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**KEMIRA**

Helsinki 10.6.1987

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FINAL REPORT

UNIDO PROJECT No. DP/PAK/83/010

UNIDO CONTRACT 84/124

Contract between the United Nations Industrial Development Organization (UNIDO) and Kemira Oy for the provision of services relating to the establishment of a Fertilizer Research and Development Institute (FR & DI) in Faisalabad, Pakistan.

K. Sarkkinen
Director for International Projects

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| cc. | UNIDO | Vienna (50 copies) |
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| | Lähdemäki | " |
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| | Karhunen | " |
| | Korvela | " |
| | Saari | " |
| | van der Meer | " |

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# 1 SUMMARY AND CONCLUSIONS

According to the contract, Kemira Oy has provided

1) project area service 35 man-months and completed the field work
2) training of FR&DI fellowship holders, 9 persons in in two groups, a total of 100 man weeks of training in Kemira Oy laboratories and
3) home office support service and full backstopping through the whole project duration 1.1.1985-15.3.1987.

The Fertilizer Research and Development Institute (FR&DI) is now in full operation doing independent research and development work in fertilizer field.

Conclusions and recommendations

1. A steering committee in NFC for the evaluation of R&D projects and for backstopping the research and development work for FR&DI is recommended.

2. Organizational changes and job descriptions recommended by the Kemira team should be implemented at the institute.

3. Information concerning the research equipment and the possibilities of FR&DI to perform demanding R&D work should be distributed to all NFC units.

4. The information inside the Institute concerning the research work and other activities should be improved by regular meetings, etc.

5. The beneficiation studies are going very well, still Kemira recommends that special emphasis should be placed on these studies which are of particular importance to NFC, and also other mineral resources than phosphates should be considered.

6. Very good progress has been made also in phosphoric acid tests, SSP granulation, NPK granulation pilot-plant and CAN caking studies which all are important subjects for the company.

7. For corrosion studies a qualified researcher with full understanding and good experience of corrosion field should be recruited.

8. An online information service with connection to international databanks is recommended.

9. More laboratory technicians or assistant chemists are urgently needed.

10. A good co-operation network within NFC companies concerning creation of new research ideas and the use of research results should be organized.

11. The institute is now well equipped for the existing fertilizer research although there is always a shortage of some routine equipment like pumps, balances, stirrers and spare parts in research laboratories. The need of additional equipment should be considered carefully in the future according to the research programme.

12. The extension of the Institute building (Phase 2) should not be considered until the Institute has been in full operation for 2-3 years.

13. Prompt completion of the fertilizer pilot plant under construction is of utmost importance.

## 2 INTRODUCTION

A contract entitled "Contract between the United Nations Industrial Development Organization and Kemira Oy for the provision of services relating to the establishment of a Fertilizer Research and Development Institute (FR&DI) in Faisalabad in Pakistan" was agreed to be effective from 29th December 1984, the UNIDO contract No. 84/124, project No. DP/PAK/83/010. Three activities were covered by this contract:

1. Project Area Services assisting the NFC in creating a Research and Development Institute capable of performing the following research:
   a) Phosphatic Fertilizer Manufacturing
   b) Testing of Domestic and Imported Raw Materials
   c) Fertilizer Anti-caking, Antifoaming Agents and other Chemicals
   d) Analytical Devices and Methods
   e) Corrosion Research.

2. Training of Pakistani Fellowship Holders in Finland.

3. Home Office Services supporting the project.

This is the Final Report of the work carried out by Kemira Oy during the period 1.1.1985-15.3.1987.

This report describes the three field missions

1) 9.2. - [illegible].3.1985
2) 8.2. - 22.2.1986
3) 2.9.1986 - 14.3.1987,

the training of two groups of Pakistani fellowship holders

I 23.9. - 29.11.1985
II 14.4. - 4.7.1986

and the home office work done throughout the whole project.

The recommendations for future activities are given in connection to the descriptions of the research projects.

## 3 THE FIRST FIELD MISSION

The mission of five Kemira experts for three weeks took place 9.2.1985 - 2.3.1985. The major targets for this mission were: to get familiar with the general situation in the project, to meet the personnel of NFC and FR&DI and to get acquainted with them, to visit NFC fertilizer plants and assess the research needs and problems for the preliminary research programme, to visit selected research organizations in Pakistan and to examine the equipment list of the Institute.

During the mission the Kemira team had 8 discussions and planning meetings in Faisalabad, 7 d fact findings at five fertilizer plants (PAK Saudi, PAK Arab, LCF Faisalabad, LCF Jaranwala, PAK China) and visit to phosphate mine in Hazara, 4 visits to the universities and research institutes and 2 discussions at NFC head office in Lahore.

Based on the first field mission the equipment list A consisting of 136 items was prepared and a preliminary research programme of 25 different R&D problems was listed. Kemira team made suggestions for modifications on drawings of the laboratory and proposals for working benches, shelves, fume cupboards etc. Also some changes in the organization of FR&DI was discussed to get a modern and effective R&D institute with less hierarchy and sufficient lab.assistant staff. A tentative training programme for Pakistani fellowship holders in Finland was planned. The operational plan of the UNIDO/Kemira Contract 84/124 was discussed thoroughly and agreed upon. The civil construction work of FR&DI was obviously late, but it was discussed with NFC that the construction work would be promoted and the laboratory would be ready for the installation of equipment at the end of October 1985.

## 4 TRAINING OF FR&DI FELLOWSHIP HOLDERS

In the first programme the training was planned for one group of 9 persons during 1.4. - 30.6.1985. Due to the lack of suitable trainees in FR&DI it was agreed during the first mission that training will be given for two groups. The first training group of 4 members: Dr. Anwar ul Haq, Mr. Ishtiaq Ahmad, Dr. Mehboob Sheykh and Mr. Muhammad Daud Ali Alvi was in Finland 10 weeks from 23.9. to 29.11.1985. The second group: Mr. Liaquat Randhawa, Dr. Saglain Bhatti, Mr. Essa Bhatti, Mr. Kamran Sibtain and Mr. Azmat Ali was in Finland 12 weeks from 14.4. to 4.7.1986.

Training for both training groups was given effectively and it was regarded as a very useful and necessary phase for the implementation of FR&DI.

## 4.1 General training

The general training consisted of introduction to the activities of Kemira in general and visits to Kemira headquarters, Kemira Engineering and Kemira's Experimental Farm, introduction to the research activities of Kemira in Espoo Research Centre and Oulu Research Laboratory, visit to Kemira Siilinjärvi phosphate mine and fertilizer plants as well as Kemira's Oulu and Harjavalta Works.

All participants got familiar with common research methods and organizing of R&D including: planning of long-range, medium-range and short-range research and budgeting of R&D projects, characteristics of productive organization, effective channels for organizational communications, steering of R&D in the company, staffing policy of R&D organization, effective management control techniques and techniques to improve productivity and innovation.

## 4.2 Individual practical training

Dr. Anwar ul Haq
- Organizing of R&D
- Pilot plant designing and scale-up
- A study of NPK pilot plant
- A study of $H_3PO_4$ pilot plant
- Production problems of Multan.

Mr. Ishtiaq Ahmad
- Theoretical evaluation of phosphate rock
- Evaluation of phosphate rock for SSP & TSP manufacture
- Bench scale manufacture of phosphoric acid by dihydrate process
- Phosphoric acid concentration test
- Ammoniation of concentrated phosphoric acid for the manufacture of MAP & DAP
- Evaluation of Pakistani phosphate rock for phosphoric acid manufacture by hemihydrate process
- NPK pilot operation.

Dr. Mehboob Sheykh
- Corrosion prevention techniques in fertilizer industry
- Anti-foaming agents and methods for their utilization
- Testing of different fertilizers and organic analysis
- Production problems at plants in Pakistan.

Mr. Mohammad Daud Ali Alvi
- Corrosion prevention techniques
- Evaluation and testing of anticorrosive chemicals
- Evaluation and testing of water treatment chemicals
- Preparation of active ingredient of biocide and flocculants for water treatment
- Blending of water treatment chemicals
- Quantitative analysis of water treatment chemicals
- Qualitative analysis of organic compounds
- Production problems at plants in Pakistan.

Mr. Liaquat Randhawa
- NPK-fertilizer pilot-plant operations and physical testing of fertilizers
- Caking tendency evaluation
- R&D Methods of manufacturing of mixed fertilizers and microscopic studies of caked/uncaked CAN
- Calculation of recipes for various grades of fertilizers
- Pilot-plant designing and scale-up
- Crystallization and designing of crystallizer starting from laboratory scale data
- Fertilizer processes for NP/NPK/PK
- Discussion about the reasons for Multan CAN caking.

