of chlorinated rubber, drying in accordance with the texture of the end product.

5. CONCLUSIONS

5.1 Mission to Vietnam

The following observations should be taken into account to reach a favourable decision about the project requested by the Institute for Chemical Industry :

- chlorinated rubber remains an essential additive for the production of corrosion-proof paints and Vietnam has the necessary raw materials for its manufacture.

- a local market exists for this kind of paint and the short and medium-term increase in the industrialization of neighbouring countries should provide further outlets.

- the Institute of Chemical Industry is a research, development and training entity capable of providing technical assistance and training for local industries in the chemistry field; in order to carry out this task, the Institute needs more modern and better adapted equipment, both in the laboratory (analysis, glassware, small items of equipment) and in the pilot plant. It has the necessary buildings and utilities for installing an additional pilot unit to complete those that already exist. The highly motivated managerial staff are capable of running this pilot plant, provided that:

  1) a turnkey pilot plant is supplied, with sufficient spare parts, because it is currently impossible to find equipment of acceptable quality for chlorination locally.

  2) the staff are trained in the country where the pilot plant was designed and constructed.

  3) small items of laboratory and analysis equipment are supplied with the pilot plant.

5.2 Mission to GDR

The Carl Schorlemmer University in Leuna Merseburg has all the buildings, scientific staff and equipment required for undertaking a pilot unit or medium sized industrial plant construction project. The proximity of the VEB AMMENDORFER factories, which produce "Chlorophane" chlorinated rubber, would be an effective advantage if cooperation could be established with this company.

The pilot unit proposed uses very traditional, quite old technology, which is tried and tested for production of chlorinated rubber with no surprises, following clearly defined parameters, but which lack flexibility for research experiments and development. The University lacks practical experience in polymer chlorination and in-depth knowledge of problems associated with natural rubber.

5.3 Mission to France and Côte d'Ivoire

IRCA has access to a network of research institutes in France and Côte d'Ivoire, along with universities and engineering companies, which enabled it to construct and operate the current liquid natural rubber pilot production unit.

In order to ensure the success of the project to install a chlorinated natural rubber pilot production unit at the Institute for Industrial Chemistry, it would be best to follow the same procedure: research and development in one or other of the institutes; design, construction, tests and dismantling of the pilot unit by a single engineering company; pilot unit assembly and start-up in Vietnam with the technical assistance of the same network. IRCA's know-how in this field should make it possible to deal with natural rubber selection, quality control and utilization problems before and during chlorination. The know-how and experience of the De Dietrich company and of Chimie Développement International in the following fields: construction of turnkey units, supply of

vitrified reactors, liquid-gas exchange and drying, backed up by the expertise of the French Petrolium Institute, offer a guarantee of success for the design and construction of the pilot chlorination plant.

6. PROJECT PROPOSAL DOCUMENT

As requested in the "Job description" in Annex I, the draft project document was drawn up by modifying the proposal in Annex II, subsequent to observations, comments and technical data gathered during the mission.

DP/VIE/86/036

Chlorinated rubber

December 1988

UNITED NATIONS DEVELOPMENT PROGRAMME

Project : Delivery of technology on experimental manufacture of chlorinated rubber.

Duration : 3 years (1989-1991).

Main target : Experimental manufacture on the pilot scale.

Secondary target : Improvement of material background for the Institute of Chemical Industry.

Classification by SRVN

Classification by UNDP

Implementating organization of SRVN government :

The Institute of Chemical Industry, General Department of Chemistry.

Executive organization of United Nations :

UNIDO

Scheduled starting time :

Preparatory works : 1989

Realization of all activities : 1989-1990-1991

Investment : Investment share of SRVN government

Investment share of UNPD

PART 1 : LEGAL CONTEXT

This preparatory Assistance Document shall be the instrument referred to as such in Article 1, Paragraph 1 of the Standard Basic Assistance Agreement between the Socialist Republic of Vietnam and the United Nations Development Programme, signed by the parties on 21st March 1978.

To reach the targets of the Standard Basic Assistance Agreement, the executive organization of the governemnt will submit to the Cooperation agency of the Government, as said in the Agreement.

PART 2 : THE PROJECT

I. Development objective

To use domestic raw materials for the production of chlorinated rubber for corrosion-proof paints under tropical, hot and humid conditions.

II. Immediate objectives

1) To manufacture chlorinated rubber on experimental equipment with a capacity of 50 kg/operation.
2) to complete some instruments in the laboratory, to study, improve and control product quality.
3) To improve technical qualifications for researchers and technicians in the field of rubber chlorination.

III. Specific notes :

IV. Background and qualifications

Vietnam is located in the tropical, hot and humid zone. All buildings, constructions, machines, household furnishings, production equipment, etc. are rapidly corroded. The simplest protective means is to paint them.

Development of the paint industry has been difficult, due to the shortage of raw materials. Synthetic resines and other materials for paints are for the most part imported.

The minimum annual demand for paint is 50 to 60 thousand tonnes (1 kg/year per capita), while the output of vegetable oil - the only domestic raw material delivered specifically to the paint industry is only 1 to 2 thousand tonnes, from which 3 to 4 thousand tonnes of oil and alkyd paints are produced.

The increase in raw materials for the paint industry is extremely urgent. According to the scheduled plan, in 1995-2000, some raw material production plants will be established to make 30,000 - 40,000 tonnes of different sortiments of

paint, in which 20,000-30,000 tonnes of paint will be corrosion-proof.

The paint sortiments produced at present are only from polymerized oils and alkyd resines, which do not meet the protective demands of use under tropical conditions. Therefore, the manufacture (experimental) of chlorinated rubber for obtaining the necessary technological data in order to develop into a chlorinated rubber production plant is an essential step in the paint industry and corrosion-proof techniques.

2) Economical and technical background

A) Raw material

The raw materials for proudction of chlorinated rubber are natural and there is excess chlorine available in the country.

Vietnam can harvest over 50,000 tonnes/year of natural rubber, half of which falls into grades 3-6, which cannot be processed into tyres and other technical equipment. According to the scheduled 1996-2000 plan, domestic natural rubber output will be 70,000 to 80,000 tonnes/year.

The caustic soda plants annually discharge considerable amounts of chlorine gas, which is not used in any industrial branch : not only spanders considerably, but also pollutes the atmosphere surrounding these plants. The use of chlorine to produce chlorinated rubber is rational.

B) Technical background and technology receiving conditions

The film-forming matter and paint-varnish research laboratory in the Institute of Chemical Industry started in 1962 and at present is a center studying technological processes, experimental manufacture of paints and varnishes, and formulation and use of many sortiments of pretective films. This laboratory has investigated the preparation of chlorinated rubber from Vietnamese natural one and waste chlorine of

caustic soda plants as well as studied its use as anticorrosion films.

Experimental results show that chlorinated rubber is a material most suitable to prepare protective paints and is of the great feasibility in Vietnam.

For over 15 years, the study on preparation, formulation and use of chlorinated rubber in the Institute has obtained only limited results, due to poorly equipped conditions, mainly, the enamelled chlorinator does not respond to the dispersion and absorption of chlorine gas in the solution. That is why the obtained products have not enough necessary technical specifications (high molecular mass, low solubility and stability, chlorine content less than 60 %, etc.); measures for improvement of economical efficiency do not exist (solvent recuperation is only 50 %) and waste toxic gas is not completely treated.

Due to the lack of experience as well as knowledge on progressive technology and lack of necessary equipment, we cannot yet prepare chlorinated rubber on the large scale to collect all technological parameters in order to propose a reasonable duction process, design and import partly machineries and equipment or commercialize it.

3) Labour

The film-forming, paint and varnish research laboratory in the Institute for Chemical Industry - main collective, studying chlorination of rubber - has many candidates of chemical science, engineers and technical workers with a high molecular chemistry speciality. Besides, there are some other helping units such as Analytical laboratory, Design bureau, etc.

4) Utility background

The Institute has in store 3 workshops with a surface of 1,500 m^2. The Institute has completed the construction of an experimental workshop with the layout of 1,000 m^2. The unit for experimental of chlorinated rubber will be arranged in this newly built workshop and use available feedstock, electricity, water and steam supply systems.

Our desire is to improve is some activities of the Institute in the field of paint/varnish research and development by means of establishment of an experimental unit, including progressive equipment and instruments for chlorination, analysis and increasing product specifications, corresponding to international commerical standard ones for exploiting effectively this product as corrosion-proof films under tropical conditions.

Without the assistance of UNDP, our Institute cannot make such prototypical equipment and grasp up-to-date chlorination technology. Hence we propose the assistance of UNDP in financial term 1986-1991.

5) Perspective results of the project

If the project is implemented, predicted results should be probably :

A) To establish an experimental unit with complete conditions for determination of technological parameters in order to commercialize afterwards.

B) To use up waste chlorine gas of caustic soda plants, to improve economic efficiency for Industry and protect the environment.

C) To equip some instruments and tools for control of chlorinated products, for formulation of protective and chemical resistant films.

D) To train and improve qualifications for technical personnel, working in paint research and production.

VI. Investment

1) Contribution of SRVN government

To supply personnel staff for the project :

- Project director (stimul. Director of the Institute for Chemical Industry)
- Engineers : 18 persons
- Technicians : 7 persons
- Workers : 15 persons
- Other officers : 4 persons

Total 44 persons

B) to invest material background :

- To build and reconstruct workshops and feedstock
- To supply labour and facilities to fit equipment and machinery
- To arrange suitable office space for chief technical advisers and other experts, to nominate secretarial and interpreter support for them
- To set up administrative staff, information service and other means
- To make power network for manufacturing unit
- To supply raw materials and other utilities serving the experimental operations.

2) Constribution of UNDP

- To send expert to formulate the project
- To designate chief technical adviser
- To nominate technical experts
- To supply equipment for manufacturing unit and instruments for control laboratory.

Home and foreign specialists participating in the project will coordinate their activities to achieve plan and targets, which were drawn up in the project. Responsibilities of home and foreign specialists are pre-determined through mutual discussion and concord, in accordance with the content of technical cooperation.

VII. Implementation

1) Work schedule

The project will be implemented in 3 phases over a period of 2 ½ years, with the following work schedule : (see annex I)

Phase 1 : premliminary research : 6 months

- Adjustment of the molecular weight of rubber through solvent phase depolymerization : 6 months.
- Improvement of the chloride content in the finished product using specific additive catalysts or ultraviolet rays.
- Stabilization of chlorinated rubber.
- Reduction of the carbon tetrachloride content in the finished product.
- Analysis.

Phase 2 : Design, construction and testing of the pilot plant :12 months

- Adjustment tests for the chlorinated rubber chlorination, precipitation and drying phases with a view to designing the corresponding reactors.
- Design, purchase or manufacture of the equipment and machines.
- Pilot plant assembly and tests.
- Partial dismantling, packaging and transport to the port.

Phase 3 : Assembling, start-up and running in Vietnam : 6 months

- Shipment to Vietnam.
- Assembly.
- Start-up and commissioning.
- Pilot plant experimentation with a view to producing chlorinated rubber.

