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DEVELOPMENT OF EXPERTISE IN WATER TECHNOLOGY MANAGEMENT
IN THE INDIAN FERTILIZER INDUSTRY

DP/IND/85/006/11-05

Technical Report: Discussions Held and Recommendations Made*

Prepared for the Government of India
by the United Nations Industrial Development Organization
acting as executing agency for the United Nations Development Programme

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Vienna

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This report describes discussions held at National Fertilisers Limited (NFL), Panipat and Fertilisers and Chemicals Travancore Limited (FACT), Cochin, India regarding Water Technology Management in November 1986.

The report is in three parts:

- i Conclusions and Summary of Recommendations for each site.

- ii NFL
 - ii Notes on discussions, principles of problems and recommendations at Panipat.
 - iii Appendices containing detailed technical points at Panipat.

- iii FACT
 - ii Notes on discussions, principles of problems and recommendations at Cochin.
 - iii Appendices containing detailed technical points at Cochin.

1.0 CONCLUSIONS AND SUMMARY OF RECOMMENDATIONSPRE-TREATMENT

The Pre-Treatment Plant was not in a fully commissioned state. It could not and was not being operated as installed and designed. This has produced downstream influences on boiler silica level, resin fouling, need for pre-chlorination and on cooling water control.

It is recommended that the system be fully recommissioned. It is a management matter, not a capital investment requirement.

DEMINERALISATION PLANT

This is working well but the anion resins have reached the end of their life and should be replaced. In my opinion there is no major organics fouling problem justifying capital expenditure on brine washing or activated carbon equipment.

It is recommended that the practice of pre-chlorinating all the site raw water should cease. This will save chlorine and prevent potential damage to the demineralisation plant and boiler.

There are no technical reservations or recommendations to make on the choice of hydrochloric versus sulphuric acid regenerant. It is a commercial decision to be taken by local management.

Proposals are made regarding condensate recovery whereby the existing polishing system is by-passed when it is in specification (saving revenue) and used in various other ways when out of specification to save wasting it.

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COOLING SYSTEM

There are genuine problems on the cooling system causing unwarranted shut-downs and loss of production.

In my opinion the two fundamental problems are lack of control on the chemistry, due to lack of communication or action following communication between the technical and production personnel and loss of biological control of the system. The former is management, the latter technical and proposals are made to appoint a coordinator who is identifiably responsible for water management. A technical programme is proposed to regain control of the biology of the cooling system and the principles involved are explained. No capital expenditure is involved but initial costs will be more until control is regained and improvements provide payback.

CHEMICAL CLEANING

Properly managed this would be a vital part of improving plant heat exchanger reliability. Coupled with better cooling water control it would lessen failure rates and reduce unplanned shutdowns and it is recommended that it is adopted as a positive maintenance technique.

FUTURE

The cooling water problems will take some time to come under control and require on site work during that time. If a review at some future date was requested by Panipat management it should not be less than 6 months from this report.

1. Summary of Conclusions and Recommendations - FACT

1. The quality of raw water available to the Udyogamandal site is a corrosive one, difficult to treat in cooling systems and one which has given problems in this area.
2. Pretreatment of this water is necessary and the new plant, currently being built, should help alleviate some problems.
3. The anti-corrosion programme chosen for the cooling water is the best but it has not been applied correctly. An explanation of the principles and recommendations for future control are given.
4. The operational policy of the demineralisation (DM) Plants on the sulphuric and ammonia installations has been developed along technically deleterious lines. These errors are explained and recommendations for future control are given.
5. Failures in the Sulphuric Acid boiler are potentially as a result of misapplication of standards following the change from softened to DM water. Recommendations for future control are given. If successful this would obviate the need for mechanical modifications e.g. raising the steam drum.
6. Potential corrosion of the sulphuric acid economiser is identified and recommendations for extra inspection are made.
7. In Several cases a clear need for chemical cleaning has been identified. This is a function of the age of the equipment and the type of water technology employed. It is recommended that FACT personnel receive training in this field to best take advantage of the techniques.
8. Some of the techniques available in the Laboratory are inadequate for the control of the site and recommendations for upgrading the service are made.

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This Report describes discussions held at NFL Plant, Panipat, India regarding Water Technology Management in November, 1986. It is essentially in three parts:

- i) Conclusions and summary of Recommendations
- ii) Notes on discussions, principles of problems and recommendations.
- iii) Appendices containing detailed technical points..

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1.0 CONCLUSIONS AND SUMMARY OF RECOMMENDATIONS

PRE-TREATMENT

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2.0 PANIPAT PLANT: Built in 1979, it is an Ammonia-Urea complex of Toyo Design utilising fuel oil as feed based on the partial oxidation route using site produced cryogenic nitrogen. The auxiliary boilers are fired with coal and steam is raised at 100 kg/cm^2 . The loop is Topsoe technology.

Water is abstracted from an irrigation canal to alternately fill one of two unequal reservoirs of approximately $113,000 \text{ M}^3$ and $150,000 \text{ M}^3$ working volume where considerable natural settlement of silt occurs. The design allows water at a rate of $1371 \text{ M}^3/\text{h}$. to be pumped to two clariflocculators where chlorine and alum are added. Clarified water flows through sand-gravel gravity filters to an underground storage tank which is the site filtered water supply. Currently, approximately 300 te/h . flow to the demineralisation plant, 70 te/h . as direct services to the plant, 2750 te/h . is used for cooling water makeup and 105 te/h . as drinking water for the NFL community. Approx. 500 te/h . is required for deashing duties which is taken from the effluent treatment plant exit.

Although the site water technology management must be a totally integrated operation, for the purposes of discussion it was broken down into the following areas: Pre-treatment, Demineralisation, Cooling systems and boiler systems. Detailed discussions were held on each of these in three and two hour sessions each morning and afternoon. Notes on each area of discussion form the detail of this report.

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3.0 PRE-TREATMENT

3.1 Introduction - Water quality analyses of raw and filtered waters are appended (Appendix 3.1.1 and 3.1.2) from which it can be seen that the design water is of high turbidity with low dissolved solids and organics. Probably due to the beneficial settlement in the raw water reservoirs the water typically reaches the plant with much lower turbidities than design although, on occasions, the sediment has been disturbed by pulling too hard on the reservoir and particularly in the early part of the rainy season when high levels of solid reach the clarifiers.

The particular problems identified by Panipat personnel were very high levels of sulphate reducing bacteria (SRB) in the incoming water (see Appendix 3.1.3), problems of colloidal silica giving higher than expected levels of soluble silica in the boiler (see 3.2) and organic fouling of the anion exchange resins in the demineralisation (DM) plant (See 4.4).

The pre-treatment plant was visited on a number of occasions during the consultancy when it was apparent that not all parts of the plant were in a fully commissioned state and discussion with the operators suggested that this was not unusual e.g. the alum solution preparation system.

During the discussions, the incoming pH of the water was around pH 8 and yet only 10 ppm of alum was believed to be being added. In theory this would only destroy 4.5 ppm of alkalinity and not reduce the pH significantly. It should be noted that at pH's above pH 7.4 some aluminium will remain in solution in the EEC standard for drinking water is < 0.05 ppm (EEC N C 214/6 to 11/75). It would be prudent to

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check the level in the drinking water to the NFL township and medical advice sought for its significance especially at times of high turbidity when high alum levels should be used e.g. upto 120 ppm.

In laboratory tests conducted on 13.11.86, 10 ppm alum added to the raw water produced negligible floc which could be removed and potentially added approximately 0.8 ppm of Al to the water. A 10 ppm alum dose at pH 6.5 produced a good floc and could be expected to produce a residual alumina concentration of < 0.05 ppm. 40 ppm alum produced identical floc in pH 6.5, 7.0, 7.5 and unadjusted (pH 8) water. Even though floc was produced some aluminium would remain in solution at the high pH.

From this it was concluded that the water was not being flocculated and clarified correctly at the time of these tests. Because little turbidity was in the water at the time, little colloidal silica would be expected to be present so no silica problem would be expected in the boiler (which was the case!). Appendix 3.1.4 shows typical results. However, pre-treatment has several effects besides reducing suspended solids and colloidal silica. Table-1 summarises the role of Pre-treatment.

TABLE-1

Role of Pre-treatment in Water Management

Correct Pre-treatment reduces	Effect if not reduced
1) Colloidal silica	High boiler water silica. Increased blowdown. Loss of efficiency. Danger of damage to turbines.

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Correct Pre-treatment reduces	Effect if not reduced
2) Suspended solids	Overloads filters. Can clog in exchange units (counter current systems would suffer damage). Increases fouling in cooling systems; makes it more difficult to run zinc/phosphate systems and to control bacteria.
3) Organic material	Fouls ion exchange anion resins. Increases fouling in cooling systems and makes it more difficult to control bacteria.
4) Bacteria	Increases problems in cooling systems in terms of biological control.
5) Iron	Fouls ion exchange resins. Increases fouling in cooling systems.

These are the reasons pretreatment is installed initially and for optimum subsequent operation of all downstream equipment it is imperative that the plant is operated correctly (see other areas for cross-references e.g. boiler silica (3.1), cooling system problems (5.3) and anion resin fouling problems (4.4)).

3.2 Colloidal Silica Problems - Occasional problems of high boiler water silica analyses despite low D.M. water valves were discussed.

This is almost certainly due to inadequate control of the pretreatment plant either in terms of pH control or alum dosage. Measurement of colloidal silica is extremely difficult and would not

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normally be recommended, but if abnormal silica levels are found in the boiler with normal D.M. silica values then immediate checks should be made on the operation of the pretreatment plant and every effort made to keep the boiler water silica level below 1.8 ppm to avoid turbine problems.

Routinely, levels of colloidal silica are reported in filtered water but this is believed to be due to limitations in the method because boiler water silica values do not agree with these values.

Problems of colloidal silica can be predicted in a general way in the sense that at the beginning of the rainy season extra vigilance will be needed and if the raw water reservoirs are disturbed. The latter could be minimised by regular cleaning of the reservoirs to remove accumulated silt.

4.0 DEMINERALISATION PLANT AND CONDENSATE RECOVERY

Specifications of demineralised water and returned condensate are in Appendix 4.0.1 and 4.0.2. Panipat personnel identified the following problems which apart from § and ¶ were discussed at length.

- a) High Condensate temperature and its effect on Resin (Cat-ion).
- b) Change over of Regenerant from sulphuric Acid to Hydrochloric Acid, from Caustic Soda to Ammonia.
- c) Organic fouling problems.
- d) Colloidal Silica problems.

Contd...

- e) Counter current and co-current generations of Resin - Advantages and Disadvantages.
- f) Acid preparation problems for regeneration.
- g) Internal damages of Distributors.
- h) Vacuum Degassing cum Blower Degassing.
- i) Carbon filter for absorption of extra chlorine from water.
- j) Problems connected with Chlorinators.
- k) Cooling Tower Dosing System
 - i) Suggestions, if any, for proper chemical dosing.
 - ii) On line monitoring instruments for cooling tower and DM Plant.
 - iii) Disposal of acid and alkali waste from DM Plant.

4.1 High Condensate Temperature and its Effect on Resin

Cation resin can safely be used upto temperatures of 120°C in the H⁺ form. There should never be a problem on the condensate recovery plant with temperatures in the region of 60°C. However, see 4.5, if the condensate was over fed back to the front end of the DM plant the combined temperature of the pretreated water and the condensate should not exceed 60°C due to problems on anion resins.

- 4.2 Choice of Regenerant - On the cation unit the choice is between sulphuric acid and hydrochloric acid. For Panipat water, the acid regeneration level could be reduced from 80 g H₂SO₄/l, to 30 g HCl/l, to maintain the same run length. The calculations in Appendix 4.2 show that at the prices supplied (data only available for the working cation units) nearly 8 lakhs/year revenue savings could be made by changing to HCl.

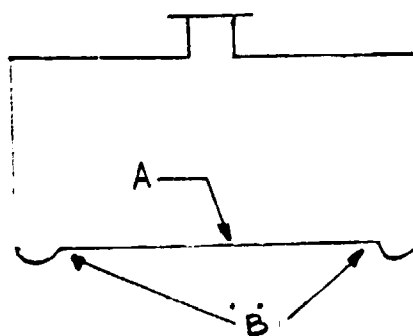
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To enable this to be done a complete new set of acid regeneration, offloading and chemical storage equipment would have to be purchased and installed as the current sulphuric acid equipment is made of incompatible materials. (See 4.3). There is no technical reason why this should not be done. It is an economic/political decision.

On the anion units the choice of regenerant discussed was that of sodium hydroxide versus ammonia. Ammonia cannot technically be used to regenerate a strong base anion resin where silica removal is required as it is inefficient at splitting the quaternary ammonium salts formed by those strong base groups on the resin. Ammonia can only be relied upon to regenerate weakly basic tertiary amino groups.

