

IFA Technical Conference

Chennai, India 24-27 September 2002

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IFA TECHNICAL CONFERENCE 2002

"Meeting Societal Challenge towards a Safer, Cleaner and More Efficient Production"

Discussions

Chennai, India, 24-27 September 2002

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EDITOR'S NOTE

This volume contains the summaries of the discussions of the papers presented at the 2002 IFA Technical Conference, held from 24 –27 September 2002 at Chennai, India.

It also contains the texts of the papers which were not available when the proceedings of the Conference were published earlier.

IFA accepts no responsibility for the data presented or views expressed in these papers and the discussions.



International Fertilizer Industry Association

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Opening Remarks: Saif Ahmed Al-Ghafli, Chairman of the Technical Committee



From left to right: K. G. Soh (Executive Secretary IFA Technical Committee); A. Vellayan (EID-Parry Ltd.); S. A. Al-Ghafli (FERTIL and Chairman, IFA Technical Committee); M. S. Swaminathan (M. S. Swaminathan Research Foundation); L. M. Maene (Director General, IFA) during the Opening Session.

Ladies and Gentlemen, good morning,

I would like on behalf of the Technical Committee to welcome you to the conference. This year marks the 75th anniversary of the founding of the Association. It is also of interest to note that this series of our conferences is now in its 55th year, the longest of its kind within the fertilizer industry. This is the first time the conference is held in a major developing country. We are indeed thankful to the members of the Fertiliser Association of India for their kind invitation to be here in this vibrant city of Chennai, the gateway to the south of this sub-continent.

Ladies and Gentlemen,

An industry exists because it fulfils society's needs. I am happy that the fertilizer industry has evolved in parallel with the need for adequate nutrition. At the infant stage of this industry, which was in the mid-19th century, the amount of fertilizers used was less than the output of a modest factory of today. A century later, in the 1950's, the level of fertilizer consumption was still modest, perhaps under 30 million tonnes worldwide. At that time, both India and China had yet to discover the benefits of crop nutrition. The Green Revolution changed all that. Later, you will have the opportunity to hear the pioneering work of Dr Swaminathan who turned the Green Revolution into a reality. Today, global fertilizer consumption is approaching 400 million tonnes.(Note: You can imagine how much 400 million tonnes means? If the fertilizers were placed on the trucks and they are lined back to back, these trucks will form a line that will circulate the Earth at least twice!!)

The massive amount of products used has, at the same time, imposed a heavy burden upon the industry. Take the case of energy consumption. Each of the 400 units or so (403 at the last count) ammonia production units around the world consumes enough energy to power a medium size city. Today, it takes an average of about 30 million BTU's to produce a tonne of ammonia. Thirty years ago, that was 80 million BTU's. That has been a very remarkable improvement in efficiency.

A problem associated with the high volume production is pollution in the form of air emission, and liquid and solid discharges. Here the industry has achieved substantial progress. In fact Greenpeace considers "The production of ammonia, a basic intermediate of nitrogenous fertilizer, as relatively clean compared with to other industrial chemical processes" (Greenpeace Technical Note 17/00,

December 2000). I am proud to note that last year over 50 member companies last year participated in the first ever emission benchmarking survey for our industry. The broad scope of the exercise, covering some 40 parameters, has enabled our members and ourselves to take an inventory of the emissions and to plan for further improvements.

Ladies and Gentlemen,

Our industry provides jobs in many ways: in production, marketing, logistics, supply of equipments and accessories, consultancy and so on. Some of the materials we handle are hazardous, in particular, during production, transportation and storage. Safety should not be compromised. It is an obligation to see that when a worker turns up for duty, he/she should return home safely by the end of the day. This year when the Committee initiated a safety benchmarking survey, some 70 members participated, a clear indication of the members' perception of the importance attached to safety.

With this preamble, it is now clear why we have chosen the theme "Meeting Societal Challenge towards a Safer, Cleaner and More Efficient Production".

The two-day conference sessions will feature some 36 papers from 15 countries and you will find that most of these papers will echo the spirit of the theme. I am very thankful to the authors of the papers who have spent many hours preparing their manuscripts and visuals in order that they can share their knowledge and experiences with the others. I would also like to take this opportunity to thank the Chairmen of sessions and their rapporteurs for their willingness to guide and add their expertise to the sessions.

I should not forget to make a note of the presence of CEO's in our midst and I wish to thank the FAI members again for their invitation and their support to ensure a successful conference. The FAI Secretariat, in particular, Dr Bhanu Swaminathan, has done an excellent job in co-ordination and should be congratulated.

On the technical visits, due to insufficient turnout, the visit to SPIC's facilities in Tuticorin has to be unfortunately cancelled. We wish to thank the Management of SPIC for their offer and preparation. As a result, the burden of the technical visits will now fall upon the shoulders of EID-Parry and Madras Fertilizers. We are grateful to their generosity.

During the conference, ladies and gentlemen, a special cultural evening will be specially arranged by the members of the FAI South region. I would like on behalf of all the participants, thank the members there, and to Shri Vellayan personally, for your warm hospitality.

Finally, I wish to remind you that the technical conference is indeed a sabbatical, held once every two years for our engineers. We know that this is an opportunity for you to immerse yourselves in innovation for the betterment of all of us.

The schedule for this conference and in keeping up with our tradition, is very tight. I know many of you have come from distant places representing over 30 countries. Ladies and Gentlemen, I do hope you will have a small window of time to get to know Chennai, its rich diversity and spicy cuisines in this land steep with tradition from its long history. I wish all of you a fruitful deliberation and good times to be spent.

Thank you.

It is now my pleasure to call upon Shri Vellayan, Vice President, FAI South region and Vice Chairman, EID-Parry, to address us.

Welcome speech: A. Vellayan, Vice President FAI Southern Region

Dr. Swaminathan, Mr. Saif Ahmed Al Ghafli, Mr. Maene, Mr. Soh, Ladies and Gentlemen,



From left to right: A.Vellayan (EID-Parry Ltd.); M. S. Swaminathan (M. S. Swaminathan Research Foundation); L. M. Maene (Director General of IFA), B. Verghese (SPIC - Southern Petrochemical Industries Corporation); K. D. Shah (Terra Nitrogen Ltd)

It is my great pleasure on behalf of FAI Southern region to welcome you all to the IFA Technical Conference being held in India for the first time. We are even more pleased because this Conference is being held in Chennai the capital of Tamil Nadu State located in South India. This part of India has had a long agricultural tradition and is home to a number of crops like paddy, sugarcane, pulses, cotton, tea, coffee, rubber, tobacco, etc.It is also, we would like to believe, a more advanced part of the country in terms of agricultural production, industrial development and social aspects like literacy, health facilities and roads and going forward is being heralded as a part of the golden triangle for the futuristic services like IT services and Biotech.

India is a vast country with a population exceeding 1 billion people. About half of its people are depended upon agriculture in some way or another. The country's agricultural production is heavily dependant upon the monsoon. When drought and famine were very common India has to import food grains to feed its millions during the 1950s.

However the far sighted approach of visionary Indian leaders like C. Subramanian and the concerted efforts of scientists like Dr. M.S. Swaminathan, lead to phenomenal growth in food grains production leaving the country not just self-sufficient, but net exporters of food-grains and other agro-based products like sugar. Today our concerns are more about food grains rotting in our warehouses rather than shortages!

We are privileged to have our midst today, Dr. M.S. Swaminathan himself to deliver the keynote address at this conference. Introducing Dr. M.S. Swaminathan is a daunting task: it would probably take an entire session if I were to go into the details of his academic and research background, his achievements and the international institutions he is involved with. Despite his very busy schedule, he has kindly consented to spare time for sharing his thoughts with us today.Suffice it to say that, as one of the key architects of the Indian Green Revolution and a champion of sustainable development, we could have chosen no better person to deliver the keynote address at this conference. My humble thanks to him for acceding to our request to deliver his address on "Nutrient Management for an Evergreen Revolution."

The theme of this Conference is "Meeting Societal Challenge towards a Safer, Cleaner and More Efficient Production". I find that as many as 37 papers are being presented at this Conference by experts from all over the world. The subjects covered include Safety and Environment, Technology

and Improvements in Efficiencies. The Conference theme and coverage appear appropriate considering the reversionary clouds hovering over us as we also the increasing awareness on environmental issues. I am sure that all of you will derive maximum benefit from the sessions being planned over the next few days.

The fertilizer industry in India had, particularly, in the South, been going through a very difficult phase. Changes in government policy and an erratic monsoon have added to the woes of the fertilizer industry. It is at this time that the industry needs all the help in terms of Technical inputs in order to reduce cost and improve productivity. Therefore, the holding of the Technical Conference at Chennai is very appropriate time and I hope all of you will derive immense benefit out of these sessions.

Most of you have travelled long distances to reach Chennai; for many, this may be their first visit to Chennai and India. I am sure you will not miss this opportunity to take a peek at the city's culture and savour its cuisine.

Chennai is a very old city that combines the modern with the traditional; its ancient temples co-exist with modern buildings housing software companies. You will find the Chennaite visiting temple early in the morning wearing the traditional Indian dhoti; the same person can be seen later in the day in Western attire conversing on his mobile phone or working on his laptop. Five star hotels with discos rub shoulders with humble wayside eateries! The city boasts of restaurants specializing in various world cuisines: Continental, Mexican, Japanese, Korean, Chinese, Thai and Mughlai food. Needless to state, India itself has a variety of cuisines and you can find them all here in Chennai.

The British who entered the country a few centuries back through Chennai have left their indelible stamp on our life and culture. English is widely spoken with many who are more proficient in English rather than their mother tongue. Historic churches like the Saint Thomas are located in Chennai. I do hope you will be able to find time to breakaway for an outing into the city to absorb its culture and sights.

Let me wish you a very pleasant and comfortable stay in Chennai.

Once again a warm welcome to the Technical Conference.

Keynote address: Prof. M.S. Swaminathan, M S Swaminathan Research Foundation

Presentation: Nutrient Management for an Evergreen Revolution

Prof. M.S. Swaminathan, M S Swaminathan Research Foundation







Top Six Countries in Terms of Irrigated Area, 1999

Country	Area ('000 ha)	% of world share
India	59000	22
China	53740	20
USA	22400	8
Pakistan	17950	7
Iran	7562	3
Mexico	6500	2

Per hectare nutrient consumption: historical and projected

Regions/Groupings	Per hect	are nutrier (kg/ha)	Growth rate (% p.a.)		
	1961/63	1995/97	2030	1961- 1997	1995/97-2030
South Asia	3	79	104	9.4	0.8
excl. India	6	99	127	8.6	0.7
East Asla	9	147	180	8.9	0.6
excl. China	15	93	106	5.8	0.4
Developing countries	7	90	107	7.9	0.5
Industrial countries	125	206	253	1.1	0.6
World	35	107	124	3.3	0.5

Fertilizer consumption, total nutrient: historical and projected

	Nutrien	t (million to	Growth rate (% p.a.)		
Regions/Groupings	1961/63	1995/97	2030	1961-1997	1995/97-2030
South Asia	0.6	18.1	27.2	10.0	1.2
excl. India	0.2	4.0	6.1	9.7	1.3
East Asia	1.7	44.4	58.8	9.7	0.8
excl. China	0.9	8.9	12.4	7.2	1.0
Developing countries	4.1	78.7	112.4	8.8	1.1
Industrial countries	24.6	46.3	58.3	1.5	0.7
World	34.3	133.9	181.6	3.9	0.9

Source: FAO (2000)

Crops	1995/97	2030	Growth 1995/97-2030 (% per annum)
Wheat	24.6	29.5	0.5
Maize	22.3	30.4	0.9
Rice	22.2	27.2	0.6
All cereals	78.2	98.8	0.7
All crops	134.0	182.0	0.9

Ehrlich 1968

- Some time between 1970 and 1985 the world will undergo vast famines - hundreds of millions of people are going to starve to death. That is, they will starve to death unless plague, thermonuclear war, or some other agent kills them first.
- The United States should announce that it will no longer ship food to countries such as India where dispassionate analysis indicates that the unbalance between food and population is hopeless.





Backbone of the Revolution

National Demonstrations in small farmers' fields led to a small govt. programme becoming a mass movement between 1964 - 68

Green Revolution Wheat Production - India now occupies the Second Position in the World

Projected total cereal requirements for India for 2020				
Country / year / Source	Food	Feed	Total	Kilograms per capita per day
India 1993 (actual)	147.12	3.71	150.83	0.47
2020 (authors' Projections) With per capita Income growth of :				

3.7 percent 0.61 246.08 50.11 296.19 Source : Prospects for India's Cereal Supply and Demand to 2020 – G.S. Bhalla, Peter Hazell, John Kerr : International Food Policy Research Institute

Crops	Produc	tion	Productivity		
	Share(%)	Rank	Ton\ha (rank)	Highest	
Rice	21	2	2.8 (51)	7.4 t/ha (Ukraine)	
Wheat	12	2	2.5 (32)	9.0 t/ha (Ireland)	
Sugarcane	23	2	65 (34)	121.4 t/ha (Peru)	
Pulses	26	1	0.6 (118)	4.8 t/ha (France)	
Cotton	14	3	0.9 (57)	4.5 (Israel)	







Scientific rationale for an Ever-green revolution

"Intensive cultivation of land without conservation of soil fertility and soil structure would lead ultimately to the springing up of deserts. Irrigation without arrangements for drainage would result in soils getting alkaline or saline. Indiscriminate use of pesticides, fungicides and herbicides could cause adverse changes in biological balance as well as lead to an increase in the incidence of cancer and other diseases, through the toxic residues present in the grains or other edible parts. Unscientific tapping of underground water would lead to the rapid exhaustion of this wonderful capital resource left to us through ages of natural farming. "

Contd ...

Scientific rationale for an Ever-green revolution

"The rapid replacement of numerous locally adapted varieties with one or two high yielding strains in large contiguous areas would result in the spread of serious diseases capable of wiping out entire crops, as happened prior to the Irish potato famine of 1845 and the Bengal rice famine of 1942. Therefore, the initiations of exploitative agriculture without a proper understanding of the various consequences of every one of the changes introduced into traditional agriculture and without first building up a proper scientific and training base to sustain it, may only lead us into an era of agricultural prosperity."

- M S Swaminathan Indian Science Congress, Varanasi, January 1968



Foundation of Ever-green Revolution Integrated Natural Resources Management

Components

- Conservation of arable land
- Soil health care
- Water conservation and management
- Integrated Gene Management
- Integrated Pest Management
- Integrated Nutrient supply
- Improved post-harvest technology
- Local level Integrated Natural Resources Management Committee

Ever-green revolution

"The problem before us is how to feed billions of new mouths over the next several decades and save the rest of life at the same time, without being trapped in a Faustian bargain that threatens freedom from security. The benefits must come from an evergreen revolution. The aim of this new thrust is to lift food production well above the level attained by the green revolution of the 1960s, using technology and regulatory policy more advanced and even safer than now in existence"

> Edward O. Wilson, 2002 The Future of life

Rice Revolution in Assam Contributory Factors

- 1999 2000 : 100, 000 shallow tube wells
- Control by Farmers' Field Management Committees
- Irrigated rice during the non-flood prone season
- Impact : Cropping intensity 180 %
 - > Yield : 4 tonnes per ha
 - > Net income : Rs.35,000 per ha
 - Surplus of 500,000 tonnes of rice

Ever-green Revolution in Assam

Strategy

- Minor irrigation through shallow tube wells and low lift pumps (taking advantage of an annual rainfall of 2800 mm)
- Soil fertility build-up through stem nodulating green manure crops before the onset of south west monsoon
- Cultivation of organic rice and jute as well as the rearing of fish (aquaculture) during south west monsoon period

Integrating Genetic Efficiency with Genetic Diversity





Biological software for sustainable agriculture

Fixing nitrogen both in stem and roots





Biological software for sustainable agriculture

Biological control

Ladybird (*Coccinella septumpunctata*) feeding on aphids



Trichogramma Egg Parasitoid to control Rice and Sugracane borers





Consequences of Improper Soil Health Management

(Lessons from Long term soil fertility experiments)

- Acidification
- Alkalinity
- Salinity
- Declining Soil organic matter
- Formation of hard pan
- · Declining Soil microbial population

Managing Multiple Nutrient Deficiencies

- Crop rotations
- FYM and Compost
- Vermiculture
- Withdrawal of nutrients from different depths of the soil surface
- Green Manure Crops
- Application of Micronutrients

Integrated Plant Nutrition System

Mobilise all available sources of nutrients and use them efficiently and synergistically based on targeted yields and agro-climatic conditions.

- Internal Farm Nutrient Sources
- External Nutrient Sources (from farm surroundings)
- Market purchased sources; including mineral fertilizers



Role of Virtual University for Efficient Nutrient Management

- Right choice of Fertilizer material
- Right quantity in relation to the inherent nutrient supply capacity of the soil
- Right method of application to reduce losses
- Right time of application in relation to crop growth period
- Right positive interaction with other farm operations like tillage, water and weed management

Clients of the Virtual University

- · Fertilizer and input supply agencies
- Agriculture Departments
- Farmer's Associations
- Village level Soil health managers and water masters (two men and two women in each village)



<u>Chairman</u>: Saif Ahmed Al-Ghafli, Ruwais Fertilizer Industries (Fertil), U.A.E. <u>Rapporteurs:</u> Antti Vuori, Kemira Oyj, Finland Hans van Baal, Stamicarbon, The Netherlands

Paper N°1: Meeting societal challenge towards a safer, cleaner and more efficient production

T.K. Jenssen, Hydro Agri, Norway

H. Van Baal, Stamicarbon, The Netherlands

- **Q.** One of the features in the Hydro Agri granulation is a more environmental friendly finishing technology. HFT is market leader. Why then has HFT decided to stop licensing this technology, which is Best Available Techniques?
- **A.** It is a business decision by Hydro to limit the licensing of Nitrogen technologies. As a result our granulation technology will for the time being not be licensed. Naturally, we will continue to service those licensing agreements already established and commitments being made.
- **Q.** In urea technology, fluid bed granulation is environmentally friendly. Why was this withdrawn from the market?
- **A.** It is correct that fluid bed granulation is environment friendly, especially compared to prilling. The withdrawal from the market of open licensing by Hydro is a business decision connected to the company's strategic focus on the nitrogen segment of the fertilizer industry.

G. Martin, Kemira Emirates Fertilizer, U.A.E.

- **Q.** You presented a strong emphasis on safety (SHE) with all of Hydro's plants. How much does ISO-9000 or ISO-14000 impact on these plant sites and what is your opinion of acquiring ISO-14000 certification? How has this contributed on your safety philosophy?
- A. We decided many years ago that ISO-9000 was something we should go for, and today our European plants are certified to ISO-9000. Not because our customers demanded it, but because we needed to clean up our procedures and improve our work methods. On ISO-14000 we have not gone so far as to certification yet. We have an internal auditing tool to check compliance with the ISO-14000 requirements, and we find this to satisfy our needs for the moment. Let us bear in mind that certification does not guarantee zero incidents. I strongly believe that management commitment, line managers' visibility in the plant and their active follow-up, and employees involvement in safety related activities, are the best "guarantees" for obtaining zero accidents.

R. Murali, Deepak Fertilisers and Petrochemicals Corporation Ltd., India

Q. What is the standard N₂O emission level from a) high pressure plant (11 bar), and b) medium pressure plant (3.4 bar) in manufacturing HNO₃?

The emission levels (NO_x) presented in the paper – do they include N_2O levels or not?