Mr. Essa Bhatti
- NPK-fertilizer pilot-plant operations and physical testing of fertilizers
- Caking tendency evaluation
- R&D methods of manufacturing of mixed fertilizers and microscopic studies of caked/uncakes CAN
- Calculation of recipes for various grades of fertilizers
- Testing of Pakistan CAN-samples of caked and uncaked fertilizers
- Crystal phase transformation studies
- Fertilizer processes for NP-NPK-PK fertilizers
- Discussion about the reasons for Multan CAN caking

Mr. Kamran Sibtain
- Discussion of Pakistani phosphate rock problems; theoretical lectures & text-book studies
- Practical work on communition and sieve analysis
- Flotation studies & practical work on Siilinjärvi rock
- Petrographic & optical microscopic studies of Siilinjärvi rock; particle size analysis methods
- Magnetic separation, x-ray diffraction and fluorescence, electron microscopic studies
- Acquaintance with bench scale $H_3PO_4$ equipment and studies
- Study of literature collected concerning beneficiation
- Studies of Pakistani rock samples.

Dr. Saglain Bhatti, Mr. Azmat Ali
- Analytical studies on nutrient analyzer and Karl Fischer titrator
- Introduction to instrumental organic analysis IR, UV, NMR, mass spectrophotometer
- Analytical studies on automatic titrator, ion analyzer, turbidometer, sulphur analyzer
- Spectrophotometric methods
- Kjelldahl automatic analyzer
- Various sample preparation methods and trace element analysis on atomic absorption
- X-ray diffraction of fertilizer and rock samples, preliminary x-ray fluorescence studies
- flame photometer analysis.

## 5 THE SECOND FIELD MISSION

The purpose of the second field mission was to plan together with the counterparts the start-up and the activities of the FR&DI for the first year of operation. The major activities during the visit were: following the civil construction work and equipment deliveries and guidance on material selection, generation of research ideas and planning of research programme for the first year of operation, checking of the recruitment of personnel and starting of the training programme in Pakistan.

The second field mission of three Kemira members for two weeks took place from 8.2. to 22.2.1986. During this mission Kemira team had four planning meetings with director Fazili and the research programme was agreed upon, also the second training group was selected and the list B1 consisting of 18 items, was prepared. Discussions concerning the organization and staffing of FR&DI was held and nine new researchers of the Institute were interviewed. Lectures of R&D methods, analytical instruments and fertilizer research were given. Kemira team leader participated in the tripartite review meeting on 13.2.1987 in Islamabad.

It was obvious that the civil work was late and it was agreed that the 3rd field work period would start in the beginning of August 1986.

# 6 THE THIRD FIELD MISSION

The third field mission of six Kemira experts started on 2nd September 1986 and continued until 14th March 1987. The major targets for this main field mission were: installation of equipment, project planning and selection, starting the research programme, coordination and control of the research projects, communication of research results and collecting new research ideas for RD&DI.

During this third field mission Kemira team accomplished 122 man weeks in the field area and all objectives of the project area were achieved. The FR&DI was capable of independent research and development work in the fertilizer field.

# 7 PRESENT SITUATION IN THE R&D WORK OF THE FR&DI

## 7.1 Inorganic section

### 7.1.1 Beneficiation research

The aim of the beneficiation research of NFC's Research Institute is to solve the concentration of the domestic ore (Lagarban) so that it will be suitable for the manufacture of phosphoric acid and fertilizers. The quality requirements of the product according to British Mining Consultants are as follows: MgO max 1 %, preferably < 0.7 %, $R_2O_3$ (i.e. $Fe_2O_3$+$Al_2O_3$) max 3 %, preferably < 2 %, $P_2O_5$ 29-34 % and recovery 70-80 %.

The economics of a possible beneficiation process will be taken into account by comparing the price of the imported raw material with the operating costs of concentration, investment of concentrator and possibilities of domestic inputs.

At the beginning, theoretical teaching was given. The researcher read books and articles, and discussions were held, also in the form of lectures. Continuous theoretical discussions about special questions of beneficiations during the mission were held.

At first earlier reports e.g. British Mining Consultants, 1982, and U.S. Bureau of Mines, 1977, were studied. In the reports the ore is considered to be difficult to concentrate. Among other methods it was suggested that the dolomite should be removed by acid leaching (sulphuric or nitric acid), which however is a technically difficult and very expensive method because of big acid consumption. (In some reports it was mentioned that flotation is expensive but generally flotation is not regarded as an expensive method at all.)

British Mining Consultants suggest magnetic separation for the removal of iron. This is a wellknown and reliable method. However, in the tests carried out by them with magnetic separators the loss of recovery was rather high.

The old and new equipment were checked. The old laboratory flotation machine is in good condition, but the old laboratory grinding mill cannot be used for wet grinding. The drawings of a new mill have been made.

Equipment

The new special equipment for beneficiation in the list A was as follows: polarizing microscope, stereomicroscope, Andreasen pipette, vibrating feeders, jaw crusher, roll crusher, hammer crusher, schwing mill, conical ball mill, screw classifier, shaking screen, flotation machine, sample divider, reagent feeder, conditioner, diaphragm pumps, pressure filter.

Of this equipment the diaphragm pumps delivered by Vacuubrand are vacuum pumps and not slurry pumps according to the specifications. However, there are other uses for vacuum pumps in the Institute, for instance in the filtration of beneficiation products as well as suction pumps in effluent motoring of the production units. Other beneficiation equipment mentioned has been in routine use.

Tests

The beneficiation desk needed some improvement. The practical beneficiation tests began with Labarban Batkanala ore. This ore type contains so much dolomite (6-9 % MgO) that it cannot be used unconcentrated for phosphoric acid or single super phosphate production. Of all the Lagarban ores this is obviously also the most difficult to beneficiate, because a part of dolomite exists as very fine inclusions in the phosphate particles. The preliminary beneficiation tests gave hints about the above mentioned difficulties. The highest percentage of the concentrate has been 31 % $P_2O_5$, but with a low recovery. When the percentarge was 28 % $P_2O_5$ the recovery was 60 %, which is lower than the target, 70-80 %. The MgO content was about 3 %, which is too high. Iron content in the concentrate was low as it is also in the feed. The percentages of the feed were 25 % $P_2O_5$ and 6 % MgO. The beneficiation has been carried out by desliming and flotation. 38 tests with Batkanala ore were carried out.

Acid leaching suggested by the British Mining Consultants has not been tested at this stage due to very high operation costs. NFC has previously made leaching tests and stated that cost of acids used (sulphuric acid) is about 400 Rs per ton of ore, which is too much. Leaching is also technically difficult to perform. However, beneficiation is a long-term research and these results are not definitive.

After the Batkanala rock, tests with the Eastern ore began. This ore turned out to be easier to concentrate than Batkanala because of the low MgO-percentage. The silica-content is high but it is however easily lowered by flotation. Generally the removal of silica is more easy than the removal of dolomite.

The 20 flotation tests have been carried out as selective flotation of phosphate or in the same way as with the Batkanala sample. The best concentrate produced is 33 % $P_2O_5$ with about 85 % recovery, the iron content was 0.2-0.3 % $Fe_2O_3$. The feed percentages were 25 % $P_2O_5$ and 3 % $Fe_2O_3$, MgO relatively low. This excellent result was achieved with a relatively few number of tests.

It has been difficult for the Institute to procure from abroad new more selective (but also more expensive) reagents. In these tests oleic acid has been used as a phosphoric collector reagent. It is a wellknown and easily available reagent on the local market.

Pilot plant

Towards the end of the third field mission, pilot plant tests were performed. The grinding mill (diameter 0.6 m, length 1 m), the screw classifier and the conditioner were placed on steel foundations in such a way that the slurry flows by gravity from the grinding mill via screw classifier and conditioner to the flotation machine (6 cells, 25 l each). In this way it was possible to run the pilot plant without slurry pumps. Later on if more flotation machines are needed also slurry pumps are required.

The material for pilot plant used was Eastern ore. The capacity of the circuit was 50-100 kg/h. The pilot plant flotation tests were carried out as selective flotation of phosphate, using oleic acid as collector, fuel oil as emulgator, sodium carbonate and sodium silicate (waterglass) as depressant of carbonates and silicates. With the flotation machine it was possible to clean the rough concentrate two times. The products flowed to cleaning inside the flotation machine by gravity without slurry pumps.