- 4.3 Acid Storage and preparation problems - The handling of sulphuric and hydrochloric acids to prepare dilute solution is well documented and Panipat personnel did not declare any specific problems. Problems have been experienced by ICI fertilizers with fumes and using a GRP bulk storage tank for HCl where cracks had been created by inadequate civil design. The tank was fabricated as indicated in the sketch in Fig.1 and the civil foundations had been specified as a flat plinth. On filling the tank the bottom area 'A' flexed and leaks occurred at 'B'. The problem was solved by supporting the area under 'A'.

Fig.1



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Secondly, at statutory inspections, the tank was drained, ladders dropped inside and the tank entered. On refilling, leaks occurred where damage had been inflicted by the presence of the inspectors.

- 4.4 Organic fouling problems - The problems of loss of capacity on the anion resins was discussed. The salient points were that the resins were 8 years old and had required brine washing when a good recovery of operating capacity was seen. The capacity deteriorated necessitating another brine wash leading Panipat personnel to believe they had a severe organic fouling problem.

Although the latter are classic symptoms of fouling, the time scale of 8 years and the lab measured deterioration in total capacity from 4.1 meq/g to between 1.8-2.7 meq/g for different charges of resin suggest that the fundamental problem is one of wornout resins which should be replaced. Improvements in the pretreatment plant would put less load of organics (and/or iron and/or bacteria-all which cause fouling of anion resins) on to the resins which in general terms, would mean less of a problem. However, in comparison to UK, European and US case histories of needing to brine wash every month, replacing resins every 2-4 years and not losing gross capacity (only operating capacity) Panipat does not have a problem. (An outline of the brine wash method used in the UK is Appendix 4.4).

In terms of replacing the resins, FF(ip) is an excellent resin and there is no reason why it should not be purchased as a recharge. The choice of an alternative is a 'shot in the dark' compared to the

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excellent service history of the FF(ip) and no change would be recommended although the alternatives were discussed at length e.g. acrylic, gel, macro-porous, iso-porous, type I, type II and their respective abilities to cope with fouling, silica removal and the general conditions at Panipat.

- 4.5 Condensate Recovery - Panipat personnel described that 175 te/h steam condensate was returned to the DM plant via two cation exchange units operating in parallel at 87.5 te/h each with a third unit on standby/regeneration. From these the condensate went to the secondary mixed beds (SMB) for final polishing before return to the boiler. Approximately 55 te/h of the condensate originates from the urea plant and has on occasions become contaminated with urea. This gave high conductivity exit the cation units and short runs on the SMB. Panipat dump the urea condensate to drain whenever this occurs but did not fully understand the chemistry or the implications of alternative actions. These were all discussed.

It is felt that urea occasionally leaks into the condensate which already contains ammonia from the boiler dosing programme (pH > 8.0) when the condensate passes through the cation columns, ammonia is exchanged for H^+ (some urea may also possibly exchange for H^+). The acid conditions will cause the urea to convert to ammonium carbonate from which the ammonia would initially be removed by ion exchange resulting in carbonic acid and then ammonium carbonate itself appearing in the polished condensate raising its conductivity and going 'out of spec.'. This load leads to short runs on the SMB.

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In this case, provided the SMB quality does not deteriorate, there is no justification in dumping the condensate which is a valuable resource.

The question that should be asked is : "Why treat the condensate at all if it is in specification ?". In a tight system the condensate will contain ammonia but nothing else. Why polish it to remove the ammonia, by consuming sulphuric acid (to regenerate the polisher) only to add ammonia to it again a little later ?.

The system increasingly used in the UK is to feed the condensate from steady state operation directly back to the boiler feed system with a flow of about 10 l/h passing through a mini-cation column (1 M long by 3 cm diameter containing ~1 l of resin. One on duty, one standby). When the condensate is good a conductivity of $< 0.5 \mu S/cm$ will be attained. If any contaminant breaks through, the conductivity will immediately rise when the condensate should be tripped out. It could be (i) fed to the inlet of the DM plant (it will be considerably better quality than the filtered water). (ii) Put back through the original cation : SMB route; (iii) Put into the cooling tower (good quality but disadvantages are high temperature and nutrient effect of ammonia/urea); (iv) Used for deashing duties.

Analysis should be carried out to determine the cause of the trip e.g. urea breakthrough, cooling water leak etc. and repairs effected as soon as possible.

A brief analysis of the cost of dumping was attempted (Appendix 4.5) which indicates a cost of approximately 53,000 R/day or nearly 2 crore/yr.

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The mini-cation columns should be designed for easy removal of the resin and when they are exhausted new resin should be charged or laboratories could carryout the regeneration in glass-ware.

- 4.6 Carbon Filters for Chlorine Removal - Panipat personnel described the problem of high level of sulphate reducing bacteria (SRB) in the incoming water and their successful attempts to reduce them by chlorinating before pretreatment. They recognised the danger of excess chlorine reaching the ion exchange resins and proposed installing activated carbon technology to simultaneously reduce the chlorine and the organics going forward.

This would be a major capital investment and the following points are relevant:

- i) There is not a major organics problem at Panipat (see 4.4).
- ii) Extensive testing work would be required to find a carbon that might work anyway e.g. coal, wood or coco-nut based carbon? Pore size? Particle size ? Regeneration method? Maintenance costs?
- iii) Operation of the pretreatment plant correctly would reduce the incoming SRB count (See 3)
- iv) Prechlorination could be carried out after the filtered water tanks i.e. do not treat the water going to the DM Plant in the first place.

On this basis it is recommended that no such technology (capital expenditure) should be contemplated.

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The danger of chlorine and DM Plants is a real one. Three things can happen(not all are in the text books, but they have been observed in ICI Fertilisers):

- i) Chlorine attacks organics in the water(or adsorbed on the resins)to produce organo-chlorine compounds. Some of these are non-ionic and pass through the DM Plant to be hydrolysed in the boiler liberating chloride which can cause corrosion;
- ii) Chlorine attacks the resins(cation and anion) shortening their life;
- iii) Chlorine can pass directly through a DM Plant eventually decomposing to chlorides which can cause corrosion.

In a test carried out on 14.11.86, 0.5 ppm of free chlorine was analysed at the cation unit outlet indicating that any or all of the above could be occurring.

The practice of chlorinating upstream of the DM plant should stop.

4.7 Vacuum Degassing versus Blower Degassing - Both types of plant are in use in plants in the UK and each has its points but on the whole blower types are preferred due to:

- i) Less maintenance
- ii) Very high reliability and hence availability

4.8 Co-current versus counter current regeneration - This subject was briefly discussed but no firm conclusions were drawn. ICI Fertilisers have experience of both technologies but on the whole prefer co-current due to:

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- i) High reliability and hence availability
 - ii) Less maintenance
 - iii) Less complicated and easier to operate
- See 4.9.

4.9 Internal Damage of Distributors - This subject was briefly discussed. Old style nozzles and laterals in co-current systems fail spontaneously from time to time. This may be due to aging (loss of plasticiser encouraging brittleness) or water hammer phenomena during valve changes, especially during regeneration. In counter-current systems tremendous problems have been experienced with upper bed laterals where bending and breakage have been common. This has been partly due to inadequate design of supports but also due to water hammer effects and piston movement of resin. Suspended solids in the incoming water are filtered out by the resin giving high pressure drop and the stresses placed on buried laterals when counter flow is introduced causes damage necessitating taking the unit out of service.

5.0 COOLING SYSTEMS

5.1 Description and problems - There are three towers labelled CT1-CT3. The basic data is shown in Appendix 5.1.1 and 5.1.2. Chemical analyses for October, 1986 for CT1 and CTII are on Appendix 5.1.3 and 5.1.4.

The problems have largely been seen on CT1 which has the most arduous duty, critical exchangers and suffers from intermittent ingress of fly ash from the nearby coal and ash handling plant. The other towers although adjacent to CT1, seem to escape this. Significant numbers of exchanger failures

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have occurred leading directly or indirectly to a high number of ammonia plant shutdowns outside the planned annual overhauls. For example, the synthesis gas compressor intercooler (details in Appendix 5.1.3) caused at least 3 shutdowns specifically for its repair and at least one emergency shutdown as well. Similar failures have occurred in GA 402, GA 404 T as specific cases discussed although the number of shutdowns they caused was not identified.

Algae growth, bacterial growth (especially sulphate reducing bacteria, SRB), loss of cooling water chemicals at times of greater ash ingress, unexpected loss of pH control from time to time, high conversion of meta to ortho-phosphate, ineffectiveness of chlorination, high volume of sludge settling in low velocity areas, high levels of cooling water chemicals in the side stream filters, failure of heat exchangers and loss of efficiency of heat transfer on exchangers were identified as problems (!)

Due to the complexity and multifarious nature of the symptoms on these systems much time was spent in discussion and this report summarises those in terms of the fundamental problem which it is believed has been identified.

5.2 Chemical control of cooling systems

5.2.1 Philosophy - The cooling water chemistry in use is a widely accepted one and is fundamentally acceptable but the limitations are equally fundamental i.e. good control within the close tolerances laid down in the specification (suggested specification in Appendix 5.2.1).

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Detailed study of the Panipat specification suggests the following philosophy: the first programme in use on the plant was HMP at 20-25 ppm plus zinc sulphate at 3-5 ppm at pH 6.3-6.8 which gave hard phosphate scales (largely due to poor pH control). To combat this, the HMP was reduced, a polyelectrolyte dispersant added and HEDP to operate in more alkaline conditions (to reduce meta to ortho conversion and minimise calcium phosphate scales). By reducing the HMP so much, its role as a corrosion inhibitor is minimised and reliance is placed on the zinc to cathodically limit corrosion. The role of the HMP is therefore much more one of scaling control. The reversion of meta to ortho is still a problem on this plant for reasons not clearly understood although the two main factors influencing reversion are pH and temperature. The lower the pH and the higher the temperature the faster the reaction. For example, micro-biological growths will produce micro-environments where the pH can vary significantly, even the bulk water pH can be depressed by nitrifying bacteria oxidising ammonia to nitrate and some heat exchangers on the ammonia plant have particularly hot skin temperatures. Section 5.3 describes the microbiological problems which must be solved first, but once they are, it could be beneficial to operate at 20 ppm HMP if the meta to ortho conversion problem comes under control. If the reversion is more a function of high temperature then it would be better to stay with 5 ppm HMP.

The key factor in applying this chemistry is to maintain the bulk water chemistry within the specifications as closely as possible. Excursions will initiate all the problems outlined in Appendix 5.1.2 which will not be totally rectified on returning to specification but will fester and lead to secondary problems e.g. high pH will cause a scale deposit, under the deposit corrosion can develop, SRBs can grow,

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failure can occur. The specifications refer to solution chemistry and require that all analyses are carried out on filtered samples. The ingress of fly ash provides high surface areas for selective adsorption of some of the treatment chemicals (i.e. Zn and HEDP) which upset this chemical control. Extra zinc and HEDP must be added to maintain solution chemistry. Some of the ash is removed in the side stream filter, complete with its adsorbed chemicals, which explains the high level found in the filter backwash.

5.2.2 Polyelectrolyte Aqua 4000 - The role of this polyelectrolyte has not become clear despite all the discussions with Panipat personnel. Without doubt an agent is required to carry out the following:

- i) Stabilise zinc ions in solution. If no agent is used zinc will precipitate at pH 7.8 and the programme will fail.
- ii) Disperse suspended solids e.g. bacteria, flocculent scale, fly ash.

These jobs may be carried out by a single polymer or a mixture of polymers and it is recommended that Panipat personnel discuss this in detail with Aquapharm and their competitors. The Aqua 4000 is an anionic polymer which is a major problem to SRB control (see 5.33) and it is suggested that a non-ionic dispersant is sought.

5.3 Biological control of cooling systems

5.3.1 Basic problem - Biological control has been lost on the systems. This is a function of several accumulated problems which has led to failure of chemical programmes and equipment. For example :

Contd...

- i) Inadequate pretreatment (see 3.1) allows higher levels of bacteria, especially SRBs, to enter the system.
- ii) Chlorine is less efficient at higher pH values and even less effective in the presence of ammonia, oil, organic material carbon (e.g. soot/fly ash particles) or any sludges (poor penetration).
- iii) Recognition of (ii) by Panipat personnel led to the use of non-oxidising biocides in a technique which they incorrectly believed would sterilise the system. Because the system was not under control (which it was believed to be) the cause of several effects were misunderstood e.g. rapid 'regrowth', "feeding on dead bacteria"; sudden pH drops caused by intrifying bacteria (but they were gone'), consistent SRB activity (ineffective biocides).