The breakdown of costs is given below :

2) Personnel required for the duration of the project

A) Qualifications, duties and length of missions for international experts

EXPERT	MONTHS	COST US $
Phase 1 : Preliminary development in the laboratory	10	87,000
1 polymer chlorination expert	6	52,200
1 organic analysis expert	4	34,800
Phase 2 : Design, construction and testing of the pilot plant	53	461,100
2 chemical engineering experts	22	191,400
2 electromechanical experts	22	191,400
1 organic chemistry expert	6	52,200
1 organic analysis expert	3	26,100
Phase 3 : Assemby, start-up and running in Vietnam	14	121,800
1 chemical engineering expert	3	26,100
1 electromechanical expert	3	26,100
1 chemist specialized in polymer chlorination	6	52,200
1 organic analysis expert		
Laboratory assembly	2	17,400
C.T.A.	12	104,400
Total	**89**	**774,300**

NB : Cost of an expert = US $ 8,700/month

B) Vietnamese experts

Vietnamese specialists = 20 x 30 months

C) Training

COURSE	COST US $
Chlorination fellowships	
2 people x 3 months	15,000
Chemical engineering fellowships	
2 people x 3 months	15,000
Operating and testing fellowships	
For the pilot unit :	
2 people x 3 months	10,000
Total	**40,000**

NB : 1 month fellowship = US $ 2,500

3) Laboratory equipment and material

UNDP contribution to the pilot rubber chlorination plant

EQUIPMENT	COST US $
Rubber chlorination	222,000
Chlorination reactors, distillation columns, condensers, circulation and transfer pumps, decanter (all treated for corrosion).	
Filtration and drying	145,000
Filter, dryer, vacuum pump (treated for corrosion)	
Effluent treatment	60,000
Absorption column, knock-down relay decanter, circulation pumps (treated for corrosion)	

Buffer tanks	60,000	
water and recycled water buffer, crude, recycled and chlorine CCL4 buffer, safety tanks, transfer pump (treated for corrosion)		
Miscellaneous	120,000	
Chlorine storage, hot water production (treated for corrosion) instruments and utilities		
Sub-total 1		607,000
Spare parts (10 x sub-total 1)	61,000	
Chemical products unavailable on site	41,000	
Sub-total 2		708,000
CIF (10 x sub-total 2)	71,000	
Unforeseens (5 % of sub-total 1)	35,000	
TOTAL		**814,000**

LABORATORY EQUIPMENT	**US $**
(unassembled, available on-site)	
Rubber quality tests	
Wallace plastometer	20,000
Mooney Viscometer	20,000
Normalized stirrer	70,000
Solution viscometer	10,000
Chlorinated rubber analysis	
Chlorine	40,000
Tetrachloride	50,000
Water analysis	30,000
Miscellaneous laboratory equipment	100,000
Total	340,000

Total imported laboratory equipment and material 1,154,000

Contribution by the Vietnamese Government

Items	Quantity
Workshop	650m²
Store (Feedstock)	300m²
Electricity network (Generator, wires)	
Steam generator and pipes	
Erection	
Test run	
Reactor	2
Tank	
Drier	
Rubber calendar	1
Rubber cutter	1

This project is in the framework of the General Department of Chemistry. The Established experimental unit will be considered as a part of the Institute of Chemical Industry. The project Director will be the Director of the Institute. At present, there is already a suitable layout for the experimental unit in the latery limit of the Institute (Cau dien background)

IX. Prior obligations and prerequisites

- To complete with all necessary formalities on investment in local money.
- To designate the Project Director.
- To nominate the project participants, approve the personnel who will be trained abroad.
- To make ready housing, transport facilities, working space and interpreters for the Chief technical adviser and other experts.
- To assure the supply of power, water, construction and production material and other materials.

- the UNDP permanent representative, in the name of UNDP will sign the project document. UNDP offers only financial assistance when all above mentioned prerequisite conditions are completed or are able to be completed. Wehn one or more of the above mentioned prerequisite conditions are not met, UNDP can independently delay or stop assistance.

PART III : SCHEDULE FOR MONITORING, EVALUATION AND REPORTS

Tripartite monitoring reviews (UNDP, UNIDO, SRVN)

The project will be monitored every year by UNDP. The first control will be carried out after one year from the approval date.

Evaluation of results

Time and measure of result evaluation of the project will be discussed by all three parties after accomplishment of the project and manufacturing comes on stream.

Periodical and terminal reports

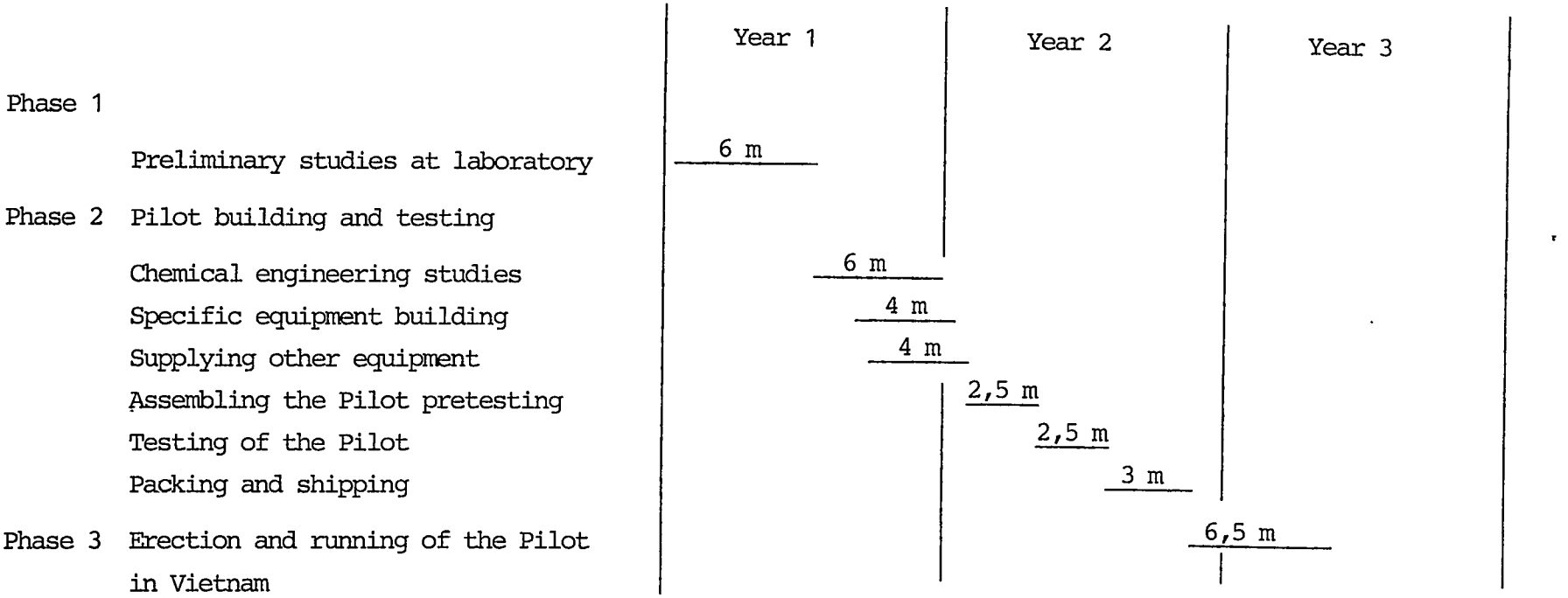
Periodical reports are written by the Chief technical adviser and the Project Director, according to UNDP procedure.

The final report will be prepared 3 months before accomplishing the project and submitted to UNDP, UNIDO and SRVN government, in accordance with existing policies and procedures.

PROJECT DP VIE 86/036/32-01

CHLORINATED RUBBER

PROGRAMME PROPOSAL



m = month

ANNEX I

2 June 1987

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

JOB DESCRIPTION

DP/VIE/86/036/11-01/J13420

Post title Expert in the Field of Natural Rubber Modification

Duration One month

Date required As soon as possible

Duty station Hanoi with work at home base

Purpose of project To develop useful outlets/applications for natural rubber and in particular corrosion-resistant paints based on chlorinated natural rubber.

Duties The expert in collaboration with counterpart authorities will specifically be expected to:

1) To meet with four Vietnamese specialists who will have previously visited rubber research centres in the Ivory Coast, France, and the United Kingdom, and will advise the specialists on the interpretation of their findings.

2) Together with the Vietnamese specialists the expert will prepare a draft research and development plan for the modification and chlorination of natural rubber and for the development of paints based on chlorinated rubber. The programme will suggest the scale on which the development work should be performed, the parameters which should be investigated as well as the specifications of equipment required.

3) The expert will visit Viet Nam to discuss the draft research and development plant with Vietnamese authorities and with the UNDP, and assist in preparation of draft project document.

The expert will also be expected to prepare a final report, setting out the findings of his mission and his recommendations to the Government on further action which might be taken.

..../..

Qualifications

Engineer or Chemist with wide experience in the development of processes for modifying and chlorinating natural rubber.

Language

English (French as working language)

Background information

There is a vital need for protective anti-corrosive paints in Viet Nam, particularly in view of the hot and humid climate. The existing paint industry produces less than a total of 10,000 tons a year of paints made using natural oils as well as some using alkyd resins. As the demand for paints is at least 50,000 tons per year, a large quantity of paints has to be imported as well as many of the raw materials for the few paints made locally. What is more, these locally-made paints are insufficiently corrosion-resistant for the Vietnamese climate. The Government has plans for the production of between 20,000 and 30,000 tons per year of anti-corrosion paints in the period 1995-2000.

Chlorinated rubber is a major constituent of many anti-corrosive paints and Viet Nam has ample supplies of the main raw materials for its manufacture. On the one hand, Viet Nam already produces approximately 50,000 tons per year of rubber and there are realistic plans and action being taken to increase this production to more than 300,000 tons per year during the next decade. On the other hand, there are at present few uses for the chlorine produced as a by-product of caustic soda manufacture in Viet Nam, and there is a problem in disposing of this poisonous by-product.

The Institute of Industrial Chemistry's film forming and paint varnish research laboratory, which was started in 1962, has to the last 15 years beeing studying the preparation, formulation and use of chlorinated rubber for paints.

Their work has been carried out on a laboratory scale or in equipment sized to make 10 kg of chlorinated rubber per batch. The have asked for UNDP assistance in setting up pilot unit with a capacity of 200-500 kg per batch.

However, neither the laboratory work, nor the work done in 10 kg batches has succeeded in making chlorinated rubber with what is considered to be a satisfactory degree of chlorination (chlorine content greater than 65%). The highest chlorine content achieved by the IIC is 61% also solvent recuperation is less than 50%, which would be a serious economic handicap to an eventual industrial process. Also the toxic waste gas is not sufficiently treated. It should be noted that IIC uses mechanical means for the necessary preparation/modification of the natural rubber before chlorination.

Independently, the Centre National de Recherche Scientifique in Hanoi has carried out some small-scale laboratory work on the chlorination of natural rubber whereby chemical means have been used to decyclise the rubber prior to chlorination. It would appear that they have achieved a satisfactory degree of chlorination and prepared paints with satisfactory properties.

The UNIDO has been the executing Agency for a number of projects carried out by members of the International Rubber Research and Development Board (IRRDB) in the Ivory Coast, France, and the United Kingdom, on the modification of natural rubber for a variety of applications, including chlorination and on chlorination itself. Degrees of chlorination as high as 68% have been obtained during this work.