Thin section samples

Because the mineral thin section machine mentioned in the list B had not arrived yet, some thin sections were ordered at the Punjab University in Lahore. Also an assistant from NFC will be trained there. The thin section samples were studied with the polarizing microscope at the NFC-institute.

Organisation

In the beneficiation research there is now one engineer as project leader and two assistants, one for flotation tests and one for material handling like crushing, grinding, dividing of material, filtration etc. This arrangement works well, and if the Institute wants to increase the research capacity, one more flotation machine should be procured and two more assistants employed. There is enough working space in the laboratory for such an expansion.

It is estimated that the beneficiation research of the Lagarban ores will take five years. Of this 2.5 years will be laboratory work and 2.5 years pilot plant work. During the pilot plant tests two engineers are needed together with at least 5 assistants.

Suggestions for research activities in the future

The beneficiation research work can continue as described above.

To speed up the research capacity, another laboratory flotation machine, a new laboratory grinding mill (wet) and also a bigger batch mill (dry), diameter 0,5 m, length 0.5 m, should be procured.

Ultra-sonic bath for cleaning of sieves, mineral thin section machines and slurry diaphragm pump are included in B1 list, which will be procured in the near future.

There is also need for more reagent feeders, slurry pumps, low and high intensity magnetic separators, air jet sieve (Alpine) and planetary mill (4 cups, for grinding of analysis samples).

Filtration capacity can be increased by using flocculating reagents, bigger funnels and installing a vacuum pump (now the vacuum is created with water ejectors).

Also new and more selective flotation reagents should be tested.

## 7.1.2
## Phosphoric acid tests

Equipment and installation

The bench scale phosphoric acid plant for dihydrate process was installed in October 1986. The work included building of stands, installation of reactors and mixers as well as the electrical installation of heating systems and temperature regulators and regulators of the dosing pumps for sulphuric acid and recycle acids. Difficulties were caused by the reactor stirrers which were lower in power than recommended in the original equipment list. As a result of these, a rotation velocity of only 150-200 rpm was achieved in the gypsum slurry instead of 300 rpm in the similar assembly in Kemira.

In the beginning of December the equipment for dihydrate process was changed for hemihydrate process. Thus a third reactor with temperature regulating elements and a slurry recycle pump was installed.

In January 1987 the hemihydrate bench scale assembly was further changed for the continuous ammoniation of phospnoric acid. For this purpose ammoniation reactors, a bottle of liquid ammonia, pressure adjusting valve and gas dosing piping were installed.

The installation of pilot phopshoric acid concentration plant was performed in October as far as parts included in the original plan for the pilot assembly were available. The work included construction of working level at 5 m height to get enough static heat for the forced circulation pump in vacuum evaporation system. There were, however, the following difficulties due to missing parts:

The compressed air driven diaphragm pumps for the forced circulation were not ordered according to the original equipment list and are not useful for the pumping of phosphoric acid. However, in the assembly of Larox pressure filter a similar pump is available with the limitation that the material is polypropylene instead of ST 316. This limits the maximum temperature to 65°C and the maximum acid concentration to 40-50 % $P_2O_5$ (if not cooled before the pump) instead of 50 % $P_2O_5$ which is required for den process TSP. For slurry process TSP and ammonium phosphates 40 % $P_2O_5$ is high enough.

The central air compressor necessary to drive the diaphragm pump was missing

The delivery of steam generator for heating the circulation acid was delayed.

For training purposes a temporary assembly was put together by using a hot water boiler instead of steam generator and a centrifugal pump instead of diaphragm pump (however the pumping was not properly running at the static head available).

Results

A. Phosphoric acid tests

Phosphoric acid tests by dihydrate process were started by using Jordan and Lagarban rocks as feed material. The tests by Jordan rock were interrupted after 2 weeks runs because of the very high corrosion due to high content of Cl in the rock (0.16 % Cl). The test time was not long enough to get the operators skilled to stabilize the run and the results were not representative.

The dihydrate runs were continued by using Lagarban/Eastern rock which is less corrosive. A fairly stable run producing 25 % $P_2O_5$ acid was achieved during the three last days of the run when the skill of operators had improved. Thus an overall $P_2O_5$-yield of 96 % and a filter capacity from 2.2 to 2.6 t $P_2O_5/m^2/d$ was achieved. This filter capacity is only 30-50 % as compared with common commercial phosphates by using a similar test equipment in Kemira research centre but corresponds well the expected figure estimated on the basis of rock impurities.

About the same filter capacities (2.2-2.6 t $P_2O_5/m^2/d$) were obtained from Eastern rock by using the hemihydrate process at 40 % $P_2O_5$ product acid level. However the $P_2O_5$ recovery was low, 80-90 %, being better than in the previous report due to stabilized run in the last experiments. The low yield is due to the sensitivity of hemihydrate process to the rock impurities. At the concentration level of 45-50 % $P_2O_5$ the filter capacity was very low, 0.5-1 t $P_2O_5/m^2/d$, and this means that it is practically not possible to get phosphoric acid suitable for den process SSP with hemihydrate process without concentration. An attempt to reduce water insoluble losses by increasing the washer from two to three was omitted because more time than expected was required to stabilize the test runs.

B. Concentration of phosphoric acid

Due to the lacking parts the concentration runs were aimed only to give training to the operators by using temporary equipment described earlier. Also the natural circulation evaporation was proved. It was found that the concentrated acid required for ammonium phosphate and TSP test can be produced by this method when waiting for the original recommended equipment (evaporation rate around 2 kg/h when compared with 5 kg/h using steam generators and Wilden diaphragm pump).

C. TSP-tests

The preliminary batch TSP tests simulating the den TSP process were performed by mixing Eastern rock + Eastern rock based phosphoric acid concen- trated up to 50 % $P_2O_5$. The results obtained until now reveal the following facts:

The $P_2O_5$-conversion into available form and the optimum acid to rock ratio are better than expected on the basis of the contents of the impurities. Results are comparable to that of the Florida phosphate based TSP. The reactivity is partly due to the fine particle size of the rock.
The part of $P_2O_5$ which is fixed to metallic impurities is higher than in TSP made of common commercial phosphates. This $P_2O_5$ is available according to the AOAC method but it can be expected to form insoluble phosphates in the soil conditions. Thus the agricultural tests are recommended.

D. MAP and DAP tests

Phosphoric acid made of Eastern rock (35 % $P_2O_5$) was ammoniated continuously in two reactors (pH 2.5 and 6.0). The ammonium phosphate slurry was divided into two parts. The part A was mixed as a batch with 50 % $P_2O_5$ phosphoric acid until pH 4.0-4.5 to get MAP simulating the MAP-production process. The part B was dried, crushed and saturated with gaseous $NH_3$ to get DAP simulating a universal DAP-production in which TVA ammoniator granulator is used.

Results:

| | MAP | DAP |
|---|---|---|
| pH (10 % solution) | 4.2 | 6.7 |
| N/P atomic ratio | 1.01 | 1.66 |
| Moisture (Fischer), % | 5.2 | 1.9 |
| $NH_3$-N tot, % dry basis | 11.7 | 16.3 |
| " ws % " | 10.4 | 14.8 |
| $P_2O_5$ tot, % " | 52.0 | 51.7 |
| " ws % " | 44.9 | 43.2 |
| " acs % " | 51.7 | 51.5 |
| Solubility, % of total | | |
| $NH_3$-N ws | 84.2 | 90.6 |
| $P_2O_5$ ws | 86.4 | 83.6 |
| $P_2O_5$ acs | 99.4 | 99.6 |

The MAP product contained 62.4 % of nutrients (dry basis) as compared to 66-68 % in commercial product. The lower content is due to the diluent effect of impurities.

In the DAP product the common content of 18 % N was not achieved because a part of the valences of $H_3PO_4$ were saturated with metallic impurities.

The portion of water soluble $P_2O_5$ of total (83-86 %) is lower than in commercial MAP or DAP products (90-95 %). This is due to the precipitation of water insoluble metallic ammonium phosphates.

Future programme for phosphoric acid tests

1. A complete tests series phosphoric acid - concentration - MAP/DAP-TSP/den for the following samples using dihydrate method:
   - Eastern phosphate (Run of mine)
   - Southern " "
   - Batkanala " "
   - Eastern concentrate high quality
   - " " low quality
   - Moroccan phosphate or
   - Florida phosphate or
   - Jordan (low Cl)

   8 tests

   Each phosphate run means at least 1 week run in three shifts or 2 weeks in two shifts to get stable run and enough acid for concentration-, MAP-, DAP- and TSP/den tests.