A. Standard emission levels of N_2O from nitric acid plants are nowadays between 1000 and 1500 ppm. German legislators have recently announced a permit level for N_2O at 800 ppm, to be

achieved several years from now. Some abatement technologies are under development, but this is still R&D and not commercialized.

On your question on N_2O emissions from nitric acid plants, EFMA issued the following guideline recently:

Oxidation pressure (bar)	Kg N ₂ 0/t HNO ₃	Comments
1	5	
3-7	7	
>8	9	directly quenched
>8	5-9	depending on the distance of the gauzes to the quench
>8	1	with SCR

The NOx emissions presented in the paper do not include N_2O emissions.

K.M. Tandon, DCM Shriram Consolidated Ltd., India

- **Q.** How do you keep your employees motivated to continuously do well in field of safety performance? Would you share your BAT booklets with others in the industry for their benefit?
- **A.** I don't have a clever answer on what motivates people. Fortunately we are all different and we get turned on for various reasons. However, we all have one thing in common we do wish to avoid accidents and injuries. For those companies continuously doing well, I think a key factor is paying respect to people's suggestions and ideas, and give clear and concise feedback. Also, participation and authority to decide on issues are important for all of us. And we need somebody to guide us enthusiastically and strongly. The BAT booklets are open for everyone. You will find them on EFMA's web page, or by contacting EFMA at the following address:

European Fertilizer Manufacturers Association Avenue E. Van Nieuwenhuyse 4, B-1160 Brussels Belgium Fax +32 2 675 39 61 e-mail: <u>hvb@efma.be</u>

S. Al-Ghafli, Ruwais Fertilizer Industries (Fertil), U.A.E.

- **Q.** I come to understand from your presentation that IFA and its members got higher responsibility of performing according to some acceptable code of conduct, well defined and agreed on. How much of control do you think could IFA have on the members, so that IFA is seen by outsiders that it is still responsible, accountable and up to its words? What could be this form of control?
- A. IFA ought to define a Code of Conduct, which should be shared and agreed with the regional associations around the world. Other industry associations use such Code of Conduct in promoting their Responsible Care and Product Stewardship. We need to do the same. Sometimes it will be necessary to react to sub-standard practices in member companies. By having an agreed Code of Conduct we will have a reference level to react to. Let us make IFA and the regional industry associations to something more than just nice meeting places for sharing experiences. Let their Boards of Directors initiate corrective actions towards member companies, if needed.

- **Q.** Possible actions of IFA platform of operators etc. could you comment on the IFA role?
- A. IFA is first and foremost a meeting place for exchanging experiences, acting as a catalyst for developing best practices in the fertilizer industry. It could take a more active role in defining best practice technical and operating standards, especially in the field of health, safety and environment. IFA should lift itself to be the spokes-body for the fertilizer industry in setting global standards on environmental protection, which is necessary for fair trading between countries, and for getting the industry's reputation right.

Paper N°2: Report on a workshop on ammonium nitrate organized by the European Commission at Ispra, Italy

Hans van Balken, EFMA, Belgium (Presented by K.D. Shah)

A. Van Brempt, Kemira S.A., Belgium

- **Q.** Based on the UN classification some AN containing grades classified before the revision are becoming now (new classification) 5.1 (oxidizing agents), e.g. straight N fertilizers with an AN content between 70 and 80% and not containing carbonates as a filler. Are such grades now also subject to ADR transportation?
- **A.** With the revised UN legislation the difference will be that there is only one single UN number for all ammonium nitrate based fertilizers, which are oxidizing agents. All those falling in Class 5.1 will be subject to ADR.

G. Martin, Kemira Emirates Fertilizer, U.A.E.

- **Q.** Report on ammonium nitrate: You mentioned "off-spec" ammonium nitrate was involved in Toulouse. What quality spec was material involved in explosion not satisfied? Porosity? Particle size? Nutrient content?
- **A.** The French authorities have published an official report on the Toulouse disaster, which includes some information about the various off-spec or reject materials. We do not know the exact off-spec parameters.

R. Murali, Deepak Fertilizers and Petrochemicals Corporation Ltd., India

Q. Ammonium nitrate is normally coated with oil/amine. How do you recycle <u>SAFELY</u> the returned "off-spec"?

How do you classify 23-23-0 AN based fertilizer containing about 50% AN?

A. Recycling of AN coated with organic coating back into the process involving hot AN solutions is not generally considered safe and, therefore, is not recommended. This is a compound fertilizer of the N:P type containing less than the 70% threshold applicable to compounds in the oxidising class. Check the parameters concerning combustible matter content and self-sustaining decomposition capability and compare with the Orange Book Definitions.

S. Al-Ghafli, Ruwais Fertilizer Industries (Fertil), U.A.E.

- **Q.** In your opinion what should AN operators review and correct in order to mitigate the risk of possible explosion?
- A. Hazard study of the manufacturing process to ensure controls are in place concerning contaminants, uncontrolled decomposition, heating of AN under confinement and other untoward conditions. In the storage and handling of product, ensure adherence to good code of practice, including safe systems for off-spec materials.

Paper N°3: Cadmium content of phosphate rocks and fertilizers

S.J. van Kauwenbergh, IFDC, USA

N. Dylevskaya, Phosagro, Russia

- **Q.** What could be the principles behind introduction of taxes on cadmium in phosphate fertilizers as one of EU option to reduce Cd content of fertilizers?
- A. The principle behind taxing the cadmium contents of phosphate fertilizers is that the added cost of taxes will increase the total costs of the cadmium containing fertilizers to the point wherein the use of cadmium containing fertilizer is economically unfavourable, the use of lower cadmium content fertilizers is more favourable, and thereby the use of higher cadmium fertilizers is discouraged and the use of lower cadmium content fertilizers is encouraged and stimulated. Taxation can be viewed as a penalty in this case; the severity of the penalty corresponding to the level of taxation. In this case, taxation can be also viewed as a means to indirectly control (as opposed to direct, perhaps more confrontational, control) the cadmium contents of fertilizers.

S. Kraishan, Indo-Jordan Chemicals Co. Ltd., Jordan

- **Q.** Is there a process to remove Cd from rock phosphate during the processing, e.g. mining, crushing, screening, or is the Cd equally distributed in the rock phosphate meaning that we cannot remove it in form of a physical or minerological process to avoid going to calcination process which is expensive?
- A. IFDC has performed several studies evaluating potential cadmium removal methods that can be implemented during mining and beneficiation. The available evidence indicates that most of the cadmium generally present in phosphate rock is cadmium substituting for calcium in the apatite structure. No simple or currently used commonly used beneficiation process appears to be suitable for cadmium removal from phosphate rock. The only viable removal options appear to be some form of calcination or total solubilization of the phosphate rock wherein the crystalline structure of the apatite is altered or destroyed and the cadmium is liberated.

N. Seetaram, Coromandel Fertilisers Ltd., India

- **Q.** Your paper described Cd content of various rocks. I would like to know about Chinese rocks on this issue. Has IFDC made any studies on these rocks?
- **A.** IFDC has not worked directly with many Chinese phosphate rock samples and does not have a significant amount of directly obtained data with respect to the cadmium contents of Chinese phosphate rocks. What data is available indicates the cadmium contents of most Chinese phosphate rocks are very low (well below 10 ppm).

A. Van Brempt, Kemira S.A., Belgium

- **Q.** A question for Mr. van Balken: The intention of the EU is to come up with new Cd limits. Have you any idea about time schedule, and the future limit of max allowed Cd content/kg P_2O_5 for EU fertilizers?
- A. H. van Balken: In a recent meeting with DG Enterprise it became clear that they will come with a proposal soon, but this has been said more often before. However, we anticipate that a proposal will be made in the beginning of next year. This proposal will be put on internet for public consultation. It is most likely that the proposal will be in line with the ERM report on the Risk Assessment studies for Cadmium in Fertilizers done by the Member States (60-40-20) with time intervals of may be 5 years. EFMA continues lobbying. EFMA is will also investigate what the consequences are (economic assessment) for costs and competiviness (e.g. with 20 mg/kg P2O5).



<u>Chairman</u>: Babu K. Verghese, Southern Petrochemical Industries Corp. Ltd., India <u>Rapporteurs</u>: R. Muthumanoharan, Jt.General Manager (Imports and Exports), SPIC Chennai, India S. Muruganandam, Chief Manager (Operation), SPIC Tuticorin, India

Paper N°5: Stretching the capacity of a 23 year-old Kellogg ammonia plant

V.S. Vaish and M.R. Patel, IFFCO, India (Presented by M.R. Patel)

M.A. Rahim, KAFCO, Bangladesh

- **Q.** Syn Gas compressor section chilling effect mentioned in the paper needs clarification?
- A. 38 to 8°C and not as mentioned in the paper.

A. Al Jabar, Saudi Arabia Fertilizer Company, Saudi Arabia

- **Q.** The cost of MT added to increase the capacity compared to the cost which will be increased if you bring from outside?
- A. More or less the cost of production of the same

G.K. Rao, Nagarjuna Fertilizer and Chemicals (NFCL) Ltd., India

- **Q.** Volume of Catalyst of pre ref.?
- A. $6m^3$ is the volume of the catalyst

R. Muthumanoharan, SPIC - Southern Petrochemical Industries Corporation, India

- **Q.** When the load of the reboiler is reduced, what is the need of adding 3 new cells in cooling tower?
- **A.** The cooling tower is common for Ammonia / Urea Plant. Although the heat daily is reduced in the reboilers, the increase in capacity of other sections warranted for increase in cooling tower capacity.

Babu Verghese, SPIC - Southern Petrochemical Industries Corporation, India

- **Q.** *How long do you think the plant life is extended?*
- A. 10 more years.

K.M. Tandon, DCM Shriram Consolidated Ltd., India

- **Q.** Did you consider installing a combustion air preheater in your reformer? How long did it take to carry out all the modifications mentioned in your paper?
- A. No answer

Paper N°6: Milling phenomenon experienced in the primary reformer of an ammonia plant at NFCL, Kakinada

Ch. Varahalu, G. K. Rao and V. Sunder, Nagarjuna Fertilizer and Chemicals Ltd., India (Presented by G. K. Rao)

K.M. Tandon, Shriram Fertilizers and Chemicals, India

- **Q.** After increasing the free space above the catalyst to 0.4 m, have you faced over heating of the exposed portion of the tube?
- A. No over heating of the portion was observed.
- **Q.** *Have you considered the catalyst grid pressure withstanding capacity, when the reformer tube pressure drop was high?*
- A. Yes, it was considered.

R.N. Bhargava, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., India

- **Q.** When the pressure drop was increasing, was there any increase in the methane content at the primary reformer exit?
- **A.** Methane content was not measured. But the process gas temperature exit PR, S/C were more than conducive and there was no doubt about methane content.

S. Muruganandam, SPIC - Southern Petrochemical Industries Corporation, India

- **Q.** *Have you observed increase in methane slip on account of reducing catalyst volume after the incident?*
- A. No increase in the methane slip was observed

S. S. Singh, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., India

- **Q.** How many days the plant was shutdown to set right the problem?
- A. The plant was back on stream after 4 days.
- **Q.** *What was the methane content at the exit of the primary reformer?*
- **A.** The methane content was between 16 and 17% at the outlet temperature of 730°C to match the capacity of PGR unit.

A. Al Jabar, Saudi Arabia Fertilizer Company; Saudi Arabia

- **Q.** *What are the methods adopted for the inspection of the primary reformer tubes?*
- A. Visual inspection, creep measurement and ultrasonic test were done.
- **Q.** *What is the expected life of your reformer tubes?*
- A. The tubes are in service since 1992 and another 5 years of life is expected.

R. Muthu Manoharan, SPIC - Southern Petrochemical Industries Corporation, India

Q. Could you establish that the "milling" was only due to faster start-up? Alternatively was there a disturbance during either admitting steam/gas? Was the quality of the pre-reduced catalyst charged good?

A. There was no disturbance while admitting steam or gas. As told earlier, the catalyst was checked both physically and chemically and found to be alright.

Paper N° 7: Shaft oil barrier seal can cause high vibration – Do you know?

Arun Kumar, National Fertilisers Ltd., India

H. Van Baal, Stamicarbon, The Netherlands

- **Q.** *Was there not a procedure for assembling its leaf spring by the application?*
- **A.** No, the procedure was not given by the supplier for similar problem was experienced by other units.

Paper N° 8: Failure and repair of the secondary reformer – an experience

V.K. Bali, IFFCO, India (Presented by S. Mathur)

G.K. Rao, Nagarjuna Fertilizers and Chemicals Ltd., India

- **Q.** What is the material of construction of secondary reformer and is it in accordance with Nelson *Curve*?
- A. The material of construction is C-1/2 MO and it is in accordance with Nelson Curve.

M.P.S. Nair, Fertilizers and Chemicals Travancore Ltd., India

- **Q.** How did you ensure that the dehydrogenation process complete?
- **A.** While welding there were no sparks and that was the indication of completion of dehydrogenation.
- **Q.** Have you considered replacement of the conical portion of the secondary reformer as an alternative?
- **A.** The alternative was checked but the alternative was likely to take 9 months leased on other's experience and hence not taken up for implementation.

K.M. Tandon, Shriram Fertilizers, India

- **Q.** Do you have UT probe to work out the shell temperature of secondary reformer, which is at 140°C?
- A. There is an external agency developed the probe for carrying out UT at the shell temperature.

V. Jayaraman, SPIC - Southern Petrochemical Industries Corperation Ltd., India

- **Q.** Can you please elaborate the procedure for dehydrogenation?
- **A.** Normally, the dehydrogenation is done by heating the metal to 350 C. In the case of failure of the secondary reformer, the dehydrogenation was done by heating up the shell to 650 C and soaked at that temperature for 1 ½ hrs. Then, the shell was cooled down gradually.

R. Muthu Manoharan, SPIC - Southern Petrochemical Industries Corperation, India

- **Q.** Were you monitoring the skin temperature of the conical portion of the secondary reformer? Soundness of insulating refractory could be confirmed by the skin temperature.
- **A.** It was not being monitored previously. However, after the failure, the temperature is being monitored regularly.

M.A. Rahim, KAFCO, Bangladesh

- **Q.** Did you experience any change in the 4-P across the catalyst bed?
- **A.** We did not experience any pressure drop across the catalyst bed of the Secondary Reformer prior to failure or at the time of observance of the leakage through the weld seam of the cone. The condition of the nickel catalyst in the 39M3 bed of 2800mm height was also found quite good on opening the vessel & inspecting the catalyst. The same catalyst was charged again subsequent to repair of the equipment.
- **Q.** How can you make UT Test monitor the cracks development trend in the in-situ condition with surface temperature of more than 145 °C
- A. Yes, periodic inspection of the Sec. Reformer Shell weld joints and selected portions of the base metal from outside is feasible by Ultrasonic Scanning in hot condition using high temperature imported grease as couplant between the 170 to 180 deg. C hot surface of the vessel and the specially designed 45 & 60 deg. shear wave Ultrasonic probes. Competent inspection agencies to carry out such High Temperature Ultrasonic Scanning job are available in India. This type of testing can give fairly good idea about the health of the vessel and crack development and propagation trend can be satisfactorily monitored and recorded in-situ for future reference.

We have carried Hot Ultrasonic Scanning of the repaired welds and base metal of our Sec. Reformer at an interval of three months in hot running condition and no significant defect has been reported till date. For calibration purpose a reference block of SA 204 Gr B material of same thickness as that of the shell, with outer surface painted with thermosensitive paint is heated to 180deg.C in a furnace to simulate the job. The DAC curve is plotted and reference gain is set accordingly.

- **Q.** Did you see the burning of the anchors or severe damage of the anchors due to excessive heat in *the combustion zone?*
- **A.** Some of the "Y" type anchors where the straight portion remains in the 200 mm thick insulating refractory layer and the "V" portion remains in the 100mm thick hot face refractory layer were found burnt at the interface zone of the two refractory layers.

This was mostly observed in the areas where the hot face refractory layer was found loose or having wide cracks or openings to allow ingrace of high temperature to the anchors in the conical region i.e. combution zone. The "V' portion of some of the anchors in these areas had also damaged and developed brittleness. In order to overcome the problem the thickness of the SS 310 matl. anchors has been increased to 10 mm in straight portion and to 8mm in "V" portion from original thickness of 6.25mm and subsequently the refractory relining work has been carried with utmost care and quality control.

Paper N°9: Consider KRES for fuel cost savings and increasing capacity in the ammonia plant

Avinash Malhotra and Stan Knez, Kellogg, Brown and Root Inc. (Halliburton KBR), USA

(Presented by Avinash Malhotra)

M.A. Rahim, KAFCO, Bangladesh

- **Q.** Have you experienced metal dusting in KRES?
- A. No metal dusting has been experienced

Y. Narula, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., India

- **Q.** What is the pressure drop of the KRES reformer tubes? What is the pressure drop across the reforming section?
- A. The pressure drop across the tube is 2.5 KSC and the section pressure drop is 3 KSC.

Sukumaran Nair, FACT, India

- **Q**. How does the cost of KRES compares with the conventional reformer?
- **A.** This would vary from case to case. The equipment of KRES can be manufactured by the vendors in the country in which the unit has to be installed. This will reduce the cost of the KRES equipment.

A.K. Maheswari, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., India

- **Q**. What is the payback period if conventional reformer is retrofitted with the KRES?
- A. The payback period will vary from case to case, depending upon the plant condition.

M.R. Patel, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., India

- **Q**. Is it required to use enriched air in KRES?
- **A.** Enriched air is not a must and excess air can also serve the purpose. Use of enriched air or excess air will depend upon the process scheme down stream of KRES.



<u>Chairman</u>: R.S. Nanda, Coromandel Fertilisers Ltd., India <u>Rapporteurs:</u> S. Lancuba, Operations Planning and Development Manager, Pivot Ltd. Australia A. A. Ramamurty, General Manager-Manufacturing, Coromandel Fertilisers Ltd., India

Paper N° 10: Experience with dry stack management of gypsum (hemihydrate calcium sulphate)

A. Chari, S. Subbiah and T.N.V. Satyanarayana, Indo-Jordan Chemicals Company Ltd., Jordan

(Presented by T.N.V. Satyanarayana)

Tibaut Theys, Prayon S.A., Belgium

- **Q.** *Are you adding any additive before filtration from hemi-hydrate to dihydrate?*
- **A.** We add to the filter anti-scaling agent. It also helps to stabilize the hemihydrate during transportation, and minimise further conversion.

P.A. Smith, Bunge Fertilizantes S.A., Brazil

- **Q.** What is the normal moisture content of the hemi-hydrate cake leaving the filter?
- A. The average moisture content is 19.96%, with a typical variation of 17 to 22% on wet basis.

R.K. Mutreja, Hindustan Fertilizer Corporation Ltd., India

- **Q.** You mentioned that dry stacking is economical because no lining is required. Is that an acceptable international practice because some leaking may take place?
- **A.** This is special for the IJC project. For example if you have high rainfall of 2000 to 2500 mm per year, there will be percolating problem through the cracks. Other factors include geological structure, presence of clay on the ground and percolation level on the ground. If material with 20% moisture is wet, it is already starving for water. Part of the water is used for conversion. The seepage level is much lower in case of hemihydrate instead of dihydrate. A general statement cannot be made.

N. Seeteram, Coromandel Fertilisers Ltd., India

- **Q.** What are: Downtime due to system? Steamfactor of the plant? P205 efficiency of the plant? P205 content in gypsum cake?
- **A.** As explained, only one conveyer is sufficient for the plant to operate, even if the other conveyors are down. There is high reliability in the system. Hence the special emphasis on the phosphogypsum conveyor.