It is therefore appropriate to acquaint Vietnamese specialists with the detail of successful work carried out in this field elsewhere and in particular in the course of the project supported by UNIDO, so that based on this knowledge and aided by an expert consultant a plan for future development by Viet Nam in this field can be prepared.

For this, it is also necessary to be informed on the requirements of the paint industry with regard to chlorinated rubber and with ways of making and applying chlorinated rubber-based paints.

ANNEX II

F/LC VIC/86/036

PROPOSAL

UNITED NATIONS DEVELOPMENT PROGRAMME

PROJECT OF SRVN

Project : Delivery of technology on experimental manufacture of chlorinated rubber from Vietnamese natural one for the production of anticorrosion paints, used in the tropical climate conditions

Duration : 3 years (1986-1989)

Main target : Experimental manufacture in large scale

Secondary target : Improvment, of material background for the Institute for Industrial Chemistry

Classification by SRVN

Classification by UNDP

Implementing organisation of SRVN government:

The Institute for Industrial Chemistry
General Department of Chemistry

Executive organisation of United Nations :

UNIDO

Scheduled starting time :

Preparatory works : 1986
Realisation of all activities :

Investment :

Investment share of SRVN government : 30,800,000 đông
Investment share of UNDP : 1,000,000 US $

SRVN government representative

Sign Date

UNIDO representative

Sign Date

UNDP representative

Sign Date

PART I

LEGAL CONTEXT

This preparatory Assistanca Document shall be the instrument referred to as such in Article 1, Paragraph 1 of the Standard Basic Assistance Agreement between the Socialist Republic of Vietnam and the United Nations Development Programme, signed by the parties on

To attain the targets of the Standard Basic Assistance Agreement, executive organisation of the government will submit to the Cooperation agency of the Government, as said in the Agreement.

PART II

THE PROJECT

I. Development objective

To use domestic raw materials for the production of anticorrosion paints in the tropical, hot and humid conditions.

II. Immediate objectives

1) To manufacture chlorinated rubber on experimental equipment with the capacity of about 200-500 kg/bath.

2) To complete some instruments for laboratory, which is able to control the quality of products, study and improve them.

3) To improve technical qualifications for research and production workers in the branch of rubber chlorination.

III. Specific notes :

IV. Background and Justification :

Vietnam is in the tropical, hot and humid zone. All building co tructio ıs, machines, household instruments, production equipments.. are rapidly destroyed. Simplest protective mean is to paint them.

- The paint industry has been developped with difficulties, due to the shortage of raw materials. Synthetic resines and other materials for paints are mostly imported.

- Yearly minimum quantity on paint demand amounts 50,000-60,000 tonnes (corresponding to 1 kg/yaer per capita), while output of vegetable oil - the only domestic raw material - specially delivered to the paint industry is only 1,000-1,200 tonnes, from which 3,000-4,000 tonnes oil and alkyd paints would be produced.

The increase of raw material for paint industry is an extremel urgent need. According to scheduled plan, in 1995-2000 period, some raw material production plants will be established to make 30,000-40,000 tonnes of different sortiments of paint, in which 20,000-30,000 tonnes of paints will be anticorrosin ones.

The paint sortiments, producing at present are only from polymerised oils and alkyd resines, which do not meet the protective demands of use in the tropical conditions. Therefore, the manufacture (experimental) of chlorinated rubber for obtaining necessary technological data in order to enlarge into a chlorinated rubber production plant is an essential step in the paint industry and anticorrosion techniques.

2) Economical technical background

a) Raw material

- The raw materials for production of chlorinated rubber is natural one and excess chlorine available in the country

- Vietnam can havest over 50,000 tonnes per year of natural rubber in which its half is 3-6th category one, unable to process into tyres and other technical details. According to scheduled 1996-2000 plan, home natural rubber output will amount 70,000 - 80,000 tonnes per year.

- The caustic soda plants discharge annually a great excess

of chlorine gas, which is not still used up in any industrial branche; not only spanders considerably, but also pollutes atmosphere surrounding these plants. Use of chlorine to produce chlorinated rubber is rational way.

b) Technical background and technology receiving conditions

The film-forming matter and paint-varnish research laboratory in the Institute for Industrial Chemistry has put into action from 1962 and at present, is a center, studying technological process, experimental manufacture of paints and varnishes, working out the formulation and use of many sortiments of protective films. This laboratory has investigated the preparation of chlorinated rubber from Vietnamese natural one and waste chlorine of caustic soda plants as well as studied its use as anticorrosion films.

Experimental results show that chlorinated rubber is a material, most suitable to prepare protective paints and of the great feasibility in Vietnam.

For over 15 years, the study on preparation, formulation and use of chlorinated rubber in the Tinstitute has obtained only limited results, due to poorly equipped conditions, mainly, enamelled chlorinator does not respond to the dispersion and absoppтion of chlorine gas in the solution. That is why the obtained products have not enough necessary technical specifications (high molecular mass, low solubility and stability, chlorine content less than 60%...) ; measures for improvement of economical efficiency do not exist (solvent recuperation is only 50%) and waste toxic gas does not completely treat.

Due to the shortage of experience as well as knowledge on progressive technology, lack of necessary equipments, we cannot still to prepare chlorinated rubber in larger scale to collect all technological parameters in order to propose a reasonable production process, design and import partly machineries and equipments to commercialize it.

3) Labour

The film-forming, paint and varnish research laboratory in the Institute for Industrial Chemistry - main collective, studying chlorination of rubber - composes of many candidates of chemical science, engineers and technical workers on high molecular chemistry speciality. Besides, there are some other helping units such as Analytical laboratory, Design bureau...

4) Utility bachground

The Institute has in store 3 worhshops with the surface of 1,500 m^2. By the plan of 1987, the Institute will complete the construction of an experimental workshop with the layout of 1,000 m^2. Unit of experimental of chlorinated rubber will be arranged in this newly built worshop and used available feedstock, electricity, water and steam supplying systems.

Our desire is to improve some activities of the Institute in the field of paint/varnish research and development by means of establishment of an experimental unit, including progressive equipments and instruments for chlorination, analysis and increasing product specifications, corresponding to international commercial standard ones for exploiting effectively this product as anticorrosion films in tropical conditions.

Without the assistance of UNDP, our Institute cannot make such prototypical equipments and grasp up-to-date chlorination technology. So we propose the assistance of UNDP in financial term 1986-1991.

5) Perpective results of the project

If the project will be realized, predicted results should be probably :

a. To establish an experimental unit with complete conditions for determination of technological paramaters in order to commercialize afterwards

b. On the basis of experimental equipments, to enable the preparation of different chlorinated resines from domestic raw materials, serving anticorrosion work in tropical climate.

c. To use up waste chlorine gas of caustic soda plants, to improve economic efficiency for Industry and protect environment.

d. To equip some instruments and tools for control of chlorinated products, for formulation of protective and chemical resistant films.

e. To train and improve qualification for technical personels, working in paint research and production.

V. Other activities of the project

To attain all targets and objectives of the project, it is necessary to carry out following activities :

1) Preparatory works

- To work out the preliminary project (The Institute for Industrial Chemistry with the help of State management agency)
- To perfect the project document (The Institute for Industrial Chemmistry with the help of State management agency and UNDP)
- To approve the project
- To designate the project director
- To choose co-ordinators and technical advisers
- To draw up legislation on responsibilities of technical advisers
- To prepare all necessary conditions to receive equipments

2) Realisation of project

- To draw up technological process and order equipments
- To build workshop and instal equipments
- To put into operation and deliver

3) Training cadres

- Project director and his colleagues make an examination mission
- To send project participants training abroad.

4 Accomplishment of the project

- To control and evaluate the realisation and obtained results

- To accomplish and submit the terminal report on project to the SRVN government and Directors of UNDP and UNIDO.

VI. Investment

1) Contribution of SRVN goverment

a/ To supply personel staff for the project :

- Project director (stimul. Director of the Institute for Industrial Chemistry)
- Engineers : 18 persons
- Technicians : 7 persons
- Workers : 15 persons
- Other officers 4 persons

Total 44 persons

b/ To invest material background :

- To build and reconstruct workshops and feedstock
- To supply labour and facilities to fit equipments and machineries
- To arrange suitable office space for chief technical adviser and other experts, to nominate secretarial and interpreter support for them
- To set administrative staff, information service and other means.
- To make power network for manufacturing unit
- To supply raw materials and other utilities, serving the experimental operations.

2) Contribution of UNDP

- To send expert to formulate the project
- To designate chief technical adviser
- To nominate technical experts
- To supply equipments for manufacturing unit and instruments for control laboratory.

VIII. Realisation

Detailed plan for the project realisation will be drawn up by Project Director and Chief technical adviser.

This plan will be worked out before starting the realisation.

The project may be adjusted appropriately, according to concret situation. Detailed plan, entirely accorded between the Project Director and Chief technical adviser, is attached to the document as a part of the project.

1/ The realisation of project includes following steps :

- Project Director and Chief technical adviser draw up together detailed plan
- To formulate a list of all imported equipments and instruments and order them
- Project Director and his colleagues make examination mission abroad
- To order equipment invoice and approve general arrangement
- To send choosen personels training in equipment supplier country
- To reconstruct workshops by layout of arranging-fitting party.
- To receive equipments and instal them
- To put separately equipments in operation
- To put all manufacturing unit in operation.

2/ Other preparatory works

Home and foreign specialists, participating in the project will co-ordinate their activities to achieve plan and targets, which were drawn up in the project. Responsibilities of home and foreign specialists are pre-determinated through mutual discussion and concord, in accordance with the content of technical co-operation.

VIII. Implementing organisation

This project is in the framework of General Department of Chemistry. Established experimental unit will be considered as a part of the Institute for Industrial Chemistry. Project Director will be Director of the Institute. At present, there is already a suitable layout for the experimental unit in the latery limit of the Institute (Cau dien background)

IX. Prior obligations and prerequisites

- To complete with all necessary formalities on investment in local money.

- To designate Project Director

- To nominate project participants, approve personels, who will be trained abroad.

- To make ready housing, transport facilities, working space and interpreters for Chief technical adviser and other experts.

- To assure the supply of power, water, construction and production materials and other materials

- Permanent representative of UNDP, in the name of UNDP will sign in project document. UNDP offers only financial assistance when all above mentioned prerequisite conditions completed or was able to complete. When one or more above mentioned prerequisite conditions do not complete, UNDP can independently delay or cease the aid.

PART III

Schedule for monitoring, evaluation and reports

) Tripartite monitoring reviews (UNDP, UNIDO, SRVN)

The project will be monotored periodically in accordance with policies and procedures , established for this purpose by UNDP. The first control will be carried out after one year from approval date.

2) Evaluation of results

Time and measure of result evaluation of the project will be discussed by all three parties after accomplishment of the project and putting manufacturing unit into stream

3) Periodical and terminal reports

Periodical reports are written by Chief technical adviser and Project Director, according to procedures of UNDP.

Terminal report will be prepared 3 months before accomplishing the project and submitted to UNDP, UNIDO and SRVN government, in accordance with existing policies and procedures.