It is believed that the problem can be summarised as follows:

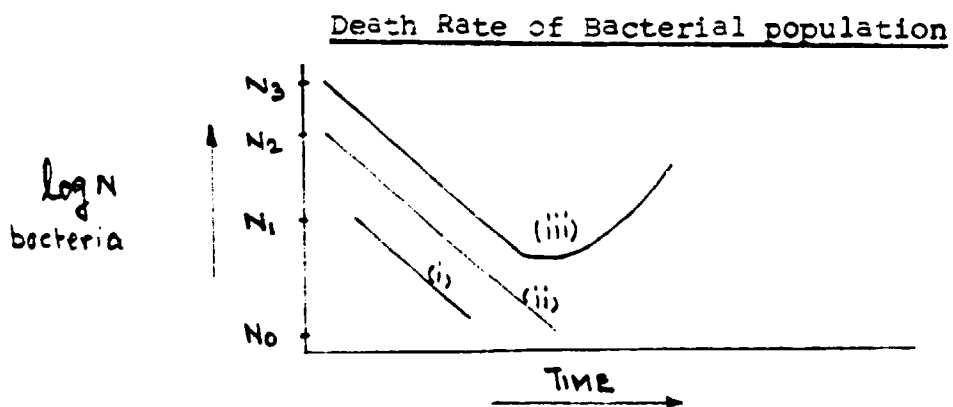
- i) Bacteria gain access to the system and grow at a rate greater than they are killed by biocide treatment.
- ii) They grow as layers and form sludges in mixtures with coal ash and other solids, especially in low velocity areas.
- iii) The sludge grows thicker with dead cells on the bottom and living cells above - aerobic at the top and anaerobic at the bottom. Differential oxygen corrosion cells are set up.
- iv) Any biocide that is added will kill the cells it contacts e.g. those in suspension and the uppermost layer on the sludge. To contact those lower in the sludge requires diffusion through the sludge.

Contd..

- v) The bacterial count in the circulating water is only a small fraction of the count in the system (and it is only the cells on the surface which really cause the problems) indicating only a moderate problem.
- vi) Anaerobic bacteria grow at the bottom of the sludge e.g. SRB, often next to the metal surface causing pitting.
- vii) The sludge layers restrict heat transfer and prevent anticorrosion chemicals gaining access to the metal surface which is the only place they can act.
- viii) Control of the system is lost.

5.3.2 Principles of Microbiological control - Death rates of bacterial populations are exponential as shown in Fig.2 reflecting the probability effect of a cell coming into contact with the biocide and actually dying.

Fig.2



It is the slope of the line that indicates the rate of death and populations (i), (ii) and (iii) in Fig.2 are being killed at the same rate. However, (ii) started as a larger population than (i) so takes a

Contd..

longer time to reach the same final number. In (iii), insufficient biocide was added to kill all the cells and the population starts to grow again, never reaching N_0 .

This outlines the two key principles :

- i) Enough toxic material must be added to kill all the cells;
- ii) It must be allowed sufficient contact time to effect the kill.

Due to the sludge formation as described in 5.3.1 the contact time to kill cells (including SRB at the bottom) could be many days, even then, the concentration that finally reaches the cell may not be enough to kill it. This contact time and concentration at the cell cannot be predicted for any given system but if varying doses are added for varying lengths of time, inspection and monitoring of the system will indicate when it is clean and lead to the selection of a cost-effective regime.

A cooling system should be operated with continuous make-up of water, continuous dosing of chemicals and continuous purge. When this occurs the best control can be maintained and the concentration of any involatile component, C , can be predicted accurately versus time in exactly the same way that decay of a radio-active element can be modelled and a half-life value expressed. In an open evaporative cooling system -

$$\text{Half-life} = \frac{0.69 V (CF-1)}{E}$$

and it can be shown that

$$C_0 = C_t \cdot e^{\frac{E}{V(CF-1)} \times t}$$

Contd...

where C_0 = concentration of component initially (t_0)
 C_t = concentration of component after a time (t_t)
 e = exponential
 v = volume of system m^3
 CF = concentration factor
 E = evaporation rate $te/h.$
 t = time hours.

Therefore, to dose once per day ($t=24$) to keep a minimum concentration (C_t) it can be calculated how much must be added (C_0). It should be remembered that the system concentration will not be zero except the first time it is dosed in such a programme. e.g. if C_t required is 10 ppm and the calculation says add 17 ppm, on day two only 7 ppm will need to be added to maintain the programme.

5.3.3 Proposed method for gaining microbiological control

The role of Aqua 4000 has been discussed in 5.2.2. Being an anionic polymer it reacts with any cationic biocide e.g. citric acid to precipitate as a floc (this was proved in a laboratory experiment on 18.11.86). Two of the most effective biocides against SRBs at the pH of the Panipat systems are cationic in nature and thus could not be expected to work reliably. In the short term it is proposed that sodium pentachlorophenate is added at 10 ppm (i.e. $C_t = 10$ ppm) once per day. Prechlorination and system chlorination may be continued as required. (There is a danger of an effect on the effluent treatment plant and this should be monitored closely). To avoid selecting resistant strains an alternate biocide should be used for one week at least once per month. Iso-thiazolones have been used by Panipat in the past and this can be continued. Although not good against SRB specifically, it is a good aerobic

bactericide and will help to gain system control. Methylene bis-thiocyanates have also been used but are not recommended as the pH is too high when the tower is on-line. Dosing should take place daily.

In the longer term, if the Aqua 4000 can be replaced by a non-ionic dispersant, a polyamine or quaternary amine biocide can be used. These are effective against SRB and have good dispersive properties (sometimes too good, an antifoam should be available when using these).

Organo-bromine compounds can be used (with or without Aqua 4000) as good controllers of SRB. Appendix 5.3.3 lists the pros and cons of the biocides.

A suggested programme would be:

Daily dosing of quaternary ammonium compound at $C_t = 10$ ppm for three weeks.

Daily dosing of organo-bromine at $C_t = 10$ ppm for one week.

Monitor by SRB count and viable plate count (VPC) but take particular notice of corrosion coupons for evidence of SRB attack.

After 4 weeks, reduce levels of each biocide by 5 ppm if corrosion coupons are free of SRB attack or increase by 5 ppm if they show attack.

Continue to monitor activity of SRB in this way, adjusting the biocide regime up and down to find the optimum, cost effective dose to meet the specifications in Appendix 5.2.1 (especially SRB) and regularly get no SRB attack on the coupons.

Contd....

Once the microbiology is under control this should (i) improve pH control (no nitrifying bacteria to plunge pH down) which may improve the phosphate reversion problem which should improve the phosphate scaling problems and subsequent under deposit corrosion problems; (ii) SRB attack - a major cause of heat exchanger failure - should cease; (iii) fouling by coal ash may be lessened - bacteria make sticky deposits, encouraging entrapment of suspended solids.

All this work demands good control of the cooling water chemistry, an understanding of the complex interplay of all the variables and a will to make it work. It is recommended that a single person is given the responsibility of co-ordinating all the aspects (lab analysis, interpretation of results and practical actions on the plants).

6.0 CHEMICAL CLEANING

A seminar was held in the Ammonia plant conference room on 18.11.86 to discuss chemical cleaning in the fertilizer industry. Most attendees were familiar with the basics but a lively discussion ensued on the philosophy and some specific problems.

The nature of the cooling water problems on the Panipat site and the history of failures suggest strongly that part of the overall management of the water cycle should include chemical cleaning of the critical heat exchanger. The following points from the discussions are valid:

- i) Clean heat exchangers foul and corrode less quickly than dirty ones;
- ii) The nature of the phosphate cooling water chemistry is that some fouling/scaling will always occur. The worse the control, the worse the problem.

Contd...

- iii) Chemical cleaning, properly planned and carried out, will not cause corrosion in a vessel.
- iv) Chemical cleaning, properly planned and carried out, will remove debris and expose holes and weaknesses.
- v) If those holes and weaknesses cause acid leaks or failure at subsequent pressure testing - so be it. They would have failed anyway in a very short time, so it is better to find the problem when already in a shutdown situation, with resources available for repair, than to go back on line and trip off one or two weeks later in an unplanned manner.

It is recommended that chemical cleaning is used as a maintenance tool to clean exchangers and, in a way, find their weaknesses.

7.0 ACKNOWLEDGEMENTS

Most discussions were held in the technical building with Messrs Sharma, Mathur, Manoharlal, Ahuja, Dubey and Dua. Meetings were also held with Messrs. G.P. Khurana, Talwar, Kashyap, Dudhe and Suri.

I would like to make particular acknowledgement to Mr. C.L. Sharma (Manager, Laboratories) who made all the necessary arrangements for the technical meetings and equally vital arrangements for my well-being throughout my time at NFL. Without his assistance and persistence the visit of this UNIDO expert would have been a technical and personal waste of time.

Thank you.

THE DESIGN AND ACTUAL QUALITY OF RAW WATER

S.No.	Parameter	Design	Actual
1.	pH	8.3	8 to 9
2.	Cond.	-	170-250
3.	Turbidity (Silica units)	90 to 2000	5-25
4.	Total Hard (as CaCO ₃)	125	70-120
5.	Ca Hard (")	90	50-80
6.	Mg Hard (")	36	30-45
7.	P alkalinity (")	8	8.0
8.	m-alkalinity (")	108	55-95
9.	EMA	-	25-40
10.	TDB	250	100-90
11.	Chloride (as Cl)	24	3-8
12.	Sulphate (as SO ₄)	40	15-25
13.	Nitrate (as NO ₃)	1.2	1.0
14.	Iron (as Fe)	1.9	0.5-0.1
15.	Sodium (as Na ⁺)	-	4.5-7.5
16.	Pot (as K)	-	1.5-4.0
17.	Silicate (as SiO ₄)	-	
	i) Soluble	6.0	5-8
	ii) Colloidal	-	1-3
18.	Dissolved oxygen (as O)	-	6-11
19.	BOD (5 days) (as O)	-	0.5-1.5
20.	COD (as O)	-	1.0-3.0
21.	KMNO ₄ value		
	i) 3 min	-	0.15-0.7
	ii) 4 hours	2.0	0.6-1.6
22.	Fluoride (As F)	2.0	NT

QUALITY OF FILTER WATER

S.No.	Parameter	Range (ppm)
1.	pH	7-8
2.	Cond	180-260
3.	Turbidity (SiO ₂ Units)	1-8
4.	Total Hard (as CaCO ₃)	70-120
5.	Ca Hard (")	50-80
6.	Mg. Hard (")	30-45
7.	p-alkalinity (")	NT
8.	m-alkalinity (")	35-85
9.	EMA	30-50
10.	TDS	110-200
11.	Chloride (as Cl)	4-8
12.	Sulphate (as SO ₄)	25-40
13.	Nitrate (as NO ₃)	NT
14.	Iron (as Fe)	0.05-0.1
15.	Sod (as Na ⁺)	4.5-7.5
16.	Pot (as K ⁺)	1.5-4.0
17.	Silicate (as SiO ₂)	5-9
	i) Soluble	5-8
	ii) Colloidal	1-2
18.	Dissolved O ₂ (as O)	6-11
19.	BOD (5-days) (as O)	0.4-1.2
20.	CCD (as O)	0.6-2.5
21.	KMNO ₄ value	
	(3 min.)	0.1-0.4
	(4 Hours)	0.4-1.0
22.	Fluoride (as F)	NT

APPENDIX 4.0.1

(A) NORMS OF QUALITY OF POLISHED WATER

S.No.	Parameter	Norm of quality (ppm)
1.	pH at 25°C	6.5-7.2
2.	Silicio acid(as SiO ₂)	0.015
3.	Hardness	Nil
4.	Conductivity at 20°C	0.2 m hos (Max.)
5.	Temperature	About 35°C
6.	Total iron	0.015
7.	Aluminium	Not precipitable
8.	Total copper	0.003
	Calcium	Nil
9.	Total CO ₂	Nil
10.	Oil	Nil
11.	KMnO ₄ value	5.0

APPENDIX 4.0.2

(B) DESIGN CHARACTERISTICS OF RETURN CONDENSATE

S.No.	Parameters	Characteristics (ppm)
1.	Temperature	60°C
2.	Turbidity	Nil
3.	pH	7-9.5
4.	Total Cation(as CaCO ₃)	1.5
5.	Total Anion(as CaCO ₃)	1.5
6.	Ionic iron(as Fe)	0.1
7.	Colloidal iron(as Fe)	0.05
8.	Copper(as Cu)	0.1
9.	Silica(as SiO ₂)	0.02-0.7
10.	Conductivity	15-25 m hos.