Over the 5-year period, the downtime is 2%. On onstream days, this pioneering plant places emphasis on high reliability. We have installed two independent belt filters. Most machineries are located in the filter section.

Even if one belt filter is down, it can still run. Hence we achieved an onstream factor if over 90%. Phosphate recovery depends on type of rocks used, but generally in excess of 90%.

R.S. Nanda, Coromandel Fertilisers Ltd., India

- **Q.** What is the P2O5 content in gypsum?
- A. The total P2O5 content varies with the system but within the gypsum it varies from 1.5% to 1.8%.

Arthur Van Brempt, Kemira S.A., Belgium

- **Q.** In the paper its mentioned that 92% of the particle size after conversion to di-hydrate (presence of board agglomerates 1 to 10 hpm)?
- A. No answer

Paper N° 11: Overall corrosion protection in a phosphoric acid fertilizer plant

Gerd Meier, SGL Acotec GmbH, Germany

G. Aviram, Rotem Amfert Negev, Israel

- **Q.** *Regarding the capacity of the dilution system of the sulphuric acid?*
- **A.** We offer a one stage, not 2-stage dilution system and the capacity so far we could meet. It is the cooler and we can build heat exchanger up to 2000m2 for the dilution system.

N. Seeteram, Coromandel Fertilisers Ltd., India

- **Q.** What is the metallic heat exchanges used for concentration section?
- A. No answer
- **Q.** Can you supply pumps for hydrofluosistic and service?
- A. No answer

Paper N° 12: Expansion of the Richards Bay

di-hydrate phosphoric acid production complex

K. Kirsten, Foskor Ltd., South Africa, J. Tytgat, SNC-Lavalin Europe, Belgium, T. Theys, Prayon S.A., Belgium (Presented by J. Tytgat)

N. Seeteram, Coromandel Fertilisers Ltd., India

- **Q.** What is the total area of the 3-filter designed and the reactor?
- **A.** We use 3 parallel belt filters, each with an area of 90 m². The plant is hydraulically designed for 2 filters in parallel. A third filter is added for operation flexibility. One filter can be in washing cycle while the other 2 still insure 100% or more capacity. When 3 filters are running in parallel more than 120% capacity can be achieved.

The total reaction volume, including the 6-compartment attack tank and the 3 digestion tanks is over 2000 m³.

- **Q.** Are you using any chemical to improve the filtration?
- **A.** Yes, a Crystal Habit Modifier is added in the reaction tank to improve crystallization and so improving the filtration.

Shiv Shulka, Monsanto DMCC, India

Q. A very good virtual tour of the plant. Was a 3-D Software used for drawing preparations for piping structural and solid models?

Which software was used?

Did it result in savings in engineering matters?

A. The software used for the 3D model is PDS, Plant Design System from Intergraph. The Module PDS-frame works was used for structural design.

The Module PDS-drawing for used for all design drawings. Major adavantage of this software is that all disciplines (civil, piping, electrical, instrumentation, etc...) can work in parallel with this integrated system in the same plant area.

This is important what scheduling of execution of engineering documents in concerned. Recent development of this software also allows us to make a virtual walk (and not only a overview as shown during the presentation) inside the plant on all levels.

Paper N° 13: Crystal habit modification in wet process phosphoric acid production

Tibaut Theys, Prayon S.A., Belgium and Benoit Roblin, SEPPIC S.A., France

(Presented by T.Theys and B. Roblin)

R.S. Nanda, Coromandel Fertilisers Ltd., India

- **Q.** You mentioned that the Montaline be used as a defoamer. Does it help to suppress foaming due to excess CO_2 generation in the attack tank?
- **A.** (T. Theys) The industrial experience demonstrate that the product is de-foaming. It is used to avoid the production of foams in the attack section during the reaction. It is used with K-09 phosphate which is a very reactive rock with CO2 content up to 7%. The foam production is very strong at the first stage of the attack. The product is used to lower the foam in the attack section and in the flash cooler.
- **A.** (B.Roblin) When we evaluated the product as an anti-foaming agent, we discovered crystal habit modifier properties as well. In regards with the de-foaming property, the Montaline will help airbubbles included in the pulp to be released then the antifoaming property will allow to kill the foams at the surface of the pulp.

B.P. Raju, Coromandel Fertilisers Ltd., India

- **Q.** This crystal modifier, is it effective for both igneous and sedimentary rocks?
- **A.** (T. Theys) Both rocks can be used, but the type of crystal habit modifier needs to be adapted accordingly.
- **A.** (B.Roblin) We worked mostly with sedimentary rocks and not much experience with the others. Nevertheless, further studies with igneous phosphate rocks are already planned.

N. Seeteram, Coromandel Fertilisers Ltd., India

- **Q.** Has this work been done on commercial scale? If so, what is the $%P_2O_5$ in the gypsum cake before and after?
- A. (T. Theys) In the pilot test, the filtration yield was improved by 0.5%.
- **Q.** *What is approximately the cost of the chemical?*
- **A.** (B. Roblin) Whatever is the reasonable cost efficiency. We highly recommend to conduct tests first, either at the laboratory or pilot level in order to estimate the most convenient dose. Each case is different.

Arthur Van Brempt, Kemira S.A., Belgium

- **Q.** *Have you any idea about the amount of Montelite left in the final concentrated acid? What is the impact on MAI granulation properties?*
- A. (B. Roblin) Unfortunately we do not know yet the breakdown. It is not so easy to quantify. Montaline is a composition of different surfactants. These surfactants are ethylene oxide polymers, they are characterized by an alkyl chain distribution and a degree of polymerization. One of the ways to dose the Montaline is to analyze the oxide content which is quite difficult to carry out due to the low dosage of the product in the pulp.

This is a pending issue. We will find ways to analyze the residue in the gypsum.

- **Q.** What is the impact on MAI granulation properties?
- A. (B. Roblin) I would say no, but we will check.

B.P. Raju, Coromandel Fertilisers Ltd., India

- **Q.** Whether effectiveness of application if crystal modifier has evaluated with respect to ingenious rock?
- **A.** (T Theys) Preliminary tests have been done in our pilot plant. They show that the crystallization was influenced by the Montaline and that filtration could be improved. These observation were only qualitative and no quantitative analysis were performed.

Shiv Shulka, Monsanto DMCC, India

- **Q.** Will this work for symposium formed in future gas di-sulfurisation section units due to reaction of Ca(OH)2 and SO2? The gypsum formed during this reaction is in the form of 20% slurry and very difficult to filter
- A. (B Roblin) We have not yet considered that process. We should do it in the future.
- **Q.** Normally gypsum produced from flue gas desulphurisation route is unfilterable. Will this process help to make it filterable, that is after using this crystal modifier.
- A. (T. Theys) This is an opportunity to work together!

(B. Roblin) Feel free to contact us for sampling

Comment Shiv Shukla: We have this gypsum slurry and the difficult part is to filter it out.

M. Alasha, SABIC, Saudi Arabia

- **Q.** Impurities such like Ca and Mg are the most disadvantageous to use H_3PO_4 in pure liquid fertilizers. Did you improve any method to reduce such impurities?
- A. (T. Theys) No, we require Mg and iron to same extend for good granulation.

Harald Franzrahe, Uhde GmbH, Germany

- **Q.** *Is the product biogradable?*
- **A.** (T. Theys) Background: In recent papers the EPA and FIPR have voiced concern over possible groundwater contamination due to flocculants and other additives in the run off and seepage from gypsum stacks.

(B. Roblin) We are very concerned by biodegradability, toxicity, eco-toxicity and labeling consequently we developed our products considering these specifications. We would be pleased to communicate data depending on the reference of your biodegradability test.

R. Murali, Deepak Fertilisers and Petrochemicals Corporation Ltd., India

- **Q.** Can crystal modifiers be used to reduce R2O₃ content (Al, Mg, etc.)?
- **A.** (B. Roblin) Sometimes Al2O3 or alumina-silicates are used to improve crystallization of calcium sulfate , Montaline can be used instead of these chemicals.
- **Q.** Will it increase water soluble P_2O_5 in acid? If yes, to what extent?
- **A.** (T. Theys) From what we have seen in the pilot test, the crystal habit modifier has a positive impact in the P2O5 water soluble losses.
- **Q.** Can montaline be used with phosphoric acid in 'AN' based fertilisers to get higher water solubility safely?
- **A.** (B. Roblin) We developed a process additives range dedicated to fertilizers manufacturing. We would be pleased to send you sample for preliminary evaluation.

Paper N° 14: Approach to optimisation of the major process parameters in WPA production

Karim Halaseh, JPMC, Jordan.

Jan Tytgat, SNC-Lavalin Europe, Belgium

- **Q.** Concerning the solid content in the reactor you mentioned that should be kept as constant as possible. Did you observe to significant difference in total P_2O_5 yield while changing these parameters?
- A. If you are running the phosphoric acid plant at 100% capacity: in that case, when the solid content is very low, you will pump too much slurry on the filter. Accordingly, you will reduce the retention time, with higher P_2O_5 losses as soluble P_2O_5 due to overloading of filters. Secondly, you will consume more defoamer in the reactor. Hence to run the plant at maximum capacity you must increase the solid content as much as possible, thereby increasing the retention time, less slurry and less water soluble P_2O_5 in the gypsum.

P.A. Smith, Bunge Fertilizantes S.A., Brazil

Q. This is a classic paper. You should be congratulated and this kind of paper has not been made at Technical Conferences for a long time. The studies were very well done and most of the results fall in line with expectations.

Two comments: This works applies specifically to Jordan phosphates which are very fine, 100% passing 100-mesh, and 70% through 75 μ . The high sulphate content is due to the fine nature. The surprising result is that the increase in temperature gives a rise to unreacted rock. Normally, reaction is a function of temperature. Aside from that unexpected result, this is a classic paper that would be referred to in the future.

A. From our observation, we note a slight increase in unreacted rock when the temperature was increased from our normal operating temperature of 72°C. However the change is not significant: from 0.85% to 1.1%. We would verify again.

Paper N° 15: Productivity improvements in Murugappa Group's phosphatic fertiliser units at Visakhapatnam and Ennore

N. Seetaram, Coromandel Fertilisers Ltd. and M.S. Srinivasan, EID-Parry, India

Jan Tytgat, SNC-Lavalin Europe, Belgium

- **Q.** What is the concentration of P_2O_5 in the reaction link?
- A. 28%
- **Q.** What is the total efficiency of the phosphoric acid plant?
- **A.** 96%

Shiv Shulka, Monsanto DMCC, India

- **Q.** Your pollution figure, are you able to obtain it on a sustained basis? 12 kg / mt on SO_2
- **A.** Yes, we have done a lot of revamp with Monsanto DMCC's know-how. The 1200 t/d plant is now running continuously with less then 1 kg/t over the last 6 months. There is no loss in production.

Anonymous

- **Q.** What is the retention time of your single reactor?
- **A.** Our reaction has a volume of 620m³ and filter area 58m², that is designed for 250 t/d plant. This has remained unchanged. The original retention time for the slurry was 6 to 7 hours. Today the residence time is around 4 hours. There is some gypsum losses. That is why I am interested on montaline since building the enlarged reactor is very expensive.
- **Q.** How much P_2O_5 is produced from one cycle of filter cloth per month.
- A. We produce around 12 000 to 13 000 t/month and change the cloth once a month.
- **Q.** *Have you faced scale formation problem on the filter cloth and grids, product pipe as you have single truck reactor.*
- **A.** It varies with rocks. We used to operate with Florida rocks, which is highly scaling. We used to do scrubbing every week in the evaporation section and 15 days in the reaction section. Now we are using Senegal, Nauru and Chinese rocks and have reduction in the scaling tendency. Currently we scrub every 2 weeks in the evaporation section.



<u>Chairman</u>: T. K. Jenssen, Hydro Agri, Norway <u>Rapporteurs</u>: Leif Rasmussen, Kemira Agro Oy, Denmark Arun Kumar, National Fertilizers Ltd., India

Paper N°16: Straight ammonium nitrate granule/prill fertilizer stabilisation: Theoretical possibilities

Bernard Laurent, Kemira SA, Belgium

Harold Lai, Mauritius Chemical and Fertiliser Industry Ltd

- **Q.** Is ammonium nitrate crystal stability still an important process issue if 20-30% filler material is added to the AN as in the production of CAN (= Calcium Ammonium Nitrate)?
- **A.** The physical properties and the resistance towards thermo cycling are generally better in CAN than in AN 33,5/34,5 N due to the larger content of filler. It can be that the filler used shows better results from a stabilisation point of view than an additive as it is added in a larger amount. It can be the case for dolomite containing impurities such as clays, gypsum or other fillers like biotite, etc.

From this point of view, it could be that the basalt you are using as CAN filler shows stabilizing properties.

But it is still relevant to add an additive if the filler does not give any resistance against thermo cycling or in the worst case if it gives poor resistance against thermo cycling like reactive limestone creating porosity.

- **Q.** Can thermo cycles increase the porosity of AN prills or granules and how?
- A. Please see chapter 4 in my presentation.

Kish Shah, Terra Nitrogen (UK) Ltd., UK

- **Q.** Could you please examine the mechanism by which China clay affects the anti caking property and thermal stability, if any?
- **A.** Clays incorporated in the prill/granule bind water and therefore increase the transition temperature by minimising the free water. It is not only the total water they can bind which is important but also the strength with which they retain it.

Some clays incorporated in the prill/granule also act as a binding agent increasing as such the product hardness.

These two properties reduce the caking.

I do not believe that China clay added to the surface in an anti-caking treatment will have any noticeable impact on the two mechanisms mentioned above.

A. A. Ramamurty, Coromandel Fertilisers Ltd. Visaphaptnam, India

- **Q.** How does the thermal stability of an AN fertiliser influence its "Explosion hazard"?
- A. Please see chapter 3 in my presentation

R. Murali, Deepak Fertilisers and Petrochemicals Corp.Ltd., India

- **Q.** What is the experience with boric acid, DAP, MAP and AS (normally called Permalene) as crystal stabiliser?
- A. Please, see chapter 5.1.3 in my presentation

Very few plants in the US are still using Permalene. To my best knowledge they are planning to change it to less complicated and more efficient stabilisers.

After unconclusive trials in Europe the Permalene has not been introduced successfully as a crystal stabiliser here.

- **Q.** *What should be the AN melt concentration (max.) at which the additive can be introduced?*
- **A.** There is no direct relation between melt concentration and additive efficiency. There is on the other hand a relation with the melt temperature.
- **Q.** What are the quality parameters of AN, if checked in the lab, for thermal cycling?
- A. Please, see chapter 2 in my presentation.

Paper N°17: Safety aspects of Ammonium Nitrate Fertilizers.

K.D. Shah, Terra Nitrogen (U.K.) Ltd., United Kingdom.

Leif K. Rasmussen, Kemira Agro Oj, Denmark

- **Q.** In the UN Classification Scheme one talks about mixture of AN, phosphate and potash What does potash mean in this context?
- **A.** Potash has the widely accepted meaning of potassium chloride typically in the mineral form, which may contain small amounts of sodium chloride, being naturally present.

Tore Jenssen, Hydro Agri, Norway

- **Q.** In what area should we focus our *R* and *D* to improve our understanding of AN safety even further?
- A. Some recent accidents have shown that our knowledge and understanding of reactions of AN with impurities and contaminants which may enter the process. However these substances would react in acidic and non-acidic conditions. EFMA recognized this gap and has commissioned a research project. This is in progress and we hope to get useful results in a year or so.

Paper N°18: Impact of impurities and pH on ammonium nitrate stability.

Antti Vuori, Kemira Oyj, Finland

Tore Jenssen, Hydro Agri, Norway

Q. Since *pH* is the most critical factor to take account of w.r.t. decomposition, assume you will confirm that the precautionary measures we need to take in the plants are:

- *i)* Availability of ammonia injection.
- ii) Availability of water for emergency dilution.
- **A.** The answer is definitely yes.

Paper N°19: AZF Toulouse Disaster

Jérôme Berthe, Grande Paroisse, S.A., France

R. Murali, Deepak Fertilizers and Petrochemical Corp. Ltd., India.

- **Q.** What was the procedure for recycling of downgrades AN before and after the incidents.
- **A.** It is impossible to describe the complete procedure for downgraded AN here. It has not been significantly changed in their facilities after the explosion. Only the authorized amount of product has been more strictly limited.
- **Q.** Are both industrial and fertilizer grades AN stored together?
- **A.** No, they were stored separately. Only the downgraded products were stored together for recycling in the complex fertilizers as nitrogen feedstock.

Harald Franzrahe, UDHE Gmbh, Germany

You mentioned that the warehouse suffered aerial bombing damage during WW II

- **Q.** Has the possibility that an unexploded bomb was the cause of the initial explosion being examined?
- A. Yes this possibility has been examined.
- **Q.** In German cities, which suffered extensive bombing, to this day unexploded bombs are still found regularly.
- **A.** Using oil prospection technique gives ("air-mag") we could not find evidence of the presence of the bomb.
- **Q.** After fifty years underground these are extremely corroded and instable. This might explain the two explosions reported.
- A. But of course, this was done after the explosion.

Paper N°20: Implementation of S.H.E.Q.Management Systems at Coromandel Fertilisers Ltd.

A.A. Ramamurty and B.P. Raju, Coromandel Fertilisers Ltd., India

Shamsher Singh, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., Kalol, India.

Q. Legal formalities and procedures fulfillment for contract labors. What are those legal formalities required. Whether these contract labors on continuous basis or intermittent. What precautions are being taken so that they do not become permanent liability for the company?

A. As per legal provision in the state of Andhra Pradesh, we are allowed to engage contract workmen in packing, activities of intermittent in nature, some maintenance activities and all civil construction work.

But all the contract workmen have to receive safety training, as per our contract procedure. Workmen so trained are given card, which they have to show at the plant entry point.

If the contractor introduces any new worker, he has to receive short-term instruction before taking up the job.

There is no legal requirement of absorbing contract workmen, so long as they are engaged in areas not specifically prohibited by the contract workmen (abolition) act of the state. The present trend is to allow companies to "outsource" point of work- the method of prescribing the work contract is important.

K. M. Tandon, Shriram Fertilizers and Chemicals, India.

- **Q.** *How do you keep your work place motivated: do better and better in safety performance.*
- **A.** We have involved all the levels of employees in implementation. We organize floor level meetings in which workmen discuss, review, and suggest changes to procedures.

We have a system of house keeping competition (quarterly), 'safe million-man hour' event celebration, suggestion scheme, and employee recognition schemes.

A 'plant safety committee' with all HOD'S chaired by plant chief, reviews safety every month.

'General safety committee' with equal participation by management and workmen representatives meets every quarter to monitor progress.

Workmen in each area are made part of terms to update points like hazard analysis, incident investigation, and 'pre startup safety review'.

M.R. Patel, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., Kalol, India.

- **Q.** *CFL has installed low NOX burner by BHEL. Can you tell us the level of NOX received with their number?*
- **A.** The low NO_X burner was a part of R and D work by M/s BHEL, Trichi, in which CFL collaborated by providing plant trials facility. Their final design reduced NOX from about 400+ PPM to about 120 PPM level and their target was less than 100 PPM.

After closure of ammonia and urea plants, there was no need for the utility boiler to operate continuously. It is run to keep on hot banks.

The low NOX conversion on DG + SET reduced NOX by 30% from earlier level.

Shiv Shukla, Monsanto-DMCC, India

- **Q.** What are the statistical quality controls being used in the process safety and quality management system? Whether safety records of contract workers are also considered for the analysis of safety data?
- **A.** For process control charts are used. For monitoring the quarterly objectives, the % non-conformance is monitored at management review meeting.