   The concentration is carried out up to 37 % $P_2O_5$. A small sample is concentrated as batch up to 50 % $P_2O_5$ to get viscosity:
   50 % $P_2O_5$ acid solid free, capillary viscometer at 25/70°C
   " " with solids, Brookfield viscometer
   The concentrated 37 % $P_2O_5$-acid is used for the ammoniation-, MAP-, DAP- and TSP/den tests.

2. As item 1 but using hemihydrate method (concentration-, MAP-, DAP- and TSP tests only for Eastern rocks)

3. A preliminary economic comparison of dihydrate and hemihydrate methods.

4. The process parameters are optimized for each Eastern/ROM-sample as follows:

| | | | | |
|---|---|---|---|---|
| run 1: | product acid | 25 %, | $SO_4$ excess | 2.0 % |
| run 2: | " | | " | 2.5 % |
| run 3: | " | | " | 3.0 % |
| run 4: | " | | " | 3.5 % |
| run 5: | product acid | 22 %, | $SO_4$ excess | opt: from runs 1-4 |
| run 6: | " | 28 %, | " | " |

5. The phosphoric acid test in item 1 is repeated by using optimum conditions from item 2 (only phosphoric acid test).

6. A study on the solids in the 50 % $P_2O_5$ acid throws light upon the possibilities to purify the concentrated acid by separation of solids. The following measurement should be made:
   - the solids content as a function of the storage time
   - x-ray diffraction analysis of solids
   - the settling properties of solids.

   The problem is how to use the sludge acid (underflow) from the clarifier. This contains generally around 15 % solids and 85 % concentrated phosphoric acid. The separation of solids from the mother liquor is very difficult in the production scale. One solution is to produce two grades of TSP:
   high quality TSP from clarified acid (overflow)
   low quality TSP from sludge acid (underflow).

### 7.1.3 Micronutrients

The target of this project is to develop micronutrient fertilizers for Pakistani soils.

The Pakistani soils are usually coarse to medium textured, low in organic matter and have a low cation echange capacity. The outstanding feature is their high alkalinity. Very low P and N, relatively low K, medium Mg and Ca and high Na contents are typical. The content of B varies widely, but in a number of soils shortage of B is apparent. Also shortages of Mn and Zn and excess of Mo are typical of many soils. Cu and Fe are usually within the normal international range.

A micronutrient mix based on Zn, Fe, Cu, Mn and B sulfates has been prepared and granulated. Pot tests with the product should be carried out as well as cost analysis of the production.

## 7.2 Pilot plant section

### 7.2.1 SSP granulation

Project target originally: to finalize granulation parameters for optimum size product with good properties. After talks with the technical manager of LCF-Faisalabad this was changed in: optimization of the existing granulation plant in LCF Faisalabad.

The planning and building of a bench scale granulator (pangranulator) were accomplished and the granulator is operating well. The obtained SSP-granules are of uniform size and good roundness even without screening. The designing of a pilot scale drum granulator with drier was originally part of this project, but after the decision that pilot-runs on SSP can be done at LCF-Faisalabad, this part was taken out of this project and handled separately by Dr. Anwar ul Haq.

Measurements were done at the granulation plant at LCF-Faisalabad and it was found that the feed of SSP to the granulator contained about 25 % lumps bigger than 4 mm due to inefficient crushing. The water supply to the granulation drum was quite irregular. It was also found that sometimes cured and sometimes uncured SSP was used for granulation which need different amounts of added water to obtain the same granule sizes. The amount of water added seemed to be much less than was used in the pangranulator tests.

One of the reasons for this was that the moisture content of the used SSP in the bench scale test was considerably lower than the SSP used in LFC. Another reason was that the SSP used in the bench scale tests was crushed up to 99.5 % below 2 mm while the SSP used in LCF was usually crushed up to about 50 % below 2 mm. Also the fact that in the bench scale tests only cured SSP was used is of importance.

Resulting from these observations it was recommended for LCF to install 3 spray nozzles in the drum. This was done but the results have still to be analyzed. Another suggestion was to increase the efficiency of the crusher to diminish the fraction above 4 mm in the feed of the granulator.

In the pangranulator studies it was observed that SSP granules which have a moisture content above 7 % have a weak hardness of 3-10 N whereas SSP granules with a moisture content below 6 % showed a hardness of 20-30 N. So it is clear that lower moisture content increases the hardness.

The optimum moisture content should therefore be determined in laboratory studies. After these results are obtained it can be decided if further suggestions to LCF should be made about increasing the efficiency of their drying drum, keeping in mind the cost of gas supply.

To test the built granulation pilot plant a one-day run with SSP was carried out. The results seemed promising but the detailed analyses have still to be done.

### 7.2.2 NPK fertilizer for tobacco

Target of the project: to develop an economic formulation for tobacco fertilizer of good grade.

Five different recipes had been calculated for the blending and granulation of 10-20-20 SOP. As raw materials diammonium phosphate (DAP), potassium sulfate (SOP), ammonium sulfate (AS), urea, single superphosphate (SSP), calcium ammonium nitrate (CAN) and filler were used. 10 kg sample of each of these recipes were granulated in the pangranulator and tested for their physical and chemical properties. The critical relative humidity seemed to be quite normal between 46 % and 61 %. The abrasion was poor (< 1.5 %) and the hardness low: 10-29 N.

No caking tendency was observed and that is promising. From the chemical analysis the $K_2O$-analysis could not yet be done because of lack of the required flame photometer. Also the Cl-analyses will have to be done. The water soluble $P_2O_5$-content varied between 67 % and 87 % which was in three cases lower than the required 80 %. The maximum obtained $NO_3$-N content was 1.2 % which was lower than the desired 50 % of total N. The cost analyses for all this recipes were done for manufacturing in Daud Khel in an NPK-plant with a capacity of 300,000 t/year. The estimated investment cost was assumed to be 250 million Rs. The total production of 20,000 t/year 10-20-20 SOP can then be done in this plant in a few weeks.

With the available price levels it seemed to be feasible to make this blending. Even a return on investment above 15 % can be obtained. Therefore it is suggested to continue this project and to test the blends also on pilot plant scale in the drum granulator. Also the possibilities to produce 10-20-20 SOP from phosphoric acid should be studied.

The draft report about cost analyses and experiences on bench scale has been written.

The programme of this project is according to the time schedule.

7.2.3
Increasing $ZnSO_4$ capacity

Targets of this project:
1) to improve the quality of $ZnSO_4$
2) to develop a continuous process for $ZnSO_4$ production.

The existing process in use in LCF-Faisalabad has been studied and during the visit on 27 January 1987 it was said that the approval has been given and the work has started on a new $ZnSO_4$ $7H_2O$-plant and the product will be white crystalline $ZnSO_4 \bullet 7H_2O$. At this moment the product is dark grey and contains about 21 % Zn. The new plant will consist of a sulfuric acid dilution tank, a Zn-ash reactor, a sand filter followed by a pressure filter, an evaporator/crystallizer which can be heated by steam and cooled by cooling water, two open pit crystallizers and a receiving vessel with pump for remaining mother liquor, which will be returned to the evaporator. The used sulfuric acid shall be diluted to about 60 %. So the targets of this project are in fact realized by the realisation of this new plant in Faisalabad. It was therefore decided that the new targets of this project should be: to find the optimal conditions for the process to be built in Faisalabad. It was decided to study on laboratory scale the following parameters on the yield of $ZnSO_4 \bullet 7H_2O$ by simulating the whole process on laboratory scale:
- the sulfuric acid concentration
- the addition time of Zn-ash
- the residence time in the reactor
- the reaction temperature.

First the acid concentration was varied at 30 %, 40 %, 50 % and 60 % while all the other conditions were kept constant. This test was performed and the results are waiting for the Zn-analyses to obtain the yield.

After these tests the rest of the parameters will be varied. The new targets should be discussed with the responsible production people.

7.2.4
NPK processes

Target of this project:
- knowledge of NPK processes
- selection of a suitable process for Pakistan.

The Odda process, pipe reactor process, Kemira NPK process and the Stamicarbon-Odda process in use by PAK-Arab Fertilizer in Multan were studied. Preliminary calculations on the cost of different grades by blending raw materials available in Pakistan were made. The plan to prepare these grades in the available pangranulator was cancelled because this project is not any more on the priority list since December 1986. A preliminary report about the literature study was made in February 1987.