PART IV

APPENDIX ON DETAILED PROJECT THROUGH SCHEDULE

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Appendix 1a

PERSONEL CONTRIBUTION OF SRVN GOVERNMENT

	Quantity	Duration	Salary	Sub-total
Project Director	1	36 months	2,000	72,000 đông
Engineer on :				
- Raw material preparative.	2	24 -	1,500	72,000 -
- Chlorination of rubber	3	24 -	1,500	108,000 -
- Distillation-Separation of products	4	24 -	1,500	144,000 -
- Solvent recovery, Washing, Purifying and Stabilizing products	4	24 -	1,500	144,000 -
- Chemical equipment/machinery	2	24 -	1,500	72,000 -
- Analysis of products	2	24 -	1,500	72,000 -
Technician on :				
- Rapid control on the spot	3	24 -	1,000	72,000 -
- Formulation of products	2	24 -	1,000	48,000 -
- Control of product specifications	2	24 -	1,000	48,000 -
Officer				
- Typewritter	1	24 -	1,000	24,000 -

(continued)

- Driver	1	24 months	1,000	24,000 đồng
- Administrative	1	24 -	1,000	24,000 -
- Interpreter	1	24 -	1,000	24,000 -
Worker				
- Electrician	2	24 -	1,000	48,000 -
- Operator	10	24 -	1,000	240,000 -
- Mechanician	3	24 -	1,000	72,000 -
Total	44			1,410,000 -

Appendix 1b

INVESTMENT SHARE ON MATERIAL BACKGROUND OF SRVN GOVERNMENT

Items	Quantity	Price	Sub-total
Workshop	650 m^2	7,000	4,550,000 đồng
Store (Feedstock)	300 m^2	4,500	1,350,000 -
Electricity network (Generator,Wires)			3,500,000 -
Steam generator and pipes			4,000,000 -
Erection			1,500,000 -
Test run			7,000,000 -
Reactor	2	1,500,000	3,000,000 -
Tank			1,500,000 -
Drier			1,500,000 -
Rubber calender	1		1,000,000 -
Rubber cutter	1		1,000 000 -
		Total	29,400,000 -

Appendix 2

CONTRIBUTION OF UNDP

Items	Quantity	Salary	Sub-total (US /$)
a) Expert's mission			
- Chief technical adviser.	12 months/p (X 4)	8,000	96,000
- Expert on :			
chlorination	4 months/p	5,000	20,000
stabilisation of product	2 months/p	5,000	10,000
analysis	2 months/p	5,000	10,000
control of paint film	2 months/p	5,000	10,000
- Other experts			20,000
- Expensive on transport, administrative...			70,000
		Total	250,000
b) Training			
- Examination mission x 2 times	8 months/p	4,000	32,000
- Training engineers on :			
chlorination	15 months/p	2,500	37,500

(continued)

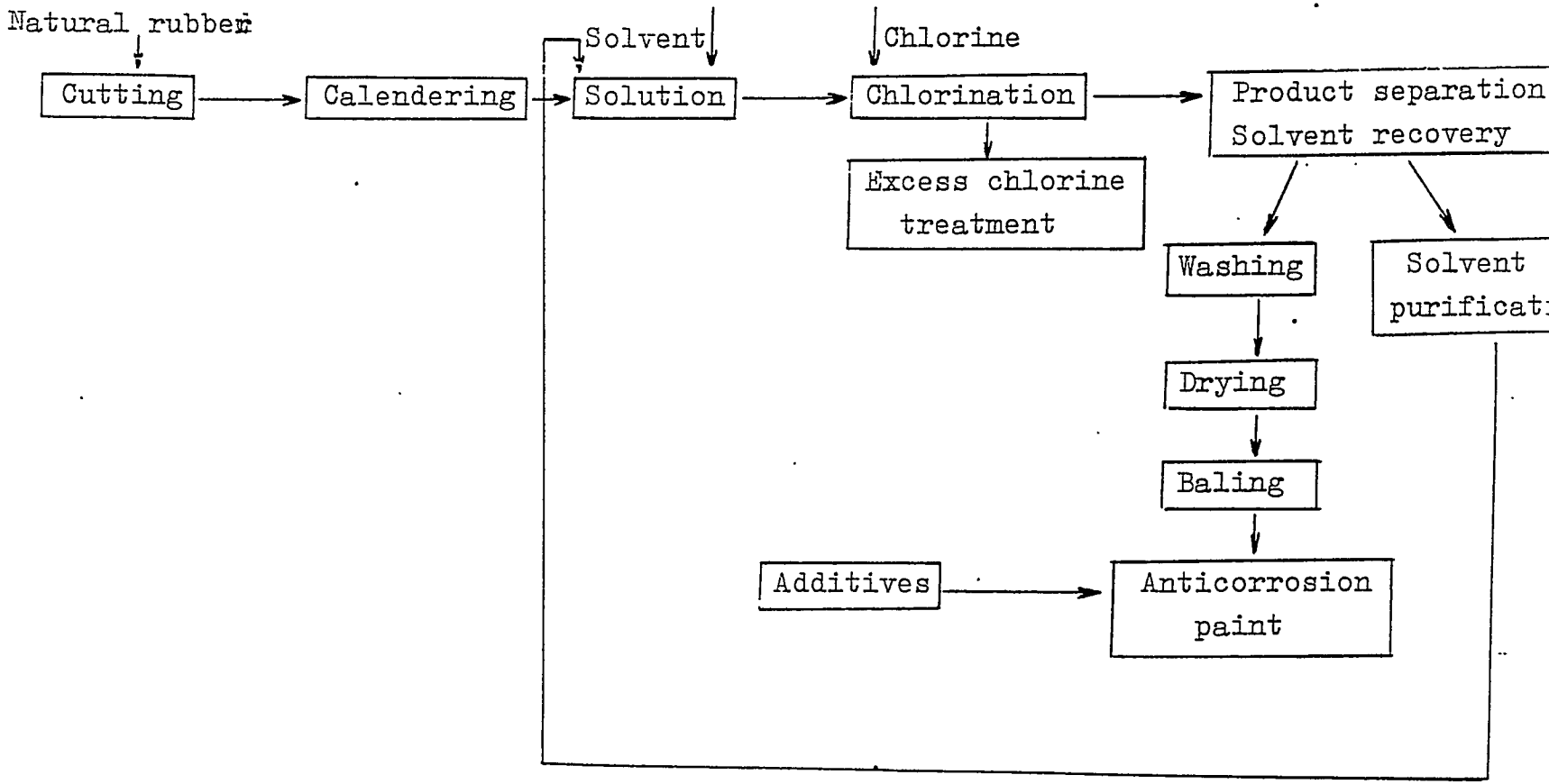
analysis	: 12 months/p :	2,500 :	30,000
modification, formulation of paint	: 12 months/p :	2,500 :	30,000
anticorrosion paint	: 9 months/p :	2,500 :	22,500
Expensive on seminar	: :	:	10,000
transport	: :	:	90,000

		Total	260,000

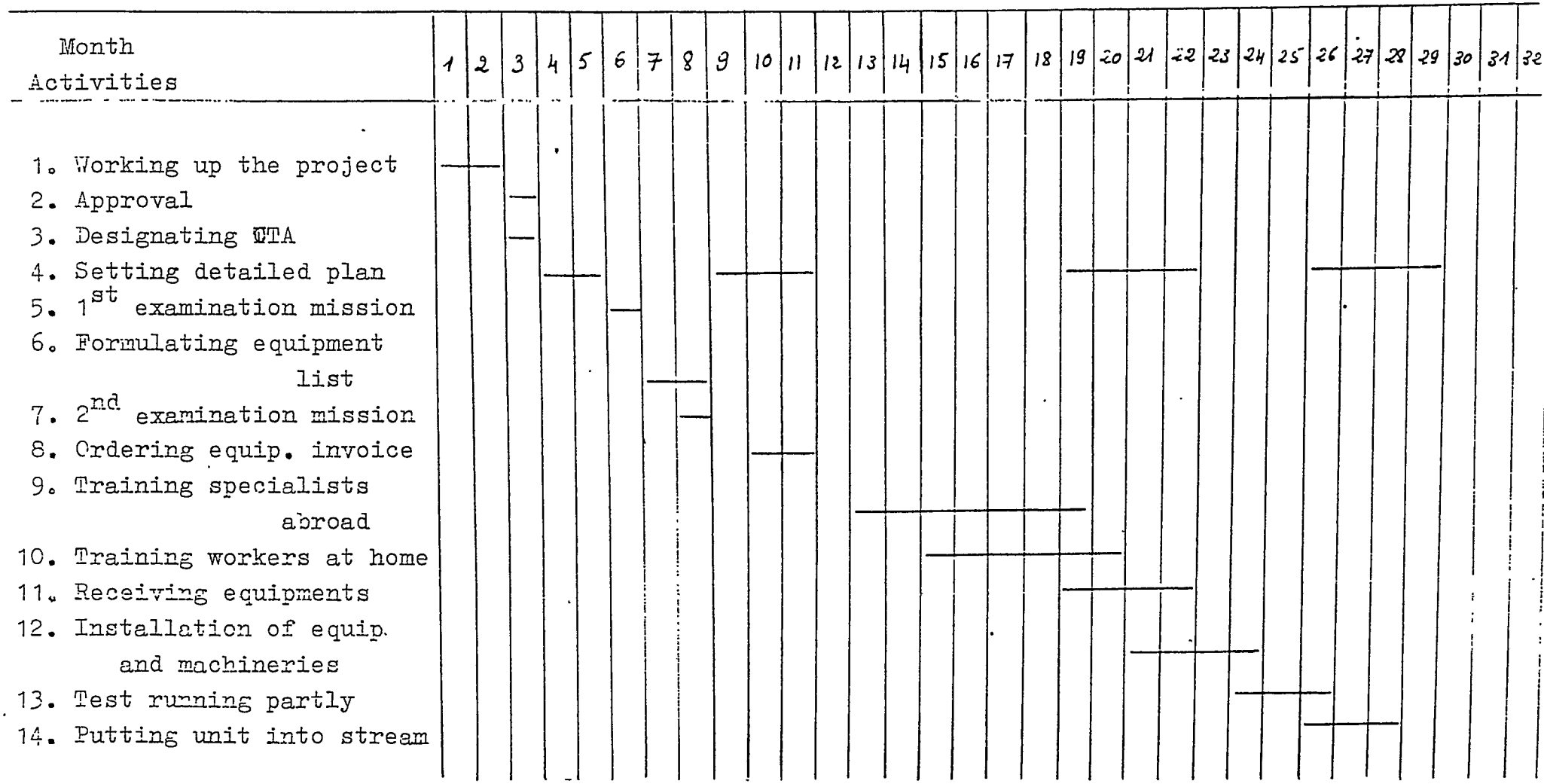
c) Equipments, Machineries

Chlorination system (including chlorination tower, pump , fan, waste chlorine treatment device....)	: :	:	250,000
Solvent separator and product recovery	: :	:	illegible
Laboratory instruments (grinder, chlorine analysator, pH-meter...)	: :	:	70,000