The safety records data is separately maintained for contract workmen and CFL employees. However, the incident investigations of accidents/incidents are a common record. Contractor's representative is associated with investigation.

For statuary authorities, we report all accidents: both contract workmen and CFL employees.

Paper N°21: Can passivation air in urea plants be minimized through the use of Safurex® and consequently improve plant safety?

Bart Gevers and Will Lemmen, Stamicarbon, The Netherlands (Presented by Bart Gevers and Hans van Baal)

Shamsher Singh, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., Kalol, India.

- **Q.** If H2 content is less than 1% in CO2 stream, can the oxygen quantity can be reduced from 0.6% by volume, which is minimum required to be maintained.
- **A.** No, the 0.6% O2 in CO2 feed is for passivity of synthesis section and so is not influenced by the H2 content.

R. Muthu Manoharan, SPIC - Southern Petrochemical Industries Corporation, India.

- **Q.** *Is it is possible to use Safurex to line the urea reactor in place of the existing titanium lining.*
- **A.** Yes, this material can be applied as well for existing as new reactors. Because of higher strength properties, the wall thickness can be reduced to approximately 5 mm, which makes that handling (forming) of plates is very well possible.

P. Orphanides, Orphanco, Greece

- **Q.** How more expensive is SAFUREX compared to other duplex steels in case of HP piping in urea synthesis.
- **A.** Duplex material is not an alternative for Safurex in oxygen free synthesis section, as duplex steel still gets active.

Price is determined by tube length (Large size available) wall thickness welding consumables and time, and downsizing and weight savings. In total; HP piping in a 2000mtpd Urea plant is in Safurex we calculated a cost saving of 60 %.



<u>Chairman</u>: Vaughn Astley, IMC Global, USA <u>Rapporteur:</u> B. Swaminathan, FAI, India S. Nand, FAI, India

Paper N°22: Pugmill granulation: a state of the art process for CAN and other ammonium nitrate based fertilisers

Harald Franzrahe and Paul Niehues, Uhde GmbH, Germany, (Presented by Harald Franzrahe)

Arthur Van Brempt, Kemira S.A., Belgium

- **Q.** *Why you are classifying pugmill as high recycle process? Recycle ratio of 1:1 is feasible.*
- **A.** The definition of high recycle and low recycle is relative. For NPK the recycle ratio is 3-4 for DAP it is 5-8 and for ammonium nitrate it is very low 1.1 to 1.2. The term has been used only to make a distinction between the significant features of the two process namely; pugmill and fluidised bed granulation. The pugmill process has relatively a very high solid recycle, while fluidised bed granulation process has relatively low solid recycle but high liquid recycle. For Uhde AN and CAN plant, the normal design recycle is between 2 to 3. Once the plant stabilizes it can then be reduced to 1 or 1.1.
- **Q.** How you can explain that water pick up occurs in the drying section if Mg(NO3)2 is used in a high recycle process?
- **A.** The problem of adding magnesium nitrate to the process is that the magnesium nitrate in the dust, dryer or drying air system tends to absorb moisture and form very large deposit in the exit of the drying drum and the air system. This leads to significant blockages. Uhde's experience in this matter is limited since only 2 or 3 plant runs were undertaken which were unsuccessful. At the moment it does not seem to work but may be with more efforts a way could be found to make it work.
- **Q.** Are there no other reasons why Uhde is not using Mg(NO3)2 as thermal stabilisation agent?
- **A.** The disadvantage of magnesium nitrate is, it introduce a very high water pick up in the product. In process where magnesium nitrate is used like fluidised bed granulation and pan granulation, the product has to be bagged immediately. Uhde is looking for substances which can stabilise the product but not increase the moisture uptake.
- **Q.** *How is the CAN granule hardness in pugmill compared with spherodizer or fluidised bed?*
- **A.** The CAN granules produced through pugmill is a very dense, hard product. It is influenced heavily by the filler added due to kneading and mixing action. The strength of the granule with dolamite filler is around 6 kg/3mm grain while the strength of granules with limestone filler is 3-4 kg.
- **Q.** What about roundness compared with spherodiser or Fluidised Bed?
- A. The roundness is not as good as fluidised bed process but is comparable and not much different.
N.Seetaram, Coromandel Fertilisers Ltd., India

- **Q.** Do you recommend this only to CAN and AN based plants?
- **A.** Pugmill or blunger is a very old development. It has been used for NPK production earlier. However, presently it is not used for NPK complex fertilizer as it involves complex reactions and mixing of varying components. Also in pugmill the air flow going though pugmill restrict the heat balance. So you have less control, where as in chemically pure compound like ammonium nitrate heat balance is easy to maintain. Consequently for NPK, pipe reactor with drum granulation is a more effective solution.
- **Q.** *How about its advantages over energy efficient pipe reactor processes?*
- **A.** Drum granulation with pipe reactor is the mainstay for NPK production. During NPK production different phosphoric acid and different additives like ammonium sulphate, ammonium nitrate is added. Drum granulation being more flexible has replaced all blunger installations.

Bjarne Christensen, Kemira Agro Oy, Denmark

- **Q.** *Have you experiences with CaCl2-2H2O or CN granulation?*
- **A.** Uhde's experience with pugmill is limited to only ammonium nitrate and calcium ammonium nitrate fertilizer.
- **Q.** Do you have pilot facility for new product testing?
- A. No answer

PAPER N°23: Process technology of urea-based NPK and practical experience of industrial plants

Guifan Chu and François Ledoux, Kaltenbach-Thüring S.A., France (Presented by François Ledoux)

N. Seetaram, Coromandel Fertilisers Ltd., India

- **Q.** *CFL has been producing urea based 28:28:0 for last 30 years but could not achieve crushing strength of the order of 3.5 kg per granule mentioned by you. Could you give reason for high crushing strength?*
- **A.** The reasons for high crushing strength are:
 - Low moisture content.
 - Use of kaolin clay as filler. Also from time to time coal ash from boiler section is used. Use of coal ash reduces the strength comparatively but is still high. For example, with the use of coal ash the strength of 2 mm granule is 2 kg and that of 3 mm granule is 3 kg. When using prilled urea, as in Coromandel, the urea prills are not crushed and therefore, weaken the granules.
- **Q.** *What acid is used?*
- **A.** Normally acid is not used. Only during the start up of the plant some acid is used. Sulphuric acid with some ammonia may be used in the rolling bed to help increase the temperature in the inter granulator. Currently, only urea solution with steam is used to adjust granulation.

Bjarne Christensen, Kemira Agro Oy, Denmark

Q. *What is the weekly capacity achieved compared to the design capacity?*

- **A.** The plant is designed for 22 hours operation per day as per IFDC recommendation. However, in future even 24 hours per day working operation would be possible. The capacity presently achieved for a significant period is 75% of the capacity on weekly basis.
- **Q.** During training was any problem faced in China like communication, culture, etc.? Please elaborate.
- **A.** Most of the communication was performed in English, a language that many Chinese engineers can speak very well. Moreover there were always one interpreter in case. Anyway KT is well experienced to work in China, where we have already 6 plants in operation and 5 under construction for NPK, ammonium nitrate, urea granulation.
- **Q.** *What brand of screen from Germany was used?*
- **A.** The screen is from Rhewum-Germany and the crusher from Aubema-Germany. Screening and crushing are two critical steps in a granulation process and reliable suppliers are absolutely required.

A.G. Margabandhu, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., India

- **Q.** How do you control the quality analysis of the product since many components are used in inputs, do you get consistent quality analysis?
- **A.** Due to residence time in the process loop and due to the time required to perform the analysis of the NPK grade, it is not realistic to adjust the grade a posteriori.

Therefore the urea solution is classically measured with electromagnetic flowmeter whereas the solid raw materials are batch weighed and then discharge to a hopper allowing to continuously feed the granulator. KT prefers that kind of system for its reliability and accuracy, much greater than with individual belt type weigh feeders for example.

In addition regular analysis (twice a shift) are performed on the final product.

Leif K. Rasmussen, Kemira Agro Oy, Denmark

- **Q.** What would be a typical set of operating parameters for water content and temperature for the product exit granulation drain?
- **A.** The whole granulation loop is performed at a low temperature compared with some other NPK processes: mainly because the dryer works at low temperature, due to urea based NPK sensitiveness to temperature, and there is a process cooler prior to screening/crushing.
- **Q.** What do you mean with pre-treatment of TSP to avoid an unwanted reaction with urea?
- **A.** The TSP is ammoniated at an accurate degree in order to avoid the release of crystallization water when urea and TSP get in contact.

K.M. Tandon, SFC, India

- **Q.** What is the investment (cost) of the granulation plant?
- A. In China, the whole investment (process plant and storages) has been around 10 million USD.

Arthur Van Brempt, Kemira S.A., Belgium

- Q. Is KT recommending a particular coating for Urea based NPK?
- A. No answer
- **Q.** Is crushing of solid Urea (prills) required in case of spec. consumption above 100 kg/t Urea?

- **A.** When solid urea is required, it should preferably be off spec prills, typically some broken prills or micro prills. If large prills to be used, then a crushing step is indeed advantageous.
- **Q.** *Please tell if there is application of bulk solution cooler in the plant, particularly in India?*
- **A.** The final cooling step in Zhongyuan plant is performed in a fluidised bed cooler. The air from the fluidised bed cooler is recycled into the process, improving the heat recovery of the process.

Paper N°24: Urea caking problems. How to avoid them

Pan Orphanides, Orphanco, Greece (Presented by Pan Orphanides)

Y. Narula, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., India

- **Q.** Is the bulk flow cooler functional anywhere especially India?
- **A.** I am not aware whether it is operating anywhere in India. But it is operating in many plants in China, Saudi Arabia and Gulf area.
- **Q.** What is the cooling water requirement of a 1000 tonnes per day plant?
- A. The cooling water requirement of a 1000 tonnes per day plant is around 80 M3.

Paper N°25: Granulation plant revamps – Methodology and design options

M.J. Bowness and D.M. Ivell, Jacobs Engineering, USA (Presented by M.J. Bowness)

Arthur Van Brempt, Kemira S.A., Belgium

- **Q.** What is your recommended mole ratio, both minimum and maximum in pipe reactor for avoiding blockage and minimisation of ammonia loss?
- **A.** The mole ratio is dependent on the product you make and how you make it. The mole ratio for DAP is 1.4 to 1.45. For MAP it is 1.4 for back titration and 0.6 for forward titration.

S. Ravi Kiran, Coromandel Fertilisers Ltd., India

- **Q.** What are the type of NPK's that can be produced in Jacob's design? Is it suitable for all grades?
- **A.** The products that have been produced are NPK's and ammonium phosphates. Urea ammonium phosphate has not been produced with pipe recator.
- **Q.** Can we get round granules with pipe reactor compared to pre-neutraliser?
- **A.** The shape of the granule is affected by granulator and type of the acid. With tank and pipe reactor similar results have been obtained.

A.G. Margabandhu, IFFCO - Indian Farmers Fertiliser Cooperative Ltd., India

Q. How do you control the back pressure in case of tank and pipe in series? What sort of a back pressure you have in case of choking or any other problem?

A. The back pressure in the pipe reactor by itself or when you have tank and pipe reactor in series will be the same. We typically design the normal operating pressure in the pipe around 2 bar and the back pressure on the pipe reactor up to 5 bar. One has to make sure of the supply of raw material in sufficient quantity and also make sure that the raw material supplied is at right pressure, both acid and ammonia going into the pipe reactor.

Paper N°26: Life extension of a vintage plant through reliability improvement systems

Babu K. Verghese, V.Jayaraman and S. Muruganandam, Southern Petrochemical Industries Corp. Ltd., India

(Presented by V. Jayaraman)

R.K. Kukreja, HLCL, India

- **Q.** What are the techniques used for preventive maintenance of electrical equipment in SPIC?
- **A.** Electrical equipment for example, rotary equipment motor is preventively checked for bearing noise, bearing condition and insulation condition. In case of problem it is removed to the workshop and the insulation is redone or the bearing is changed. In the case of modules, during shutdown or in planned frequency, the unit is stopped and the module is pulled out, cleaned and the condition of the module is checked.
- **Q.** What is a problem bank in SPIC?
- **A.** In SPIC a register is kept in each of the plant. Whosoever identifies a problem writes it in the register. This register is checked by the Head of the Department everyday and if needed engineering advice is taken on the identified problems.



<u>Chairman</u>: Tibaut Theys, Prayon S.A., Belgium <u>Rapporteurs</u>: Jan Tytgat, SNC-Lavalin Europe, Belgium Benoît Roblin, SEPPIC, France

Paper N°28: Increased energy recovery in a sulphuric acid plant by heat recovery system (HRS)

Shiv Shukla, Monsanto - DMCC, India.

T.N.U. Satyanarayana, Indo Jordan Chemicals Co. Ltd., Jordan

- **Q.** How does HRS plant compare with conventional plant with respect to the performance of converter in terms of efficiency and life of catalyst?
- **A.** Over a period of time, you are expecting that the catalyst performance may go down, but that would not happen because this energy recovery in the acid circuit is independent of that and the only thing which is to affect cool water performance is any mist carry out. Thus great care is taking in designing the distributors. That is why the tower has two sections : top section and bottom section.

Bottom section is the one which is operated at high temperature and top section operated at almost like your inter pass absorption tower so that you do not have a lot of mist coming out and once you do not have much of mist entering your converter bed, you do not have any effect of HRS system on your converter performance.

- **Q.** What is the maximum acid inlet and gas temperature allowed in conventional DLDA plant which is not equipped with HR9?
- A. Now here in a sense what we are taking about it is not the question of maximum gas temperature which we can take but it is a question of maximum acid temperature that we can take. Basically in a conventional plant we try to recover as much as energy possible through the gas. So there, the objective would be to go down to the lowest possible temperature without having any condensation of sulphuric acid.

Now in the case of a HRS, gas temperature would be still same as a normal plant, only acid side temperature will be increased so that we can recover this energy from the acid circuit which otherwise goes to the cooling tower.

G. Aviram, Rotem Amferi Negev, Israel

- **Q.** In case the production of steam is balanced with the consumption of other plants, what can be done with the extra stream from HRS?
- **Q.** *Is it economical to install turbo-generator to convert the steam to electric power?*
- A. That is a very important question.

The first thing is to understand the steam balance of a complex and as I said that with the rising power cost nowadays, with 8 c/KWH it is viable to recover the energy in the former power. So basically what we are trying to do is: what ever steam is produced in HRS can be used in generating power but that power is only viable if your power cost is something like 8 to 10

c/KWH. If it is anything lower than that, unfortunately in today scenario it will not be economically viable.

So we are talking about future scenario when the energy cost are going to go up and up.

I think that it is the trend in energy sector. It will make sense to recover much and much energy and convert it to power even though you may not have the use of steam.

Nugroho Christijanto, PT. Petrokimia Gresik, Indonesia

- **Q.** Is it possible to install HRS to SA DCDA existing plant without any major modification to the existing plant. Does steam injection to the gas flow still have benefit like in the HRS system please explain.
- A. No answer

N. Seeteram, Coromandel Fertilisers Ltd., India

- **Q.** What is the mechanical condition of the plant HRS system installed in 10 years back. What is the expected life of HRS system?
- A. No answer

Neel Naidoo, Foskor Ltd., South Africa

- **Q.** Mr. Shukla's presentation showed HRS heaters without anodic protection. How is this achieved?
- A. No answer
- **Q.** How do you control the acid strength to prevent corrosion problems?
- A. No answer

R. Muthu Manoharan, SPIC - Southern Petrochemical Industries Corperation, India.

- **Q.** You indicated that the cost of retrofit to HRS process is 1.8 million USD, does it include all the cost including installation of the electrical distribution?
- A. No answer

Paper N°29: Sulphur fertilizers - New products add to conventional sources to offer a wide range of options

D.L. Messick and C. de Brey, (Presented by D.L. Messick), The Sulphur Institute, USA

Miss Kabbaj, OCP, Morocco

- **Q.** Is there any agronomic effectiveness differences between sulphur coated fertilizers and sulphur incorporated fertilizers?
- **A.** If the sulphur get into the soil, then it gets the oxidation, then you are fine, it does not matter if it is coated or contained in an NPKS. I do not know any differentiation that has been demonstrated in the field.

A.A. Ramamurthy, Coromandel Fertilisers Ltd., India

Q. India being a net importer of elemental sulphur, would it be economical to make elemental sulphur for the soil application in India?

A. We think so. Trade channel are set up predominantly coming from the Middle East (oil and gas product) and other place where you can get the material.

Also technologies is available for elemental sulphur containing materials.

Arthur Van Brempt, Kemira S.A., Belgium

- **Q.** *From an agronomical point of view, is CaSO4 as efficient as ammonium sulphate?*
- A. Calcium sulphate and ammonium sulphate; we have done a lot of trials comparing the two.

The sulphur contained materials perform equally.

What we found is that if gypsum is not used in a lot of market has to do with its physical properties because it has to be formed in a certain way to go into a bulk blending or whatever. Transportation cost is also affecting it. Thus it is not hardly used outside of mine or places where it is produced.

The solubility of both products is different in the laboratory but in field it does not seems that there is a big difference.

- **Q.** Is there knowledge related to safety available for mixtures of ammonium nitrate and elemental sulphur?
- **A.** I do not know. I was not aware of this mixture. I have to ask to a colleague to answer you the question.
- **Q.** Are blenders mixing granulated elemental sulphur with ammonium nitrate fertilizers?
- A. No answer

A.A. Ramamurthy, Coromandel Fertilisers Ltd., India

- **Q.** What are the sources of process technology for sulphur fertilizer especially Bentonite-mixed "S" and for coating ammonium-phosphate based fertilizers?
- A. No answer

Paper N°30: An overview of radiation legislation that impacts on the phosphate and fertilizer industry

A.J. Van der Westhuizen, Foskor Limited, South Africa

T.N.U. Satyanarayana, Indo Jordan Chemicals Co. Ltd., Jordan

- **Q.** What are the guidelines of radiation level on wet and dry gypsum stacks?
- **A.** IAEA is working on that now. I was part of the technical group that started Monday at IAEA. and continuing till Friday.

One of the main consideration was: should old gypsum stack be considered as "a practice or an intervention". One is preventing that a dose exceed a certain value, the other is reducing dosage.

I believe that it should be considered as intervention. Old stack is supposed to be build under best practice. Any new change in legislation should not be held against you. By the end of the week, more information will be available.

- **Q.** Do you have any experience/data whether there will be change in radiation level if the stack is left in 10 20 years?
- A. Gypsum material is subject to the same specific activity criteria as your product.

Like for example, in Europe it is around 1 Bq/g for uranium and thorium. But in Germany you have to add the uranium and the thorium and the sum must be equal or less than 1 Bq/g. So level will vary from country to country.

Paper N°31: Putting really cool ideas to work in phosphates

Vaughn Astley, IMC Global, USA

V. Jayaraman, SPIC - Southern Petrochemical Industries Corporation, India

- **Q.** Why do you purify (by reverse osmosis) : a) the gypsum pond water?b) where is it used?
- A. Actually it is a process that is in development. We have done a lot of bench work and some pilot work. At Poiny Point the problem is critical. There is a surplus of water and it is on Tampa Bay. A release of water would be really bad news. We did a presentation for The Florida Institute of Phosphate Research. Instead of going to a pilot plant running at 100 GPM, they devote us 1.5 million dollars to implement a unit at Poiny Point of 1500 GPM.

But to answer your question we would expect the P2O5 concentrated strain to go into the DAP plant.