### 7.2.5 NPK granulation pilot-plant

To be able to do research on compound fertilizer and on SSP granulation the design of a fertilizer pilot unit (capacity 100-200 kg/hour) with granulation drum was carried out. The construction work was started in December 1986 and the following equipment was ready and is tested in a test run with success:
- a granulation drum with adjustable spray nozzles
- a drying drum with heater, outlet box and blower
- the connecting chute between granulation drum and drying drum
- the construction work on the ground level and the first level.

The recycle elevator, recycle belt and feeding belt were designed and ordered but not yet installed. In the future the detailed design and the construction of the screens, the crusher and the reactor system will have to be done. Also the construction of the second and the third level, and of the roof can then be started.

The pilot plant when completed will be of paramount importance for fertilizer studies.

### 7.2.6 Urea quality

During the first field mission in February 1985 it was pointed out by one NFC unit that the physical quality, especially the prill strenght of urea, should be increased.

The usual way to increase the strenght of prilled urea is to add some formaldehyde (HCHO). Usually formaldehyde is added in the form of special compound called UF-85 which contains 15 % $H_2O$ and 85 % urea + formaldehyde.

Before being in a position to judge if this method of strengthening the prills is right for the manufacturing units, some methods to make uniform size prills or particles on the laboratory scale had to be developed. First attempts with punctured rubber sheet or iron plate with uniform holes were not successful enough. Therefore a simple oil prilling method was developed and very uniform and spherical particles were made.

The results with the first UF-sample were as follows:

| | prill strenght |
|---|---|
| 0 % addition | 11 N |
| 1 % " | 14 N |
| 1.5 % " | 14 N |
| 1.75 " " | 14 N |

The increase in prill strenght was also only marginal and later it was found out that the sample which was believed to be UF-85 actually was normal UF-glue intended for particle board manufacture.

Meanwhile efforts were made to find out the manufacturers of formaldehyde and related products in Pakistan, and it was found out that Wah Nobel Chemicals Ltd. is able to make formaldehyde and also UF-85. A new sample for laboratory testing was asked from them as well as the prices of the material in bigger quantities.

The final cost analysis of the UF-treatment shall be made in connection with a urea plant after a plant trial taking into consideration the net profits of reduced recycle and production capacity increase (the cost of UF-85 deducted).

The product from the test trials should also be tested in agricultural field test because it has been claimed that formaldehyde has some efficiency as urease inhibitor and through this property there would be an extra benefit for the farmers (reduced $NH_3$ loss).

## 7.3 Organic section

### 7.3.1 Water treatment chemicals

Cost analysis for own MBT-based microbiocide blending was prepared. The savings are marginal. The questionnaire concerning the availability and prices in Pakistan of other commonly used microbiocides so far without results.

Information was gathered on the prices and amounts of all water treatment chemicals used in NFC units for treatment of cooling water (microbiocides, dispersants, descalers, corrosion inhibitors). The gathered material has to be further processed into process descriptions and material balances to find out the actual potential areas upon which the research should be concentrated. This work has been started but not finished.

The boiler feed water treatment should be included in this project; for instance the testing and comparison of different ion exchange resins for water purification.

### 7.3.2 Lubricating oil purification

Target of this project:
Development of methods for purification of used lubricating oils of NFC units on minimum possible costs.

A study has been completed on the amount and the quality of lubricating oils in use in different factories of NFC. The following samples of used oils have been received:

| | |
|---|---|
| - Shell Clavius 15 | PAK-American |
| - Valvata 460 | PAK-American |
| - Machine oil PBS Clavius 46 | PAK-China |
| - Machine oil T-78 | PAK-China |
| - Compressor oil Perfectos | PAK-China |
| - Clavius oil | PAK-Arab |
| - Tellus oil | PAK-Arab |

Of these oils a low-vicosity oil (Clavius 15) and a high-viscosity oil (Valvata 460) have been tested by removal of water and treatment with Fuller earth.

The low-viscosity oil (Clavius 15) could be reclaimed easily and the tested specifications were within the specifications of fresh oil. The only property that was not yet measured properly is the pour point. Also the colour of the reclaimed oil was the same as in the fresh sample.

The high-viscosity oil (Valvata 460) was difficult to filter at lower temperatures after treatment with Fuller earth because of the high viscosity. At higher temperatures the filtration however succeeded. The acid number of this oil is higher than the acid number of the fresh oil so treatment with hydroxide will be necessary. A considerable profit is expected when reclamation is successful, as 25,000 l per year of this oil is used in PAK-American. Also Clavius oils are used in considerable amounts: 9,000 l/year in PAK-American and 21,000 l/year in PAK-Arab and reclamation seems to be easy and profitable. However, because of the question of warranty of equipment by suppliers who demand the use of specified lubricating oils and the difficulty that oil blending is regulated by governmental licences it was decided in December 1986 to defer the project. Because the analyses outside the Institute were not allowed, the project has been totally stopped since December 1986. Kemira team recommended that this decision should be reconsidered because of good economical potential of the project and the fact that reclaimed oils are used in many ammonia plants in other countries without any damages. Also PAK-American and PAK-Arab were still interested in this project. On the review meeting on 12 March 1987 it was realized that the project should be continued. So also the other received samples will be treated and analyzed to study if the used procedure is also suitable for other oils.

7.3.3
Slow release urea

Nitrogen is used predominantly as urea. Pakistani soils, however, are predominantly alkaline soils and no urease inhibitors are used. Therefore there arises the question how much N is lost to the atmosphere as free $NH_3$. More research work in the agricultural institutes and universities is needed on this subject and this is also important topic for FR&DI (to investigate manufacturing methods for slow release urea N and to investigate possibilities to introduce urease inhibitors to the product of existing urea plants and to fabricate corresponding products in FR&DI pilot for field trials in agricultural institutes and experimental farms).

The price of ureaform (38-39 % N) is too high for standard fertilizer. Locally available cheap dissolution rate retarders should be sought.

Sulphur coated urea should also be considered as a technical possibility because of shortage of S in Pakistani soils.

7.3.4
Corrosion studies

1. Equipment and research methods

Installation of the computerized PAR potentiostat was done and the instructions to use the computer. The software consists of the following programmes:
1) potentiodynamic
2) polarization resistance
3) tafel plots
4) cyclic polarization
5) corrosion behaviour diagrams
6) reactivation
7) galvanic corrosion
8) galvanostatic
9) corrosion potential vs. time

Corrosion cell which was purchased alongwith can be used for all other programs except 7 which requires another type of arrangement.

The system works excellently. Without going to details it is possible to
1) calculate the corrosion rate by programmes 1, 2, 3
2) find out the corrosion mechanism from 1, 4 and 5
3) get necessary information for anodic or cathodic protection of metals from 1, 4 and 5
4) study the parameters of pitting by 4 and 5
5) find out the efficiency and mechanism of inhibitors by 1 and 3 etc.

Conventional methods

ASTM standards and recommended practices were provided by Kemira and most important of them explained.

Further equipment needed

Digital multimeter for measuring currents, resistance and potential.

Set of caliper rule, micrometer, measuring tape etc. for geometrical measurement of corrosion specimens.

Magnifying glass for observation of surfaces of solid materials before and after exposure.

Dye penetrants for crack detection for corrosion failure analysis.

Field identifying set for stainless steel family.

Reference materials

Standard and specialty stainless steel samples of sheet and rod with specifications are urgently needed as well as proprietary alloys and some special metals.

Research activities in corrosion field

Questionnaire of corrosion cases for all NFC units was prepared and three units responded. Based on the answers to this questionnaire corrosion study of PAK-Arab CN-brine was started.

Generally the situation concerning corrosion research equipment is good. For the corrosion research personnel participation in the specialized lectures in corrosion in university is suggested. Also the possibility to employ new experienced personnel in corrosion testing should be considered.

## 7.4 Analytical section

### 7.4.1 Training

Of the four researchers of the analytical section Dr. S.A.Bhatti, Mr. A.Ali and Mr. M.E.Bhatti have been trained in Finland during 12 weeks. The fourth researcher Mr. A.Kazmi has been trained in Japan one week by Hitachi Co. In connection with the installation of the nutrient analyzer and atomic absorption spectrophotometer training has been given by the experts of suppliers.

It can be said with satisfaction that the researchers in the analytical section are now familiar with basic methods needed in fertilizer research.