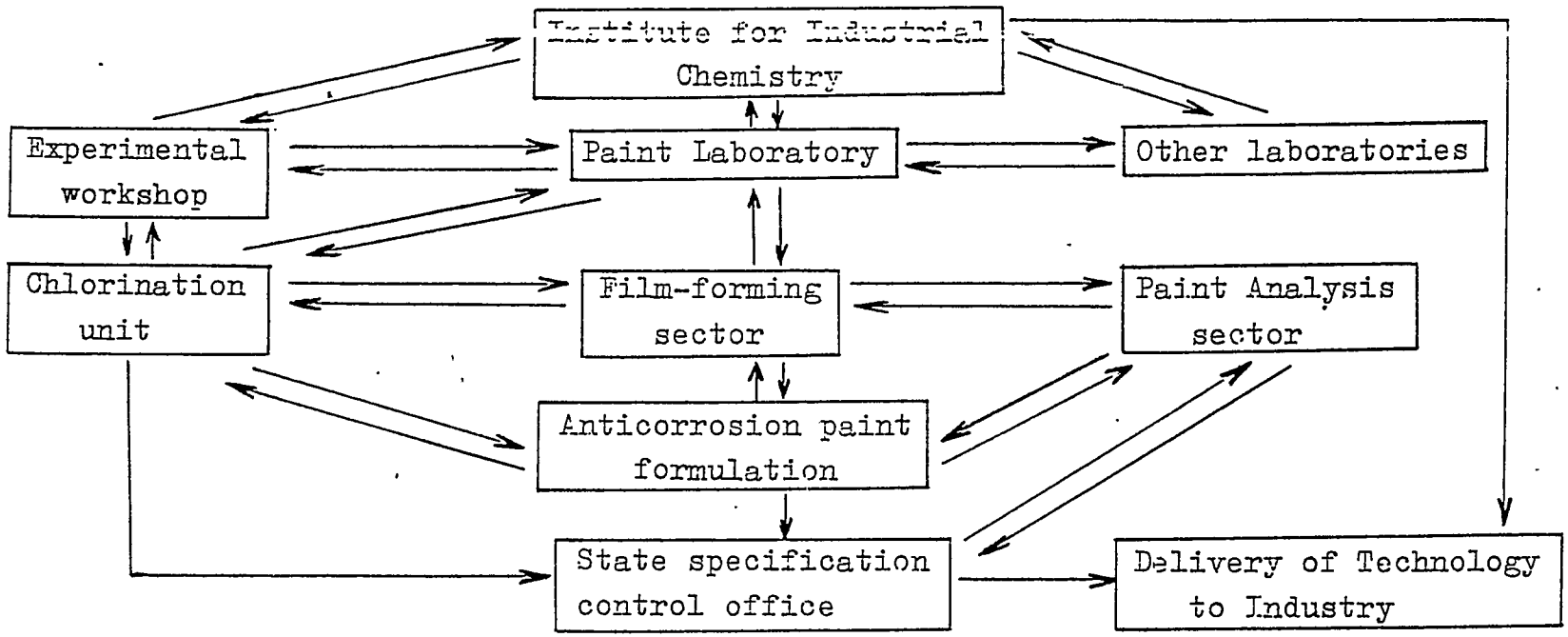
SCHEDULE OF EXPERIMENTAL MANUFACTURE OF CHLORINATED RUBBER



MASTER SCHÉDULE OF THE PROJECT



ORGANISATION SCHEDULE OF EXPERIMENTAL MANUFACTURE OF CHLORINATED RUBBER



CONTENT

PRELIMINARY PROJECT

PART IV. Appen dix on detailed project through schedule

1. Table on SRVN government investment share
   a/ Personel
   b/ Material background
2. Appendix on UNDP contribution
   a/ Expert's mission
   b/ Training
   C/ Equipments, machineries
3. Schedule of experimental manufacture of chlorinated rubber
4. Master schedule of the project
5. Organisation schedule of experimental manufacture of chlorinated rubber

ANNEX III

INSTITUTE FOR INDUSTRIAL CHEMISTRY

IN 1955, the Chemical Laboratory of Ministry of Industry and Trade was established on the basis of a newly-received and poorly-equipped laboratory of the former Indochina Mines Service, the number of its staff is approximately 20 technicians of primary and secondary levels. This is the initial germ of nowaday Institute for Industrial Chemistry.

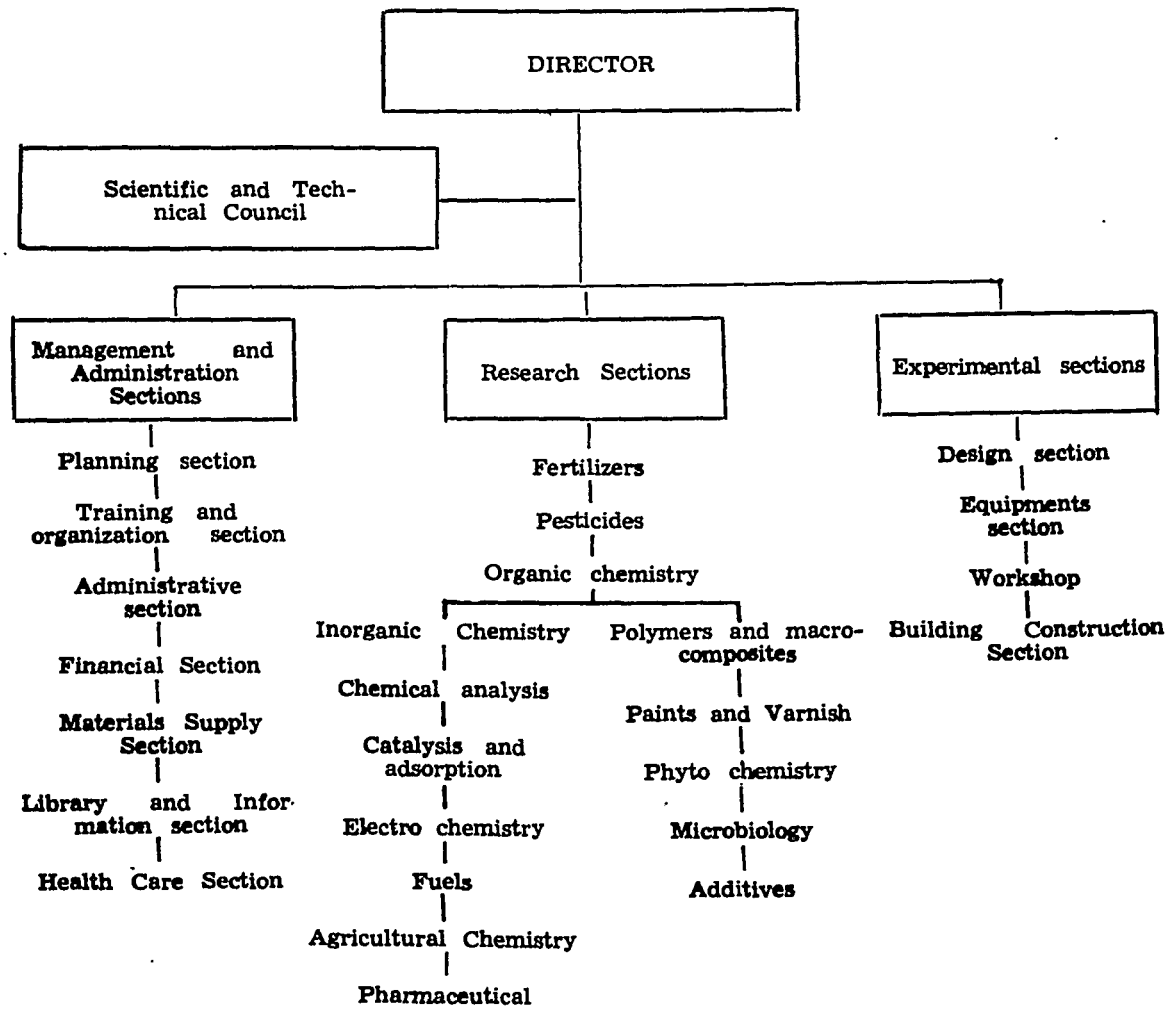
The Institute for Industrial Chemistry is a leading scientific and technical research centre of chemical industry of the country.

For above 30 years, the institute has passed through considerable steps on the way of development.

From the beginning, the research subjects for production of fused phosphate fertilizers, phosphate treated by brine vertical furnace cement refractory bricks, enamel tiles has been successfully implemented at Thanh hoa, Ha Bac, Phu Tho, Son Tay, Ha dong provinces. And through the activity of chemical products, the Institute has actively participated in the restoration and development of our national economy for the period of 1955 — 1959.

In the days of the anti — American aggressors resistance war we have to evacuate our Institute to Vinh Phu province. And in difficult conditions, laboratories were also set-up to continue scientific research activities. At the same time, many working groups were sent to factories such as Vietri Chemical Factory, Haiphong Cementary, Lamthao SuperPhosphate Plant... to carry out in continuing the technical subjects of research serving on due time to the struggle against American aggressors and to the development of production.

ORGANIZATIONAL CHART OF INDUSTRIAL CHEMISTRY INSTITUTE



The Institute for Industrial Chemistry has taken a more important role and actively contributed to our national economy. Many study and research results and scientific-technical progresses have been applied to agricultural and industrial production.

Many units within the Institute have been received the highest awards from the Government.

— In 1961: Fertilizers Section awarded Labor Medal 3rd class

— In 1970: Inorganic Chemistry Section, N.2 Group awarded Labor Medal 3rd class

— In 1975: Pesticide Section awarded Felicitation Diploma of the Government.

— In 1978: Pesticide Section awarded Labor Medal 3rd class.

— In 1979: Chemical Analysis Section awarded Felicitation Diploma of the Government

From the first days after the liberation of the whole country, the Institute has been sending many chemicals and equipments and many qualified officers also including Candidates of Science, Engineers and Managers have been assigned to go to the Central and Southern area of the country for contributing their efforts in management and administration of newly-received chemical factories and building-up the new ones there. Now, among them, many persons became Directors or Deputy-directors of Research Centres. Companies, Factories in Hochiminh City, Danang City and in other provinces.

The process of building-up and development of the Institute was closely linked with the efforts in training of their qualified staff. From the sixties, many groups of officer have been sent to Socialist countries to study, to participate in training and fellowship programmes for improving their knowledges. The total number of staff is actually 225 persons, including 3 professors, 2 doctors of science, 22 candidates of science, 28 qualified engineers and above 30 high level technicians and workers. Three post-graduated students have upheld successfully candidate of science title.

Thirty years have marked by the outstanding development of the Institute for Industrial Chemistry in number and qualification of their staff and their achievements in serving to the national economy. These are well-guided steps on the way to match with higher functional requirements of the Institute.

In 1985, the Institute had the honour of being awarded Labor Medal 2nd class.

Director	: Engineer	LE VAN NGUYEN
Deputy directors:	Candidate of Science	NGUYEN THI TAI ANH
	Professor	DO HUY DINH
	Candidate of Science	CAO VAN TU
	Candidate of Science	NGUYEN HUY PHIEU

Địa chỉ: VIỆN HÓA HỌC CÔNG NGHIỆP
Tổng Cục Hóa Chất
2 Phạm Ngũ Lão Hà Nội
CHXHCN Việt Nam
Tel: 53930

Address: INSTITUTE FOR INDUSTRIAL CHEMISTRY
General Department for Chemistry —
2 Pham Ngu Lao Street, Hanoi
SRV

Biên tập: PTS Ngô Huy Du

Ảnh: Đặng Đình An — Đào Đức Hoàng
Nguyễn Xuân Lăng

Trình bày: Họa sĩ Trịnh Trí

Ảnh bìa 1: Viện Hóa Học công nghiệp — cơ sở Hà Nội.

Ảnh bìa 4: Viện Hóa Học công nghiệp — cơ sở Cầu Diễn.