APPENDIX 3.1.3

BACTERIAL COUNTS IN CT-1 AND CT-2 CIRCULATION WATERS AND MAKEUP WATER

Month	CT-1		CT-2		m/u water	
	VPC	SRB/100 ml	VPC	SRB/100ml	VPC	SRB/100ml
Aug.85	5,500	460	21,000	270	215	50
	to	to	to	to	to	to
	1,34,000	790	2,45,000	1800	1190	140
Sept.85	31,000	110	21,000	50	880	120
	to	to	to	to	to	to
	1,14,000	1100	1,25,000	1300	1910	140
Oct.85	20,000	70	20,000	20	125	20
	to	to	to	to	to	to
	2,04,000	700	1,25,000	130	1090	240
Nov.85	46,000	80	44,000	220	140	MT
	to	to	to	to	to	to
	1,10,000	790	1,80,000	490	575	20
Dec..85	6,000	20	12,000	50	68	MT
	to	to	to	to	to	to
	3,80,000	340	1,02,000	120	210	20
Jan.86	26,000	40	14,000	40	60	20
	to	to	to	to	to	to
	1,20,000	740	3,40,000	790	200	40
Feb.86	18,000	220	29,000	50	130	20
	to	to	to	to	to	to
	4,49,000	2400	2,12,000	1300	253	90
March,86	5,000	110	1,600	140		
	to	to	to	to		
	2,52,000	1550	1,80,000	1100		
April 86	S/D	S/D		S/D		
May,86	87,000	340	32,000	240	230	20
	to	to	to	to	to	to
	4,10,000	1300	3,50,000	1100	610	110
June,86	67,000	1300	52,000	120	80	50
	to	to	to	to	to	to
	2,49,000	2400	2,51,000	2400	600	90
July,86	9,000	170	13,000	230	110	20
	to	to	to	to	to	to
	2,67,000	1300	3,61,000	1750	650	90

OCT-86

APPENDIX 3.1.4

Date	Boiler No	Deserator Inlet		Deserator Outlet				C.B.D.H.S.G. Plant							
		PH	SiO ₂	PH	Cond	SiO ₂	N ₂ H ₄	NH ₃	PH	Cond	SiO ₂	P.A.H	m.NL	PO ₄	Fe
1-10-86	2	6.5	0.012	8.6	17.0	0.016	0.1	1.2	8.6	24.0	0.52	1.5	5.0	2.25	0.11
	11								8.5	20.0	0.52	1.0	4.0	2.28	0.13
	11								8.45	19.0	0.45	1.0	3.0	1.71	0.12
2-10-86	2	6.5	0.013	9.1	19.0	0.016	0.08	2.0	8.6	24.0	0.45	2.0	5.0	2.28	0.13
	11								9.5	22.0	0.75	8.0	17.0	7.10	0.14
	11								8.6	20.0	0.37	1.5	4.0	1.7	0.11
3-10-86	2	6.5	0.016	8.8	15.0	0.027	0.24	1.6	9.3	55.0	0.75	5.0	12.0	5.1	0.14
	11								9.5	77.0	0.27	7.0	15.0	5.7	0.13
	11								8.8	24.0	0.60	2.0	6.0	2.8	0.10
4-10-86	2	6.5	0.01	9.0	23.0	0.017	0.17	-	9.5	70.0	0.65	7.0	15.0	5.77	0.14
	11								10.2	105.0	1.20	11.0	23.0	9.12	0.15
5-10-86	2	6.5	0.01	8.9	12.0	0.014	0.10	-	9.6	75.0	0.90	9.0	19.0	7.25	0.13
	11								8.6	26.0	0.45	2.0	5.0	1.41	0.12
	11								8.8	44.0	0.60	3.0	8.0	2.28	0.15
6-10-86	2	6.5	0.01	8.6	12.0	0.018	0.12	1.2	9.4	53.0	0.67	6.0	13.0	4.30	0.10
	11								9.3	49.0	0.30	5.0	11.0	4.00	0.12
7-10-86	2	6.5	0.012	9.0	20.0	0.018	0.2	-	9.8	74.0	0.75	8.0	12.0	7.5	0.18
	11								9.7	72.0	0.60	7.0	15.0	5.77	0.12
	11								9.1	35.0	0.45	3.5	9.0	4.25	0.14
	11								9.0	30.0	0.37	3.0	8.0	2.45	0.10
8-10-86	2	6.5	0.01	9.5	24.0	0.015	0.12	2.8	9.6	22.0	0.45	8.0	17.0	7.12	0.12
	11								9.3	51.0	0.45	5.0	12.0	5.12	0.14
9-10-86	2	6.5	0.015	9.1	18.0	0.018	0.00	2.6	9.0	52.0	0.37	3.0	8.0	3.42	0.15
	11								9.8	25.0	0.45	8.0	17.0	7.12	0.11
	11								9.5	65.0	0.60	6.0	14.0	5.77	0.12
10-10-86	2	6.5	0.01	8.9	11.0	0.014	0.18	1.6	8.7	22.0	0.37	2.0	6.0	2.25	0.14
	11								9.1	20.0	0.45	2.0	8.0	2.42	0.11
	11								10.2	107.0	0.60	11.0	23.0	9.60	0.14
11-10-86	2	6.5	0.01	8.9	12.0	0.016	0.20	1.6	9.2	32.0	0.45	3.5	9.0	3.25	0.12
	11								10.1	105.0	0.90	11.0	23.0	9.60	0.12
12-10-86	2	6.5	0.013	9.0	12.0	0.016	0.12	1.6	10.2	125.0	0.90	13.0	27.0	10.20	0.16
	11								10.3	140.0	1.05	14.0	29.0	13.60	0.16
	11								8.7	22.0	0.35	2.0	6.0	2.25	0.12
	11								8.9	28.0	0.35	2.5	7.0	3.25	0.13
	11								9.0	31.0	0.40	2.0	8.0	3.25	0.15

Date	Es No	Deaerator inlet-		Deaerator outlet-					C.B.D.						
		PH	SiO ₂	PH	Cond.	SiO ₂	NH ₃	NH ₂	PH	Cond.	SiO ₂	P. Alk.	M. Alk.	PO ₄	Fe
13.10.86	I	6.5	0.015	9.0	15.0	0.02	0.32	1.6	9.2	45.0	0.90	5.0	12.0	5.12	0.1
	II								10.2	152.0	1.25	15.0	31.0	13.60	0.13
	III								9.0	28.0	1.05	3.0	8.0	3.42	0.11
14.10.86	I	6.5	0.01	9.0	20.0	0.014	0.45	3.6	8.9	24.0	0.77	7.0	7.0	2.28	0.16
	II								9.2	31.0	0.65	7.5	8.0	3.42	0.1
	III								9.8	54.0	1.05	6.0	13.0	4.57	0.20
15.10.86	I	6.5	0.01	9.0	12.0	0.014	Trace	1.96	9.0	35.0	0.37	2.5	7.0	2.85	0.14
	II								9.1	38.0	0.75	4.0	9.0	3.42	-
	III								8.5	15.0	0.45	1.0	4.0	0.86	0.12

Unit: NEL YANIPAT
 Month of: October 86

AQUA-HARM
 AQUAPHARM CHEMICAL CORP. LIMITED
 DATA OF COOLING TOWER - 3

Inlet Temperature: _____
 Outlet Temperature: APPENDIX-5.1.3

DL	PRODUCTS ADDED (Consumption in Kg.)					Make up M ³ /hr	Flow down M ³ /hr	Consumption		Tot-H/ Ca-H	TDS	Cond	pH	Cl	F. Cl ₂	Sulfate (ppm, 50 ²)	Metho Blank Dist (ppm)	Org. PO ₄	Meta/ Oxide PO ₄	CC (ppm) Limon (ppm Fe)	NH ₃ (ppm NH ₃)	Zn	Turb	VPC (ppm) ml.	
	Acid kg.	Li ₂ kg.	SP kg.	SP kg.	Adjust 321			M ³	CMH																
1	-	180	-	350	-	9030	800	332	222		592	860	7.9	62	.8		3.5	2.74	3.7	4.07	-	.6	5.5	-	
2	-	150	-	400	-	8430	500	340	232		639	940	7.9	72	1.0		2.61	4.68	2.96	4.0	-	.6	9	-	
3	-	50	-	350	-	9100	500	336	230		625	920	8.0	64	.6		3.95	4.26	2.75	3.92	-	.6	9	6500	
4	15	140	-	350	-	9660	400	336	230		632	930	7.7	72	1.0		3.5	4.72	2.3	3.92	-	.6	9	-	
5	20	140	-	350	-	9385	400	374	254		680	1000	7.6	92	.6		3.95	4.74	2.75	4.4	-	.6	11	-	
6	80	140	-	350	-	9150	600	366	248		714	1050	7.9	88	.6		4.4	4.80	2.3	4.37	-	.7	9	-	
7	240	160	-	350	-	10050	700	408	276		744	1100	8.1	90	.5		3.61	4.21	3.2	4.89	-	.6	7	-	
8	40	60	-	350	-	10275	500	414	282		748	1100	8.1	88	.5		3.5	4.8	3.65	4.71	-	.75	6.5	1400	
9	40	140	-	350	-	10735	-	414	284		737	1100	8.1	85	1.0		3.95	4.27	2.75	4.69	3.1	.70	6.5	-	
10	50	60	-	350	-	9770	400	466	314		804	1200	8.2	80	.2		3.5	4.74	2.75	5.42	-	.5	7.0	2400	
11	100	130	-	350	-	9840	400	440	306		737	1100	8.2	74	.5		3.5	5.74	3.65	4.78	-	.7	6.5	-	
12	100	120	-	350	-	9320	400	440	284		726	1100	8.0	67	1.0		2.17	5.1	4.01	5.51	1.41	.6	6	-	
13	-	-	-	350	-	8650	400	446	310		804	1150	7.9	68	.6		3.95	4.26	2.75	4.85	-	.6	7.5	-	
14	35	50	-	350	-	8750	200	452	308		780	1300	7.69	71	.2		3.06	3.5	5.51	4.96	-	.6	10	-	
15	110	65	-	350	250	10080	1050	446	310		804	1150	7.9	68	.6		3.95	4.26	2.75	4.85	-	.6	7.5	1900	
16	100	120	-	350	-	9680	-	398	268		714	1000	7.7	58	.5		3.5	4.27	3.2	4.33	-	.6	9	-	
17	-	200	-	350	-	6920	100	300	252		680	1000	7.6	55	.6		3.06	6.28	4.09	4.2	-	.9	9.5	-	
18	-	-	-	350	-	6400	10	270	270		670	990	7.7	52	1.2		3.5	5.74	3.65	4.46	-	.8	8.0	-	
19	-	-	-	350	-	8570	-	284	284		737	1100	7.8	66	.5		3.06	5.74	4.09	4.8	-	.8	14	-	
20	-	200	-	350	-	8850	-	450	298		804	1200	7.6	70	.5		3.5	5.32	2.76	5.06	5.8	.9	14	-	
21	-	150	-	350	-	8200	300	460	302		878	1300	7.4	90	NT		4.4	3.85	3.7	5.27	-	1.0	8	-	
22	-	9	-	350	-	8160	400	468	306		666	1275	7.5	90	NT		4.4	5.87	4.17	-	-	1.0	6.5	-	
23	-	260	-	350	-	6750	350	438	296		836	1230	8.0	90	NT		4.86	7.5	4.66	5.4	5.95	1.25	6	-	
24	180	50	250/35	350	-	9055	2150	472	310		871	1300	7.5	100	NT		5.77	7.44	3.74	5.06	-	1.4	8.5	-	
25	200	150	-	350	-	8030	500	387	250		770	1150	7.6	86	NT		5.77	8.94	4.38	4.28	-	1.75	8	-	
26	-	80	-	350	-	7480	400	424	274		871	1300	7.5	110	1.0		7.62	7.88	3.84	4.68	-	1.6	7.5	-	
27	-	60	-	350	-	4460	650	428	278		837	1250	7.4	100	1.0		5.7	7.03	3.24	4.68	-	1.5	10	-	
28	-	60	-	350	-	3000	875	414	264		778	1050	7.8	90	NT		5.31	3.45	5.18	-	-	1.2	8	-	
29	-	-	-	350	-	1950	150	926	210		598	880	7.3	82	1.6		5.91	6.43	3.74	-	-	1.6	6	-	
30	-	-	-	-	-	1350	300	900	200		-	830	7.4	74	NT		6.22	3.0	5.73	-	-	1.4	5	-	
31	-	-	-	-	-	1750	-	238	152		382	570	7.1	52	.6		4.85	6.85	3.24	-	-	1.3	5	-	
MW																									