### 7.4.2
### Purchase and installation of equipment

In the purchase procedure there were three counterparts: UNIDO, NFC and Kemira. The co-operation did not work properly due to the long distances causing confusion and delay. Much closer co-operation would be recommended in similar projects.

All equipment except flame photometer were installed by 1.3.1987.

### 7.4.3
### Main equipment and methods

Most probably the atomic absorption spectrophotometer (Varian 1475) with lamps for 20 elements will be the "workhorse" in the analytical section of the Institute like in many analytical laboratories around the world today.

The automatic nutrient analyzer (Skalar 104) with separate channels for $NO_3$- and $NH^{+4}$-determinations is also a valuable tool for the fertilizer research. The high quality spectrophotometer (Hitachi 150-20) with a data processor is good for various spectrophotometric methods.

Among the minor analytical equipment it can be mentioned the memo titrator (Mettler DL 20 RC), water determination with the Karl Fischer method (Mettler DL 18) and the ion analyzer (Orion Research EA 920).

For organic research there are the gas chromatograph (Hitachi 263-50), the liquid chromatograph (Hitachi 655 A-11) and the IR-spectrophotometer (Hitachi 270-30).

### 7.4.4
### Personnel

In the analytical section there are four researchers and four assistants but no laboratory technicians. The situation cannot continue like this, i.e. all the routine determinations are made only by the analysts. The role of analyst should rather be as follows: supervision and training, method development, to be a link between the analytical section and other sections as well as other units of the company. At least five laboratory technicians are needed immediately: 1 for atomic absorption and flame photometer, 1 for nutrient analyzer, 1 for organic determinations and 1 for physical testing. Later on this minimum number of laboratory technicians can be increased as required.

7.4.5
Future activities

Co-operation inside NFC

The analytical section of the Research Institute is now the best equipped analytical laboratory in NFC. In addition to that, the section has analyst resources to help the control laboratories in their analytical problems. Close co-operation should be established between all the analytical laboratories of NFC and the analytical section of the Research Institute In this co-operation the analytical section could be the co-ordinating laboratory and the terms of co-operation could be as follows, for instance:

1. Evaluation of present situation, what are the problems.
2. Analytical methods should be made uniform inside NFC and files should be created, where all the methods are collected.
3. Checking the accuracy of nutrient determinations by sending fertilizer samples from NFC plants to the Institute frequently.
4. Creating a store of standard reference material and control samples for the checking accuracy.
5. Transfer of analytical personnel even into other laboratories in cases where extra help is needed.
6. Organizing at least once a year meetings for analysts of the Institute and for the heads of control laboratories. That is the proper forum for solving current problems and introducing new methods as well as instruments.

Co-operation outside NFC

Co-operation is the key word in analytical work, not only inside NFC but also outside NFC. Although the analytical section is a well-equipped analytical laboratory, it will need some help from outside the company. The natural counterparts for co-operation in soil and plant analysis are University of Agriculture and Nuclear Institute of Agriculture and Biology both in Faisalabad. For temporary needs of x-ray diffraction and fluorescence analysis as well as DTA and TGA-analysis a suitable laboratory shoud be found. For the microscopic studies of geological samples the Geological Department of Punjab University is highly recommended.

Need of additional equipment

The analytical section is well equipped for the basic inorganic determinations needed by fertilizer research. The need of additional equipment should be considered in the future very carefully and the possible procurements should be based on real and permanent needs. For temporary needs the analytical section should co-operate with other laboratories as long as possible. After the research activities in the Institute have started with many projects in the area of inorganic chemistry there will be need of some additional equipment like x-ray fluorescence and diffraction spectrometer, carbon furnace as an accessory for atomic absorption spectrophotometer and perhaps some element analyzer like carbon and sulphur analyzers.

7.4.6
Projects

The main task of the analytical section is to give the best possible analytical service to the research projects of the Institute. If there is some capacity in addition to that main task, the analysts of the section should do their own projects. The project schedule of this kind of project should be flexible to avoid disturbing the main task. There were three projects intended directly for the analytical section: 1. Analytical problems of production units, 2. Trace element analysis, 3. Gas effluent analysis. Until now, because of installations and routine determinations, there has been lack of time to start practical work on these projects. The project "Trace element analysis" should be initially restricted to harmful elements like Cd in fertilizer produced by NFC.

7.5
Physical testing laboratory

Equipment, Physical laboratory

All equipment was installed and operational except for the shattering test and the capillary viscometer.

The shattering could not yet be done because of lack of compressed air. The capillary viscometer misses still the capillars. The calorimeter has been twice calibrated and will be fully ready for use after two further calibrations. The principles of the stalagmometer, pitot tube apparatus, thermal decomposition apparatus and the particle size analyzer (Andreasen pipettes) have been worked through and this equipment is ready for use. The torsion viscometer and the ball viscometer have been used and also measurements with the conductivity meter and the stroboscope have been carried out.

7.5.1
Analysis and quality control of NFC and imported fertilizers

Target: to get information about the quality of the available fertilizers in Pakistan.

Since 1st January 1987 about 300 determinations were performed on samples of SSP, urea, CAN, home garden fertilizer, rock phosphate and tobacco fertilizer to obtain the following properties:

- moisture content
- critical relative humidity
- granule hardness
- abrasion
- angle of repose
- bulk density
- caking tendency
- flowability
- screen analysis
- dust content
- granule roundness.

Also urea samples were collected from the local market of the following production units:

- Daud Hercules Ltd. "Babarsheer"
- Exxon Chemical Ltd. "Angro"
- Fauji Fertilizer Company (FFC) "Sona"
- NFC PAK-China "Kissan urea"
- NFC PAK-Arab "Kissan urea"
- NFC PAK-Saudi "Kissan urea"
- imported urea from Romania.

The following observations were made:

- All the urea had a screen analysis of more than 99 % smaller than 3.15 mm, so the hardness test has to be done on prills of 2 mm only and is therefore not comparable with the hardness results of NPK.
- The caking tendency of Angro was higher than that of others, which showed none or slight caking tendency.
- PAK-Saudi and Angro contained clearly more dust than the others but only the imported urea had a low dust content.
- The roundness of the granules from Daud Hercules was the lowest (25 %) followed by Angro (45 %). All the others showed a roundness of 85-95 %.
- The abrasion of PAK-China urea is the highest. Only for Daud Hercules, Exxon, and imported urea from Romania the abrasion figure was good.
- The imported urea had a bigger average size and less granules below 1 mm than all the others.
- The granule hardness will have to be measured still.
- The prill strenghts.

Home garden fertilizers showed a strong caking tendency immediately after production (52 % for a moisture content of about 6 %) but showed no caking tendency any more after drying until a moisture content of less than 2 %.

### 7.5.2 CAN caking studies

In spite of remarkable improvement in physical quality of PFL CAN, the product still frequently has caking tendency. Therefore a more detailed study of the problem was started. There are two reasons for the caking of CAN: the hygroscopity due to CN and the volume increase as well as disintegration of the prills because of $AN_{IV->III}$ crystal transformation.

The research work concentrated on the following points:

1. To find out the function between retention time in the mixing vessel of molten AN with by-product lime and CN formation on the other hand.
2. To find out the function between the amount of $KNO_3$ added and the stability range of AN III.
3. To find out the conversion efficiency of $K_2SO_4$ into $KNO_3$ in molten AN at constant retention time.
4. To see the effect of various coating agents and coating methods, on the caking tendency of CAN.

The method used for measuring crystal transformations was dilatometric temperature-volume determinations using turpentine oil as dilatometric liquid. Temperature was changed slowly (1°C/3 min), results were recorded as volume-temperature curves (V-T) also in derivative form ($\frac{\Delta V}{\Delta T}$ $_{-T}$).

The main conclusions from the results of this research can be drawn as follows:

a) Most important reduction in the caking tendency will be achieved when the stability is reached by maintaining the required amount of $KNO_3$ in the product. Only after this condition is fullfilled the normal anticaking methods will become efficient. There seems to be possibility also to improve the efficiency of the programme against normal caking, that is caking due to moisture absorption, minor changes in the temperature etc.

For instance, it seems obvious that coating with inert powder after application of oil amine will decrease the caking. However, more oil amine samples and more powders should be tested to find out the best combination in this respect.

b) The amount of added $K_2SO_4$ is too small considering the coldest temperatures in Multan during the period from November to February.

c) To reach the full crystal stability by $K_2SO_4$ addition only (to form enough $KNO_3$ for full stability down to 2-3°C, the minimum temperatures) will be too costly without compensation in the product price for added $K_2O$.

d) The required amount of $K_2SO_4$ will be smaller if the product storage will be provided with doors and these doors are kept closed during the night in cold season.