- **Q.** *Have you not thought of using the pond water for using it in the P.A. Plant for slurrying gypsum (Pumpability purpose)?*
- **A.** It depends of the water balance of the circuit. Usually you keep recycling the water or if your balance is negative you add water. The reverse osmosis system is necessary is really only applicable if you have to get rid of water of the system.

Sulphate control being very important, is any reliable free acid analyzer developed and used at present or it is still by hourly sampling, analysis and feeding to control logic system?

When I was working for Albright and Wilson I used to market a sulphate analyzer. In A and W they had 3 production units and they dedicated a person to maintain the analyzer. When it was utilized in Florida it was not very successful. We believe that analyze of sulphate is one of the easiest, simple and cheapest thing to do. It is easy to maintain, it is a quick technique. Analyze takes less than 2 minutes. We utilize turbidimetry. We looked in the past at refractive index, conductivity and other techniques but none of them was satisfactory.

A lot of the process control that we do is using the process data to tell you what the result is. In minerals, for instance, we use "virtual analyses". We use the process to tell what the result is then we turn it around to control the process. So just like when you can use the process to predict SO2 emission rates by virtual analyses then you use that predicted number to control the process. So you do not need an instrument, you need an instrument engineer.

On that topic P.A. SMITH of BUNGE add the following comments:

The problem with on-line analyzer is that you end up calibrating it very often and you end up analyzing every 2 hours to check that it is working. The problem is: if it is totally on-line and there is an error the result is a disaster so you end-up to check if it is working properly.

ASHLEY goes on:

In our production plant, the process parameters are so stable that sulphate analysis can be done every 1.5 to 2 hours.

N. Seeteram, Coromandel Fertilisers Ltd., India

- **Q.** Sulphate control being very important, is any reliable free acid analyses developed and used at present or is it still by hourly sampling analysis and feeding to control logic system?
- A. No answer

Paper N°32: Urea Phosphate: manufacturing and uses of this dynamic fertilizer

Gerald Martin, Kemira Emirates Fertilizer, U.A.E.

Anonymous

- **Q.** What is the crystal content of the mother liquor?
- A. The mother liquor that we purge out and make a by-product with, is to go from 10 to 15%.

P. Orphanides, Orphanco, Greece

- **Q.** *Is P44 produced with trace-elements?*
- **A.** No we have not. We use only the 3 elements, but some micro elements can be found brought by the acid to the amount that it is a benefit.

Mohammed Akasha Khaleel, SABIC, Saudi Arabia

- **Q.** Why do you think UP does not spread worldwide like other NP materials DAP, MAP, etc. ...?
- **A.** I believe on one side that it is due to the tradition of using the same fertilizer from one generation to the other. Changing behavior of farmer is very difficult.

Second reason, it is more expensive than MAP or DAP.

You have to educate the growers by showing them experimentation. Market is growing fast especially in Middle East and Mexico.

- **Q.** *How did you handle the hygroscopicity problem of UP?*
- **A.** We do not add any anti-caking agent. We keep the relative humidity inside the building as low as possible. The plant is located in the middle of a desert.
- **Q.** Why didn't you go to use technical grade H3PO4 instead of the process acid? You are using prill area + acid, why did you have scrubbing unit to catch ammonia? Are you purifying H3PO4 before using it to remove Mg++ and Ca++?
- A. No answer



<u>Chairman</u>: Bjarne Christensen, Kemira Agro Danmark <u>Rapporteurs</u>: Bernard Laurent, S.A. Kemira, Belgium Veikko Harjula, Kemira Agro Oyj, Finland

Paper N°33: 4 bar absorber system

M.Rashid Al Rashid and Hussain Imam Bukhari, Ruwais Fertilizer Industries (Fertil), U.A.E.

(Presented by Ahmed Al Hossany)

K.M. Tandon, Shriram Fertilizer and Chemicals, India

- **Q.** *How did you choose 4 bar pressure for the scrubber?*
- **A.** Due to installation of new scrubber, ammonia and CO2 venting is less. So, 4 bar pressure is the optimum cost and less expensive than installing higher pressure.

Ans.B. Gevers from Stamicarbon: The lowest pressure has been selected to reduce the investment cost meanwhile achieving the environmental goal. Naturally, higher is the pressure, better is the catchment efficiency.

Remark: B. Christensen: CO2 recovery is not a true environment achievement as it is released in any case but NH3, yes.

Paper N°34: Approaches to limiting the content of environmentally harmful impurities in phosphate fertilizers

Nadya Dylevskaya, Phosagro, Russia (Presented by N. Dylevskaya and B. Levin)

Alfred Lowey-Ball, Ecophos S.A., Belgium

- **Q.** *What is the cost estimate for Cd removal technology plant? What technology was considered?*
- A. Our estimates are about 3 million USD with a production of 50.000 t P_2O_5 /year. We use the co-crystallization process which has been mentioned by Mr van Kauwenbergh.

Anonymous

- **Q.** The tables in your paper contains no sources of reference. Can you tell us where they were obtained from?
- **A.** Our sources are the book by P. Becker, issues of Phosphorous and Potassium, certificates issued by producers, our own data based on studies made.
- **Q.** Define "toxicity", especially with reference to phosphate rocks. Does the definition conform to Code of FAO?
- **A.** Our methodology was based on toxicity indicators of the elements in water and foodstuffs. We did not try to bring our toxicity index into line with FAO requirements.

It is an approach to single out different kinds of phosphate rocks.

Karim Halaseh, JPMC, Jordan

- **Q.** What is the percentage of Ti and Ln_2O_3 in Russia Kola apatite?
- A. We produce 2 grades of phosphate rock (Kola apatite). The content of TiO_2 in one grade is 0,4-0,5 %. In the super grade the content is 0,2 %. The Ln_2O_3 content is about 1 %.
- **Q.** *My reference is* 1.5% Ln₂O₃ *at which level it affects the hemi-dihydrate process.*
- **A.** OK but we have the technology to refine it to 1%.

Remark by Tore Jenssen Hydro Agri, Norway

At this moment, there is no proposal, no compulsive limit from EEC. EEC has asked for risk assements from European countries. These were done but nothing came out of it as there were different opinions. 60 ppm/kg P_2O_5 is a starting point.

The duty on Russian rock has no link with environment: Cd content but well on structure cost.

Paper N°35: New technologies to produce high quality fertilizers efficiently without environmental impact

F. Lopez de Azcona and S. Carrillo, Incro S.A., Spain (Presented by S. Carillo)

Bjarne Christensen, Kemira Agro Oy, Denmark

- **Q.** What is the ambient air requirement to efficient operation of the EM-system?
- **A.** For efficient operation you need at least some months with relative humidity around 70 %. On humid months you can accumulate in a pond. For efficient continuous operation in extremely humid weather it is better to install an EMC-unit.
- **Q.** Do you have any experience with nitric acid in the X-PR.
- A. Yes, we have installed several PRS for AN production, although design is different from X-PR.

Abdur Rahim, KAFCO, Bangladesh

- **Q.** What will be the cost per ton of recovered condensate of the effluent by atmospheric evaporation without addition cost for heat?
- A. Evaporation cost is 2-3 €/ton and to recover condensate price is about twice (5E/T). It depends if you consider the cost of the cooling tower.

G. K. Rao, Nagarjuna Fertilizers and Chemicals Ltd., India

- **Q.** What is the crushing strength of granule at CFL, India in your test run?
- A. 2,5 3 kp/cm² for 20-20-0 and 3 4 kp/cm² for 14-35-14
- **Q.** Your pipe reactor test run was demonstrated only 20-20-0, what other grades are possible to produce?
- **A.** 14-35-14, DAP, 15-40-10, 16-20-0 and many others. In principle any grade can be produced if suitable raw materials are available.

H Lai, Mauritius Chemical and Fertilizer Industry Ltd., Mauritius

- **Q.** What is the cost of the evaporation unit and max concentration of the result concentrate for a typical fertilizer application?
- A. One E-MC unit for 2 2,5 m³/h evaporation costs are 200.000 \in (400-600l/h without heat). Concentration depends on application, but it may go up to the pumpability of the solution.

P. Orphanides, Orphanco, Greece

- **Q.** The 14 35 14 NPK is produced with weak H_3PO_4 or only concentrated?
- A. 70 80 % concentrated and 20 30 % diluted to minimize recycle ratio

Arthur Van Brempt, Kemira S.A., Belgium

- **Q.** By co-neutralisation of H_2SO_4 / P_2O_5 in the X- PR: ratio of quantity of needed NH_3 for full neutralization is fed to the pipe and the granulator bed?
- **A.** In these grades, because of such a huge quantity of sulphuric acid and phosphoric acid and everything goes to the pipe reactor, most of the ammonia (80 90 %) goes to the pipe reactor and rest to the granulator bed. We keep a N/S and N/P ratio between 1,4-1,6
- **Q.** *NH3 losses from the drum granulator (total from PR and bed granulation)?*
- **A.** Ammonia losses are practically the same as the traditional pipe reactor.No problems were encountered with the scrubbing section.

Paper N°36: Environmental management issues of Indian fertiliser plants

M.P. Sukumaran Nair, The Fertilisers and Chemicals Travancore Ltd, India

No comments or questions.

Paper N°37: Environmentally efficient management of water resources in the German potash industry

K.D. Müller, M. Strube, K+S Kali GmbH and H.G. Bäthge, K+S Aktiengesellschaft, Germany (Presented by H.G. Bäthge)

Bjarne Christensen, Kemira Agro Oy, Denmark

- **Q.** What is the total water use in K+S? and what is the total disposal? Can waste brines be used in deicing during the winter period?
- **A.** About 80 mio m³ of water per year are used. From this 80 90 % is cooling water, which is released unchanged into a watercourse. The rest are waste brines. A part of the waste brines is concentrated to magnesium chloride solutions by evaporation of water. These magnesium chloride solutions are used in special industries and as deicing solution for a special wet-salt technique used in Germany during the winter period. About 12 mio m³ of brines are discharged approx. 50 % into watercourses and the other into deep wells.

R.K. Mutreja, Hind Lever Chemicals Ltd., India

- **Q.** In case brine is pumped to a geological suitable horizon, will it not pollute the fresh wateraquifers?
- **A.** It must be ensured that usable water-aquifers are not influenced by salt water. Therefore a comprehensive monitoring system is applied. Extensive geological and hydrogeological investigations must be carried out before a license or permit could be given to the discharger.
- **Q.** Does the German law allow pumping of brine deep into soil?
- A. Indeed, there is a problem with the German Water Act. The pollution of groundwater is there forbidden. Further more the definition of groundwater in Germany includes all natural flowing water below the surface assuming that all these waters are fresh water. But this is really not the case. At many places, especially in northern Germany, a lot of salt deposits could be observed, which have been risen up in the past million years near to the surface. These deposits are covered with an anhydrite layer (so called cap-rock) and in this layer a naturally nearly saturated salt solution is present.

As a conclusion: In very special cases of mining industry (e.g. oil and gas industry and potash industry) the discharge of waste water into special unsuitable groundwater levels could be allowed. But these waste water must not have any content of toxic or other components as the mined components.

The EU water framework directive considers the problems of mining industry (other than German law) and has an exemption clause in Art. 13 especially for the mining industry. Discharge of waste water from the mining industry could be allowed under the above mentioned requirements.

EXPERIENCE WITH DRY STACK MANAGEMENT OF GYPSUM (HEMIHYDRATE CALCIUM SULPHATE) (a)

A. Chari*, S. Subbiah** and T.N.V. Satyanarayana***, Indo Jordan Chemicals Company Ltd.,Jordan

1. Preamble

Indo Jordan Chemicals Company Ltd. (IJC), a joint venture company promoted by M/s. Southern Petrochemical Industries Corporation Ltd. (SPIC) - India, M/s. Jordan Phosphate Mines Co. Ltd. (JPMC) - Jordan, and The Arab Investment Company SAA (TAIC) - Saudi Arabia, has established a phosphoric acid complex in the Special Industrial Free Zone in Eshidiya, Jordan.

The phosphoric acid complex consists of a 2,000 MTPD sulphuric acid plant, based on Monsanto's Double Conversion and Double Absorption process, a 700 MTPD P2O5 phosphoric acid plant based on Hydro Agri's single stage hemihydrate process, and associated utilities and off-sites.

IJC commissioned its plants early 1997 and crossed 100% capacity utilization of its plants for three years in succession during 1998 - 2000. However, the capacity utilization was affected during the year 2001, due to recession in the phosphoric acid market.

Considering that the location of phosphoric acid plant is in dry desert area where virtually there is no rainfall and the water resources are precious, dry stacking of hemihydrate calcium sulphate, the byproduct from the phosphoric acid plant was selected.

This paper describes the operating experience of five years with Dry Stack management of gypsum (hemihydrate calcium sulphate), with specific reference to the following.

- Basis of selection of dry disposal method.
- Brief description of the system.
- Design features incorporated to have better operational flexibility.
- Characteristics of hemi-hydrate-calcium-sulphate produced/stacked.
- Experience with the first stack.
- Problems faced with stacking equipment.
- Reliability and capacity improvement jobs carried out.
- Advantages and limitations of dry stacking.

2. Basis of Selection of Dry Disposal Method

The selection of mode of disposal of filter cake (gypsum), wet or dry, for any phosphoric acid plant greatly depends on three factors, viz, the location of the plant, the climatic conditions and the investment cost (although the operating cost cannot be neglected).

2.1 Location of the plant

The plant is located in the desert area close to the phosphate mines in the southern part of Jordan, with no inhabitants within 50 km. The space required for stacking gypsum does not pose as such any constraint.

^{*} Managing Director

^{**} Assistant Managing Director (site)

^{***} Manager (Process Engineering)

Email: ijcshdya@go.com.jo

⁽a) received on 10/06/02

There are no water bodies in and around the plant. The static water level in the region is 279 m below the ground. In the geological structure of the area, there is practically an impermeable layer of more than 10 m of hard coquina. From the location point of view, both wet and dry modes of discharge are feasible.

2.2 Climatic conditions

The climatic conditions are extreme with ambient temperature varies from minus 7.4 deg C in winter to 42 deg C in summer. The air is dry with relative humidity between 29% and 57% during the year. The evaporation rate is quite high during summer. The rainfall is extremely low at 44 mm per year. Though the climatic conditions are favorable for both wet and dry modes of discharge, dry discharge of gypsum is more suitable considering the fact that the water resources are limited in the project location.

2.3 Investment cost

The initial investment cost will be lower for dry disposal system when compared to wet discharge since around 50,000 m2 area per year needs to be provided with lining for wet discharge for a 700 MTPD P2O5 plant, which is costlier compared to the cost of extension of the conveyor in case of dry discharge.

One of the key factors for the selection of hemihydrate process was minimization of water consumption since only half molecule of water is attached to CaSO4. Taking the advantage of the geographical factors, disposal of gypsum in dry mode has been judiciously selected in order to further reduce the water consumption and to minimize the investment cost of the project.

3. Brief Description of the System

Phosphoric acid is produced using different grades of Jordanian rock phosphate by a single stage hemihydrate process. The reaction takes place in a battery of three reactors in series. The slurry containing 40 - 42 % P2O5 and hemihydrate gypsum from the reaction system is filtered in two totally independent streams of belt filters of 60% capacity each. The filter cake is transported by a series of belt conveyors in dry mode and stacked (The process scheme for Gypsum Handling system is enclosed as Exhibit -1).

The filter cake from each belt filter is fed to a common belt conveyor (M 451-10) through the independent discharge chutes, which are provided with automatic hammering devices. The filter cake from this belt conveyor can be diverted in two ways. If the filter cake is of normal quality, it is fed to another belt conveyor (M 451-20). If the filter cake is of poor quality, it is diverted to emergency stack directly, from which it is transported to the main stack using pay loaders and trucks.

The filter cake from the second conveyor (M 451-20) can be diverted in two ways. During normal operation, the filter cake is fed to stack gypsum conveyor (M451-30). This conveyor consists of a fixed part which is anchored to the ground, and a part moving on rails which is linked to the belt tensioning device with counter weight. The initial length is 184 m with the last 80 m sloped at 10 0. The length of the conveyor can be extended periodically in steps up to maximum of 50 m per step without addition of belt and structural supports. The conveyor can be extended for a maximum of 1000m total length.

The filter cake from the stack gypsum conveyor (M451-30) is fed to a gypsum stacker (M 451 –40). This is equipped with a boom which can slew on a range of 180 0, which enables to feed the filter cake on either side of the stack, since the stack gypsum conveyor (M 451-30) is located at the center line of the stack. From the gypsum stacker (M 451-40), the filter cake is fed to gypsum swiveling

conveyor (M 451-50), which is mounted on caterpillars, and equipped with a fixed boom and a retractable belt thrower device slewing on 180 0, that is totally movable.

Finally, the filter cake is thrown from the thrower device at a trajectory of 12 - 16 m, and stacked automatically by suitable positioning of the gypsum stacker and the gypsum swiveling conveyor, and by using pay loaders / bulldozer as and when required.

Each stack is designed to meet three years of plant operation and subsequently a new stack is to be used.

4. Design Features Incorporated for Better Operational Flexibility

Based on the experience of operating phosphoric acid plants of SPIC and JPMC, special emphasis has been given during the design to maximize the onstream factor of the phosphoric acid plant. Accordingly, two totally independent streams of filtration have been provided. To maintain the same level of operational flexibility, the following features are incorporated in the design of gypsum handling / stacking system as well.

Conservative design of stack with 2,880,000 m3 capacity for three years of operation of phosphoric acid plant.

Emergency diversion at the outlet of first gypsum conveyor (M 451-10), should the filter cake be very wet / sticky during plant start ups / upsets. The storage capacity is for about 16 hrs, and the material is trucked to the main stack.

Diversion of filter cake at the downstream of second gypsum conveyor (M 451-20) to an auxiliary stack using movable conveyor (M456-10), should there be any blockage of chutes or maintenance of stacker conveying system. The storage capacity is for about 24 hrs, and the material is trucked to the main stack.

Bypass arrangement for the gypsum swiveling conveyor (M 451-50), and the material is stacked using pay loaders /bulldozer, for short duration.

Modular tables, hydraulic jacks and additional belt with winch and tension device for the stack gypsum conveyor (M451 - 30) for easy extension and minimum down time during each extension.

Level adjusting device to allow to compensate the slope of the ground level (5 0) to maintain the slewing ring horizontal for the gypsum stacker (M451 - 40).

A local mobile control room, which is shifted automatically along with the stack gypsum conveyor (M 451-10) during each extension, near to the stacking area for better monitoring.

Video monitoring device in the central control room to monitor the stacking system.

5. Characteristics of Hemi–Hydrate-Calcium-Sulphate Produced / Stacked

As envisaged in the design stage, different types / grades of rock phosphate (both wet and dry) such as 70-72 BPL, 60-65 BPL and dryer fines, etc, have been processed during last five years of operation. The filter cake at the exit of belt filters contains 18-22% W/W free moisture, with an average value of 20% W/W. Though filtration was normal, variation in the cake / crystal characteristics was noticed which was obviously due to the type of rock processed and the plant parameters.

5.1 Conversion of hemi- to dihydrate during transportation

Samples are collected from the filter feed slurry, the cake at the discharge of belt filters and at the outlet of thrower device, and analyzed for hemi hydrate content.

The typical results are tabulated below.