MAKE UP WATER CHARACTERISTICS

REMARKS:
CORROSION

Date: 7.18.86, 10.1.86, 14.6.86, 16.10.86, 20.10.86
 CR 2.6 2.5 2.45 2.85 2.20

By Sampson Detrad 11.9.86 to 10.10.86 = 2.31

Unit : NFL PANIPAT
 Month of : October '86

ACCAP WATER
 AQUAFORM CHEMICALS PRIVATE LIMITED
DATA OF COOLING TOWER-II

Inlet Temp : _____
 Outlet Temperature : APPENDIX-5.1.4

Dt.	PRODUCTS ADDED (Consumption in kg)					Make up M ³ /hr	Blow down M ³ /hr	Cond. cycle		Tot-H ⁺ Ca-H	TDS	Cond	pH	Cl	F. Cl ₂	NH ₃ (ppm)	M-H ⁺ (ppm)	Org. PO ₄	Meth ⁺ Oxide PO ₄	Meth ⁻ (ppm)	Silica (ppm SiO ₂)	Zn	Turb	VPC (SAB)	SFC	
	Acid	Cl ₂	NaOH	CaOH	Ammonia			On TH	On CaH																	
1		60		200		2200	300	242	164		514	890	7.6	56	1.0	-	5.89	883	4.4	6.18		5.6	4.5	-	-	
2		60		200		2775	500	262	176		550	900	7.9	68	1.0	-	3.18	9.73	4.4	5.18		1.75	5.0	-	-	
3	-	25		200		2200	500	308	210		610	1000	8.0	68	NT	-	3.69	7.8	5.77	5.69		1.75	7.5	200	2400	
4	15	65		200		2575	400	320	218		671	1100	7.8	78	0.3	-	3.77	10.77	4.66	5.12		1.75	7.5	-	-	
5	20	65		200		3100	400	286	194		620	1000	7.7	80	0.8	-	3.03	7.5	4.4	4.21		1.5	8.5	-	-	
6	-	60		200		2350	600	312	212		651	1050	7.7	86	0.2	-	3.7	9.68	5.31	4.68		1.6	6.5	-	-	
7	80	80		200		2375	700	330	220		671	1050	7.8	88	NT	-	4.07	6.2	6.2	6.72		1.6	8.0	-	-	
8	240	140		200		2925	500	358	240		744	1200	7.6	106	0.2	-	4.21	8.37	7.15	5.79		1.6	14.0	400	2200	
9	40	60		200		3200	-	336	226		775	1250	7.7	126	1.0	-	3.93	7.44	5.31	4.0		1.5	9.0	-	-	
10	40	80		200		2965	400	298	202		713	1100	7.8	95	0.5	-	3.42	6.44	4.96	4.19		1.5	7.0	9600	470	
11	50	40		200		2750	400	356	242		823	1300	8.0	100	NT	4.5	4.07	7.11	5.31	4.74		1.5	11.0	-	-	
12	100	70		200		3350	400	334	212		804	1200	7.9	98	0.1	22.8	4.0	6.97	3.95	2.82		1.5	6.0	-	-	
13	100	150		200		2675	400	358	244		806	1300	7.0	126	NT	-	4.07	10.23	2.57	3.92		2.0	7.0	-	-	
14	-	-		200		2810	200	342	210		900	1225	7.7	128	NT	-	4.55	10.05	7.62	3.34		2.4	5.0	-	-	
15	25	30		200	150	3100	1050	306	208		744	200	7.0	108	NT	-	3.26	7.5	6.2	3.7		1.7	14.0	1500	915	
16	110	30		200		3200	-	260	175		600	1050	7.4	86	0.5	-	2.83	8.55	4.4	4.6		1.6	9.0	-	-	
17	100	160		200		2350	400	308	204		682	1100	7.6	112	0.6	-	3.46	10.09	4.4	5.6		1.8	6.0	-	-	
18	-	80		200		3050	400	322	214		671	1100	7.5	110	0.5	-	3.6	11.93	4.8	5.19		2.0	6.0	-	-	
19	-	-		200		3200	-	316	210		670	1100	7.3	104	0.5	-	3.53	11.1	5.77	4.03		2.4	7.0	-	-	
20	-	-		200		2750	-	358	238		775	1050	7.3	126	0.5	335	4.0	4.61	9.52	2.37		2.7	4.5	-	-	
21	-	60		200		3305	300	322	208		737	1200	7.3	130	NT	-	3.8	10.8	8.57	4.84		2.6	4.5	-	-	
22	-	50		200		2825	400	334	214		712	1200	7.4	118	NT	-	-	11.73	8.1	4.76		2.6	12.0	-	-	
23	-	45		200		2700	550	296	190		710	1060	7.8	104	NT	3877	3.56	10.4	6.7	4.68		3.0	4.0	-	-	
24	180	105	59/5	200		2700	2150	316	208		701	1150	7.8	82	NT	-	3.37	10.7	5.31	4.66		2.5	15.0	-	240	
25	200	40		200		2925	500	210	140		589	950	7.7	60	NT	-	2.18	7.5	4.86	4.19		1.6	7.0	-	-	
26	-	50		200		1800	400	238	156		651	1050	7.9	68	NT	-	2.56	9.0	4.66	4.2		1.8	7.0	-	-	
27	-	-		200		1195	650	274	174		682	1100	7.6	70	NT	-	3.12	10.9	5.3	8.31		1.9	7.0	-	-	
28	-	-		200		-	875	260	164		612	950	7.4	60	NT	-	-	5.85	3.15	4.2		2.0	4.5	-	-	
29	-	-		-		-	150	-	-		-	-	-	-	-	-	-	-	-	-		-	-	-	-	
30	-	-		-		1950	900	-	-		-	-	-	-	-	-	-	-	-	-		-	-	-	-	
31	-	-		-		-	-	-	-		-	-	-	-	-	-	-	-	-	-		-	-	-	-	
MW																										

MAKE UP WATER CHARACTERISTICS

REMARKS : Date 7.10.86 to 10.10.86 CR 1.8 11.1 19 265 11.21 Coupon period 11.9.86 to 10.10.86 79

(A) SPECIFICATIONS OF CIRCULATION WATER OF COOLING TOWERS
CT-1 AND CT-2

S.No.	Parameters	Specification (ppm)
1.	pH	7.5-8.0
2.	Turbidity	15
3.	Conductivity	1200 mhos
4.	TDS	700
5.	Zn ⁺⁺	2.0
6.	Meta phosphate	5.0
7.	Ortho-phosphate	5.0
8.	Organo-phosphonate	10.0
9.	Chlorides	100
10.	Free chlorine	0.2 to 0.5
11.	Total Hardness	600
12.	Calcium Hardness	380
13.	Magnesium Hardness	220
14.	Cycles of conc.	3.0
15.	Corrosion rate	2.0 to 3.0 mpy.

(B) NORMS OF BOILER FEED WATER

S.No.	Parameters	Norms (ppb)
1.	TDS	100
2.	Total Iron	10
3.	Total copper	5
4.	Total SiO ₂	20
5.	Oxygen	7
6.	Hydrozine	10-20
7.	pH	8.8-9.2

Appendix to 4.2

Regeneration level on existing plant	: 350 Kg 2% H ₂ SO ₄ 350 Kg 4% H ₂ SO ₄
Resin volume	: 8.75 M ³
Regeneration level	: 80 g H ₂ SO ₄ /lr.
Approx.capacity expected with water of 31% Mg, 55% Ca, 14% Na+k, 66% alkalinity.	: 30.1 g CaCO ₃ /lr.
Approx.throughput of water per regen on design water of 120 ppm cations.	: $\frac{32.8 \times 8.75}{120} \times 1000 = 2392$
Approx use of H ₂ SO ₄ per year(inc.MB)	: 975 te/yr.
Approx number of regenerations per year on cation with 8 hours online time.	: 1200
Approx amount of acid used on cations per year	: 840 tes.
Cost at 1200 R/te for 98% acid delivered	: $1200 \times 840 \times \frac{100}{98} = 10.3$ lakhs
On HCl regeneration level required for 32.8 g CaCO ₃ /lr.capacity	: 30 g HCl/lr.
With 1200 regenerations per year acid use	: $\frac{30 \times 8.75 \times 1200}{1000} = 315$ tes/yr.
Cost at 250 R/te for 33% acid delivered	: $250 \times 315 \times \frac{100}{33} = 2.4$ lakhs

APPENDIX TO 4.4

Outline of brine wash method

Run unit to end point (i.e. resin in exhausted state)

Backwash at normal regeneration rate

Add 2 Bv of 10% w/v NaCl + 2% w/v NaOH at ambient temperature.

Leave to soak for as long as possible (6-12 hours minimum)

Displace with 3 Bv of water

Carryout backwash, settle and caustic inject stages of normal regeneration.

Repeat caustic inject stage.

Carryout displacement and rinse stages of normal regeneration.

Return to service.

APPENDIX TO 4.5

Flow rate of urea condensate	: 55 te/h.
Approx cost raw water	: 0.77 R/te.
Approx cost of DM water @ 20°C	: 10 R/te.
Approx cost of condensate @ 60°C	: 30 R/te*
Cost/day of condensate if dumped 55 x 24 x 30	: 39,600 R.
Cost/day of extra make-up due to lost condensate. 55 x 24 x 10	: 13,200 R
	<u>52,800 R/day</u>

* Estimate based on UK value of condensate being 3 times DM value.

APPENDIX TO 5.1

5.1.1 Cooling towers on Panipat plant

Name	CT1	CT2	CT3
Plant served	Ammonia	Urea	Urea condenser
Type	Induced draught, cross flow, open evaporative		
Number of cells	8	3	1
Circulation rate m ³ /h.	17500	-	-
$\Delta T^{\circ}C$	11	10	-
Hot return temp. $^{\circ}C$	44		
Volume m ³	7500	3500	-
Evaporation te/h.	350	150	-
Concentration factor	3	3	3
Side stream filtration	2.3%	2%	Nil
Half-life,	29.6	32.2	-

APPENDIX TO 5.1

5.1.2 Cooling water chemistry in use in November 1986

Chemical parameter	Role	Effect if Hi	Effect if Lo
Hexa-metaphosphate (HMP)	Cathodic corrosion inhibition + Limited dispersancy of calcium as positively charged colloidal particles within defined pH limits.	Increased corrosion protection. Increased danger of higher $O-PO_4$ giving scaling.	Serious pitting.
Organo-phosphonate (HEDP)	Calcium scaling inhibitor within defined pH limits.	Minimal	Excessive scaling.
Zinc	Cathodic corrosion. Inhibition within defined pH limits.	Danger of excessive $Zn(OH)_2$ precipitating as foulant.	Serious pitting.
Aqua 4000	Anionic dispersant. Role unclear from all discussions (see text)	?	?
Ortho-phosphate	Not added deliberately but formed by hydrolysis of HMP.	Excessive PO_4 scaling.	Desirable state.
pH	Critical for allowing other agents to work correctly.	Precipitates Zn giving $Zn(OH)_2$ (see above) causes calcium carbonate and phosphate scales.	Causes fast hydrolysis of HMP to $O-PO_4$ causes corrosion.

APPENDIX TO 5.1

5.1.3 Synthesis Gas compressor Intercooler 3rd stage

Water in shell, carbon steel, 2 pass, U-tube bundle

Design water velocity : 1.1 m/s
Design gas inlet/outlet temp. : 114°C/43°C
Design water inlet/outlet temp. : 33°C/43°C *

Failure mechanism:

Deposits, SRB corrosion and possibly vibration

Deposit analysis[†]:

PO ₄	: 36.9%
Ca	: 10.9%
Fe	: 7.5%
Zn	: 16.5%
Loss on ignition	: 32%
Acid insoluble	: 1.8%

Presence of algae, bacteria and coal particles

Failure History:

Commissioned - 1979

Failed : January, 1981
March 82, May, 82, June, 82, June, 82
April 83, November, 83, November, 83
June, 84, June, 84, July, 84.

Changed April 85.

* A 0°C approach temperature is impossible but this was the data in a report. (Possibly a typing error).

+ This adds up to greater than 100% because some components will be included in two analyses e.g. some Zn will volatilise on ignition.

APPENDIX TO 5.2

5.2.1 Suggested specification of cooling water chemistry

Parameters	Max	Min	Average
HMP ppm as PO ₄	50	3 (15)	5 * (20)
HEDP ppm as PO ₄	40	8	10
Zinc ppm as Zn	3	1	2
Aqua 4000	See text		10
O-Phosphate ppm as PO ₄	5	0	
pH	8.0	7.5	7.7
Total bacteria (UPC/ml)	1,00,000		20,000
SPB (per 100ml)	300	Nil	100
Calcium ppm CaCO ₃	600	50	250
Concentration factor	5	1	3

* The Panipat specification was 5 ppm average. See text for explanation of possibility of 20 ppm.

APPENDIX TO 5.3.1

5.3.3 Selection of Biocides

Chemical	Advantage	Disadvantage
Methylene bis-thiocyanate		Not effective at pH ≥ 7.5
Na pentachlorophenate	Effective over a long period.	Effects sewage treatment.
Polyamine Quaternary amine	Easily biodegradable Good dispersant Low toxicity to man	Adsorb on suspended particles. Can cause foaming Incompatible with anionic polymer.
Organo-bromine	Easily biodegradable Compatible with all other treatment chemicals.	Not good against algae.
Iso-thiazolone	Biodegradable Good dispersant	Not good against SRB.