## 7.6
## Instruction in computer use

At the end of the field mission no IBM Personal Computer was available as originally planned. Because the use of computers in research work is of great help in calculations, graphics and report writing it was important to make all the researchers familiar with some useful modern software.

Fortunately an Apple IIe computer was connected with the corrosion measurement system. For this system three programs have been bought:

| | |
|---|---|
| Multiplan | for spreadsheet calculation |
| Applewriter | a word processor |
| Visiplot/Visitrend | a graphics package. |

Several weeks have been spent on the teaching of the basics of programming in the language Applesoft Basic and of the use of Multiplan as tool in cost analysis and as easy report writer. In general it should be said that the goal is not achieved and only about 5 persons can use these programs still with a lot of difficulty.

8
PROGRESS OF THE PROJECTS OF FR&DI IN REVIEW MEETINGS
26.11.1986 and 12.3.1987

| SECTION/Project | Techn.probability of success x) 26.11.86 | 12.3.87 | Progress of research work xx) 26.11.86 | 12.3.87 |
|---|---|---|---|---|
| INORGANIC SECTION | | | | |
| 1. Beneficiation research | 0.5 | 0.6 | + | +++ |
| 2. Phosphoric acid tests | 0.9 | 1.0 | ++ | +++ |
| 3. TSP-tests | 0 | 1.0 | 0 | ++ |
| 4. MAP and DAP tests | 0 | 0.9 | 0 | ++ |
| 5. Micronutrients | 0.5 | 0.7 | - | + |
| PILOT PLANT SECTION | | | | |
| 1. SSP granulation | 0.9 | 1.0 | ++ | +++ |
| 2. NPK fertilizer for tobacco | 0.8 | 0.9 | + | ++ |
| 3. Increasing $ZnSO_4$ capacity | 0.6 | 0.8 | - | + |
| 4. NPK process | 0.6 | 0.6 | + | + |
| 5. Concentration of $H_3PO_4$ | 0 | 0.8 | 0 | + |
| 6. NPK granulation pilot plant | 0.7 | 1.0 | + | +++ |
| 7. Urea quality | 0.8 | 0.8 | - | ++ |
| ORGANIC SECTION | | | | |
| 1. Water treatment chemicals | 0.7 | 0.9 | + | + |
| 2. Lubricating oil purification | 0.8 | 0.9 | ++ | + |
| 3. Anticaking chemicals | 0 | 0 | 0 | 0 |
| 4. Antifoaming chemicals | 0 | 0.5 | 0 | + |
| 5. Slow release urea | 0 | 0.5 | 0 | + |
| 6. Corrosion studies | 0.7 | 0.7 | + | ++ |
| ANALYTICAL SECTION | | | | |
| 1. Analysis & quality control of NFC and imported fertilizers | 1.0 | 1.0 | + | ++ |
| 2. CAN caking problems | 0.8 | 0.9 | ++ | +++ |
| 3. Analytical problems of production units | 0.9 | 0.9 | 0 | 0 |
| 4. Trace element analysis | 1.0 | 1.0 | 0 | 0 |
| 5. Gas effluent analysis | 0.8 | 0.8 | 0 | 0 |

x) 1 = 100 % technical success
0 = not technically possible

xx) +++ very good
++ good
\+ reasonable
\- unsatisfactory
0 not started

# 9
# OUTPUTS OF THE PROJECT

9.1 A well established Fertilizer Research and Development Institute comprising the following facilities:

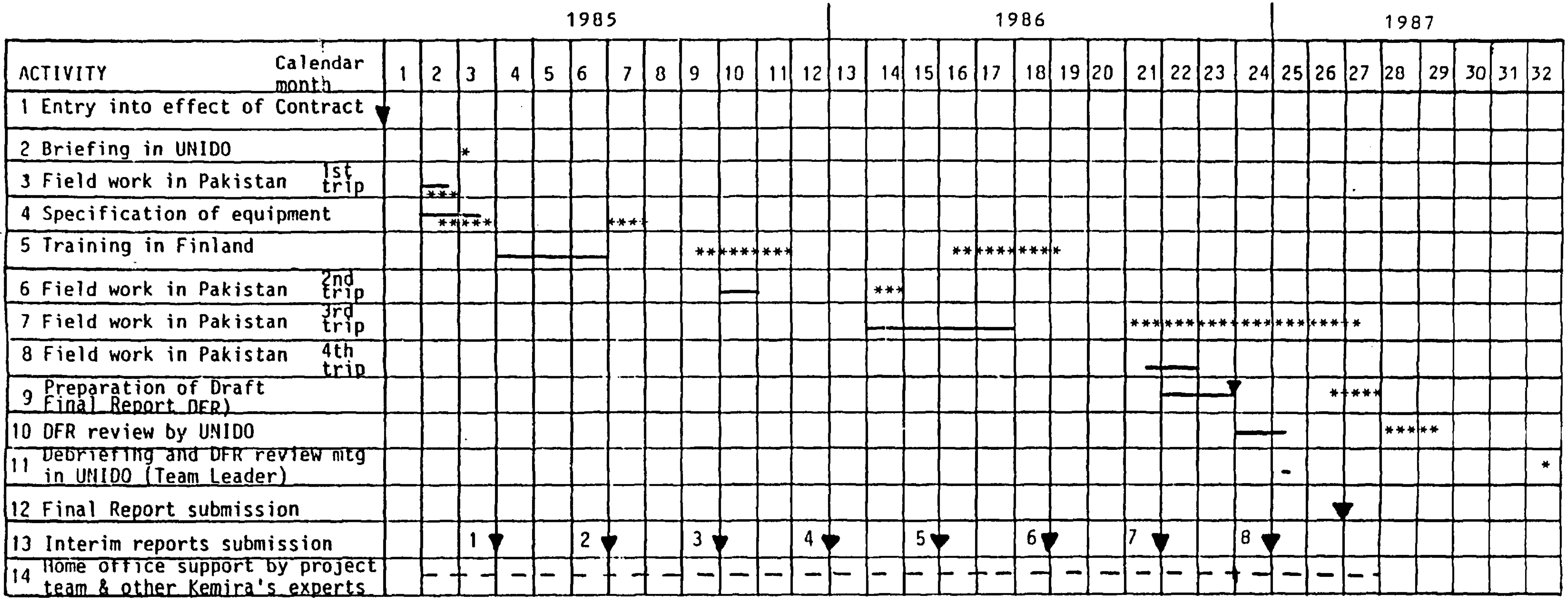
a) Raw materials testing laboratory, inorganic.
b) Fertilizer product testing laboratory.
c) Unit operations pilot-hall provided with semi-pilot and pilot-scale equipment and machines.
d) General (physico-chemical) analytical laboratory and instrument laboratory.
e) Pilot-plant for NPK granulation.
f) Library and documentation section (facilities not totally ready yet).

9.2 18 engineers and researchers trained in Pakistan by Kemira experts and 9 of them also in Finland through fellowship assignments in the following fields of specialization:

a) Testing, evaluation and processing of rock-phosphates for the fertilizer industry.
b) Testing of imported raw materials like rock phosphate and other mineral nutrients, also chemicals such as anticaking, antifoaming, cooling water treatment and proposing ways and means for local mixing of these chemicals.
c) Beneficiation tests and methods for beneficiation of phosphate ore.
d) Testing and evaluation of NFC and imported products and assessing their suitabilities for local climatic conditions and applications.
e) Testing and recovering of waste materials and by-products and suggesting ways and means of their economic utilization.
f) Purification, selection and testing of lubricating oils for use in fertilizer industry.
g) Testing of corrosion and corrosion resistant materials.
h) Suggesting technological improvements to be made in the manufacturing units and adopting advanced technologies for the production of straight, compound and blended fertilizers to suit conditions and specific requirements in Pakistan.
i) Computer use in research and development work.
j) Planning of research projects, research programmes and research budgets.
k) Evaluation and selecting of the research projects.
l) Staffing the research projects and motivating the researchers.
m) Coordination and control of research and development work.
n) Reporting of projects and R&D results.
o) Long-range planning of R&D.