TEST	FILTER FEED	EXIT OF BE	ELT OUTLET OF	REMARKS
NO.	SLURRY	FILTER	THROWER	
1	98.28 %	98.20 %	98.12 %	New Stack
2	99.68 %	99.58 %	99.50 %	New Stack
3	99.85 %	99.82 %	99.78 %	New Stack

From the above, it is noted that the conversion from hemi to dihydrate during filtration and transportation is low (maximum 3% was observed earlier), though the hemi content in filter feed slurry, at times, was as low as 95 %.

5.2 Conversion of hemi- to dihydrate during storage

In order to check the degree of conversion, two cake samples were collected. One sample was stored under controlled ambient conditions inside the room (indoor) and the other one in natural atmosphere (outdoor). The content of hemi hydrate in the cake from each sample is analyzed every day for ten days, and the results are plotted in Exhibit -2.

It is noted that the rate of conversion was rapid in case of cake stored indoor and followed polynomial of third order as below.

Y = -0.187 X3 + 3.902 X2 - 27.394 X + 96.64

The rate of conversion of the sample stored out side was lower and followed polynomial of fifth order as below.

Y = -0.0054 X5 + 0.1195 X4 - 0.810 X2 - 9.565 X + 100.07

The data on the permeability of the cake stored indoor and outdoor is also measured and furnished in Exhibit -3. The permeability of the cake stored outdoor is maintained almost constant, while that of the cake stored indoor increased rapidly and crossed 200 cm2.

Based on the above experimental results, it is inferred that the climatic conditions have significant influence on the conversion from hemi to dihydrate.

5.3 Characteristics of filter cake stacked

Several samples have been collected from both sides of the gypsum stack at top and sides at a depth of around one foot (circa 30cm), and analyzed for hemihydrate content, permeability and specific surface area. The data is enclosed as Exhibit -4.

The average hemihydrate content varied between 8% - 27% W/W, while the permeability varied between 7 - 25 cm2 and the specific surface area varied between 1200 - 2000 cm2 / gm.

5.4 Size distribution of filter cake stacked

The typical size distribution of filter cake stacked during the years 1998 to 2002 is plotted and brought out as Exhibit -5. It is noted that more than 95% of the filter cake staked, passed through 150 micron mesh.

6. Experience in the first Gypson Stack

Though the stack was designed for three years of plant operation, it was used for five years (1997-2002) with total estimated quantity of 2,062,750 m3 filter cake, corresponding to 1,011,654 MT P2O5 production, due to the increased width of the stack (110-115 m actual vs. 100 m design), the natural compaction and the cushion provided in the design (such as the bulk density, moisture content). The second stack, adjacent to the first one, is in operation since April 2002. The observations made during the five years of operation with first gypsum stack are summarized hereafter.

6.1 General observations

- There was no major problem faced with the stack. No percolation of liquid was noticed either within the layers of the stack or at the ground level. The surface area of the stack, both on top surface and on side walls, was dry (Exhibit -6).
- The undisturbed areas (side walls and a part of top surface) varied from hard to very hard, while the top surface, where vehicle movement was present, was dry and powdery.

6.2 Settlement

Uneven settlement of stack was noticed especially adjacent to the stack gypsum conveyor, due to natural compaction, stacking equipment and partial conversion of filter cake to dihydrate, Due to this the slope of the stack could not be maintained at 5 0 as envisaged in the design. The actual elevation of the stack at the central axis versus the projected / theoretical elevation of the stack is shown in Exhibit -7.

6.3 Cracks

Cracks up to 5 - 10 m deep and 20 - 150 mm wide were noticed (Exhibit -8) since second year of operation, especially at end of the stack, which is most likely due to self weight of filter cake at the edge of the stack and conversion to dihydrate. As such no problem has been encountered during stacking, since material was filled and compacted as the stack was periodically advanced.

7. Problems Faced with Stacking Equipment

During commissioning and initial stage of operation, frequent problems were encountered with the stacking equipment. Some of them are highlighted below.

- 1. Frequent blocking of conveyor discharge chutes, leading to tripping of the conveyors. It was found that the cross section of the chutes was not adequate to take in to account the trajectory of the material discharged at different plant loads. The chutes were modified suitably.
- 2. Off centering of stacker gypsum conveyor. This was due to settlement of gypsum stored and frequent spillage of material. Periodical level measurements were taken for correction and self aligning rollers were provided.
- 3. Premature failure of conveyor belts. The belts were replaced in stages with chemical and oil resistant belts of higher strength.
- 4. Derailing of stacker gypsum conveyor due to high wind speeds. Additional supports / rollers were provided.
- 5. Problems with advancing of gypsum stacker conveyor. This was due to settlement and ineffective hydraulic jacks. The portable hydraulic jacks were replaced with self mounted hydraulic jacking system.
- 6. Conventional problems associated with belt conveyors and heavy equipment. Traditional periodical maintenance and preventive maintenance methods were adopted.

8. Reliability and Capacity Improvement Jobs Carried Out

In additional to troubleshooting, some modifications were carried out, in order to improve the reliability of the stacking system as well as to handle the higher quantity of filter cake generated at higher plant loads. Some of the major modifications carried out are described below.

- 1. Replacement of original conveyor belts (multiply polyamide +polyester + rubber lining) with oil resistant ROS grade with higher strength EP 630/3.
- 2. The speed of the conveyors was increased to 1.6 m/sec to handle higher quantity of filter cake.
- 3. The gear boxes and motors for some equipment were replaced with higher capacity.
- 4. The diameter of the head drums was increased from 500 to 560 mm for improved reliability.
- 5. The thrower device was replaced with the one of higher capacity.
- 6. Automatic hammering devices were installed for the critical conveyor discharge chutes to minimize the choking tendency.
- 7. Chutes were provided with motorized ram for the two diversion chutes for ease of operation / quick action.
- 8. Rubber flaps were installed for the belt filter outlet chutes in addition to the hammering devices to minimize the build up of material, which is found very effective.

9. Advantages and Limitations of Dry Stacking

9.1 Advantages

- Transportation of gypsum in dry mode is simpler when compared to handling of gypsum slurry, which poses problems of pumping and plugging of pipelines.
- The initial cost for dry stacking is less compared to wet stacking, as the lining of the ponds and the complex under drain systems are not required.
- The problems of percolation and flooding of acidic water from the dry gypsum stacks are less in most of the cases, and virtually absent if the stacks are located in dry climates.
- The down time of phosphoric acid plant will be lower in case of dry stacking, if suitable provisions are made for diversion.
- The specific consumption of water will also be lower, which is most important factor, where water resources are limited.

9.2 Limitations

- Hemihydrate cake becomes hardened if stored for few days, and requires to be shifted at the earliest from the temporary storage to permanent storage.
- Dust problems are encountered during movement of vehicles and during windy days, which requires spraying of water.
- The operational flexibility with respect to water balance in phosphoric acid plant is less in dry stacking, especially during routine washings and plant shut downs, particularly when wet rock phosphate is used as feed.
- No recovery of P2O5 from filter cake is possible as in some of the wet gypsum ponds.

10. Conclusion

On the basis of the operating experience for five years, the overall performance of dry stacking of hemihydrate calcium sulphate has been good as evidenced by the fact that the down time of phosphoric acid plant due to Gypsum handling system was 2% only, though maintenance problems with the equipment have been faced.

The selection of dry or wet stacking for any project is closely linked to geographical factors as well as project economics. In IJC's case, considering the limited water resources in Jordan, both factors have played an equally important role, and the experience confirms the appropriateness of the decisions made.

EXHIBIT 1



EXHIBIT -2



EXHIBIT -3





EXHIBIT -5



EXHIBIT - 6



Longitudinal view of the stack



Rear view of the stack at maximum height



EXHIBIT - 8



Long crack near the edge of the stack



Wide crack near the edge of the stack

OVERALL PROTECTION IN A PHOSPHORIC ACID PLANT (a)

Gerd L. Meier SGL ACOTEC GmbH D – 86405 Meitingen - Germany

Fertilizer plants are designed to operate continually at high yield. Unfortunately corrosion is limiting the success in the plant performance and is using up a large portion of your maintenance budget.

From the sulfur burning to the storage of the produced phosphoric acid, in each of the manufacturing steps the most economical and reliable material and the proper application is required to limit the corrosion attack and guarantee the maximum operating rate without interruption.

Servicing the fertilizer market for decades with continuous improvement of products and technologies in partnership with the worldwide leading engineering companies and owners of phosphoric acid plants has established SGL ACOTEC to be the competent partner for all question and problem solutions to optimize the corrosion protection from one source.

The product- and service portfolio covers all your major demands. Our "System-Approach" optimizes the material selection, the equipment supply and all interfaces within the complete process of phosphoric acid dealing with corrosive media. This applies for new plants, plant extensions as well as the all around service for service, maintenance and up-grades.

What are the major materials and products required to manage the corrosion problems in the phosphoric acid plant:

- > Fire resistant brick lining in the sulfur burning furnace
- > Acid proof brick lining of the sulfur absorption tower
- > Rubber lining and/or polymer coating and lining of the H_2SO_4 storage tank
- \succ H₂SO₄ dilution system
- Rubber lining and carbon brick lining in the attack tank
- Rubber lining, carbon brick lining, graphite evaporators and FRP interconnecting piping in the concentration stage
- Rubber lining of the phosphoric acid storage tanks and transport trucks, railroad cars and container ships
- ➢ Graphite or metal lined pumps are required in all production steps

Email: <u>Gerd.Meier@sglcarbon.de</u> Received on: 28/08/2002 For the "White Acid" production you require:

- Rubber and carbon brick lined tanks with special metal heaters for an additional treatment to upgrade H₃PO₄
- Graphite and/or noble metal heat exchangers and FRP columns are used in the H₃PO₄ reconcentration
- The HF de-fluorination process requires graphite heat exchanger, PTFE-lined column, FRP duolaminate column and vessels
- Piping, armatures in rubber lined, FRP duo-laminate and pumps in graphite or noble metal combine all production steps
- > The concrete floors of all plant sections have to be protected with synthetic resin coating and tiles.



REFRACTORY MATERIALS

In the first production process sulfur is burned to sulfur dioxide in the furnaces. At temperatures of up to 1500°C it is necessary to apply special refractory materials to protect the furnace shell of carbon steel. The fire proof bricks are formed to the exact shape and joint together with fire-resistant cements, fireproof mortars and fibers. Manufacturers and operators of sulfuric acid plants worldwide require a reliable and competent partner in everything to do with the planning and implementation of complex and efficient schemes for optimum protection.

ACID PROOF BRICK LINING

The drying and absorption towers of sulfuric acid plants require an efficient protection against corrosion. The first measures involves applying a sealing layer to the steel jacket. SGL uses special developed rubber sheeting like the quality BS or thermoplastic polymers ([®]Repanol) for this purpose, depending on individual requirements. The sealing layer has to be protected from the effect of heat, direct contact with acid and also mechanical stresses. The towers are completely lined with acid-proof ceramic bricks, often several layers thick, and the accompanying laying and jointing materials are based on silicate cement such as [®]Keranol. The aim here is to develop individual solutions where the key factor are life-time and cost-efficiency.

HIGH PERFORMANCE SELF-SUPPORTING DOMES

SGL has been very successfully using self-supporting domes in the packed towers of sulfuric acid plants. The use of supporting grid with its acid-proof ceramic dome bricks makes it possible to dispense with any form of supporting pillar structure. This construction material offers two advantages:

- > it allows a substantial cost-saving on supporting elements
- the efficiency of the plant is markedly improved because the gas flow is no longer impaired by the supporting pillars.

The ability of the gas stream to permeate the tower packing is aided by the special construction of the bricks. The free passage through the SGL self-supporting domes is at least 55 percent, from diameters larger 7 meters even 60 percent.

SGL supply a comprehensive performance package for all diameters from 0,5 to 10 m:

- early comprehensive consultation
- detail planning and design
- structural analyses including all construction drawings
- supporting framework for the construction of the dome
- if requested, turn-key installation of the dome by qualified SGL fitters or installation supervision



Self-supporting dome

SULFURIC ACID DILUTION UNITS

For some processes in the fertilizer plant the highly concentrated sulfuric acid has to be diluted to the required concentration. SGL offers skid mounted units

- with plate, block or shell and tube heat exchangers and pumps in [®]DIABON, proven resistance to corrosion and high temperature
- > manual, semi-automatic or fully automatic controlled and monitored units
- > all flow rates and concentration possible
- ➢ single or multi-stages units



PLASTIC COMPONENTS

In sulfuric and phosphoric acid plants the key components (evaporator, filter, cooling towers, storage tanks, gas ducts and piping systems) are subjected to many different corrosion-related parameters. The chemical, mechanical and thermal stresses differ from case to case and need special adapted concepts for each situation in order to protect the plants.

SGL uses high-quality ®KERAVERIN or KERA components very successfully to give effective and long-lasting protection against corrosion. KERAVERIN is a fiber glass-reinforced plastic (FRP) with a thermoplastic lining. Equipment fabricated in KERA, which is a thermoset material, is based on phenolic or furan resins reinforced by glass or carbon fibers.

The advantages are self-evident.

- The plastic components have excellent chemical, thermal and mechanical resistance.
- The low weight makes for ease of handling while also ensuring simple, reliable processing.
- Potential weak points such as flanged joints on lined metal pipelines can be eliminated or greatly reduced by using welding and laminating processes to join the plastic component systems.
- The high resistance to vacuums, based on the high-strength bond between the liner and FRP laminate substrate.
- \Rightarrow The result is a reduction in maintenance expenditure and high plant reliability. This ensures optimum cost-efficiency.

KERAVERIN can be supplied with following materials as internal liner:

• PVC, FEP, PVC/C, E-CTFE, PP, PFA, PVDF

Where advisable because of the prevailing conditions, SGL can supply a modified PTFE as an innovative lining material in a FRP composite. The result of continuous further development work, this product provides greatly improved protection against chemical and thermal stresses.





KERAVERIN reaction vessel

KERAVERIN piping

LICUFLON columns and vessels are used successful for absorption, extraction, rectification and distillation of products containing hydrofluoric acid or highly concentrated, hot phosphoric acid. The load bearing steel parts are lined with a 4 mm skived PTFE sheet in loose lining technique.

The butt welding of the PTFE sheet without any thermoplastic welding material allows the production of any diameter up to 3,5 m. Column internals in DIABON, like trays, liquid distribution, feed pipes, support grids, etc. are designed to meet the process guarantee. LICUFLON column are in operation up to 250 °C.



LICUFLON columns

FLOOR COATING SYSTEMS

Effective anti-corrosion and surface protection measures in sulfuric and phosphoric acid plants are by no means confined to original plant segments. Durable floor coating in the whole area of the plant is absolutely essential for safety and also environmental protection. A suitable coating can prevent undesired penetration by chemical substances into the concrete floor slab, as well as any resulting corrosive attack on the concrete or pollution of the groundwater.

In our own facilities in several continents, SGL uses the latest technology to produce a range of synthetic resin coatings ideally suited to treating the floors of acid plants. The synthetic resins possess equally high chemical and thermal resistance properties. They can be applied in the form of spreading, casting, brushing or spraying compounds. The synthetic resins can be used to coat not only the floor area but also any walls likely to be exposed to corrosive stresses. In addition, the coatings – those applied with ®CEILCOTE, ®KERAPLAN or ®KERACID, for example – can function as a sealing layer in any specially installed secondary containment systems or run-off trenches constructed from acid-proof ceramic or carbon tiling that is bonded and jointed with synthetic resin cements.

PROPERTIES OF SYNTHETIC RESIN MATERIALS (COATINGS)

- Chemical resistance and impermeability
- Resistance to atmospheric corrosion and ageing stability
- Mechanical and/or thermal tolerance under load
- Crack-bridging capabilities
- Physiologically non-hazardous
- Electrically discharging
- Antiskid properties and facility of decontamination
- Visually pleasing appearance







Polymer Lining in Concrete Settling Tanks

SURFACE PROTECTION WITH RUBBER LININGS

When phosphoric acid is produced both steel and concrete surfaces need to be properly shielded from the influence of the aggressive media.

SGL has been using a number of special rubber lining systems to cope with problematic process situations. These linings are notable for their high effectiveness and reliability, as well as long-lasting resistance to the various influences. These features make them highly cost-efficient.

As a result, the lined segments are completely stable to vacuums and elevated temperatures up to a constant 125°C for the rubber material as well as for the bonding system.

The crucial factor is the use of a bonding system tailored to the individual lining material. This is the only way that an optimum bond can be created between the rubber lining and the surface of the respective segment of the plant, or between two extruded rubber sheets.

Different rubber linings, both hard and soft, are required. SGL's [®]VULCOFERRAN or [®]KERABUTYL products can be used wherever the steel and/or concrete surfaces of vessels and pipelines need to be protected effectively against corrosion. An additional soft rubber layer can be applied to the first rubber sheeting as protection against abrasive wear.

All the SGL rubber sheet manufacturing sites extrude the rubber sheet in the roller head machine to the standard thickness from 3 to 6 mm. The most successful quality grades in the phosphoric acid industry are the soft rubber in bromobutyl VULCOFERRAN 2206 and KERABUTYL BS and the hard rubber VULCOFERRAN 2194 since 1982. In this period more than 200.000 square meter are successfully applied in this industry. These qualities are suitable to be applied on site or in work shop. The lining is vacuum resistant and the rubber is chemically bonded to steel.



Roller head extruder for rubber sheets

Lining of a storage tank with rubber sheets and carbon bricks

CARBON BRICK LININGS

In the reactors and other vessels used in phosphoric acid plants, the application of a rubber lining is not the only measure needed for lasting and effective protection against corrosion. Other measures Carbon brick linings may be installed to prevent abrasion of rubber linings, along with the appropriate laying and jointing materials based on synthetic resins – especially furan resins. Carbon bricks give the necessary protection against abrasive wear, and at the same time resist the hydrofluoric acid occurring in the production process.

SGL has an ideal basis for ensuring the highest quality standards: to be the largest carbon and graphite manufacturer world-wide.



Rubber lined tank with brick protection

DIABON HEAT EXCHANGER

Heat exchangers in phosphoric acid plants perform important functions as heaters, coolers, condensers, evaporators or absorbers in various production phases, depending on the circumstances. This DIABON heat exchangers are available in shell and tube, block or plate design. Special design features are combined with materials with high resistance to virtually all organic and inorganic substances. Together they ensure optimum operational reliability, high heat transfer levels and great cost-efficiency.

SGL heat exchangers have synthetic resin-impregnated graphite tubes in standard quality ([®]DIABON-.NS1) or alternatively wrapped with highly pre-tensioned fibers on the outside surface (DIABON-HF1). This is a design feature that not only brings a marked improvement in operational reliability but also noticeably expands the range of use of shell-and-tube heat exchangers in critical applications. The reinforcement does not impair resistance to corrosion because the chemical resistance of the reinforcement is identical to that of synthetic resin-impregnated graphite. Owing to the extreme elasticity of the carbon fibers the tension on the reinforcement is retained even under sharply fluctuating load or stress surges without material fatigue. As this reinforcement with carbon fibers markedly improves the mechanical properties of graphite components and the reliability, most evaporators in the phosphoric acid plants are now supplied by SGL with carbon fiber-reinforced tubes and tube sheets.





Fracture behavior of glass-, standard graphite tube and fiber reinforced graphite tube



DIABON P2O5 evaporator

DIABON plate heat exchanger

PUMPS

Pumps are imperative to circulate and transport the aggressive media sulfuric and phosphoric acids. Corrosion resistance and reliability are the main features required. DIABON pumps meet these demands in addition to temperature and dimensional stability. They enable pumping liquids with volumetric flow rates from 5 to 2000 m³/h and discharge heads up to 100m. SGL pumps are fitted with single or double mechanical seals. The first mag-driven pumps are tested and in service.