ANNEX IV

A) WORK ~~HAVE~~ HAS BEEN DONE.

I) Experiments in the latex phase.

To the point, we haven't any solvent as local material. Therefore we had tried to research producting the chlorinated rubber in the latex phase of all reactions steps of experimental producing process.

a Natural latex rubber, which has been taken out of rubber stre trees, contains about 30°/o of dry subtances.

a1- ~~It was put~~ some additive chemical agents were put in to modify rubber as anhydric acetique; Coban acetate, H_2O_2 , emulgator and with or without ulviolet ray.

a2- The reaction solution has been warmed up to 70° - 80°C with frequent stir by serious times (from 4- 10h)

a3- The finished reaction solution was stood still to separate itselft in two phases. The first phase contained reaction product of modified rubber, which was then coagulated by alcohol as C_2H_5OH (90°) and washed by warm water and at least dried by over in the temperature of 60°C under vacurim-condition.

a4- Dried substance was combined with dry-oil (unsatate oil) by 120°C and received end product, which was able to formulate good film of verni.

b b1 The product of first phase of a_2 was mixed with HCl of 20°/o concentration to set up the reaction-solution, which had the acid concentration of 8 N.

b_2 The reaction of rubber-solution and chlorine gas under the condition of temperature of from 40 ° -to 80°C was carried out from 10 - 16h and an ultraviolet lamp.

b_3 This work achieved a product of white powder, which has the chlorine cintent from 56 - 61 °/o, its dissolubility were positive in aromatical hydrocarbon solvents in the cases of carrying out experiments of lab. scale of 200 g of chlorinate rubber once.

b4 - On the other hand, if the experiments was were fulfiled on

the scale of over 200 g once, we received the product of chlorine content of also 56 - 61°/o but its dissolubity were bad in solvents.

b_5 - To solve the bad dissolubility, we put dried powder of chlorinated rubber in tetrachlorid carbon in the concentration of 10°/o in weight.

b_6 - Chlorine gas has been leading to the point, when the reaction solution show quite clear.

On the way, we receive results with chlorinated rubber, which contain 60 -61°/o of chlorine, and dissolubility is good in aromatic solvents.

Through carrying out the experiments in later phase, we noticed that :

1) During the reaction process, the dispersived particles bend up each other to bigger, some time it might be as big as a marble. This unwilling phenomena was dangerous for stir.

2) The dispersived solution had been destroying slowly during leading time in chlorine gas. The contact between tow phases: solid particles and water went more smaller and after smaller to the time of reaction.

3) Noticing the point of endreaction rapidly was rather difficult.

4) What kind of equipment is suitable to use in cases, ultraviolet light as a reagent katalysator.

5) We have been always thinking about the method to measure quickly and the chlorine content during the process.

6) The experimental work in two steps of chloranation-reaction was complicated and uneconomic.

2) Experiments in the solvent phase.

- Natural rubber in the form of sheet or crum, block was depo-

lymerred by three miles machine to the soft indicate of 5
- depolymered rubber was cut in small pieces and dissolved in CCl_4 with the concentration of 5°/o in weight in equipment, whi which has stir, condense.
- To have desirious viscosity of the reaction-solution we put Copper salt (Custearate or $CuSO_4$ in BuOH; or $CuCl_2$ in BuOH) to this reaction solution and carried out in temperature of 80°C to the point, when we have got the disirous viscosity.
- Chlorine gas was led with serious speed, At first 100g chlorin 150g dry rubber. This speed would be slower after at least 100g chlorine/300 g dry rubber.
- It was shown that the viscosity of solution go down during the process. At the finished point of reaction the solution is quite clear.

This experiment was carried out in the equipment size of 10 kg chlorinated rubber per/batch.

The chlorine content of end product of both in lab and in work done was only from 59 - 61°/o Cl_2.

Work of equipment size of 10kg product has the recuperation of solvent in less than 50°/o.

Toxic waste gas is not sufficiently treated.

Through experimental work we noticed that:

1) The recuperation of solvent was quite slow.
We suppose that the used equipment is not smooth and even by closing of equipment, the solvent and chlorine gas go out very much during the process.
2) We weren't able to know rapidly the chlorine content of product during the process in order to decide the end point of process.
3) We are always thinking about the analysable method, which is used in the practice to notice immediately parameter of reactions.
4) We always like to know, what kind of pumpe is suitable to use.

5) How do people treat economically the waste gas.

We have done the experiments on equipment in size of 10kg per batch in 1979 - 1982 . Since 1982 we have been only studying of the chlorination of serious kind of rubber in order to use the slow quality kind to produce chlorinated rubber.

Experiments show that even using of the 5th and 6th quility of rubber we can receive dissolable product, but we must need the step to separate dissolved subtances.

This work only has been doing only in lab, because we have only porzelan, a hungarian reaction-equipment made out of porzelan, which was broken by someone in 1982.

The product of experiments by using 5th quality kind of rubber has chlorine content 59-61%.

We haven't still yet do with stabilisators of chlorine products.

Capacity of chlorine in Viêt Nam

Factory	at present		after 1995	
	Capacity	no use		
Viêt trì	3000	1500	8900	5000
Biên hoà	2300	1500	7064	
Bãi bằng (fac.of paper)	4000	2000	6600	
Da nẵng	200	200	445	
Dông nai	800	400		
Thanh binh	480	0	1600	
Thanh hướng	480	0	1600	
Biên hoà	480	0	1600	

Capacity of rubber

Total 1988	45 - 46.000 tons
- 1990	55.000 -
- 1995	139.000 -
- 2000	237.000 -

Quality of concentrated latex.

Dry content (DRC)	= 60,0°/o
Total subtance content(TSC)	6I,5
TSC - DRC	2,0
Amoniac content of latex	I,6
Mechanical stable time (MST)	475
KOH indicate	0,8
VFA	0,2

Quality of rubber in form of block

I- mixtured subtance	°/o	≤ 0,05	0,05	0,10	0,2	0,5
2- Ash content	°/o	≤ I,00	I,00	I,00	I,00	I,00
3- Vaporous content	°/o	≤ I,00	I,00	I,00	I,00	I,00
4- N_2 content	°/o	≤ 0,60	0,60	0,60	0,60	0,60
5- Soft indicate (Po)		≥ 30	30	30	30	30
6- PRP		≥ 60	60	50	40	30
7- Colour indicate		≤				
8- Raw materials		liquid-latex		sub.latex		

In I980 we built a pilot unit to produce chlorinated rubber with a capacity of IO kg per batch in a factory of the general department of chemistry. We achieved the product with a highest chlorine content of 6I°/o and solvent recuperation in less than 50°/o. The toxic waste gas was not sufficiently treated. The technology included the following process stages :

1. Breking
2. Dissolution
3. Chlorination
4. Solvent recovery
5. Hydrogen chloride absorption
6. Drying
7. Packing

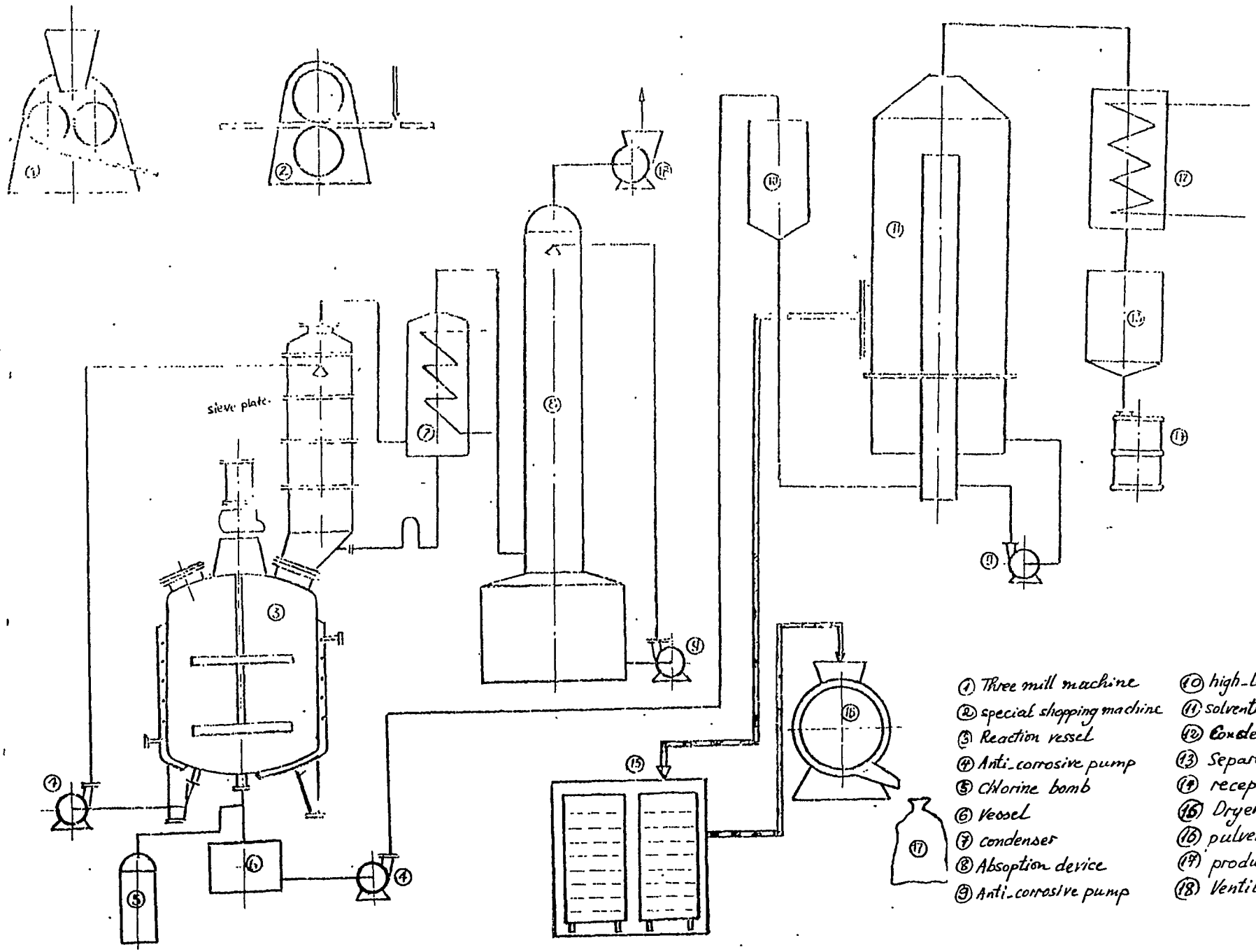
In the producing process we discovered some disadvantages of our unit. Firstly we hadn't a suitable reaction equipment so reaction's time was too long. Secondly solvent recovery equipment could only be usedin a pilot unit with small capacity. We got had these disadvantages because partly we did not had anti-corrosive equipment and machine.