9.3 During the field mission technical reports were completed by R&D researchers under the guidance of Kemira experts on R&D work carried out in the Institute on the following subjects:

- Beneficiation studies of local rock
- Phosphoric acid tests of different rocks
- Water treatment chemicals
- Micronutrients
- Lubricating oils purification
- Tobacco fertilizers
- Increasing urea quality
- CAN caking studies
- NPK processes
- Pre-feasibility study on $MgSO_4$ production




Original plan ————

Realization of project work *********

**KEMIRA ENGINEERING**

| | |
|---|---|
| CLIENT | UNIDO, Vienna |
| PROJECT | DP/PAK/83/010 |
| DRAWING TITLE | 3 Time schedule<br>24.1.1985 |

APPENDIX 1

## CONTRACTOR'S PERSONNEL

The effective work done by Kemira
through the whole project duration
1.1.1985 - 15.3.1987

| NAME | FUNCTION | PROJECT AREA SERVICE man months | HOME OFFICE SERVICE man months |
|---|---|---|---|
| Antero Hörkkö | Team leader and expert of R&D and fertilizer manufacturing | 6.75 | 6 |
| Erkki Aalto | Phosphate ores and other fertilizer raw materils expert | 5.75 | 3 |
| Jukka Karhunen | Phosphate beneficiation and mineral processing expert | 5.75 | 2 |
| Timo Korvela | Fertilizer manufacturing and research and corrosion expert | 6.25 | 3.5 |
| Arie van der Meer | Fertilizer testing and manufacturing and computer use expert | 5.0 | 1.5 |
| Esko Saari | Analytical methods and instruments and mineralogical expert | 6.25 | 3.0 |
| Reino Lähdemäki | Project coordinator and home office support | - | 6.0 |
| | Total | 35.75 | 25.0 |

LIST OF MANUALS, PUBLICATIONS AND TEXTBOOKS
(Suggested for Purchase/Subscription)

FERTILIZER LITERATURE

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| Chemistry in Production of High-analysis mixed Fertilizers | Takashi Akiyama, Japan Research Institute for Phosphate Resources, 188 p., Hatano. 1986. |
| CRC Handbook of Chemistry and Physics | 62th ed. 1981 - 1982 |
| Dangerous Properties of Industrial Materials | 6th ed. 1984 by N. Irving Sax |
| Farm Chemicals Handbook '86 | Meister Publ. Co. Willoughby, Ohio. 1986. |
| Fertilizer Manual 1980 | UNIDO document ID/250. 353 p. |
| Manual of Fertilizer Processing | Francis T. Nielsson, Marcel Dekker Inc. 525 p. New York & Basel. 1986. |
| Minerals Yearbook 1983 Vol. 1: Metals and Minerals | 1984. |
| Minerals Yearbook 1980 Vol. 3: Area Reports, international. | Bureau of Mines, USA. 1982. |
| Official Methods of Analysis of the Association of Official Analytical Chemists | 13rd ed., ed. William Horwitz. 1980. |
| Perry's Chemical Engineers' Handbook | 6th edition. 1984. |
| Phosphates and Phosphoric Acid. | Pierre Becker. 1983. |
| Principles of Plant Nutrition | Mengel, Konrad & Kirkby, Ernest A., International Potash Institute. 593 p. Bern. 1987. |
| Process Technologies for Nitrogen Fertilizers | UNIDO document ID/211. 67 p. 1978. |
| Process Technologies for Phosphate Fertilizers | UNIDO document ID/209. 50 p. 1978. |

| | |
|---|---|
| Raw Material Committee Meeting Held in Kyoto Oct 15 - 16, 1985. Proceedings vol. 1 - 4 | IFA. U.s. IFA A/85/130a-d. Paris. 1985. |
| Technology and Manufacture of Ammonia | Samuel Strelzoff. 1981. |
| Test Methods for Measuring the Physical Properties of Fertilizers | Heikki Takala. Kemira Oy. 1986. |
| The Condensed Chemical Dictionary | 10th ed. Gessner G. Hawley. 1981. |
| Urea Survey, World Analysis and Prospects 1983 - 1993 | 1983. |
| World Survey of Phosphate Deposits | 4th ed. Ed. M.C. Mew. 1980. |


JOURNALS AND MAGAZINES


| | |
|---|---|
| Chemical Engineering | New York, N.Y. |
| Chemical Engineering Progress | New York, N.Y. |
| Chemical Processing | Chicago, Ill. |
| Contact the IFA Newsletter | Paris |
| Current Contents, Agriculture Biology and Environmental Sciences | Philadelphia, Pa. |
| Farm Chemicals and Croplife | Willoughby, Oh. |
| Fertilizer Focus | Trade, Transportation, Technology/Richmond Surrey. |
| Fertilizer International | London |
| Fertilizer Research | An International Journal on Fertilizer use/Hagve. |
| Fertilizers and Agriculture | IFA. Paris. |
| Hydrocarbon Processing | Houston, Tex. |
| IFDC Report | International Fertilizer Development Center, Muscle Shoals, Al. |
| Nitrogen | London. |
| Phosphorus and Potassium | London. |

| | |
|---|---|
| Process Engineering | London. |
| Processing | London. |
| Soil Science | Baltimore, Md. |
| Soil Science Society of America | Madison, Wis. |
| Soils and Fertilizers | Farnham Royal, Bucks. |
| Sulphur | British Sulphur Corp., London. |

ANALYTICAL PUBLICATIONS

| | |
|---|---|
| Analytical Methods for Atomic Absorption Spectrophotometry | Perkin-Elmer. Norwalk, Connecticut, USA. 1982 |
| Basic Methods for Determining Elements in Fertilizer Industry | |
| Micronutrients and the Nutrient Status of Soils | A global study by Mikko Sillanpää. FAO Soils Bulletin. Rome. 1982 |

MINERAL LITERATURE

| | |
|---|---|
| Benefication of Mineral Fines | P. Somasundaran & N. Arbiter, Printed by Edward Brothers Inc., USA. 1979 |
| Coagulation and Flocculation | John Bratby. Uplands Press Ltd., England. 1980. |
| Flotation Agents and Processes | M.W. Ranney. Noyes Data Corp. USA. 1980. |
| Flotation. A. M. Gaudin Memorial Volume. | Vol. 1 & 2, M.C. Fuerstenau, American Institute of Mining, Metallurgical and Petroleum Engineers, Inc., New York. 1976. |
| Mineral Processing Technology | 2nd ed. B.A. Wills, Pergamon Press, 1981. |
| Principles of Flotation | R.P. King, South African Institute of Mining and Metallurgy. 1982. |

## PHOSPORIC ACID LITERATURE AND PUBLICATIONS


| | |
|---|---|
| Phosphoric Acid, Raw Materials, Technology and Economics of the Wet Process | Pierre Becker. Marcel Decker Inc., New York, 1983. |
| Phosphoric acid, Part I - II | A.V. Slack, Marcel Decker Inc., New York, 1968. |
| Phosphoric Acid, Outline of Industry | 2nd ed. The British Sulphur Corp. Ltd., London. 1984. |
| The Role of Phosphorus in Agriculture | American Society of Agronomy etc., Madison, Wis. 1980. |
| Effect of Impurities in Wet-Process Phosphoric Acids on DAP Grades | F.P. Achorn, E.F. Dillard, A.W. Frazier, D.G. Sulladay, IFA Technical Meeting, Vienna. 1980. |
| From Bench Scale and Pilot Plant Test to Industrial Applications, Proven Extrapollution Techniques | P. Becker, Fert. Ind. Round Table, Washington 1981. |
| General Properties of Florida Phosphate Rock for Phosphoric Acid and Fertilizer Use | D.W. Lenshon, Society of Mining Engineers, June 1967, 192 - 196. |
| Impurities in Phosphate Rock - Good or Bad? | McClellan, G.H. and J.R. Lehr Fert. Ind. Round Table. Atlanta, Ga. 1982. |
| Phosphate Products from Indigenous Resources | McClellan, G.H. and Schulz, J.J.(IFDC), Paper presented at British Sulphur Co's Eight International Conf., London 1985. |
| Phosphoric Acid by Direct Sulphuric Acid Digestion of Florida Land-Pebble Matrix | J.C. White, Bureau of Mines (USA), Report No.8086, 1975. |
| Phosphoric Acid Manufacture, Updating Process Technology | Phosphorus & Potassium. No. 143, May-June 1986. |
| Removing Impurities to Improve Phosphoric Acid Quality | Phosphorus & Potassium, No. 194, 1987. 30 - 32. |
| Removing Magnesium from Wet Process Acid with Fluosilicate | Phosphorus & Potasium No. 79 1975., 25 - 27 |
| Solving problems in Chemical Processing of Low Quality Phosphate Rock | Engineering and Mining Journal 178, 1977. 104 - 108. |