Alternative to DIABON pumps SGL can also offer exotic metal lined pumps in Titanium, Hastelloy, Zirconium or Nickel.

The advantages are:

- most competitive manufacturing process
- materials microstructure are more homogeneous and have higher strength with better corrosion resistance
- ➢ full vacuum design
- ➢ short delivery time



DIABON pump

SUMMARY

SGL know-how, experience and competence in corrosion resistant materials, process equipment and services is your benefit in the phosphoric acid plant. We offer a complete system from a single source – from optimized material selection through interface management, production, delivery and installation right up to the warranty. You can save yourself time and trouble in the future by taking advantage of a partner, who offers comprehensive, system-based range of products and services.

EXPANSION OF THE RICHARDS BAY DIHYDRATE PHOSPHORIC ACID PRODUCTION COMPLEX (a)

K. Kirsten, Foskor Limited, Republic of South Africa J. Tytgat*, SNC-Lavalin Europe, Belgium T. Theys, Prayon, Belgium

5.2 Production data

The phosphoric acid plant was mechanically completed during Q3 2002. Full weak acid production was progressively started on one belt filter at a time until all three were operating smoothly in parallel. The concentration unit and the merchant acid production were put into service shortly after weak acid production started.

The Performance Test (PT) was successfully finalized early November 2002.

Table 3: Production capacity

	Average obtained capacity during PT	Guaranteed capacity
Weak acid	1166 MTD P ₂ O ₅	1100 MTD P ₂ O ₅
Concentration units	2350 MTD	2195 MTD (*)
	Evaporated matter	Evaporated matter

(*) Due to recirculation of phosphoric acid slurry from the concentrated clarifier, the average concentration of weak acid, fed to the concentration unit, is increased from 27 up to about 29,4% P_2O_5 . However, the design capacity is based on the quantity of matters to be evaporated based on the nominal capacity of 1,100 MTD starting from 26% P_2O_5 .

Table 4: Analysis of Phosphoric Acid

Average Chemical Analysis during the Performance Test: % weight on solids free basis

	Weak Acid ex-filters	Merchant grade acid ex final settler
P ₂ O ₅	27.05	54.47
CaO	0.30	-
SO ₃	1.75	2.22
F	1.65	0.27
Solids	-	0.25 (1)
Temperature	About 65 °C	43.8 °C

(1) While using flocculant in the clarifiers

The concentrated acid leaving the export tanks is per the merchant acid quality standards imposed by Foskor limited:

- Min 54% P_2O_5
- Max 0.6% solids
- Max 45 °C (or max 35 °C depending on shipping requirements)
DEVELOPMENT OF A METHOD OF A COMPLEX ESTIMATION AND CLASSIFICATION OF BIOLOGICAL AND ECOLOGICAL HAZARDS ASSOCIATED WITH PHOSPHATE ROCK AND FERTILIZERS.

Boris Levin, Research Institute for Fertilizers, PhosAgro, Moscow, Russia (a)

The method of estimating biological and ecological risks associated with phosphate rock is based upon the components transmitted through the circuits up to the foodstuffs. The method allows classification of the hazards associated with the commercial grades of phosphate rock and their products. The method takes into account the complex ecological hazards of phosphate rock and its products on the basis of two cycles of pollution: in agricultural soils (1) and surface and ground waters (2) (Fig.1). In this respect, the danger of the second cycle of pollution is incomparably higher, than that of the first cycle due to stricter sanitary requirements. The offered method is based on fundamental data of toxicity of chemical elements accumulated by biochemistry, agrochemistry and medicine, and is open for new inquiry.



There are two basic reasons for the urgency to derive a complex and accurate estimation of ecological and biodanger of phosphate rock and its products:

1. The leading manufacturers in the advanced countries are orientating towards a high standard in the field of environment and safety for the human health.

2. An enormous database on toxicity of chemical elements and compounds has been accumulated that allows the creation of a complex method of relative classification of products, intermediates and raw materials by criteria of ecological and biodanger.

Additionally, there is extensive practical experience in the field of processing phosphate rock by acid methods developed by the foremost engineering companies(Prayon, Rhone Poulenc / Krebs / Speichim, IMI, Norsk Hydro, etc.). Transmission of the basic and impurity components during sulphuric acid decomposition of the phosphate rock has been investigated in detail (table 1). The share of such acid decomposition is more than 85 % of the world production of phosphorus fertilizers. The lack of data concerning impurity distribution for nitric and hydrochloric acid decomposition can be easily compensated by analytical researches.

The method of the estimation of the ecological and biodanger of phosphate rock should correspond to the below requirements:

- 1. To integrate approach and to take into account the influence of all toxic elements;
- 2. To be based on the authentic and representative data on chemical structure of phosphate rock, toxicity of elements, extreme allowable concentration in ground, water, foodstuffs, and parameters of distribution of elements during processing;
- 3. To have heuristic properties, that can be revamped and be adaptive to new data and knowledge;
- 4. To be simple and verifiable, i.e. available for checking the relative biodanger of phosphorus fertilizers by accessible methods.

Recently there is too much attention concentrated on the problem with cadmium. Certainly, this problem have an objective basis, but it results in an unjustified increase of risk from other toxic elements and put out of focus other elements, which are not less dangerous.

The analysis of the special literature, devoted to questions of human resistance against influence of hazardous elements, allows allocation of groups of chemical elements on the degree of toxicity upon man. Practically all specified toxically elements are present in commercial grades of phosphate rock and appropriately in the products after their processing.

Table 1. Distribution	indexes for	impurities in	h by-products	and	finished	products	at sulphuric	acid
decomposition.								

Elements	Apatite	Phosphorite
Cd	~60	50-80
Hg	~20	~10
As	~80	70-90
Pb	20-30	10-20
Sr	~3	-
Cu	~40	~60
Ni	~75	~50
Zn	30-35	50-86
Co	~60	-
Cr	~80	~90
v	~30	-
Mn	~90	-
F	~55	40-60

Further these elements together with useful nutritious components (N, P2O5, K2O) pass into the soil solutions and are distributed on a circuit: partially acquired by plants, partially immobilized by organic part of the soil, partially in ground and superficial waters. Thus, it is necessary to a complex estimation of the biodanger phosphate raw material to reflect real depots - stores that are in contact with alive organisms. For the most widespread types of fertilizers (MAP, DAP, NPK) the key feature is the presence of toxic elements in their active form as against chemically or mechanically immobilized in phosphate rock.

The algorithm of an estimation of various types of phosphorus fertilizers toxicity (from ordinary up to complex), after taking into account the entry of toxic elements not only from phosphate rock is given in Fig. 2.

Fig. 2. The algorithm of the estimation of various type phosphorus fertilizers toxicity.



To summarize the source of pollution of fertilizers it is necessary to include sulphur raw material (pyrites, the natural sulphur), potassium raw material (potassium chloride, potassium sulphate), and nitric raw material and intermediate products (ammonium sulphate, ammonia, nitric acid). Thus the integrated index of the phosphorus fertilizers toxicity is as follows:

I₂ = (I sulphur + I phosphate + I MOP, SOP + I N) raw material

In this work a limiting case is considered, when the integrated factor is determined by an extreme index of phosphate rock toxicity. The legitimacy of such approach is proven by that in the general case $I_D \ge I_{phosphate \ raw \ material}$ in which both requirements: cleanliness and the safety of the phosphate raw material, should be set by rigid, limiting criterion.

Applying the general approach to an estimate for the biodanger of raw material, in the case of phosphate raw material the following criteria are used:

1. Pollution cycle: soil – agricultural products – foodstuffs.

$I_{p.r.m} = \frac{m \sum x_i C_i / MCL_{i \text{ soil}}}{C P_2 O_5}$

m – input indices of phosphate raw material for fertilizer production, t/t;

Ci – concentration of i-element in phosphate rock, mg/kg;

xi – distribution index for impurities in by-products and finished products (table 1);

 $\mathbf{\tilde{N}}$ **P2O5** – concentration of the P2O5 component in phosphate rock;

MCL*i soil* – maximum concentration level of i-element in soil, mg/kg;

2. Pollution cycle: soil – ground water – surface water – drinking water.

$$I_{p.r.m.} = \frac{m \Sigma x_i C_i / MCL_{i water}}{C P_2 O_5}$$

MCLi water - maximum concentration level of i-element in water, mg/kg.

The data used are given in the Table 2, or are taken from the open publications and certificates of the companies - producers of the commercial grades of phosphate raw material. The overall results are shown in Fig. 3.

The calculation of the integral index of biological toxicity helps make up 3 groups of phosphate rocks:

- 1. Phosphate rocks with a low level of toxicity. They include apatite concentrates from Russia (JSC Apatit, Kovdor benefication plant), South Africa (Phalaborwa). This group is regarded as the elite. To process these rocks various well-known technologies and equipment can be used without any restrictions or additional removal technologies for harmful impurities. The value of the rocks is increasing today as a result of tighter environmental control.
- 2. Phosphate rocks with a moderate toxicity level. They include phosphorites of medium grade from Jordan, Morocco and USA. To meet environmental requirements in countries with strict regulations these rocks should be treated additionally to reduce the concentration of contaminants. Application of removal technologies involves a rise in operating costs.
- 3. Phosphate rocks with a high level of toxicity. These are low-grade phosphates from Tunisia, Algeria and high-grade rocks from Togo, Senegal, and Morocco. Fertilizers traditionally produced fall short of the safety requirements in economically advanced countries. To meet these environmental standards the purification of finished products is needed with expensive technologies employed (ion exchange, ion flotation, etc.)

		Mor	оссо	Toqo	Senegal	Jo	rdan	Tunisia	Algeria	USA	Israel	Syria	Russ	ia	South Africa
No	Element	Bu Cræ	khouribga	Benin	Taiba	Eshidia	El Hassa	Cafsa	Djebel Onk	Florida	Nahai Zin	Kneifiss	Kola	Kovdor	Phalaborva
1	Cđ	34,1	12,8	85,8	73,0	5,5	5,4	34,1	15,3	9,4	23,0	13,0	0,1	0,8	1,8
2	Hg	0,1	0,1	-	0,3	0,0	0,2	0,1	2,2	0,1	0,5	0,1	0,0	0,1	0,2
3	As	5,8	8,8	-	3,0	6,0	12,2	16,7	12,5	8,7	4,0	20,0	0,6	2,7	9,3
4	РЬ	1,0	2,4	-	4,8	3,4	6,5	7,7	11,0	18,0	4,0	6,5	1,3	6,1	11,8
5	Cr	130,0	300,0	30,0	70,0	75,0	80,0	81,0	200,0	91,0	60,0	-	2,5	30,0	8,0
6	Sb	-	-	-	-	-	-	-	12,0	-	-	-	1,5	2,7	-
7	Ba	-	-	-	100,0	-	-	-	-	-	-	-	850,0	300,0	-
8	F*	4,2	4,0	3,8	3,7	3,8	3,8	4,0	3,5	3,8	3,5	3,5	3,1	1,0	7,4
9	Cu	14,0	43,0	-	60,0	21,0	20,0	23,5	19,8	9,0	30,0	20,0	46,0	20,0	120,0
10	Ni	40,0	41,0	-	40,0	11,0	35,3	28,0	18,6	30,0	35,0	59,0	7,9	21,0	27,3
11	Zn	90,0	230,0	-	-	150,0	230,0	260,0	185,0	95,0	400,0	330,0	26,0	50,0	14,0
12	Co	-	-	-	1,0	6,0	15,5	18,0	17,0	-	-	8,0	4,0	5,0	-
13	Mn	200,0	-	200,0	20,0	11,6	60,0	36,0	25,0	290,0	6,0	6,5	340,0	600,0	200,0
14	Sr	-	150,0	30,0	460,0	-	2000,0	1925,0	2022,0	-	2500,0	1900,0	28000,0	2400,0	5000,0
15	v	122,0	21,0	-	140,0	60,0	55,7	62,0	45,0	70,0	130,0	-	100,0	36,0	12,7

Table 2. Concentrations of hazardous elements in phosphate rocks taken into account, ppm

* concentration in %

Fig. 3. 3D - Diagram of relational classification of biological and ecological hazard of phosphate rock. 2nd pollutions cycle.



Radioactivity is an additional factor, which helps to distinguish the 1st group from the other two. All types of apatite concentrates have a low radioactivity level, by far lower than the levels required by radiological safety standards. By contrast, phosphorite concentrates are enriched with radioactive isotopes and have a high radioactivity level. Russia has enough reserves of high-quality contaminant-free apatite concentrates to provide regions under high environmental requirements with eco-friendly

phosphate rocks and apatite concentrate-based finished products. Apatite concentrates from Russia and South Africa have the lowest integral toxicity and radioactivity levels of all phosphate raw materials. Phosphate fertilizers produced meet the strictest environmental and sanitary demands without any additional purification phases and operating costs involved. The value of apatite concentrates and their fertilizer derivatives becomes far more evident despite their small share in world production and trade – about 13%. Another option for the use of apatite concentrates and environmentally clean phosphate fertilizer is an increase in capital costs to equip operating plants with additional phases for contaminant removal and a consequent rise in operating costs.

References:

- 1. Harmful chemical substances. prof V.A. Filov, Saint Petersburg «Chemistry», 1988.
- 2. Rikhvanov L.P. The contents of heavy metals in soils. 1993.
- 3. Fomin G.S. Soil quality assurance and ecological safety under the international standards, VNIIstandart, 2001.
- 4. Heavy metals in system soil plants fertilizer. Ovcharenko M.M. Moscow 1997.
- 5. Yagodin B.A., Zhukov Y.P., Kobzarenko V.I. Agrochemistry. M.: Kolos, 2002.
- 6. Kabata-Pendias A., Pendias H Microelements in soils and plants. 1989.
- 7. Chernykh N.A., Milaschenko N.Z., Ladogin V.F. Ecological and toxically aspects of pollution soils by heavy metals, M.: Agroconsult, 1999.
- 8. Kazak B.G., Angelov A.I., Kiperman Y.A. Ecological and geochemical estimation of phosphate raw material and fertilizers, Gornyi vestnik, 1996, spec. release 76.
- 9. The control of chemical and biological parameters of an environment. prof Isaev L.K., Saint Petersburg: Crismas+, 1998
- Steven J. Van Kauwenburgh. Cadmium and other potential hazards. Fertilizer International, 380, 51, 2001
- 11. Estimating risk from contaminants contained in agricultural fertilizers /Draft Report/ Office of Solid Waste U.S. Environmental Protection Agency and Center for Environmental Analysis Research Triangle Institute Research, 1999
- 12. F.H. Oosterhuis, Institute for Environmental Studies (IVM), Vrije Universiteit, Amsterdam, F.M. Brouwer, Agricultural Economics Research Institute (LEI), The Hague,H.J. Wijnants, DHV Environment and Infrastructure (DHV-MI), Amersfoort. A possible EU wide charge on cadmium in phosphate fertilizers: Economic and environmental implications. Final report to the European Commission, 2000.
- 13. A.M. Kharikov, V.V. Smetana, Heavy metals and Radioactivity in phosphate fertilizers: short term detrimental effects. Technical conference. New Orleans, USA, 2000, 426.
- 14. John J. Mortvedt, James D. Beaton, 6 heavy metal and radionuclide contaminant in phosphate fertilizers, Scientific Committee On Problems of the Environment, V. 54, 1996.
- 15. Williams C.H., David D.J., The accumulation in soil of cadmium residues from phosphate fertilizers and their effect on the cadmium content of plants, Soil Sci., 121, 86, 1976.
- 16. Andersson A., Hahlin M., Cadmium effect from fertilization in field experiments, Swed. J. Agric. Res., 11, 3, 1998.
- 17. R.M. Vermeul, C.G. H. van Ede, P.C.M. Mutsaers, N.W. Kolmeijr Hydro Agri Rotterdam, Netherlands, IFA Technical conference, Jordan, 1994.
- 18. I.J. Lin, M. Shorr, A challenge for the phosphate industry: Cd removal, Phosphorus & Potassium 208, 27, 1997.
- 19. J Mastrantoni, C. Nerantzakis, The cycling of Cd, coming from fertilizers in the Greek environment. 2000.
- 20. Halase K.G., Toshinskii V.I, Loboiko A.Y., Chemical industry, 1993, №12, 25 (625)

- Вредные химические вещества. Под редакцией проф. В.А. Филова, Ленинград «Химия», 1988.
- 2. Рихванов Л.П. Содержание тяжелых металлов в почвах. 1993.
- Фомин Г.С. Почва контроль качества и экологической безопасности по международным стандартам, ВНИИстандарт, 2001.
- Тяжелые металлы в системе почва растения удобрение. Под редакцией Овчаренко М.М. Москва 1997.
- 5. Ягодин Б.А., Жуков Ю.П., Кобзаренко В.И. Агрохимия. М.: Колос, 2002.
- Кабата-Пендиас А., Пендиас Х. Микроэлементы в почвах и растениях. М.: Мир, 1989, 439с.
- Черных Н.А., Милащенко Н.З., Ладогин В.Ф. Экотоксикологические аспекты загрязнения почв тяжелыми металлами, М.: Агроконсалт, 1999.
- Казак В.Г., Ангелов А.И., Киперман Ю.А. Эколого-геохимическая оценка фосфатного сырья и удобрений, Горный вестник, 1996, спец. выпуск. с. 76.
- Контроль химических и биологических параметров окружающей среды под ред. проф. Л.К. Исаева, СПб.: Крисмас+, 1998
- Steven J. Van Kauwenburgh. Cadmium and other potential hazards. Fertilizer International, 380, 51, 2001
- Estimating risk from contaminants contained in agricultural fertilizers /Draft Report/ Office of Solid Waste U.S. Environmental Protection Agency and Center for Environmental Analysis Research Triangle Institute Research, 1999
- 12. F.H. Oosterhuis, Institute for Environmental Studies (IVM), Vrije Universiteit, Amsterdam, F.M. Brouwer, Agricultural Economics Research Institute (LEI), The Hague, H.J. Wijnants, DHV Environment and Infrastructure (DHV-MI), Amersfoort. A possible EU wide charge on cadmium in phosphate fertilizers:

Economic and environmental implications. Final report to the European Commission, 2000.