दि फ़र्टिलाइजर्स एण्ड केमिकल्स, ट्रावन्कोर लिमिटेड
The Fertilisers And Chemicals, Travancore Limited

F A C T

Report on visit of D G Hooper on behalf of UNIDO to
Fertilisers and Chemicals Travancore Ltd., India
to discuss Water Technology Management

November 24th - 26th, 1986.

Prologue

This report is essentially in three parts:

- (i) Summary of conclusions and recommendations.
- (ii) Notes on the discussions held with F&CT personnel including general data, concepts and reasons for reaching certain conclusions and recommendations.
- (iii) Appendices containing detailed technical information used in the discussions or forming the technical recommendations.

Contents - FACT

1. Summary of conclusions and recommendations.
2. Introduction.
3. Sulphuric Acid Plants.
 - 3.1 Boiler Problems.
 - 3.2 Chemical Cleaning.
4. Ammonia Plants.
 - 4.1 Demineralisation Plant on Reform Unit.
 - 4.2 Reform Plant - Flue Gas Boiler (FGB).
 - 4.2.5 Reform Plant - Reform Gas Boiler (RGB).
5. Cooling Systems.
 - 5.1 Introduction.
 - 5.2 Root cause of problems.
 - 5.3 Recommendations for Cooling Systems.
 - 5.4 Microbiological Aspects.
6. Chemical Cleaning.
7. Analytical Control.
8. Acknowledgements.

1. Summary of Conclusions and Recommendations

1. The quality of raw water available to the Udyogamandal site is a corrosive one, difficult to treat in cooling systems and one which has given problems in this area.
2. Pretreatment of this water is necessary and the new plant, currently being built, should help alleviate some problems.
3. The anti-corrosion programme chosen for the cooling water is the best but it has not been applied correctly. An explanation of the principles and recommendations for future control are given.
4. The operational policy of the demineralisation (DM) Plants on the sulphuric and ammonia installations has been developed along technically deleterious lines. These errors are explained and recommendations for future control are given.
5. Failures in the Sulphuric Acid boiler are potentially as a result of misapplication of standards following the change from softened to DM water. Recommendations for future control are given. If successful this would obviate the need for mechanical modifications e.g. raising the steam drum.
6. Potential corrosion of the sulphuric acid economiser is identified and recommendations for extra inspection are made.
7. In Several cases a clear need for chemical cleaning has been identified. This is a function of the age of the equipment and the type of water technology employed. It is recommended that E.C.T personnel receive training in this field to best take advantage of the techniques.
8. Some of the techniques available in the Laboratory are inadequate for the control of the site and recommendations for upgrading the service are made.

2. Introduction

Fertilisers And Chemicals Travancore Ltd. (FACT) has evolved on the Udyogamandal site since 1945. It now consists of two Oil Gasification Plants, a cryogenic Oxygen Plant, two cold gas quenching Ammonia Synthesis Loops, a naphtha steam reform Ammonia Plant, two Sulphuric Acid Plants, one Phosphoric Acid (100 te/d, P_2O_5), a two stream Ammonium Chloride Plant (75 te/d), a two stream Ammonium Sulphate (600 te/d) and two NPK granulation Plants (350 + 150 te/d). Three auxiliary boilers are available to balance the site steam supply but this usually runs in a surplus.

New Sulphuric Acid and Ammonium Sulphate Caprolactam Plants are under construction as is a new water pretreatment plant. An effluent treatment plant takes all the site liquid effluent which is treated before being put to a brackish water river. Further details are in Appendices 2.1 to 2.6.

The water supply is from a fresh water river with an analysis (Appendix 2.7) showing it to be a high turbidity, low TDS water with only moderate organics. In drought conditions the salinity can rise significantly and in the monsoon the organics can rise. It is a naturally corrosive water.

3. Sulphuric Acid Plants

3.1 Boiler Problems

The boiler system was uprated and converted to demineralised water feed (from softened) in 1980. Frequent failures have subsequently occurred in the top coil bundle requiring renewal every two years, causing excessive lost production and damage to the plant itself. Figure 1 in appendix 3.1 is a sketch of the boiler system.

Appendix 3.1 - Sketch of Sulphuric Acid Plant Boiler System

Figure - 1

Demineralised water is pumped through a pressure deaerator in which oxygen is reduced ~~from~~ ^{to} 0.04 ppm. Hydrazine is added to maintain 0.2 ppm in the feedwater and the water passes through the economiser at pH 6.5-7.5 to the steam drum where trisodium phosphate is added. Typical control parameters and analyses of deposit from the region of the boiler failures are shown in Table 1.2 in Appendix 3.1. The points discussed were the relatively high values of iron, silica and TDS in the boiler water as the boiler failure mechanism is creep of the tubes following loss of heat transfer due to the accumulation of mainly iron and silica.

In a demineralised (DM) water boiler, the levels of impurities which can be tolerated are much lower than in the same boiler (independent of pressure or heat flux) on softened water feed. With DM feed of less than 1 ppm TDS and 0.8 ppm SiO_2 the boiler, with 20 ppm phosphate (35 ppm as Na_3PO_4) should not have more than 100 ppm TDS and 40 ppm SiO_2 . The vital questions therefore are to find out what the rest of the TDS are, what trouble they may be causing and how to limit them. Some analyses were initiated during

: 3 :

the visit, but not all the results were available at the time of writing this report. However, studies of the way the DM Plant was being operated were discussed on the basis of data collected by F&CT personnel.

The DM Plant was being run to a conductivity end point of 30 $\mu\text{S}/\text{cm}$ exit the strong anion bed. Figure 2 in Appendix 3.1 shows the unseen effect (in terms of conductivity) of silica leakage under such a philosophy. Although conductivity was not recorded, it can be assumed to have finally broken through at around 22 hours, reaching 30 $\mu\text{S}/\text{cm}$ at 23 hours. Figure 2 Appendix 3.1 is a sketch of how the conductivity would be ^{expected} expressed to behave. For a DM Plant of this type the cation unit should be regenerated at a sodium concentration of 0.5 ppm and the anion at a silica value of 0.5-1.0 ppm (or regenerated as a linked pair on the first limiting ion). From Figure 1 Appendix 3.1 this would be after ~~ca~~ 13 hours on line. It is totally false economy to run the plant on due to the theoretical danger of silica scaling in the boiler. Additionally, again not recorded, bicarbonates break through from the resin at the same time as silica and will pass to the boiler. They could be part of the high TDS, possibly linked with sodium which may also have leaked from the ^{run} overness cation.

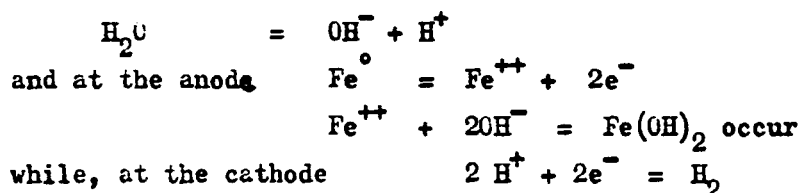
It is recommended that some way of "catching" the end point is developed and regeneration initiated at that time. For example (best method to be assessed by F&CT personnel):

- i) Frequent (hourly) manual analysis of silica
- ii) On-line analysis of silica.
- iii) Use of a lower conductivity set point to catch the sodium breakthrough (e.g. if normal conductivity is less than 0.2 $\mu\text{S}/\text{cm}$ set trip at 2 $\mu\text{S}/\text{cm}$ exit the strong anion. This will only successfully limit the silica concentration if the cation and anion are closely balanced in operation on the F&CT water).

: 4 :

iv) Measure conductivity exit the strong anion after passing the sample through a small column of cation ^{resin} iron in the H⁺ form. This sensitises the measurement by removing traces of sodium from the water such that, when silica and bicarbonate breakthrough occurs, a sharp increase in conductivity is seen which can be used to to raise a signal or alarm.

The high iron figure may be due to corrosion of the feed and economiser system as the old high pressure phosphate pumping system (from the softened water days) is still in commission. Thus, although the water is deaerated and hydrazine dosed, without alkali it is still highly corrosive at a pH less than 8.0. The corrosion mechanism is not really a "chemical" effect but a true electrochemical one where :



Metal is thus lost and, in practice, it appears as fairly uniform corrosion because an infinite number of cathodes and anodes exist at the same time (and are even liable to change their polarity). Polarisation and passivity never occurs because flowing water removes the Fe(OH)₂ and H₂. It is recommended that some alkali, e.g. the phosphate is added immediately after the deaerator to raise the pH ^{at deaerator} less than 8.0. Critical inspection should be made of the economiser and feed lines at the next shutdown including thickness measurements at selected points to ascertain its condition.

The values in Table 3, Appendix 3.1 are recommended as control parameter: for the boilers to lessen the risk of scaling on high heat transfer surfaces. These should be reviewed after 1-2 years operation.

3.2 Chemical cleaning

It would be standard practice exchanging water chemistry e.g. softened to DN feed, to chemically clean a boiler to remove any old deposits from one regime before attempting to impose another to avoid

chokes and failures. The analysis in Table 2 Appendix 3.1 shows that some calcium and magnesium are still in the boiler. This may be due to old deposits or a further penalty of overrunning the DM Units. Experience is showing that failures occur in the top coil after $1\frac{1}{2}$ to 2 years operation. Improvements in the water quality may help this situation but that cannot be guaranteed (the uprating of the boiler may have created a particularly localised hotspot that still leads to excessive scale formation). However, if it will last more than 1 year, chemical cleaning to return the tubes to an "as new" condition should prolong their life further and it is recommended that chemical cleaning is carried out at the next shutdown in 1987. An outline method is given in Appendix 3.2 but this should not be treated as a complete specification. See 6.

4. Ammonia Plants

4.1 Demineralisation Plant on Reform Unit

The DM Plant was being run to a conductivity end point of $0.5 \mu\text{S}/\text{cm}$ exit the mixed bed unit. The original design was for 720 M^3 between regenerations (daily regeneration) but EACT personnel found they could get 3000 M^3 before the conductivity varied. They routinely run to 2000 M^3 throughput or the $0.5 \mu\text{S}/\text{cm}$ end point.

The discussions in 3.1 are relevant to this plant as well and were reiterated i.e. potential breakthrough of silica and resultant scaling of heat transfer surfaces. Silica analysis is not routinely carried out on the boilers and no study has been carried out on this DM Plant so no conclusions could be drawn. A one-off analysis had shown 3 ppm SiO_2 in the boiler which is not unacceptable.

4.2 Reform Plant - Flue Gas Boiler (FGB)

This boiler has gradually deteriorated in performance throughout the life of the plant, the design and attained temperatures being shown in Table 1, Appendix 4.2. The general water chemistry of the boiler is shown in Table 2, Appendix 4.2.

After discussion it was concluded that nothing fundamental appeared to be wrong in the water chemistry except that insufficient analysis was being done to fully control and understand the boiler. It is recommended that the suggestions in Table 3 Appendix 4.2 are followed for future records.

There is a need to clean the boiler as it looks as though it has gradually fouled over the years. This may be due to silica or iron oxides laid down on line, or corrosion occurring off-line. The only way to be certain it is to cut a tube out and section it although it is accepted that this would be very difficult to do and is probably unjustified. The boiler is not a difficult one to clean and lends itself to mechanical or chemical methods i.e. jetting or gas lift circulation with HCl and HF⁹ or other fluoride. See 6. The excess heat escaping this boiler is expensive energy and much of it should be recovered after cleaning giving a direct payback on the exercise. Chemical cleaning is recommended as the preferred technique.

4.5 Reform Plant-Reform Gas Boiler (RGB)

Problems have been experienced on the gas side of this boiler necessitating frequent changes but, to date, nothing from the water side. It is hoped that this will continue but good control of the waterside is important because RGB's the world over are usually the ones causing the most problem in single stream ammonia plants. In the absence of any detailed information it is recommended that the RGB is controlled to the same specification as the FGB. (Table 3, Appendix 4.2). These are high for world RGB's on DM water and may need to be reviewed in 1-2 years time.

5. Cooling Systems

5.1 Introduction

The symptoms described by all the plants operating cooling systems were similar and discussion of all the details suggest a common problem.

Typical analyses of the ammonia synthesis and reform ammonia plants cooling waters are shown in Table 1 in Appendix 5.1. Analyses of deposits from heat exchangers are shown in Tables 2-3 in Appendix 5.1. These values

were discussed in connection with inspection of the ammonia vaporiser on the synthesis plant which was being wirebrushed and washed on 24.11.86 and the scrapped interstage cooler from the reform ammonia plant.

5.2 Root Cause of Problems

The incoming water (appendix 2.7) is very "thin" and corrosive and contains some suspended solids. Despite concentration in the cooling systems it is still highly corrosive (Negative langelier Index) and requires corrosion inhibitors at all times to prevent damage.