Now we intend to build a new pilot unit with capacity of 2oo-500 kg per batch (2oo kg per batch.) We'll use the improved method which is taken out of the old method. For example: to shorten the reaction's time, we'll design a new equipment using a anti-corrosive pump to raise the surface-substance-exchange. We also add to this unit some devices to reduce the manuel labour. (See the following cheme)

The caoutchouc bales will be breaked by a three mill machine(I) then They will be shopped in pieces by a special shopping device (2) Those pieces will be put into a stirred vessel (3) that called the reaction vessel. The solvent CCl_4 will be poured into it. The stirred vessel starts to work to dissolve the pieces of caoutchouc. Water steam is used in this case to raise the temperature of the fluid caoutchouc to 6C°C and 65°C.Then chlorine from chlorine pressed vessel is put under the caoutchouc solution. It will go through this solution up. Here there is a reaction

which called the chlorinated process of rubber. In the reaction process the revulutionary pump 4 is working to rice the reaction surface and to reduce reaction time. One part of chlorine combines with rubber and the other picks the solvent up and gets away. Chlorine and boiling solvent will go through the condenser (7) The boiling solvent will form the fluid solvent and will go back into the vessel. Chlorine will go through pipe into column (8) Here it is absorbed by caustic soda. The viscosity of the solution is tested. When the solution has a certain viscosity, the reaction finishes. So we receive the solution product. The pump (4) is used to transport the solution product intc vessel (IO). This product from vessel IO is put into the device (II). Here the product and the solvent are separated. We gain the product and recovered solvent. The product is dryed in the dryer (I5) then it is put into pulveriz-machine and is packed. The recovered solvent goes back into reaction-vessel.

We've intended to build the pilot unit many times, but we haven't carried out ~~it~~ because we have got a lot of difficulties. We have had the corrosion-resistant equipment and machine such as anti-corroaive pumps and reaction-vessel.


sieve plate
① Three mill machine
② special shopping machine
③ Reaction vessel
④ Anti_corrosive pump
⑤ Chlorine bomb
⑥ Veosel
⑦ condenser
⑧ Absoption device
⑨ Anti_corrosive pump
⑩ high_level_vessel
⑪ solventrecovery device
⑫ Condenser
⑬ Separation vessel
⑭ receptacle
⑯ Dryer
⑯ pulverize_machine
⑰ product_bag
⑱ Ventilator

SOLVENT RECOVERY SYSTEM

The solvent recovery equipment was a device which we discovered during our thought and experiment process, that's why the device couldn't be found in any book. We knew spray dryer was used to recover solvent in modern production process of chlorinated rubber. But the spray dryer needed a corrosion-resistant geer pump. We hadn't this special pump so we had to design the following device. Although an old method was used we gained good results in the process steg:

- We received the clean chlorinated rubber without solvent.
- Solvent recuperation was about 90°/o (we wanted to note that in the whole process we achieved solvent recuperation of only 50°/o, because our reaction vessel was leaking)
- Acid content of product was low because product was easy to wash in the device.

The following scheme showed the working process of the device which was used by us at that time :

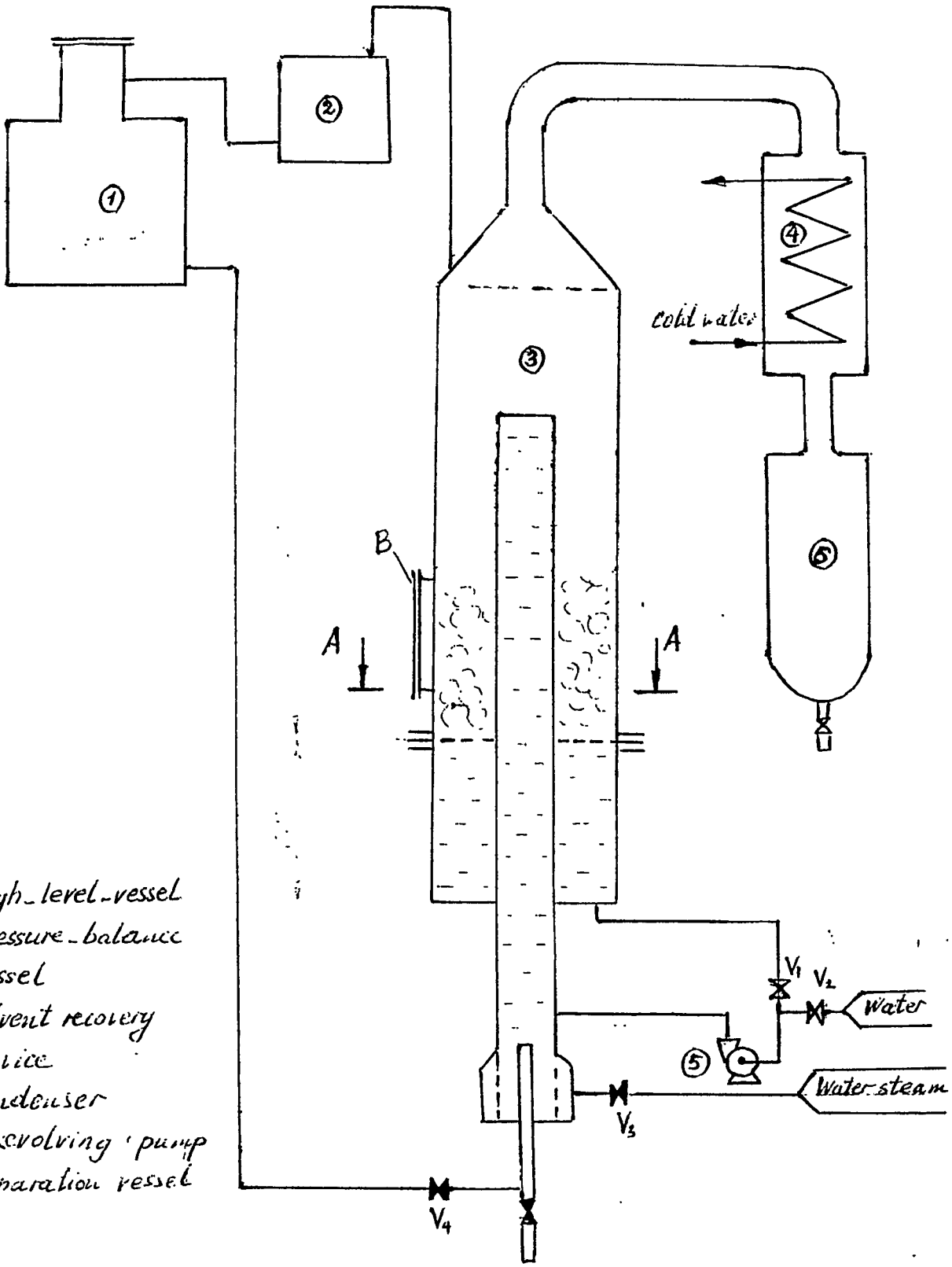
The chlorinated rubber solution was put into vessel (I) The pump (5) was used to fill the device (3) with water up to level A-A (valve V_1 was shut; V_2 was opened). After that the pump (5) started to put the water in the device into circulation (V_1 was opened; V_2 was shut). At this time water steam was put into the device (3) to heat the water (V_3 was opened). After 15 minutes the whole water of the device could achieve the essential temperature, so we opened the valve V_4 slightly to put the solution into the device.

The solution met water steam and a turbulent water stream there (in the great middle pipe of the device) The turbulent water stream tore drops of the solution so that the solvent could be able to steam faster under effect of the heat of water steam. In brief due to influence of water steam and turbulent water stream solvent of solution steamed out and chlorinated rubber formed a comik form without CCl_4 then

it went over the pipe and fall douwn to the sieve of the device. When the solvent steam and the water steam went through a condenser (4) they are condensed into the fluid solvent and water. Then they flowed down the vessel (6). Water and solvent are insolubla so it's easy to pick solvent CCl_4 out.

After the vessel (I) of solvent was emptied, the door was opened then chlorinated rubber was pick up.

Vessel (I) and device (3) were connected by a pipe to balance the pressure between (I) and (3). The balance of the pressure was an important condition for a steady stream of solution. The steady steam affected the quality of product (the product without solvent).



① High-level-vessel
② Pressure-balance vessel
③ solvent recovery device
④ Condenser
⑤ revolving pump
⑥ separation vessel