- A.M. Kharikov, V.V. Smetana, Heavy metals and Radioactivity in phosphate fertilizers: short term detrimental effects. Technical conference. New Orleans, USA, 2000, 426.
- John J. Mortvedt, James D. Beaton, 6 heavy metal and radionuclide contaminant in phosphate fertilizers, Scientific Committee On Problems of the Environment, V. 54, 1996.
- Williams C.H., David D.J., The accumulation in soil of cadmium residues from phosphate fertilizers and their effect on the cadmium content of plants, Soil Sci., 121, 86, 1976.
- Andersson A., Hahlin M., Cadmium effect from fertilization in field experiments, Swed. J. Agric. Res., 11, 3, 1998.
- R.M. Vermeul, C.G. H. van Ede, P.C.M. Mutsaers, N.W. Kolmeijr Hydro Agri Rotterdam, Netherlands, IFA Technical conference, Jordan, 1994.
- Lin, M. Shorr, A challenge for the phosphate industry: Cd removal, Phosphorus & Potassium 208, 27, 1997.
- J Mastrantoni, C. Nerantzakis, The cycling of Cd, coming from fertilizers in the Greek environment. 2000.
- К.Г. Халасе, В.И. Тошинский, А.Я. Лобойко, Химическая промышленность, 1993, №12, с. 25 (625)

Presentation: Report on a workshop on ammonium nitrate organized by the European Commission at Ispra, Italy

Hans van Balken, EFMA, Belgium (Presented by K.D. Shah)







AMMONIUM NITRATE WORKSHOP

> ORGANISED BY EU COMMISSION FOLLOWING TOULOUSE > EXPERTS FROM MEMBER STATES AND

EFMA INVITED TO GIVE FOUR PAPERS



ISPRA WORKSHOP ON AN SAFETY PRESENTATIONS

- > Ammonium nitrate: Production & Economic Importance. Properties & Potential Hazards. K.Shah Terra Industries
- A review of Past Ammonium nitrate Accidents & Lessons Learned. D. Heather. FMA
- Ammonium Nitrate Safety Tests. J.Chys. Norsk Hydro
- Best Practices for Storage and Handling of Ammonium Nitrate Fertilizers. J.A.M. van Balken EFMA
- ≻Main points arising from the EFMA papers

(http://mahbsrv.jrc.it/Proceeding.html)









EC FERTILIZER LAW CHANGES IN « REFONTE »

> SCOPE OF ART. ON STRAIGHT AN (>28%), WIDENED TO STRAIGHT AND COMPOUND

CHECKS ON DETONABILITY STILL OPTIONAL BUT WILL BECOME MANDATORY

>IT ADRESSES TO ISSUE OF TRACEABILITY

> STATUS CHANGE FROM DIRECTIVE INTO REGULATION



LEGISLATION: PRODUCTION AND STORAGE

 FIRST SEVESO DIRECTIVE ADOPTED IN 1982
 DESIGNED TO ADDRESS MAJOR HAZARDS FROM CHEMICAL INDUSTRY (SEVESO, FLIXBOROUGH)

REVISED A FEW TIMES, GOING THROUGH RADICAL

>AN WAS COVERED BY TWO ENTRIES



COMAH (SEVESO II) DIRECTIVE SCOPE

"Installation" shall mean a technical unit within an establishment in which <u>dangerous substances</u> are <u>produced</u>, used, handled or stored. It shall include all the equipment, structures, pipe work, machinery, jetties, <u>warehouses</u> or similar structures, floating or otherwise, necessary for the operation of the installations".



COMAH DIRECTIVE MAIN PROVISIONS

- >Identification of Establishments (art. 3)
- > General obligations of the Operator (art. 5)
- > Notification (art. 6)
- > Major accident prevention policy (art. 7)
- >Domino effects (art.8)
- > Safety reports (art. 9)
- Modification of installation, establishment, storage (art. 10)
- >Emergency plans (art. 11)
- >Land-use planning (art.12)
- > Public information (art.13)
- >Accident reporting (art. 14)
- >Competent Authority and Inspections (art. 16 & 18)0





COMAH (SEVESO II) DIRECTIVE AMMONIUM NITRATE NOTE 1

This applies to ammonium nitrate and ammonium nitrate compounds in with the nitrogen content as a result of the ammonium nitrate is more than 28% by weight (compounds other than referred to in note 2) and to aqueous ammonium nitrate solutions in which the concentration of ammonium nitrate is more than 90% by weight.

12



COMAH (SEVESO II) DIRECTIVE AMMONIUM NITRATE NOTE 1

This applies to ammonium nitrate and ammonium nitrate compounds in with the nitrogen content as a result of the ammonium nitrate is more than 28% by weight (compounds other than referred to in note 2) and to aqueous ammonium nitrate solutions in which the concentration of ammonium nitrate is more than 90% by weight.

12



COMAH (SEVESO II) DIRECTIVE AMMONIUM NITRATE NOTE 2

This applies to simple ammonium nitrate based fertilizers which comply with Directive 80/876/EEC and to composite fertilizers in which the nitrogen content as a result of the ammonium nitrate is more than 28% by weight. (a composite fertilizer contains ammonium nitrate with phosphate and/or potash)

13





Amendment 53

-15,75% in weight or less and unrestricted combustible materials,

and which are capable of self-sustaining decomposition according to the UN Trough Test (see United Nations Recommendations on the Transport of Dangerous Goods: Manual of Tests and Criteria, Part III, sub-section 38.2). 15

20/11/2002



20/11/2002

Amendment 53 ANNEX ,POINT 3 A (NEW) Annex I, Part I, Notes 1 and 2 (Directive 96/82/EC) Ammonium nitrate (1250 / 5000): fertiliser grade

This applies to straight ammonium nitrate-based fertilisers and to ammonium nitrate-based compound/composite fertilisers in which the nitrogen content as a result of ammonium nitrate is

-more than 24,5% in weight, except for mixtures of ammonium nitrate with dolomite, limestone and/or calcium carbonate with a purity of at least 90%,

-more than 15,75% in weight for mixtures of ammonium nitrate and ammonium sulpi

-more than 28% in weight for mixtures of ammonium nitrate with dolomite, limestone and/or calcium carbonate with a purity of at least 90%,

and which fulfil the requirements of Annex II of Directive 80/876/EEC (as amended and updated).

20/11/2002



Amendment 53 ANNEX ,POINT 3 A (NEW) Annex I, Part I, Notes 1 and 2 (Directive 96/82/EC)

Ammonium nitrate (350/2500): technical grade

This applies to ammonium nitrate and preparations of ammonium nitrate in which the nitrogen content as a result of the ammonium nitrate is

-between 24,5% and 28% in weight, and which contain not more than 0.4% combustible substances,

-more than 28% in weight, and which contain not more than 0.2% combustible substances.

-aqueous ammonium nitrate solutions in which the concentration of ammonium nitrate is more than 80% by weight.

20/11/2002



Amendment 53 ANNEX ,POINT 3 A (NEW) Annex I, Part I, Notes 1 and 2 (Directive 96/82/EC)

Ammonium nitrate (10/50): "off-specs" material and fertilisers not fulfilling the detonation test

This applies to -material rejected during the manufacturing process and to ammonium nitrate and preparations of ammonium nitrate, straight ammonium nitrate-based fertilisers and ammonium nitrate-based compound/composite fertilisers referred to in Notes 2 and 3, that are being or have been returned from the final user to a manufacturer, temporary storage or reprocessing plant for reworking, recycling or treatment for safe use, because they no longer comply with the specifications of Notes 2 and 3.

-fertilisers referred to in Notes 1 and 2 which do not fulfil the requirements of Annex II of Directive 80/876/EEC (as amended and updated).

20/11/2002



18 1942 Am pitrate containing not more than 0.2%

efma	1942	Ammonium nitrate, containing not more than 0.2% combustible substances, including any organic substance calculated as carbon, to the exclusion of any other substance.	5.1.0	(Ye
<i>`</i>	2067	Ammonium nitrate fertilisers: uniform non seyfregating initrates of ammonium nitrate with added priater which is inorganic and chemically inert (overdis ammonium nitrate, containing not less than 90% of combutition material (including organic material calculated ar carbon), or containing less than 90% but nore than 70% of ammonium nitrate and not more than 70% of combustible material.	5.1.0 IG	
	2068	Ammonium nitrate fertilisers: uniform hon segregating motures of anmonium nitrate with calcium carbonate and/or dolomite, containing more than 80% but less than 90% of ammonium nitrate and no more than 0.4% of total combustible material.	5.1.0.	
	2069	Ammonium nitrate fertilisers: uniform o on segregating motures of animonium nitrate/ammonium sulphate containing more than 45% but not more than 70% of ammonium nitrate and not more than 0.1% of total combustible material.	5.1.0.	
	2070	Ammonium mittels fortilisers uniform non-engregating mistures of introgen phosphate or infrogen potash types or complete fortilisers of introgen phosphate)otash type, containing hore than 70% of ammonium nitrate and not more than this of total added combustible material or containing not more than 45% of ammonium nitrate with unrestricted combustible material.	5.1.0	
28/09/1999 9:1	420/11/20	002		21

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fma	-	
UN Number	Description	Class Division
222	Ammonium nitrate, containing more than 0.2% combustible substances, including any organic substance calculated as carbon, to the exclusion of any other substance. explosive	1.1.2. res
223	Ammonium nitrate fertilisers which are more liable to explode than ammonium nitrate, containing more than 0.2% combustble substances, including any organic substance calculated as carbon, to the exclusion of any	1.1.2.





RATIONALISATION IN DEC. 2000

TOO MANY UN NUMBERS CONCERNING AMMONIUM NITRATE (MORE THAN 10)

> CONFUSING AS SEVERAL ENTRIES WITH SAME SHIPPING NAME

6

23

COMPOSITION BASIS RESTRICTED

COMPOSITIONS NOT REFLECTING CURRENT PRODUCT RANGE



RATIONALISATION IN DEC. 2000

> TOO MANY UN NUMBERS CONCERNING AMMONIUM NITRATE (MORE THAN 10)

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>COMPOSITION BASIS RESTRICTED

> COMPOSITIONS NOT REFLECTING CURRENT PRODUCT RANGE

23





UN Name and No description		Class or Divisi an	Subsidar y risk	UN packin g group	Special Provision s	Lizaited quantitie s		Special Provision 1	Portable tank intructio n	Portable tank Special Provision
more than 0.2 total combustil material, inclu any organic substance, calculated as carbon to the	AMMONIUM NITRATE with	5.1	<u> </u>	ш	306	1 KG	P002 IBC08			
	substance, calculated as carbon to the exclusion of any other added					b)	>90%; < >70%, < >80%, < carbonat	90% +ot 90% Ca-	her inor	
2067	AMMONIUM NITRATE BASED FERTILIZER	5.1		ш	106 306 307		(2067,20 >45%,<7		40	
2071	AMMONIUM NITRATE BASED FERTILIZER	9			186 193		>70%);<			







ISPRA WORKSHOP ON AN SAFETY MAIN POINTS EFMA PAPERS II

> Two main commercial products: HD: fertilizer application LD/HP: ANFO explosives

Ebili . Alti O explosives

- Products classified in UN scheme as oxidiser
 Potential hazards AN (based) well researched and understood
- LD AN significantly more sensitive to detonation
 EU: several criteria to ensure high level of safety (including detonation test for EC grade AN)
- (including detonation test for EC grade AN)
- History: major accidents <1950 due to unsafe practice. Practices abandoned, product not banned



ISPRA WORKSHOP ON AN SAFETY MAIN POINTS EFMA PAPERS I

- Safer practices >1950: far fewer accidents; extreme low number of fatal injuries.
- Effective criteria/tests in current legislation; need for further testing questionable.
- Detailled guidance for handling/storage available from Industry
- Review of EFMA guidance document will include
 Off-spec material
 - >Unforeseen causes

http://mahbsrv.jrc.it/Proceeding.html



EFMA STATEMENT OF AN SAFETY

"Although the cause of the explosion is not known yet and although EFMA is not in a position to validate or comment on the first official French investigation report, one fact is undisputed: ammonium nitrate fertilizer conforming with E.U. and French national legislation was NOT involved. The explosion rather involved material classified as off-spec., consisting of off-spec, low density/high porosity ammonium nitrate (technical grade), together with off-spec. high density/stabilised ammonium nitrate (fertilizer grade).

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Such off-spec. material which is not intended to enter the commercial supply chain as a straight nitrogen fertilizer needs to be clearly distinguished from ammonium nitrate fertilizer that is within specification".



ISPRA WORKSHOP ON AN SAFETY OUTCOME

EXISTING CATEGORIES FOR AN IN COMAH DIRECTIVE SHOULD BE REVIEWED WITH A VIEW TO PARTICULARLY INCLUDE « OFF-SPEC » MATERIAL

>INDUSTRY TO ISSUE GUIDANCE DOCUMENT ON « OFF-SPEC » MATERIAL

>INDUSTRY TO UP-DATE EFMA 1992 HANDBOOK TO INCLUDE:

- a) « OFF SPEC » MATERIAL
- b) UNFORESEEN CAUSES
- c) MULTI-STAKEHOLDER INVOLVEMENT

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d) IMPLEMENTATION



CONCLUSION

> IN THE EU A COMPLEX SET OF LEGISLATION IS AVAILABLE THAT REGULATES TRANSPORT AND STORAGE OF DANGEROUS GOODS

>THERE IS A CONTINUOUS PROCESS TO REVISE THIS LEGISLATION, ESPECIALLY AFTER A MAJOR ACCIDENT

> MORE MEASURES ARE ENVISAGED IN NEAR FUTURE

> EFMA CONTINUOUSLY WILL IMPROVE HSE ASPECTS OF FERTILIZERS

> EFMA WILL ALSO CONTINUE TO BE ACTIVELY ENGAGED IN THE REGULATORY PROCESS.

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Presentation: Putting really cool ideas to work in phosphates

Vaughn Astley, IMC Global, USA



Inderving the Future Inderving



Technology Development

- Focus on How TD Started,
- How We Put Really Cool Ideas to Work
- A Few Examples of Projects

Technology Development

• So How Did We Get Started?

So What Was The Problem

- A \$2 Billion Company With No Long Term Technical Focus.
- IMC Needed a Way to Develop New Products and Apply Latest Technologies

So What Was The Problem, Really

- Company Had a Lot of Ideas but Could not Manage to Study Them All
- No Project Evaluation Process
- Many Areas Were Backlogged With Existing Work Load and Short Term Projects

How do we Begin to Evaluate All the Potential Projects and Ideas

- Need Increased Resources – Separate from Day to Day Operations
- Must Also Look to Outside for Help – Infusion of Technology
 - Don't Reinvent or Develop Stuff

Who or What is Out There

- In-House
- Industrial Contract Organizations
- O Universities

In House

Cons Stalled projects

 Short term demands Short of staff

Pros

- Dedicated inquisitive people
- Knowledge base
- Promotable group in training
 Proprietary information controllable
 Pride in accomplishments

- Long hours are not charged for
 Can appreciate impact of work on other operational areas
- If fully occupied, cheaper than contract

Industrial Contract Organizations

- Cons
 - Lack of secrecy
 - Lack of proprietary protection
 - Lack of detailed specific plant knowledge
 - Usually costs more than In-House
 - Need oversight to keep direction and costs in check
 Scope of work and goal must be well defined

 - Frequent project overview is required
- Pros
 - Responsive
 - Wide general knowledge base
 - Specialized knowledge and experience in area of study
 - Flexible, varied manning availability
 - Can expeditiously stop funding

Universities

- Cons •
 - Slow •
 - Lack of secrecy •
 - Lack of proprietary protection
 - Lack of detailed specific plant knowledge
 - Need oversight to keep direction and costs in check
 - Preference for work that can be published

• Pros

- Inexpensive
- No pre-conceived ideas

So What Did We Do?

- Board Presentation Form an In-house Group, but With the Majority of Work Carried Out With Outside Assistance
- Called It "Technology Development" Rather Than "R & D"
- Essential to Fund It From Corporate
 - No Charges to Local Operations
- Report to Chief Operating Officer
- No Rules

Technology Development

Never Exceed Annual Budget

Or At Least Not By Much

No Rules, except

Goal and Objective of Technology Development

- Responsible for Developing New Technology to Lower Costs by \$10/ton DAP
- Explore Other Sources of Income
- Evaluate Projects or Proposals From Conceptual to Commercialized
- Find and Challenge the Paradigms
- To Funnel Projects Into Present Process & Project Engineering Structure
- To Take on Longer Term Projects Requiring Development

The Technology Development Group

- Formed Sept. 27, 1995 by JV Policy Board (Yes, We're Almost 7 Years Old)
- Mission is to Implement New Technology to Increase the Profitability of the Corporation
- We Were Told by Many, the Areas Where We Did Not Need to Look, or Should Not Work

The Technology Development Group

Senior Management Told Us Where We Were <u>Not</u> To Look For These Improvements Here's the List

Areas Excluded From Technology Development

How Do We Work

- Work Performed Internally (Mostly Computer, Pilot Plant and Field)
- Contract with Experts (Over 100)
- Provide Tools for Others in Organization That Have Valuable Ideas But Cannot get The Tools
- Random Acts of Kindness
- We Welcome All Ideas/Problems/If Onlys

Our Focus

- 1996 Worked on 5 years Out
- 1997 Worked on 4 years Out
- 1998 Worked on 3 years Out
- 1999 Worked on 2 years Out
- 2000 Worked on 2001
- 2001 Short Term Implementations
- 2002 Working on now, and 5 years Out

Luckily, We were going Short Term when **DAP** price Fell

Talking Points

- Permanent
- Leveraglines
- Kuger
- Dilbert
- Reck Piling Programs
- DAT Process Controls Hane
- · P205 Recovery
- aidance Tegnoslatti Ar
- Smart Fortilizers

- - Thurnessed Cloy

🗧 📴 🖬 Database

- O Woll-• 6m
- Ganneyona
 Attack Controls
- Treads in Mining
- Pond Water Remediation · Donna

Pond Water – Reverse Osmosis

- Previous Efforts (~1980's)
 - Failed Because of Irreversible Membrane Fouling
- Pretreatment is Essential
 - Pond water is SuperSaturated Solution
 - Saturation Must be Relieved to Allow **Removal of Water Without Precipitation**

Pond Water – Reverse Osmosis

- Pre-Treatment developed that allows Pond Water to be processed through R. O. System
 - ~75% of Feed volume meets (exceeds) discharge requirements
 - Sludge volume reduced ~75% - ~70% of P₂O₅ Recovered in Concentrate
- Process Chemistry confirmed at bench & Pilot scale (Including R.O. System)
- Patent Applications Filed (Have Provisional)

Pond Water – Reverse Osmosis

- OPre-Treatment to De-Saturate System
- OBut Majority of P2O5 Retained in Solution
- P₂O₅ Recovered as Concentrate Economic Value ~7 to 8% P2O5
- OPermeate is Essentially Pure Water

PROCESS CONTROLS

- Development started in 1988
- Process Control Computers
- Basic program language
- Rules and Fuzzy Logic
- Adaptive Controls
- On-Line Controls in 1990

PROCESS CONTROLS



PHOSPHORIC ACID PLANT ON-LINE CONTROLS

- Sulfate Control
- Phosphoric Acid Strength Control
- Filter Feed Control
- Rock Rate

PHOSPHORIC ACID PLANT ON-LINE CONTROLS

Sulfate control

- -Sulfate target
- -Acid to rock ratio
- -Total flow deviation control
- -Adjust ratio after every sample

PROCESS CONTROLS PHOSPHORIC ACID FLOWSHEET Filter Wash Water Sulfuric Acid Bypass Water Rock Recyc Acid Filtrate 27% Acid Gyp Slurry

Filter

Attack









PHOSPHORIC ACID PLANT ON-LINE CONTROLS

- Sulfate Control
- Phosphoric Acid Strength Control
- Filter Feed Control
 - No. 3 Filtrate density target
 - Density target sets filtration recovery
 - Filter feed rate set based on No.3 filtrate density





PHOSPHORIC ACID PLANT ON-LINE CONTROLS

- Sulfate Control
- Phosphoric Acid Strength Control
- Filter Feed Control
- Rock Rate
 - -Rock rate set based on filter feed level

PROCESS CONTROLS





The PhosAcid Control Strategies







PHOSPHORIC ACID PLANT ON-LINE CONTROLS

Control Limits and Logic for

- -Upset Conditions
- -Data entry errors
- -Rate and flow limits
- -Sensor drift and scaling













DAP APC Objectives

- Improve DAP Plant Controls - Stabilize the process
 - -PC based controls
 - -Additional instrumentation
- Determine optimum operating parameters
 - -Reactor mole ratio and gravity
- Improve Granulation



DAP2 APC Benefits

- Improved plant process control
 - Reduced P2O5 losses
 - Reduced ammonia losses
 - Improved DAP quality









Which Rock Would You Pick Up First?



<u>Notes</u>