The anti-corrosion programme chosen of zinc:chromate:phosphate is the best one available but it was not being controlled correctly and was in fact probably making the situation worse rather than better. The underlying philosophy of the treatment needs to be understood to explain such a statement.

Chromate is an anodic inhibitor capable of excellent inhibition in neutral pH's at levels of 100 to 10000 ppm. It works by laying down an insoluble film of ferric and chromic oxides. The higher levels are needed when pitting ions are present at high levels e.g. chlorides. The lower level of 100 ppm is required to ensure that thermodynamically iron (chrome VI) chromate is precipitated as the main reaction rather than the reduction of chrome VI to chrome III with oxidation of iron to iron II or iron III thus causing corrosion, often as pits, rather than inhibiting it. Zinc is a cathodic inhibitor capable of excellent inhibition in neutral and acid pH's at levels of 1-10 ppm soluble zinc. It works by being precipitated as a film of zinc hydroxide in the region of the cathodic reduction of oxygen. Phosphates work in a combination of ways depending on their concentration and molecular weight. Ortho-phosphates act as anodic inhibitors at high concentration by producing iron phosphate films while polyphosphates are predominantly cathodic. Polyphosphates react with divalent cations e.g. Ca^{++} , Zn^{++} to form positively charged colloidal particles which inhibit scale formation. These colloids migrate to cathodic areas where they build up a film which stifles the reaction and so inhibits corrosion. 2-100 ppm as PO_4 are required for corrosion protection.

The synergistic interactions of all three mean that lower concentrations of each agent are needed to produce an overall better effect as the complexes of chrome, iron, phosphate and zinc formed on the metal surfaces are better than the individual component films. Thus programmes with chromate levels of 20-30 ppm, zinc of 1-2 ppm and phosphate of 10-20 ppm are effective, relatively cheap and relatively easy to dispose of the effluent treatment systems. However, should parts of the programme fail for any reason the synergism is lost and the "rules" of single inhibitors apply. For example, Table 1, Appendix 5.1 shows that in the ammonia plant there is negligible phosphate and no zinc at all. Under such conditions the 20 ppm CrO_4 will act as a pitting agent and promote corrosion further. No cathodic inhibition will occur and at the neutral to acid pH's in operation widespread corrosion will take place producing large amounts of iron oxides and hydroxides. These will settle out as fouling sludges in low flow areas where they will build up as encrustations, hardening by oxidation and dehydration. Occluding the metal surfaces will lead to reduction in heat transfer and differential aeration at the metal surface, initiating further electrochemical corrosion cells and so continuing the cycle. The total absence of zinc indicates a demand at cathodic sites and to begin to break the above vicious circle this demand must be met.

Much discussion centred on the need for dosing phosphate in FACT systems containing such low levels of calcium and alkalinity where scaling is not a problem but the danger of zinc precipitation with ortho-phosphate could further deplete the soluble zinc. My experience has been that even at pH 6.5-7.5, with ortho-phosphate upto 10 ppm I have been able to maintain 1-2 ppm soluble zinc in cooling waters and successfully control corrosion and scaling. Thus the recommendations are to retain all three agents to give more insurance of maintaining the synergism.

5.5 Recommendations for Cooling Systems

Corrosion cannot be stopped totally nor can it be so readily controlled in dirty systems as in clean ones i.e. the inhibitors must get to the parent metal surface to inhibit corrosion. If they have to diffuse through debris they may not arrive at the surface at a sufficient rate or concentration to inhibit the reactions.

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Initially it is recommended that dosing of polyphosphate, chromate and zinc is carried out once per shift in sufficient quantities to satisfy the solution requirements of each item. The recommended control parameters are in Table 1, Appendix 5.3.

The demand for zinc may be staggeringly high and continued shiftly addition may apparently not satisfy it. However, provided the added zinc is being consumed by the cathodic sites it should continue to be added. It is to check that this is where the zinc is going that a specification for soluble and total zinc is given. If the total is high and soluble non-existent a zinc dispersant will be required but this can be reviewed at a future date. In most cases at pH 6.5-7.2 no dispersant is required.

Once the cathodic sites start to be inhibited the whole corrosion process should slow. However, debris will spall from the surfaces as passive films are established both revealing fresh surfaces which will consume inhibitors and producing higher levels of suspended solids which could settle in other areas exacerbating the problem we are trying to solve. Specialised dispersants for iron are available eg from Aquapharm, and their use should not be ignored. Sales advice should be sought from the suppliers but great care should be exercised in their use.

The state of the equipment is in an advanced stage of tuberculation and corrosion so more fouling and failures must be expected. To fully bring the equipment back to a good condition it will be necessary to change or chemically clean key exchangers and interconnecting pipe work and subsequently vigorously maintain the chemical programme recommended. The mechanical cleaning being carried out on the ammonia vaporiser, while removing a lot of material, was not efficiently cleaning the tubes. They were left with deposit on them and in a rough state. Even perfect water chemistry control would have difficulty in establishing a passive film and minimising further fouling and corrosion on such tubes. Chemical cleaning or jetting at 8-10000 psi is recommended.

The deposit analyses in Table 3, Appendix 5.1 shows that silt and organics are further significant components. With the commissioning of the new water pretreatment plant not far away, no proposals are made to deal specially with these materials as their ingress will be significantly reduced with the new plant.

5.4 Microbiological Aspects

Algae are a minor problem and potentially some of the organics referred to above are bacterial in origin but otherwise only one problem has been seen due to microbiological activity and that has been the bacterial oxidation of ammonia to nitrite/nitrate in the reform Ammonia Plant leading to significantly reduced pH's. The addition of 50 ppm of proprietary methylene-bis-isothiocyanate every 10 days appears to solve this problem although there are indications that fouling (reduced heat transfer) may be worse with this better control. Table 5, Appendix 5.1 shows analyses before and after biocide addition which indicate less silt but more iron, aluminium and organics.

It is believed that, at the higher pH, aluminium and iron have precipitated as hydrated complexes rather than being washed out of the system. These complexes, as in the pre-treatment Plant, will be associated with organics. Dispersants may be advantageous in moving such foulants from the system.

Side stream filtration is an effective manner for removing suspended solids from circulating cooling water (including algae) and it is recommended that trials should be conducted with a possible view to specifying a process design for use at RCT. Improved corrosion control and water quality from the pre-treatment Plant should lessen the fouling problems but dust ingress from the atmosphere will always remain.

6. Chemical Cleaning

Chemical cleaning is an integral part of water technology management both in terms of rectifying wrongs and also as a routine maintenance technique against the inevitable build up of foulants and corrosion films.

In the context of the Udyogamandal installations it has been mentioned several times for different problems and it is recommended that F&CT Personnel become trained in the use of the techniques. Such training is beyond the scope of this consultancy visit although an outline of some of the methods available, the flexibility, power, cost-effectiveness and danger have been given. Training courses are available through ICI if required.

It is recommended that a small team (two or three people) from the technical services are trained in Chemical Cleaning Technology and are always involved with the technical, planning and supervision aspects of carrying out cleans. These people would rapidly become in-house experts in the technology and enable F&CT to confidently exploit the benefits. For example, approximately 56 lakhs were spent by ICI on Chemical cleaning in the last year where paybacks in terms of identifiable increased production (heat transfer efficiency) and improved plant availability (reduced outage for repair) were measured in hours or months rather than years.

7. Analytical Control

The evolution of the Udyogamandal site has been commented upon showing the increased numbers and complexity of Plants. In concert with this there is a need for improved and more sophisticated analytical techniques to monitor and control the newer plants. This is especially true on making the step change from softened water to DM water Chemistry, the units of materials that cause problems change from ppm to ppb (10^{-6} to 10^{-9} gram per litre) and the analysis of deposits become much more demanding in terms of troubleshooting.

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The discussions were limited on a number of occasions when analytical techniques were not available or insufficiently accurate to give the information required. For example, with the water technology currently used by FACT at Udyogamandal, at least the following instrumental techniques should be available and it is recommended that they are obtained.

Analysis	Equipment/Technique	Range required
pH	Electrode/MV meter	0 - 14 ± 0.1
Na	Electrode/MV meter or AA *	Down to 0.005 ppm
Fe, Al, SiO ₂ , Ca, Mg.,	AA *	0.02 ppm

AA * - Atomic Absorption or Emission

8. Acknowledgements

The technical discussions were held with personnel from the Technical Services Department and production units under the guidance of the Chief Superintendent (Technical Services), Mr. P. Ramakrishnan to whom I extend particular thanks for his personal help and organisation of private comforts as well as technical requirements.

Personnel involved in the discussions included:

Mr. T. Nandakumar, Mr. Kochukrishnan, Mr. T.P.S. Nair, Mr. A.S.G. Nayar, Mr. John Davis, Mr. Job Joseph and Mr. Hemachandran.

Appendix 2.1

OIL GASIFICATION PLANTS

Feed stock	:	Naphtha and O ₂
Duty	:	Produce Hydrogen by partial oxidation of Naphtha.
Water Services:		
Demin.	:	One stream
Cation	:	anion
Boilers:		
Type	:	2 off waste heat water in shell. 5 bar steam.
Cooling:		
Type	:	2 off countercurrent, induced draught, open evaporative separate hot well and cold well.
Duty	:	Admiralty Brass condenser on turbo alternator.
Chemical control	:	None

Appendix 2.2

OXYGEN PLANT

Feed stock : Air
Duty : Provide O₂ and N₂ for site
for oxidation of naphtha
and nitrogen for ammonia

Water Services

Cooling:

Type : 1 off countercurrent, induced
draught, open evaporative
separate hot well and cold well.
Duty : Compressor coolers
Chemical control : Chromate, Phosphate and zinc.
No biocide

Appendix 2.3

AMMONIA SYNTHESIS (2 STREAM)

Feed stock : Hydrogen from gasification and
Nitrogen from Oxygen Plant.
Duty : Synthesis Ammonia (ICI loops)
80 + 140 te/d

Water Services

Cooling:

Type (i) : Once through cascade systems.
(ii) : 2 off countercurrent, induced draught
open evaporative separate hot well
and cold well (returns are open channels)
Duty (i) : Ammonia converter effluent cooler
(ii) : Compressor cooling, inter coolers,
Ammonia vapouration.
Chemical control (i) : None
(ii) : Chromate, Phosphate and Zinc.
No biocide.

Specifications Evaporative = 43 te/h circulations = 1600 M³/h
 Δ T^oC = 4^oC CF = 3 - 4

Problems

Poor Chemical control ; ingress of debris to return channels;
sludge deposition; loss of heat transfer; frequent cleaning of
intercooler by taking compressors off line (monthly). See 5.

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Appendix 2.4

STEAM REFORM AMMONIA PLANT

Feed stock : Naphtha, air and water
Duty : Produce 150 te/d ammonia
Commissioned : 1971

Water Services

Demin. Single stream filter: Cation : Anion : MB CCR systems

Resins 225 N(ip) 225/FF(ip)

Final quality $\leq 0.5 \mu S/cm$ $\leq 0.2 ppm SiO_2$

quantity : 30 m³/h

Boilers : Flue gas boiler (see appendix 4.2)
Reform gas boilers
Shift converter boiler 2.5 te/h
Gas preheater

Pressure : 55 bar

Chemical Control : Hydrazine + Trisodium Phosphate

Cooling:

Type : 1 off countercurrent; induced draught,
open evaporative separate hot and cold well.

Duty : Various exchangers in Plant.

Chemical control : Chromate, Phosphate and zinc.
Biocide; Methylene bis-isothiocyanate,
50 ppm every 10 days.

$\Delta T = 8^\circ C$ Circulation=1620 m³/h

CF = 5 - 5

Problems

Fouling of boiler (see 4.2)

Cooler failures; fouling and loss of heat transfer (See 5)

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Appendix 2.5

SULPHURIC ACID PLANTS (2 OFN)

Feed stock : Sulphur
Duty : Produce 160 + 600 te/d Sulphuric Acid (Gisum)

Water Services

Demin: Two stream filter cation : Anion CCR system
Resins 225 : N(ip)
Quality < 0.2 μ S/cm < 0.8 ppm SiO₂
Quantity 60 M³/h

Boilers : 3 coil, natural circulation, flue gas duct,
water in tube, waste heat boiler.
15.3 bar steam 25.6 te/h @ 204°C

Chemical control : Hydrazine + Trisodium Phosphate

Cooling:

Type : Once through, cascade systems
Duty : Cool SO₂ gas before absorption
Chemical Control : None

Problems:

Changed from softened water to demineralised when uprated
1980. Boiler failures. Repeated failures since (See 3.1)

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Appendix 2.6

PRE-TREATMENT PLANT

Water source	: River controlled by upstream damming.
Analysis	: Appendix 2.7
Flow rate	: 3000 m ³ /h
Filtration	: 10 off gravity filter. Fluted underberbe with ~ 5cm. balls: $\frac{3}{4}$ " pebbles; gravel: sand. Water backwash only on excess head of water.
Chlorination	: Full flow after filtration.
Drinking water:	
Source	: Filtered water from pretreatment plant
Treatment	: Lime + Alum dosed Sedimentation : Filtration Chlorination.