Solvent recovery system

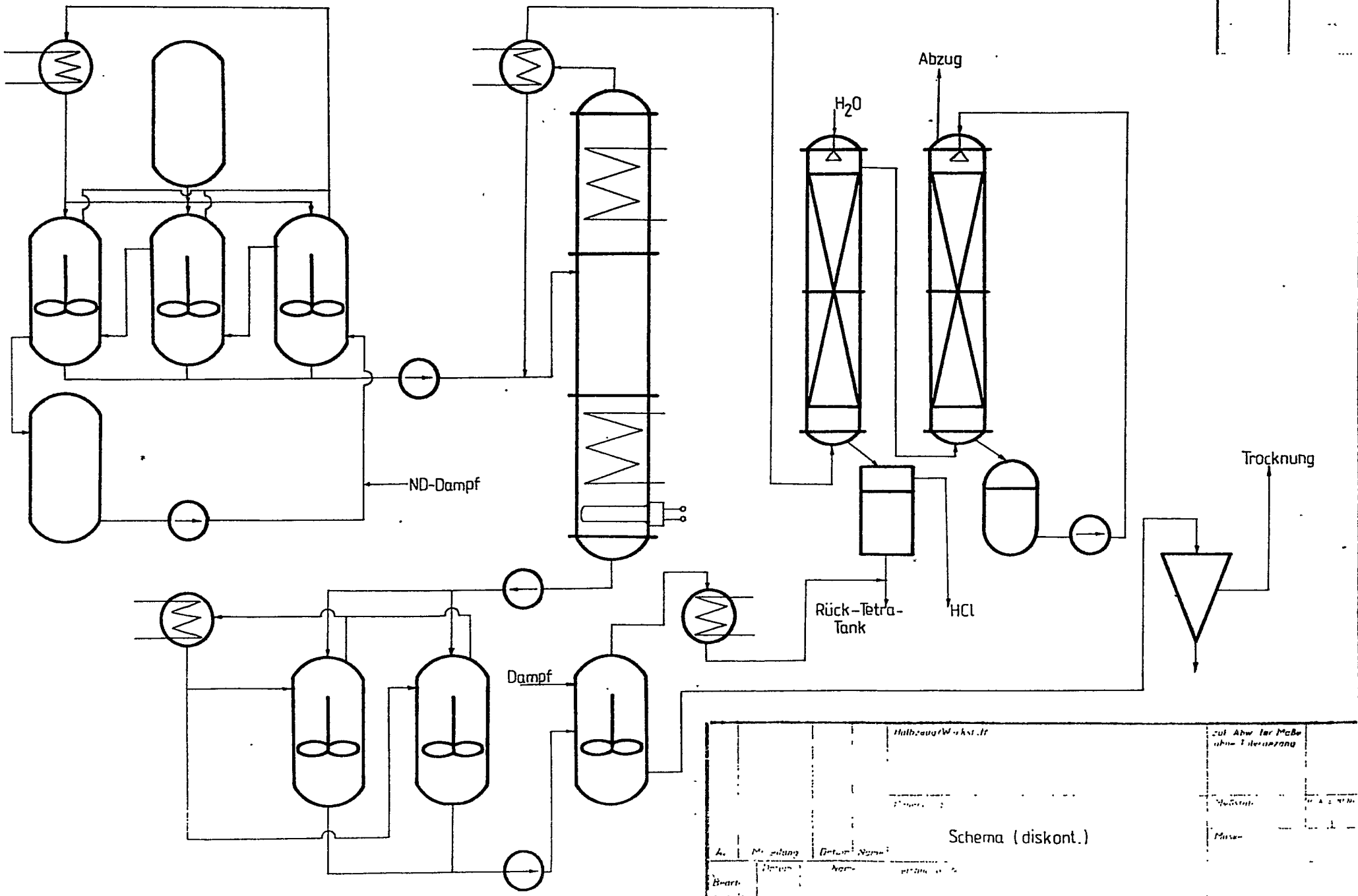
ANNEX V

No.	Equipment and machine	Supplies		Price US$
1	- Stirred vessel (glass lined steel) V: 2501	EHW-Thale	5	140,000
2	- Stirred vessel (glass lined steel) V:25001	EHW-Thale	1	50,000
3	- Chlorination columm (glass) 300x3000	VEB Jenaer	1	5,000
4	- Columm 100x2000 (glass)	VEB Jenaer Glaswerke	2	6,000
5	- Separator (glass) 300x1000	VEB Jenaer Glaswerke	2	3,000
6	- Coroboncendenser F:5m2	Chemickombi-nat Bitterfeld	3	15,000
7	- Filtration device	TH-Merseburg	1	1,000
8	- Coroboncondenser F: 11m2	Chemickombi-nat Bitterfeld	1	8,000
9	- Plunger membrane pump 200 l/h	Salewedel	2	15,000
10	- Plunger membrane pump 1000 l/h	Salewedel	2	8,000
11	- Centrifuge pump	Erfurt	2	4,000
12	- NaOH-Circulation tank (glass lined steel)		1	5,000
13	- Glass pipe (with Gaskets and flanges)	VEB Jenaer Glaswerke		10,000
14	- Dryer F:20m2	Ofenban Aken	2	20,000
15	- Tetra-tank	Rudisleben	1	5,000
16	- Electro-measuring device	GRW-Teltow		25,000
17	- Lab-apparatus	MLW		80,000
	Total price			400,000 US$

All apparatus and equipment for the pilot unit will be designed, and delievered, and the technology will be transferred with following conditions

The project must pay the designing, engineering prices (see as know-how price) about 375,000 US Dollars.

If the project will have a pilot unit with the continous technology producing chlorinated rubber, the price of know-how must be 1,100,000 US Dollars.



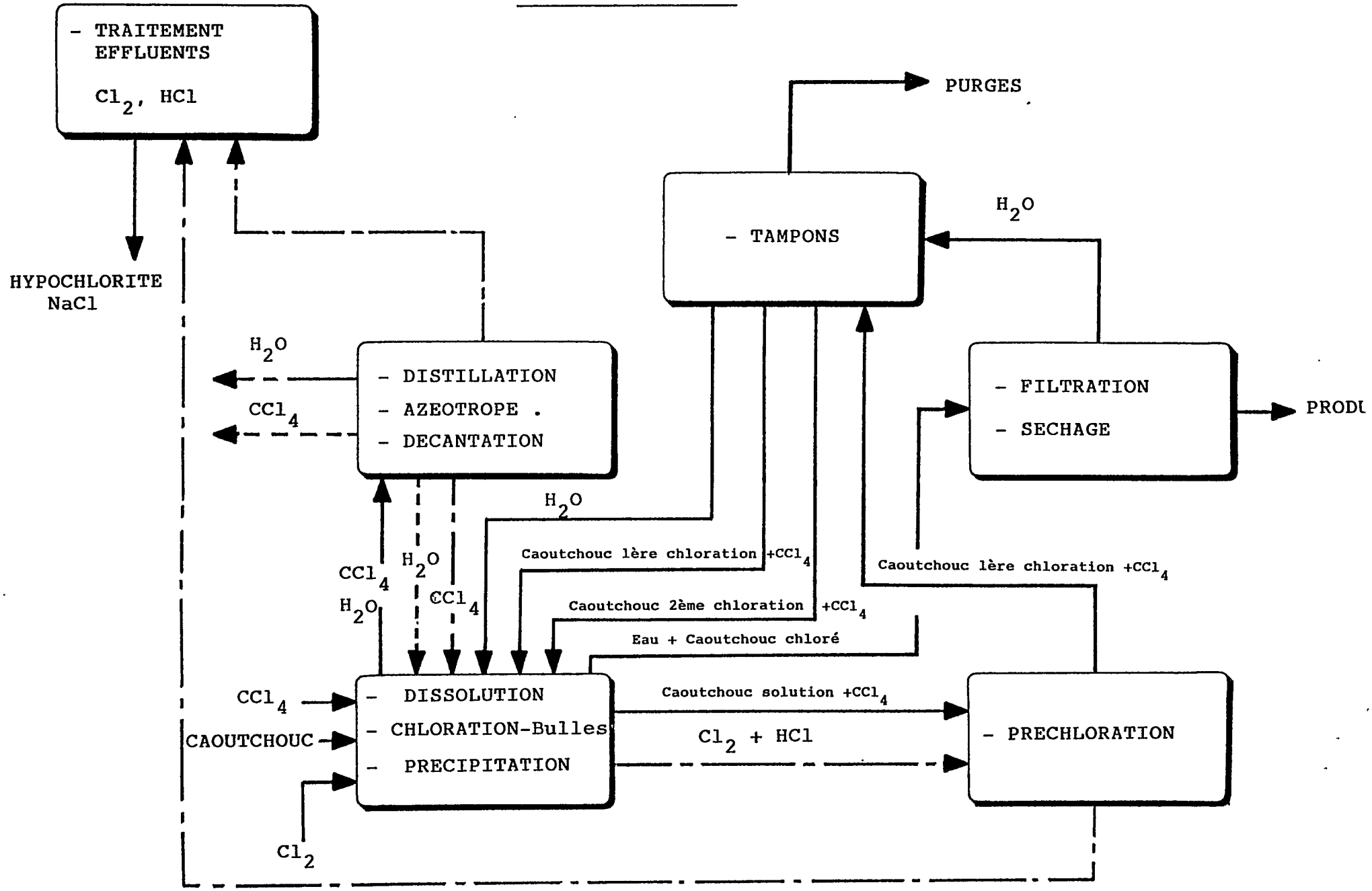
Schema (diskont.)

ANNEX VI

PILOTE DE CHLORATION DU CAOUTCHOUC

De Dietrich
Division Equipement Chimique
Chimie Développement International

SCHEMA DE PROCEDE



République Française

Institut Français du Pétrole

Centre d'études et de développement industriels
Solaize

N° 3175
REÇU LE 28 MAI 1990

Monsieur DE LIVONNIERE
INSTITUT DE RECHERCHE SUR LE CAOUTCHOUC
42, rue Scheffer
75116 - PARIS

national 78.02.20.20
international +(33) 78.02.20.20
Poste _____

SO40. JPW/MD. n°L80A

Objet : Chloration d'élastomères
Projet IRCA - DE DIETRICH - VIETNAM

Solaize, le 22 mai 1990

Cher Monsieur,

Nous poursuivons nos consultations à l'I.F.P. concernant l'affaire citée en référence.

M. DAWANS, Directeur de la Direction de Recherches "Matériaux et Chimie Appliquée", qui détient une bonne part des acquits de l'I.F.P. en matière de chloration profonde des polymères (en particulier polybutadiène 1-2) se propose de vous rencontrer pour discuter avec vous de certains points qui mériteraient d'être approfondis :

1 - l'interdiction d'utilisation du CCl_4
2 - la détection du CCl_4 résiduel
3 - l'utilité d'une dépolymérisation partielle préalable à la chloration
4 - les produits de la concurrence.

Voici ses coordonnées :
M. François DAWANS
INSTITUT FRANCAIS DU PETROLE
1 & 4, avenue de Bois-Préau
B. P. 311
92506 - RUEIL MALMAISON Cédex
Téléphone : (1) 47 49 02 14 Télécopie : (1) 47 49 04 11

M. DAWANS s'est entretenu préalablement du sujet avec M. PAUTRAT (IRAP). Cette rencontre devrait permettre d'examiner si le know-how disponible à l'I.F.P. est suffisant pour entreprendre le développement du procédé. Si tel n'était pas le cas, il paraît très peu probable que l'I.F.P. entame de nouvelles recherches sur le sujet.

Nous vous prions d'agréer, Cher Monsieur, l'expression de nos sentiments distingués.

J. P. WAUQUIER

Autoroute Lyon-Vienne
Echangeur de Solaize-Vernaison

Institut Français du Pétrole-CEDI
Boîte Postale 3

Télex IFP SOL 340257 F

liste des equipements de laboratoire pour la chloration des NR

Description	FF	US $
2 Armoire pour distribution de chlore	77000	
2 Reacteur complet de 1 l		
1 Reacteur complet de 3 l		
Refrigerant		
4 chauffages thermostat à circulation		
4 Viscosimetres Ubbelhode	3500	
Refrigerants		
35 Unité de distillation de solvant	10500	
bain thermostatique		
etuve		
pompe à vide		
dosage du chlore	55000	
titrimetre		
pompe		
34 Agitateurs magnetiques chauffant complet	4000	
verrerie de laboratoire : flacons, ~~becher~~ bechers, et ~~burettes~~ et..		
chauffe ballon	2500	
2 Balance electronique d'analyse 0-162 g	22300 x 2	
Thermocouple		

		FF
-6-86	Tronc à outil	2500
03-85		1300
	PH mètre	2500
	Electrode	500
-02-86	Etuve ventilée	10000
-7-86	Thermostat à immersion	3200
	Conisiomètre Mooney	
	Plastomètre Wallace	
	Tronc outil	1h00
	Perceuse	1484
-11-87	Rétroprojecteur	4200
-11-87	HPLC	160000 F
10-87	+ Microordinateur	21 0001
	+ Colonne gel	420
12-87	Alimentation stabilisée	35400
-10-87	Etuve à vide	18200
10-87	Infrarouge	188000
0>87	Réacteur thermostaté complet 1L	1h0002
3 84	avec agitation	
-10-87	Projecteur	10700
10-87	Balance de précision Sartorius	31200
	Turbidimètre	15000
	Thermomètre à contact	2700
10-87	Balance 0-160g	22300
10.86	Tronc (découpe Labo Mooney)	9000
9-86	Thermocouple	2300
n-88	Pompe à vide avec pièges	7000
//	Appareil de distillation	6000

		FF
	Matériel	
8.87	Noix	
	Tige	
	Capsule	4300
	pipette	
	fiole	
	pompe à vide	2400
	agitateur à variateur	4500
	cuve verre	1200
	Thermomètre	200
	Réfrigérant	500
	Ampoule à décanter	500
	gant	
02.87	Thermostat à immersion	5280
	Balance Mettler	18000
11.88	Electrode pH-mètre	700
	100 erlenmeyer Erlenmeyer	12000
0.34	Verrerie de labo	12000
10.86	Plaque chauffante	5330
11.76	Ballon + erlen + erlenmeyer + Soxhlet (SOXHLET)	5500
10.86	Cristallisoirs boîte de Petri verre de montre	6200
4.86	Balance mettler avec imprimante	27000
6.86	Multimètre numérique	800
07.86	PH-mètre portable	2200
	Etalon pH	340
1	PH-mètre LCD	3300
	électrode + cable	800
11	Dichromate 6000g	9200

5.85	matériel labo distillation	10500
"	Autre matériel thermomètre collier tube	13000
02.85	Gants protection	2600
08.84	Thermostat à immersion	2850
12.84	Valve régulateur air comprimé	1500