Appendix 3.1

Table - 1 TYPICAL CONTROL PARAMETERS IN SULPHURIC ACID PLANTS

Analysis	Boiler	Feed
Phosphate as PO_4	20 - 50 ppm	
Silica as SiO_2	40 - 60 ppm	
Iron as Fe	0.1 - 0.4 ppm	
TDS mg/l	400-800 ppm	
P -value $mgCaCO_3/l$	10 - 50 ppm	
pH	10.0 - 10.5	6.5 - 7.5
Hydrazine		0.2

Table - 2 DEPOSIT ANALYSIS FROM ENDED COIL OF SULPHURIC ACID PLANT BOILER

<u>Analysis</u>	<u>%</u>
Iron as Fe_2O_3	80.52
Silica as SiO_2	11.68
Calcium as CaO	1.08
Magnesium as MgO	1.44
Phosphate as PO_4	2.41

Figure - 2 EXPECTED PATTERN OF CONDUCTIVITY TIME

Appendix 3.1

Table - 5 SUGGESTED BOILER AND FEED WATER STANDARDS
FOR SULPHURIC ACID PLANTS

Analyte	Boiler	Feed
Phosphate as PO_4	20 - 40 ppm	
Silica as SiO_2	2 - 40 +ppm	
Iron as Fe	<0.2 * ppm	<0.05 ppm
TDS as mg/l	55 -100 ppm	
pH	9.5 - 10.5	8.0 - 9.0
Hydrazine as N_2H_4		0.1 - 0.2 ppm

* Iron will not be present in solution at this pH. The aliquot for this analysis should be taken first from the plant sample which has been thoroughly shaken.

+ These figures assume there is no colloidal silica in the feed water. If there is these figures still stand as maxima in the boiler - Colloidal silica will be controllable once the new pretreatment plant is commissioned.

Although the maximum recommended is 40 ppm, the normal would be expected to be in the range of 2 - 10 ppm with good Plant operation.

Appendix 3.2

SUGGESTED APPROACH TO CHEMICAL CLEANING OF
SULFURIC ACID BOILER

1. Plan job thoroughly from Chemical, Engineering and management aspect
2. Set-up external pumping/tank system to enable heating to 80°C and pumping at rates equivalent to 1 to a minimum of 5 feet/second.
3. Flush to drain at rates equivalent to > 5 feet/second.
4. Use 5% W/V HCl at 80°C with 0.1% Moline 215 inhibitor. Circulate at a rate equivalent to 1-5 feet/second, without heating, for about 6 hours or until no further increase in iron concentration occurs in any one hour. Continely test effectiveness of inhibitor by a float test with steel wool balls. If the concentration of iron reaches 1.5% dump the acid and start again. (Ensure sufficient acid + inhibitor are available for this before starting).
5. Add 0.5% ammonium bifluoride to the acid and circulate for a further one hour. Drain.
6. Refill with 0.1% citric acid. Ambient temperature. No inhibitor.
7. Flush to drain at rates equivalent to > 5 feet/second.
8. Refill with ~~dey~~^{DM} water and heat to 60°C. Add 0.5% citric acid and circulate, without heating or inhibitor for one hour.
9. Add ammonia to create pH >10 .
10. Add 0.5% sodium nitrite and circulate for two hours.
11. Drain and flush with DI water until nitrite free.
12. Fill with DI water dosed with ~ 50 ppm ammonia (pH >10.5) to carry out pressure test if required and/or to store for up to several days until the end of the shutdown.

Appendix 4.2

Table - 1 DESIGN AND ATTAINED TEMPERATURE ON THE FGB

	Design	Attained
Gas inlet	828 °C	775 - 800 °C
Gas outlet	288 °C	350 - 400 °C
Water	241 °C	244 °C 241

Table - 2 GENERAL WATER CHARACTERISTICS ON THE FGB

<u>Analyte</u>	<u>Range</u>
pH	9 - 10
M - alkalinity	60 - 100 ppm CaCO ₃
Phosphate as PO ₄	50 - 55 ppm

Table - 3 SUGGESTED ANALYSES FOR CONTROL OF FGB

<u>Analyte</u>	<u>Range</u>	<u>Frequency</u>
pH	9 - 10	Shiftly
Phosphate as PO ₄	20 - 40 ppm	Daily
Silica as SiO ₂	2 - 10 ppm	Daily
TDS mg/l	55 - 100 ppm	Daily

Appendix 5.1

Table -1 TYPICAL ANALYSIS OF COOLING WATER IN ALUMINA PLANTS

Analyte	Synthesis plant	Reform Plant
pH	6.2 - 7.0	6.5 - 7.0
Alkalinity ppm CaCO ₃	20	24
Chloride as Cl	13 - 17	15 - 24
Total Hardness ppm CaCO ₃	70 - 130	72
Ortho-Phosphate ppm PO ₄	4	5
Poly-Phosphate ppm PO ₄	2	Nil
Chromate ppm CrO ₄	20	20
Zinc ppm Zn.	Nil	Nil
Concentration Factor	3 - 4	3 - 5

Table - 2 DEPOSIT IN ALUMINA VAPORISER ON SYNTHESIS PLANT.
SAMPLED ON 24.11.1986

<u>Analyte</u>	<u>%</u>
SiO ₂ + acid insolubles	3 - 4
Cr ₂ O ₃	4
PO ₄	3 - 4
Fe ₂ O ₃	70

Appendix 5.1

Table - 3 DEPOSITS IN REFORM AMMONIA PLANT

Analyte	Inter stageCooler+ % April 1986	Tower Basin + % April 1986	Tower Basin * % November 1986
Loss at 230 - 500°C	29.56	15.58	19.8
SiO ₂ + acid insoluble	16.0	58.16	25.2
Fe ₂ O ₃	29.98	10.58	25.5
Al ₂ O ₃	0.26	0.15	15.77
Cr ₂ O ₃	5.12	4.12	0.4
CaO	0.65	4.06	ND
MgO	4.25	5.12	5.4
SO ₃	1.79	0.76	ND
P ₂ O ₅	4.98	3.28	5.3
Zn	ND	ND	ND

ND = Not Determined

+ Before biocide addition pH of water typically pH 4 - 6.2

* After biocide addition pH of water typically pH 6.5 - 7.2

Appendix 5.3

Table - 1 SUGGESTED CONTROL PARAMETERS FOR COOLING SYSTEMS

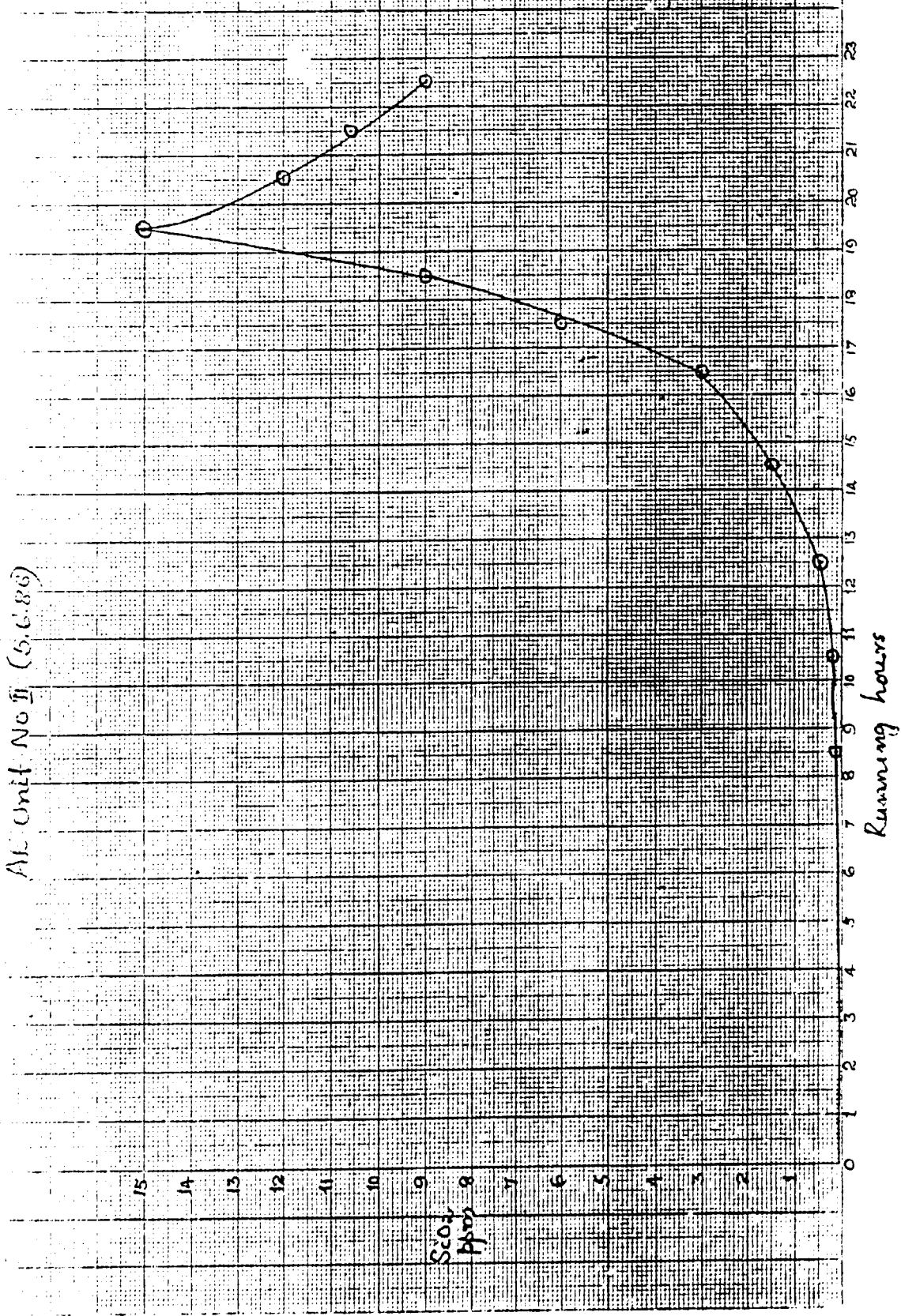
<u>Analyte</u>	<u>Level</u>
pH	6.5 - 7.2
CrO ₄ ppm	20 - 30
Sol. Zinc ppm	1 - 2
Total Zinc ppm	1 - 5
M - PO ₄ ppm	10 - 20
O - PO ₄ ppm	5
Ca ppm CaCO ₃	50 - 500
Concentration factor	3 - 6

rr/

Fig 2. Appendix 3.1

Stress-strain curve

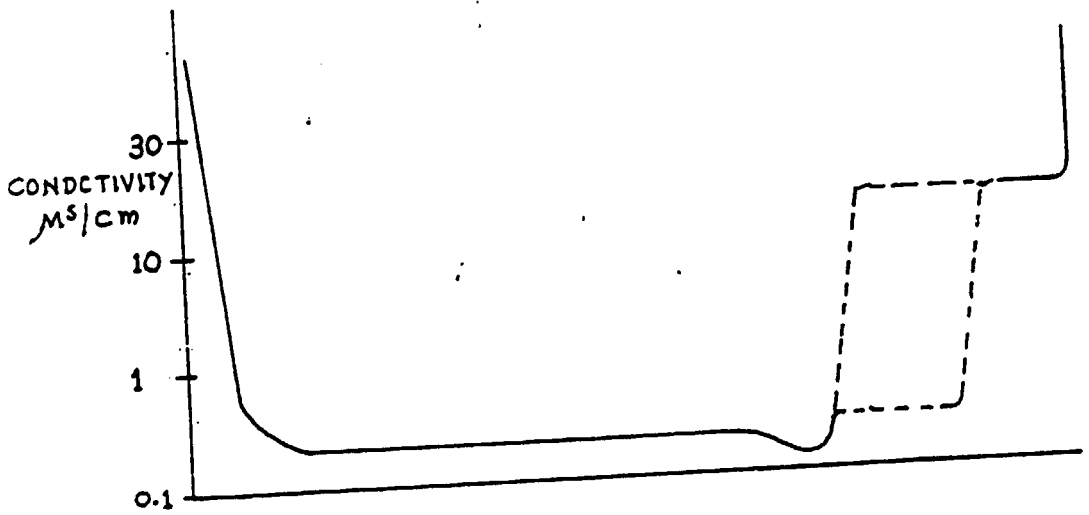
AL Unit No II (S.C.88)



Stress
Proy 8

EXPECTED PATTERN OF CONDUCTIVITY
RESUS-TIME

FIGURE- 2



SKETCH OF SULPHURIC ACID PLANT BOILER

ANNEX B-1

FIG - 1

